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# SECTION 11

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## PRACTICAL LABORATORY INFORMATION

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## 11.1 COOLING

**TABLE 11.1** Cooling Mixtures

The table below gives the lowest temperature that can be obtained from a mixture of the inorganic salt with finely shaved dry ice. With the organic substances, dry ice ( $-78^{\circ}\text{C}$ ) in small lumps can be added to the solvent until a slight excess of dry ice remains or liquid nitrogen ( $-196^{\circ}\text{C}$ ) can be poured into the solvent until a slush is formed that consists of the solid-liquid mixture at its melting point.

| Substance                  | Quantity of substance, g | Quantity of water, mL | Temperature, $^{\circ}\text{C}$ |
|----------------------------|--------------------------|-----------------------|---------------------------------|
| Ammonium nitrate           | 100                      | 94                    | $-4.0$                          |
| Sodium nitrate             | 75                       | 100                   | $-5.3$                          |
| Sodium thiosulfate 5-water | 110                      | 100                   | $-8.0$                          |
| Sodium chloride            | 36                       | 100                   | $-10.0$                         |
| Sodium nitrate             | 50                       | 100                   | $-17.8$                         |
| Sodium bromide             | 66                       | 100                   | $-28$                           |
| Magnesium chloride         | 85                       | 100                   | $-34$                           |
| Calcium chloride 6-water   | 100                      | 81                    | $-40.3$                         |
|                            | 100                      | 70                    | $-55$                           |

| Substance                | Temperature, $^{\circ}\text{C}$ | Substance        | Temperature, $^{\circ}\text{C}$ |
|--------------------------|---------------------------------|------------------|---------------------------------|
| Ethylene glycol          | $-13$                           | Acetone          | $-77$                           |
| 1,2-Dichlorobenzene      | $-17$                           | Ethyl acetate    | $-84$                           |
| Carbon tetrachloride     | $-22.9$                         | 2-Butanone       | $-87$                           |
| Bromobenzene             | $-31$                           | Hexane           | $-95$                           |
| Methoxybenzene           | $-37$                           | Methanol         | $-98$                           |
| Bis(2-ethoxyethyl) ether | $-44$                           | Carbon disulfide | $-112$                          |
| Chlorobenzene            | $-45$                           | Bromoethane      | $-119$                          |
| <i>N</i> -Methylaniline  | $-57$                           | Pentane          | $-130$                          |
| <i>p</i> -Cymene         | $-68$                           | 2-Methylbutane   | $-160$                          |

**TABLE 11.2** Molecular Lowering of the Melting or Freezing Point*Cryoscopic constants.*

The cryoscopic constant  $K_f$  gives the depression of the melting point  $\Delta T$  (in degrees Celsius) produced when 1 mol of solute is dissolved in 1000 g of a solvent. It is applicable only to dilute solutions for which the number of moles of solute is negligible in comparison with the number of moles of solvent. It is often used for molecular weight determinations.

$$M_2 = \frac{1000w_2K_f}{w_1 \Delta T}$$

where  $w_1$  is the weight of the solvent and  $w_2$  is the weight of the solute whose molecular weight is  $M_2$ .

| Compound                           | $K_f$ | Compound                           | $K_f$      |
|------------------------------------|-------|------------------------------------|------------|
| Acetamide                          | 4.04  | Diphenylamine                      | 8.60       |
| Acetic acid                        | 3.90  | Diphenyl ether                     | 7.88       |
| Acetone                            | 2.40  | 1,2-Ethanediamine                  | 2.43       |
| Ammonia                            | 0.957 | Ethoxybenzene                      | 7.15       |
| Aniline                            | 5.87  | Formamide                          | 3.85       |
| Antimony(III) chloride             | 17.95 | Formic acid                        | 2.77       |
| Benzene                            | 5.12  | Glycerol                           | 3.3 to 3.7 |
| Benzonitrile                       | 5.34  | Hexamethylphosphoramide            | 6.93       |
| Benzophenone                       | 9.8   |                                    |            |
| Bicyclohexane                      | 14.52 | <i>N</i> -Methylacetamide          | 6.65       |
| Biphenyl                           | 8.0   | 2-Methyl-2-butanol                 | 10.4       |
| Borneol                            | 35.8  | Methylcyclohexane                  | 14.13      |
| Bornylamine                        | 40.6  | Methyl <i>cis</i> -9-octadecenoate | 3.4        |
| Butanedinitrile                    | 18.26 | 2-Methyl-2-propanol                | 8.37       |
| Camphene                           | 31.08 | Naphthalene                        | 6.94       |
| Camphoquinone                      | 45.7  | Nitrobenzene                       | 6.852      |
| <b>D</b> -(+)-Camphor              | 39.7  | Octadecanoic acid                  | 4.50       |
| Carbon tetrachloride               | 29.8  | 2-Oxohexamethyleneimine            | 7.30       |
| <i>o</i> -Cresol                   | 5.60  | Phenol                             | 7.40       |
| <i>p</i> -Cresol                   | 6.96  | Pyridine                           | 4.75       |
| Cyclohexane                        | 20.0  | Quinoline                          | 1.95       |
| Cyclohexanol                       | 39.3  | Succinonitrile                     | 18.26      |
| Cyclohexylcyclohexane              | 14.52 | Sulfuric acid                      | 1.86       |
| Cyclopentadecanone                 | 21.3  | 1,1,2,2-Tetrabromoethane           | 21.7       |
| <i>cis</i> -Decahydronaphthalene   | 19.47 | 1,1,2,2-Tetrachloro-               |            |
| <i>trans</i> -Decahydronaphthalene | 20.81 | 1,2-difluoroethane                 | 37.7       |
| Dibenz[ <i>de,kl</i> ]anthracene   | 25.7  | Tetramethylene sulfone             | 64.1       |
| Dibenzyl ether                     | 6.27  | <i>p</i> -Toluidine                | 5.372      |
| 1,2-Dibromoethane                  | 12.5  | Tribromomethane                    | 14.4       |
| Diethyl ether                      | 1.79  | 1,3,3-Trimethyl-2-oxabicyclo-      |            |
| 1,2-Dimethoxybenzene               | 6.38  | [2.2.2.]octane                     | 6.7        |
| <i>N,N</i> -Dimethylacetamide      | 4.46  | Triphenylmethane                   | 12.45      |
| 2,2-Dimethyl-1-propanol            | 11.0  | Water                              | 1.86       |
| Dimethyl sulfoxide                 | 4.07  | <i>p</i> -Xylene                   | 4.3        |
| 1,4-Dioxane                        | 4.63  |                                    |            |

## 11.2 DRYING AND HUMIDIFICATION

TABLE 11.3 Drying Agents

| Drying agent                                    | Most useful for  | Residual water,<br>mg H <sub>2</sub> O per liter<br>of dry air (25°C) | Grams water<br>removed per gram<br>of desiccant        | Regeneration,<br>°C |
|---|--|---|--|---------------------|
| Al <sub>2</sub> O <sub>3</sub>                  | Hydrocarbons   | 0.002–0.005   | 0.2  | 175 (24 h)          |
| Ba(ClO <sub>4</sub> ) <sub>2</sub> <sup>a</sup> | Inert gas streams  | 0.6–0.8   | 0.17   | 140                 |
| BaO   | Basic gases: hydrocarbons, aldehydes, alcohols                     | 0.0007–0.003  | 0.12   | 1000                |
| CaC <sub>2</sub> <sup>b</sup>                   | Ethers   |   | 0.56   | Impossible          |
| CaCl <sub>2</sub> <sup>c</sup>                  | Inert organics   | 0.1–0.2   | 0.15 (1 H <sub>2</sub> O)<br>0.30 (2 H <sub>2</sub> O) | 250                 |
| CaH <sub>2</sub> <sup>d</sup>                   | Hydrocarbons, ethers, amines, esters, higher alcohols              | 1 × 10 <sup>-5</sup>  | 0.85   | Impossible          |
| CaO   | Ethers, esters, alcohols, amines                                   | 0.01–0.003  | 0.31   | Difficult, 1000     |
| CaSO <sub>4</sub>                               | Most organic substances  | 0.005–0.07  | 0.07   | 225                 |
| Dow Desiccant 812 <sup>e</sup>                  | Most materials   | (5–200 ppm)   |  | No                  |
| K <sub>2</sub> CO <sub>3</sub>                  | Most materials except acids and phenols                            |   | 0.16   | 158                 |
| KOH   | Amines   | 0.01–0.9  |  | Impossible          |
| LiAlH <sub>4</sub> <sup>f</sup>                 | Hydrocarbons   |   | 1.9  | Impossible          |
| Mg(ClO <sub>4</sub> ) <sub>2</sub> <sup>a</sup> | Gas streams  | 0.0005–0.002  | 0.24   | 250 (high vacuum)   |
| MgO   | All but acidic compounds   | 0.008   | 0.45   | 800                 |
| MgSO <sub>4</sub>                               | Most organic compounds   | 1–12  | 0.15–0.75  | Not feasible        |
| Molecular sieves: 4X                            | Molecules with effective diameter >4Å                              | 0.001   | 0.18   | 250                 |
| 5X  | Molecules with effective diameter >5Å                              | 0.001   | 0.18   | 250                 |
| 9.5% Na-Pb alloy <sup>d</sup>                   | Hydrocarbons, ethers   | (For solvents only)   | 0.08   | Impossible          |
| Na <sub>2</sub> SO <sub>4</sub>                 | Ketones, acids, alkyl and aryl halides                             | 12  | 1.25   | 150                 |
| P <sub>2</sub> O <sub>5</sub>                   | Gas streams; not suitable for alcohols, amines, ketones, or amines | 2 × 10 <sup>-5</sup>  | 0.5  | Not feasible        |
| Silica gel                                      | Most organic amines  | 0.002–0.07  | 0.2  | 200–350             |
| Sulfuric acid                                   | Air and inert gas streams  | 0.003–0.008   | Indefinite   | Not feasible        |

<sup>a</sup> May form explosive mixtures when contacting organic material. <sup>b</sup> Explosive C<sub>2</sub>H<sub>2</sub> formed. <sup>c</sup> Slow in drying action.  
<sup>d</sup> H<sub>2</sub> formed. <sup>e</sup> Used as column drying of organic liquids. <sup>f</sup> Strong reductant.

A saturated aqueous solution in contact with an excess of a definite solid phase at a given temperature will maintain constant humidity in an enclosed space. Table 11.4 gives a number of salts suitable for this purpose. The aqueous tension (vapor pressure, in millimeters of Hg) of a solution at a given temperature is found by multiplying the decimal fraction of the humidity by the aqueous tension at 100 percent humidity for the specific temperature. For example, the aqueous tension of a saturated solution of NaCl at 20°C is  $0.757 \times 17.54 = 13.28$  mmHg and at 80°C it is  $0.764 \times 355.1 = 271.3$  mmHg.

**TABLE 11.4** Solutions for Maintaining Constant Humidity

| Solid Phase   | % Humidity at Specified Temperatures (°C) |       |       |       |       |       |       |
|---|---|-------|-------|-------|-------|-------|-------|
|   | 10  | 20    | 25    | 30    | 40    | 60    | 80    |
| K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>                       |   |       | 98.0  |       |       |       |       |
| K <sub>2</sub> SO <sub>4</sub>                                      | 98  | 97    | 97    | 96    | 96    | 96    |       |
| KNO <sub>3</sub>  | 95  | 93    | 92.5  | 91    | 88    | 82    |       |
| KCl   | 88  | 85.0  | 84.3  | 84    | 81.7  | 80.7  | 79.5  |
| KBr   |   | 84    | 80.7  |       | 79.6  | 79.0  | 79.3  |
| NaCl  | 76  | 75.7  | 75.3  | 74.9  | 74.7  | 74.9  | 76.4  |
| NaNO <sub>3</sub>   |   |       | 73.8  | 72.8  | 71.5  | 67.5  | 65.5  |
| NaNO <sub>2</sub>   |   | 66    | 65    | 63.0  | 61.5  | 59.3  | 58.9  |
| NaBr · 2H <sub>2</sub> O  |   | 57.9  | 57.7  |       | 52.4  | 49.9  | 50.0  |
| Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> · 2H <sub>2</sub> O  | 58  | 55    | 54    |       | 53.6  | 55.2  | 56.0  |
| Mg(NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O               | 57  | 55    | 52.9  | 52    | 49    | 43    |       |
| K <sub>2</sub> CO <sub>3</sub> · 2H <sub>2</sub> O                  | 47  | 44    | 42.8  |       | 42    |       |       |
| MgCl <sub>2</sub> · 6H <sub>2</sub> O                               | 34  | 33    | 33.0  | 33    | 32    | 30    |       |
| KF · 2H <sub>2</sub> O  |   |       |       | 27.4  | 22.8  | 21.0  | 22.8  |
| KC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> · 1.5H <sub>2</sub> O | 24  | 23    | 22.5  | 22    | 20    |       |       |
| LiCl · H <sub>2</sub> O   | 13  | 12    | 10.2  | 12    | 11    | 11    |       |
| KOH   | 13  | 9     | 8     | 7     | 6     | 5     |       |
| 100% Humidity: Aqueous Tension (mm Hg)                              | 9.21                                      | 17.54 | 23.76 | 31.82 | 55.32 | 149.4 | 355.1 |

**TABLE 11.5** Concentrations of Solutions of  $\text{H}_2\text{SO}_4$ ,  $\text{NaOH}$ , and  $\text{CaCl}_2$  Giving Specified Vapor Pressures and Percent Humidities at  $25^\circ\text{C}$ 

| Percent humidity | Aqueous tension, mmHg | $\text{H}_2\text{SO}_4$ |          | $\text{NaOH}$ |          | $\text{CaCl}_2$ |          |
|------------------|-----------------------|-------------------------|----------|---------------|----------|-----------------|----------|
|                  |                       | Molality                | Weight % | Molality      | Weight % | Molality        | Weight % |
| 100              | 23.76                 | 0.00                    | 0.00     | 0.00          | 0.00     | 0.00            | 0.00     |
| 95               | 22.57                 | 1.263                   | 11.02    | 1.465         | 5.54     | 0.927           | 9.33     |
| 90               | 21.38                 | 2.224                   | 17.91    | 2.726         | 9.83     | 1.584           | 14.95    |
| 85               | 20.19                 | 3.025                   | 22.88    | 3.840         | 13.32    | 2.118           | 19.03    |
| 80               | 19.00                 | 3.730                   | 26.79    | 4.798         | 16.10    | 2.579           | 22.25    |
| 75               | 17.82                 | 4.398                   | 30.14    | 5.710         | 18.60    | 2.995           | 24.95    |
| 70               | 16.63                 | 5.042                   | 33.09    | 6.565         | 20.80    | 3.400           | 27.40    |
| 65               | 15.44                 | 5.686                   | 35.80    | 7.384         | 22.80    | 3.796           | 29.64    |
| 60               | 14.25                 | 6.341                   | 38.35    | 8.183         | 24.66    | 4.188           | 31.73    |
| 55               | 13.07                 | 7.013                   | 40.75    | 8.974         | 26.42    | 4.581           | 33.71    |
| 50               | 11.88                 | 7.722                   | 43.10    | 9.792         | 28.15    | 4.990           | 35.64    |
| 45               | 10.69                 | 8.482                   | 45.41    | 10.64         | 29.86    | 5.431           | 37.61    |
| 40               | 9.50                  | 9.304                   | 47.71    | 11.54         | 31.58    | 5.912           | 39.62    |
| 35               | 8.31                  | 10.21                   | 50.04    | 12.53         | 33.38    | 6.478           | 41.83    |
| 30               | 7.13                  | 11.25                   | 52.45    | 13.63         | 35.29    | 7.183           | 44.36    |
| 25               | 5.94                  | 12.47                   | 55.01    | 14.96         | 37.45    |                 |          |
| 20               | 4.75                  | 13.94                   | 57.76    | 16.67         | 40.00    |                 |          |
| 15               | 3.56                  | 15.81                   | 60.80    | 19.10         | 43.32    |                 |          |
| 10               | 2.38                  | 18.48                   | 64.45    | 23.05         | 47.97    |                 |          |
| 5                | 1.19                  | 23.17                   | 69.44    |               |          |                 |          |

Concentrations are expressed in percentage of anhydrous solute by weight.

**Source:** Stokes and Robinson, *Ind. Eng. Chem.* **41**:2013 (1949).

**TABLE 11.6** Relative Humidity from Wet and Dry Bulb Thermometer Readings

| Dry bulb<br>temperature,<br>°C | Wet bulb depression, °C |     |     |     |     |     |      |      |      |      |      |      |
|--------------------------------|-------------------------|-----|-----|-----|-----|-----|------|------|------|------|------|------|
|                                | 0.5                     | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5  | 4.0  | 4.5  | 5.0  | 5.5  | 6.0  |
|                                | Relative humidity, %    |     |     |     |     |     |      |      |      |      |      |      |
| −10                            | 83                      | 67  | 51  | 35  | 19  |     |      |      |      |      |      |      |
| −5                             | 88                      | 76  | 64  | 52  | 41  | 29  | 18   | 7    |      |      |      |      |
| 0                              | 91                      | 81  | 72  | 64  | 55  | 46  | 38   | 29   | 21   | 13   | 5    |      |
| 2                              | 91                      | 84  | 76  | 68  | 60  | 52  | 44   | 37   | 29   | 22   | 14   | 7    |
| 4                              | 92                      | 85  | 78  | 71  | 63  | 57  | 49   | 43   | 36   | 29   | 22   | 16   |
| 6                              | 93                      | 86  | 79  | 73  | 66  | 60  | 54   | 48   | 41   | 35   | 29   | 24   |
| 8                              | 93                      | 87  | 81  | 75  | 69  | 63  | 57   | 51   | 46   | 40   | 35   | 29   |
| 10                             | 94                      | 88  | 82  | 77  | 71  | 66  | 60   | 55   | 50   | 44   | 39   | 34   |
| 12                             | 94                      | 89  | 83  | 78  | 73  | 68  | 63   | 58   | 53   | 48   | 43   | 39   |
| 14                             | 95                      | 90  | 85  | 79  | 75  | 70  | 65   | 60   | 56   | 51   | 47   | 42   |
| 16                             | 95                      | 90  | 85  | 81  | 76  | 71  | 67   | 63   | 58   | 54   | 50   | 46   |
| 18                             | 95                      | 91  | 86  | 82  | 77  | 73  | 69   | 65   | 61   | 57   | 53   | 49   |
| 20                             | 96                      | 91  | 87  | 83  | 78  | 74  | 70   | 66   | 63   | 59   | 55   | 51   |
| 22                             | 96                      | 92  | 87  | 83  | 80  | 76  | 72   | 68   | 64   | 61   | 57   | 54   |
| 24                             | 96                      | 92  | 88  | 84  | 80  | 77  | 73   | 69   | 66   | 62   | 59   | 56   |
| 26                             | 96                      | 92  | 88  | 85  | 81  | 78  | 74   | 71   | 67   | 64   | 61   | 58   |
| 28                             | 96                      | 93  | 89  | 85  | 82  | 78  | 75   | 72   | 69   | 65   | 62   | 59   |
| 30                             | 96                      | 93  | 89  | 86  | 83  | 79  | 76   | 73   | 70   | 67   | 64   | 61   |
| 35                             | 97                      | 94  | 90  | 87  | 84  | 81  | 78   | 75   | 72   | 69   | 67   | 64   |
| 40                             | 97                      | 94  | 91  | 88  | 85  | 82  | 80   | 77   | 74   | 72   | 69   | 67   |
| Dry bulb<br>temperature,<br>°C | Wet bulb depression, °C |     |     |     |     |     |      |      |      |      |      |      |
|                                | 6.5                     | 7.0 | 7.5 | 8.0 | 8.5 | 9.0 | 10.0 | 11.0 | 12.0 | 13.0 | 14.0 | 15.0 |
|                                | Relative humidity, %    |     |     |     |     |     |      |      |      |      |      |      |
| 4                              | 9                       |     |     |     |     |     |      |      |      |      |      |      |
| 6                              | 17                      | 11  | 5   |     |     |     |      |      |      |      |      |      |
| 8                              | 24                      | 19  | 14  | 8   |     |     |      |      |      |      |      |      |
| 10                             | 29                      | 24  | 20  | 15  | 10  | 6   |      |      |      |      |      |      |
| 12                             | 34                      | 29  | 25  | 21  | 16  | 12  | 5    |      |      |      |      |      |
| 14                             | 38                      | 34  | 30  | 26  | 22  | 18  | 10   |      |      |      |      |      |
| 16                             | 42                      | 38  | 34  | 30  | 26  | 23  | 15   | 8    |      |      |      |      |
| 18                             | 45                      | 41  | 38  | 34  | 30  | 27  | 20   | 14   | 7    |      |      |      |
| 20                             | 48                      | 44  | 41  | 37  | 34  | 31  | 24   | 18   | 12   | 6    |      |      |
| 22                             | 50                      | 47  | 44  | 40  | 37  | 34  | 28   | 22   | 17   | 11   | 6    |      |
| 24                             | 53                      | 49  | 46  | 43  | 40  | 37  | 31   | 26   | 20   | 15   | 10   | 5    |
| 26                             | 54                      | 51  | 49  | 46  | 43  | 40  | 34   | 29   | 24   | 19   | 14   | 10   |
| 28                             | 56                      | 53  | 51  | 48  | 45  | 42  | 37   | 32   | 27   | 22   | 18   | 13   |
| 30                             | 58                      | 55  | 52  | 50  | 47  | 44  | 39   | 35   | 30   | 25   | 21   | 17   |
| 32                             | 60                      | 57  | 54  | 51  | 49  | 46  | 41   | 37   | 32   | 28   | 24   | 20   |
| 34                             | 61                      | 58  | 56  | 53  | 51  | 48  | 43   | 39   | 35   | 30   | 26   | 23   |
| 36                             | 62                      | 59  | 57  | 54  | 52  | 50  | 45   | 41   | 37   | 33   | 29   | 25   |
| 38                             | 63                      | 61  | 58  | 56  | 54  | 51  | 47   | 43   | 39   | 35   | 31   | 27   |
| 40                             | 64                      | 62  | 59  | 57  | 54  | 53  | 48   | 44   | 40   | 36   | 33   | 29   |

**TABLE 11.7** Relative Humidity from Dew Point Readings

| Depression of<br>dew point,<br>°C | Dew point reading, °C |    |    |    |    |
|-----------------------------------|-----------------------|----|----|----|----|
|                                   | − 10                  | 0  | 10 | 20 | 30 |
|                                   | Relative humidity, %  |    |    |    |    |
| 0.5                               | 96                    | 96 | 96 | 96 | 97 |
| 1.0                               | 92                    | 93 | 94 | 94 | 94 |
| 1.5                               | 89                    | 89 | 90 | 91 | 92 |
| 2.0                               | 86                    | 87 | 88 | 88 | 89 |
| 3.0                               | 79                    | 81 | 82 | 83 | 84 |
| 4.0                               | 73                    | 75 | 77 | 78 | 80 |
| 5.0                               | 68                    | 70 | 72 | 74 | 75 |
| 6.0                               | 63                    | 66 | 68 | 70 | 71 |
| 7.0                               | 59                    | 61 | 63 | 66 | 68 |
| 8.0                               | 54                    | 57 | 60 | 62 | 64 |
| 9.0                               | 51                    | 53 | 56 | 58 | 61 |
| 10.0                              | 47                    | 50 | 53 | 55 | 57 |
| 11.0                              | 44                    | 47 | 49 | 52 |    |
| 12.0                              | 41                    | 44 | 47 | 49 |    |
| 13.0                              | 38                    | 41 | 44 | 46 |    |
| 14.0                              | 35                    | 38 | 41 | 44 |    |
| 15.0                              | 33                    | 36 | 39 | 42 |    |
| 16.0                              | 31                    | 34 | 37 | 39 |    |
| 18.0                              | 27                    | 30 | 33 | 35 |    |
| 20.0                              | 24                    | 26 | 29 | 32 |    |
| 22.0                              | 21                    | 23 | 26 |    |    |
| 24.0                              | 18                    | 21 | 23 |    |    |
| 26.0                              | 16                    | 18 | 21 |    |    |
| 28.0                              | 14                    | 16 | 19 |    |    |
| 30.0                              | 12                    | 14 | 17 |    |    |

### 11.3 BOILING POINTS AND HEATING BATHS

**TABLE 11.8** Organic Solvents Arranged by Boiling Points

| Name                     | BP, °C | Name                      | BP, °C |
|--------------------------|--------|---------------------------|--------|
| Ethylene oxide           | 10.6   | 1-Propanol                | 97.2   |
| Chloroethane             | 12.3   | Heptane                   | 98.4   |
| Furan                    | 31.4   | 1-Chloro-3-methylbutane   | 99     |
| Methyl formate           | 31.5   | Ethyl propionate          | 99.1   |
| Diethyl ether            | 34.6   | 2-Butanol                 | 99.6   |
| Propylene oxide          | 34.5   | Formic acid               | 100.8  |
| Pentane                  | 36.1   | Methylcyclohexane         | 100.9  |
| Bromoethane              | 38.4   | 1,4-Dioxane               | 101.2  |
| Dichloromethane          | 39.8   | Nitromethane              | 101.2  |
| Dimethoxymethane         | 42.3   | Propyl acetate            | 101.5  |
| Carbon disulfide         | 46.3   | 2-Pentanone               | 101.7  |
| 1-Isopropoxy-2-propanol  | 47.9   | 3-Pentanone               | 102.0  |
| Ethyl formate            | 54.2   | 2-Methyl-2-butanol        | 102.0  |
| Acetone                  | 56.2   | 1,1-Diethoxyethane        | 102.7  |
| Methyl acetate           | 56.3   | Butyl formate             | 106.6  |
| 1,1-Dichloroethane       | 57.3   | 2-Methyl-1-propanol       | 107.9  |
| Dichloroethylene         | 60.6   | Toluene                   | 110.6  |
| Chloroform               | 61.2   | <i>sec</i> -Butyl acetate | 112.3  |
| Methanol                 | 64.7   | 1,1,2-Trichloroethane     | 113.5  |
| Tetrahydrofuran          | 66.0   | Nitroethane               | 114.1  |
| Diisopropyl ether        | 68.0   | Pyridine                  | 115.2  |
| Hexane                   | 68.7   | 3-Pentanol                | 115.6  |
| 1-Chloro-2-methylpropane | 68.9   | 4-Methyl-2-pentanone      | 115.7  |
| 1,1,1-Trichloroethane    | 74.0   | 1-Chloro-2,3-epoxypropane | 116.1  |
| 1,3-Dioxolane            | 74–75  | 1-Butanol                 | 117.7  |
| Carbon tetrachloride     | 76.7   | Acetic acid               | 117.9  |
| Ethyl acetate            | 77.1   | Isobutyl acetate          | 118.0  |
| 1-Chlorobutane           | 77.9   | 2-Pentanol                | 119.3  |
| Ethanol                  | 78.3   | 1-Bromo-3-methylbutane    | 119.7  |
| 2-Butanone               | 79.6   | 1-Methoxy-2-propanol      | 120.1  |
| 2-Methyltetrahydrofuran  | 80.0   | 2-Nitropropane            | 120.3  |
| Benzene                  | 80.1   | Tetrachloroethylene       | 121.1  |
| Cyclohexane              | 80.7   | Ethyl butyrate            | 121.6  |
| Propyl formate           | 80.9   | 3-Hexanone                | 123    |
| Acetonitrile             | 81.6   | 2,4-Dimethyl-3-pentanone  | 124    |
| 2-Propanol               | 82.4   | 2-Methoxyethanol          | 124.6  |
| 1,1-Dimethylethanol      | 82.4   | Octane                    | 125.7  |
| Cyclohexene              | 83.0   | Butyl acetate             | 126.1  |
| Diisopropylamine         | 83.5   | Diethyl carbonate         | 126.8  |
| 1,2-Dichloroethane       | 83.7   | 2-Hexanone                | 127.2  |
| Thiophene                | 84.2   | 1-Chloro-2-propanol       | 127.4  |
| Trichloroethylene        | 87.2   | 2-Chloroethanol           | 128.6  |
| Isopropyl acetate        | 88.2   | 3-Methyl-1-penten-2-one   | 129.5  |
| 1-Bromo-2-methylpropane  | 91.5   | 1-Nitropropane            | 131.2  |
| 2,5-Dimethylfuran        | 93–94  | Chlorobenzene             | 131.7  |
| Ethyl chloroformate      | 94     | 1,2-Dibromoethane         | 131.7  |
| Allyl alcohol            | 96.6   | 4-Methyl-2-pentanol       | 131.7  |
| 1,2-Dichloropropane      | 96.8   |                           |        |

**TABLE 11.8** Organic Solvents Arranged by Boiling Points (*Continued*)

| Name                                       | BP, °C  | Name                               | BP, °C  |
|--|---------|------------------------------------|---------|
| 3-Methyl-1-butanol                         | 132.0   | 2-Octanol                          | 179     |
| Cyclohexylamine                            | 134.8   | 1,2-Dichlorobenzene                | 180.4   |
| 2-Ethoxyethanol                            | 134.8   | Ethyl acetoacetate                 | 180.8   |
| Ethylbenzene                               | 136.2   | Phenol                             | 181.8   |
| 1-Pentanol                                 | 138     | 2-Ethyl-1-hexanol                  | 184.3   |
| <i>p</i> -Xylene                           | 138.4   | Aniline                            | 184.4   |
| <i>m</i> -Xylene                           | 139.1   | Benzyl ethyl ether                 | 185.0   |
| Acetic anhydride                           | 140.0   | Diethyl oxalate                    | 185.4   |
| 2,4-Pentanedione                           | 140.6   | 1,2-Propanediol                    | 188     |
| Isopentyl acetate                          | 142     | Bis(2-ethoxyethyl) ether           | 188.4   |
| Dibutyl ether                              | 142.4   | Dimethyl sulfoxide                 | 189.0   |
| 4-Heptanone                                | 143.7   | 1,2-Ethanediol diacetate           | 190.2   |
| <i>o</i> -Xylene                           | 144.4   | Benzonitrile                       | 191.0   |
| 2-Methoxyethyl acetate                     | 144.5   | 2,5-Hexanedione                    | 191.4   |
| 1,1,2,2-Tetrachloroethane                  | 146.3   | 2-(2-Methoxyethoxy)-ethanol        | 194.1   |
| 3-Heptanone                                | 147.8   | <i>N,N</i> -Dimethylaniline        | 194.2   |
| Tribromomethane                            | 149.6   | 1-Octanol                          | 195.2   |
| Nonane                                     | 150.8   | 1,2-Ethanediol                     | 197.3   |
| 2-Heptanone                                | 151     | Diethyl malonate                   | 199.3   |
| Isopropylbenzene                           | 152.4   | Methyl benzoate                    | 199.5   |
| <i>N,N</i> -Dimethylformamide              | 153.0   | <i>o</i> -Toluidine                | 200.4   |
| Methoxybenzene                             | 153.8   | <i>p</i> -Toluidine                | 200.6   |
| Ethyl lactate                              | 154.5   | 2-(2-Ethoxyethoxy)-ethanol         | 202     |
| Cyclohexanone                              | 155.7   | Acetophenone                       | 202.1   |
| Bromobenzene                               | 156.2   | 1,2-Dibutoxyethane                 | 203.6   |
| 1,2,3-Trichloropropane                     | 156.9   | 1-Phenylethanol                    | 203.9   |
| 1-Hexanol                                  | 157.5   | <i>m</i> -Toluidine                | 203.4   |
| Propylbenzene                              | 159.2   | Benzyl alcohol                     | 205.5   |
| Cyclohexanol                               | 161.1   | Camphor                            | 207     |
| Bis(2-methoxyethyl)ether                   | 160     | 1,3-Butanediol                     | 207.5   |
| Isopentyl propionate                       | 160.2   | 1,2,3,4-Tetrahydro-naphthalene     | 207.6   |
| 2-Heptanol                                 | 160.4   | $\gamma$ -Valerolactone            | 207–208 |
| Pentachloroethane                          | 160.5   | <i>o</i> -Chloroaniline            | 208.8   |
| 2-Furaldehyde                              | 161.8   | Nitrobenzene                       | 210.8   |
| 2,6-Dimethyl-4-heptanone                   | 168.1   | Ethyl benzoate                     | 212.4   |
| 4-Hydroxy-4-methyl-2-pentanone             | 169.2   | 3,5,5-Trimethylcyclohex-2-en-1-one | 215.2   |
| 2-Furanmethanol                            | 170.0   | Naphthalene                        | 217.7   |
| Ethoxybenzene                              | 170     | 2-(2-Ethoxyethoxy)ethyl acetate    | 218.5   |
| 2-Butoxyethanol                            | 170.2   | Acetamide                          | 221.2   |
| Diisopentyl ether                          | 173.4   | Methyl salicylate                  | 223.0   |
| Decane                                     | 174.2   | Diethyl maleate                    | 225.3   |
| 1,3-Dichloro-2-propanol                    | 174.3   | 1,4-Butanediol                     | 230     |
| Cyclohexyl acetate                         | 174–175 | Propyl benzoate                    | 231.2   |
| 1-Heptanol                                 | 175.8   | 1-Decanol                          | 230.2   |
| Furfuryl acetate                           | 175–177 | Phenylacetoneitrile                | 233.5   |
| 1,3,3-Trimethyl-2-oxabicyclo-[2.2.2]octane | 177.4   | Quinoline                          | 237     |
| 4-Isopropyl-1-methylbenzene                | 177.1   | Tributyl borate                    | 238.5   |
| Isopentyl butyrate                         | 178.6   | Propylene carbonate                | 240     |
| Bis(2-chloroethyl) ether                   | 178.8   |                                    |         |

**TABLE 11.8** Organic Solvents Arranged by Boiling Points (*Continued*)

| Name                                  | BP, °C  | Name                                | BP, °C  |
|---------------------------------------|---------|-------------------------------------|---------|
| 2-Phenoxyethanol                      | 240     | Isopentyl salicylate                | 277–278 |
| Bis(2-hydroxyethyl) ether             | 245     | 1-Bromonaphthalene                  | 281.1   |
| Dibutyl oxalate                       | 245.5   | Dimethyl <i>o</i> -phthalate        | 283.7   |
| Butyl benzoate                        | 250     | 2,2'-(Ethylenedioxy)-<br>bisethanol | 285     |
| 1,2,3-Propanetriol<br>triacetate      | 258–259 | Glycerol                            | 290     |
| 1-Chloronaphthalene                   | 259.3   | Diethyl <i>o</i> -phthalate         | 295     |
| Isopentyl benzoate                    | 262     | Benzyl benzoate                     | 323.5   |
| <i>trans</i> -Ethyl cinnamate         | 271.0   | Dibutyl <i>o</i> -phthalate         | 340.0   |
| Bis[2-(methoxyethoxy)-<br>ethyl]ether | 275.3   | Dibutyl decanedioate                | 344–345 |
| 1-Methoxy-2-nitrobenzene              | 277     |                                     |         |

**TABLE 11.9** Molecular Elevation of the Boiling Point*Ebullioscopic constants.*

Molecular weights can be determined with the relation:

$$M = E_b \frac{1000 w_2}{w_1 \Delta T_b}$$

where  $\Delta T_b$  is the elevation of the boiling point brought about by the addition of  $w_2$  grams of solute to  $w_1$  grams of solvent and  $E_b$  is the ebullioscopic constant. In the column headed "Barometric correction" is the number of degrees for each millimeter of difference between the barometric reading and 760 mmHg to be subtracted from  $E_b$  if the pressure is lower, or added if higher, than 760 mm. In general, the effect is within experimental error if the pressure is within 10 mm of 760 mm.

The ebullioscopic constant, a characteristic property of the solvent, may be calculated from the relation:

$$E_b = \frac{RT_b^2 M}{\Delta_{\text{vap}} H}$$

where  $R$  is the molar gas constant,  $M$  is the molar mass of the solvent, and  $\Delta_{\text{vap}} H$  the molar enthalpy (heat) of vaporization of the solvent.

| Compound              | Barometric correction | $E_b$ ,<br>°C kg · mol <sup>-1</sup> |
|-----------------------|-----------------------|--------------------------------------|
| Acetic acid           | 0.0008                | 3.22                                 |
| Acetic anhydride      |                       | 3.79                                 |
| Acetone               | 0.0004                | 1.80                                 |
| Acetonitrile          |                       | 1.44                                 |
| Acetophenone          |                       | 5.81                                 |
| Aniline               | 0.0009                | 3.82                                 |
| Benzaldehyde          |                       | 4.24                                 |
| Benzene               | 0.0007                | 2.64                                 |
| Benzonitrile          |                       | 4.02                                 |
| Bromobenzene          | 0.0016                | 6.35                                 |
| Bromoethane           |                       | 1.73                                 |
| 1-Butanol             |                       | 2.17                                 |
| 2-Butanone            |                       | 2.28                                 |
| cis-2-Butene-1,4-diol |                       | 2.73                                 |
| D-(+)-Camphor         | 0.0015                | 4.91                                 |
| Carbon disulfide      | 0.0006                | 2.42                                 |
| Carbon tetrachloride  | 0.0013                | 5.26                                 |
| Chlorobenzene         | 0.0011                | 4.36                                 |
| 1-Chlorobutane        |                       | 3.13                                 |
| Chloroethane          |                       | 1.77                                 |
| Chloroform            | 0.0009                | 3.80                                 |
| Cyclohexane           | 0.0007                | 2.92                                 |
| Cyclohexanol          |                       | 3.5                                  |
| Decane                |                       | 6.10                                 |
| 1,2-Dibromomethane    | 0.0016                | 6.01                                 |
| 1,1-Dichloroethane    |                       | 3.13                                 |
| 1,2-Dichloroethane    |                       | 3.27                                 |
| Dichloromethane       |                       | 2.42                                 |
| Diethyl ether         | 0.0005                | 2.20                                 |
| Diethyl sulfide       |                       | 3.14                                 |
| Dimethoxymethane      |                       | 2.12                                 |
| N,N-Dimethylacetamide |                       | 3.22                                 |
| Dimethyl sulfide      |                       | 1.85                                 |
| Dimethyl sulfoxide    |                       | 3.22                                 |

TABLE 11.9 Molecular Elevation of the Boiling Point (*Continued*)

| Compound                              | Barometric<br>correction | $E_b$ ,<br>$^{\circ}\text{C kg} \cdot \text{mol}^{-1}$ |
|---------------------------------------|--------------------------|--|
| 1,4-Dioxane                           |                          | 3.00   |
| Ethanol                               | 0.0003                   | 1.22   |
| Ethoxybenzene                         |                          | 4.90   |
| Ethyl acetate                         | 0.0007                   | 2.82   |
| Ethylene glycol                       |                          | 2.26   |
| Formic acid                           |                          | 2.36   |
| Glycerol                              |                          | 6.52   |
| Heptane                               | 0.0008                   | 3.62   |
| Hexane                                |                          | 2.90   |
| 2-Hydroxybenzaldehyde                 |                          | 5.87   |
| Iodoethane                            |                          | 5.27   |
| Iodomethane                           |                          | 4.31   |
| 4-Isopropyl-1-methylbenzene           |                          | 5.92   |
| Methanol                              | 0.0002                   | 0.86   |
| Methoxybenzene                        |                          | 4.20   |
| Methyl acetate                        | 0.0005                   | 2.21   |
| <i>N</i> -Methylaniline               |                          | 4.3  |
| 2-Methyl-2-butanol                    |                          | 2.64   |
| 3-Methyl-1-butanol                    |                          | 2.88   |
| 3-Methylbutyl acetate                 |                          | 4.83   |
| <i>N</i> -Methylformamide             |                          | 2.2  |
| Methyl formate                        |                          | 1.66   |
| 2-Methyl-1-propanol                   |                          | 2.14   |
| 2-Methyl-2-propanol                   |                          | 1.99   |
| Naphthalene                           | 0.0014                   | 5.94   |
| Nitrobenzene                          |                          | 5.24   |
| Nitroethane                           |                          | 2.46   |
| Nitromethane                          |                          | 2.09   |
| Octane                                |                          | 4.39   |
| 1-Octanol                             |                          | 5.06   |
| Pentyl acetate                        |                          | 4.71   |
| Phenol                                | 0.0009                   | 3.54   |
| Piperidine                            |                          | 3.21   |
| Propanoic acid                        |                          | 3.27   |
| 1-Propanol                            |                          | 1.66   |
| 2-Propanol                            |                          | 1.58   |
| Propionitrile                         |                          | 1.97   |
| Pyridine                              |                          | 2.83   |
| Pyrrole                               |                          | 2.33   |
| Pyrrolidine                           |                          | 2.32   |
| Quinoline                             |                          | 5.62   |
| Tetrachloroethylene                   |                          | 6.18   |
| Tetrachloromethane                    |                          | 5.26   |
| 1,2,3,4-Tetrahydronaphthalene         |                          | 5.58   |
| Toluene                               | 0.0008                   | 3.40   |
| <i>p</i> -Toluidine                   |                          | 4.51   |
| Trichloroethylene                     |                          | 4.52   |
| Trichloromethane                      | 0.0009                   | 3.80   |
| 1,1,2-Trichloro-1,2,2-trifluoroethane |                          | 5.93   |
| Triethylamine                         |                          | 3.57   |
| Water                                 | 0.0001                   | 0.512  |
| <i>o</i> -Xylene                      |                          | 4.25   |

**TABLE 11.10** Substances Which Can Be Used for Heating Baths

| Medium                      | Melting point, °C | Boiling point, °C | Useful range, °C | Flash point, °C | Comments                            |
|-----------------------------|-------------------|-------------------|------------------|-----------------|-------------------------------------|
| Water                       | 0                 | 100               | 0–100            | None            | Ideal                               |
| Silicone oil                | – 50              | —                 | 30–250           | 315             | Somewhat viscous at low temperature |
| Triethylene glycol          | – 7               | 285               | 0–250            | 165             | Noncorrosive                        |
| Glycerol                    | 18                | 290               | – 20 to 260      | 160             | Water-soluble, nontoxic             |
| Paraffin                    | 50                | —                 | 60–300           | 199             | Flammable                           |
| Dibutyl <i>o</i> -phthalate | – 35              | 340               | 150–320          | 171             | Generally used                      |

## 11.4 SEPARATION METHODS

**TABLE 11.11** Solvents of Chromatographic Interest

| Solvent                | Boiling point, °C | Solvent strength parameter    |   | Viscosity, $\text{mN} \cdot \text{s} \cdot \text{m}^{-2}$ (20°C) | Refractive index (20°C) | UV cutoff, nm |
|------------------------|-------------------|-------------------------------|---|--|-------------------------|---------------|
|                        |                   | $e^\circ$ (SiO <sub>2</sub> ) | $e^\circ$ (Al <sub>2</sub> O <sub>3</sub> ) |  |                         |               |
| Fluoroalkanes          |                   |                               | −0.25                                       |  | 1.25                    |               |
| Pentane                | 36                | 0.0                           | 0.0   | 0.24 <sup>15°C</sup>   | 1.358                   | 210           |
| Hexane                 | 69                | 0.0                           | 0.0   | 0.31   | 1.375                   | 210           |
| 2,2,4-Trimethylpentane | 99                |                               | 0.01  | 0.50   | 1.392                   | 215           |
| Decane                 | 174               |                               | 0.04  | 0.93   | 1.412                   | 210           |
| Cyclohexane            | 81                | −0.05                         | 0.04  | 0.98   | 1.426                   | 210           |
| Cyclopentane           | 49                |                               | 0.05  | 0.44   | 1.407                   | 210           |
| Diisobutylene          | 101               |                               | 0.06  |  | 1.411                   |               |
| 1-Pentene              | 30                |                               | 0.08  | 0.24 <sup>0°C</sup>  | 1.371                   |               |
| Carbon disulfide       | 46                | 0.14                          | 0.15  | 0.36   | 1.626                   | 380           |
| Carbon tetrachloride   | 77                | 0.14                          | 0.18  | 0.97   | 1.466                   | 265           |
| 1-Chlorobutane         | 78                |                               | 0.26  | 0.43   | 1.402                   | 220           |
| 1-Chloropentane        | 98                |                               | 0.26  | 0.58   | 1.412                   | 225           |
| <i>o</i> -Xylene       | 144               |                               | 0.26  | 0.81   | 1.505                   | 290           |
| Diisopropyl ether      | 68                |                               | 0.28  | 0.38 <sup>25°C</sup>   | 1.369                   | 220           |
| 2-Chloropropane        | 35                |                               | 0.29  | 0.33   | 1.378                   | 225           |
| Toluene                | 111               |                               | 0.29  | 0.59   | 1.497                   | 286           |
| 1-Chloropropane        | 47                |                               | 0.30  | 0.35   | 1.389                   | 225           |
| Chlorobenzene          | 132               |                               | 0.40  | 0.80   | 1.525                   |               |
| Benzene                | 80                | 0.25                          | 0.32  | 0.65   | 1.501                   | 280           |
| Bromoethane            | 38                |                               | 0.37  | 0.40   | 1.424                   |               |

|                      |     |      |       |                      |       |     |
|----------------------|-----|------|-------|----------------------|-------|-----|
| Diethyl ether        | 35  | 0.38 | 0.38  | 0.25                 | 1.353 | 218 |
| Diethyl sulfide      | 92  |      | 0.38  | 0.45                 | 1.443 | 290 |
| Chloroform           | 62  | 0.26 | 0.40  | 0.57                 | 1.443 | 245 |
| Dichloromethane      | 41  |      | 0.42  | 0.44                 | 1.425 | 235 |
| 4-Methyl-2-pentanone | 116 |      | 0.43  | 0.42 <sup>15°C</sup> | 1.396 | 335 |
| Tetrahydrofuran      | 66  |      | 0.45  | 0.55                 | 1.407 | 220 |
| 1,2-Dichloroethane   | 84  |      | 0.49  | 0.80                 | 1.445 | 228 |
| 2-Butanone           | 80  |      | 0.51  | 0.42 <sup>15°C</sup> | 1.379 | 330 |
| 1-Nitropropane       | 131 |      | 0.53  | 0.80 <sup>25°C</sup> | 1.402 | 380 |
| Acetone              | 56  | 0.47 | 0.56  | 0.32                 | 1.359 | 330 |
| 1,4-Dioxane          | 101 | 0.49 | 0.56  | 1.44 <sup>15°C</sup> | 1.420 | 215 |
| Ethyl acetate        | 77  | 0.38 | 0.58  | 0.45                 | 1.372 | 255 |
| Methyl acetate       | 56  |      | 0.60  | 0.48 <sup>15°C</sup> | 1.362 | 260 |
| 1-Pentanol           | 138 |      | 0.61  | 4.1                  | 1.410 | 210 |
| Dimethyl sulfoxide   | 189 |      | 0.62  | 2.47                 | 1.478 | 265 |
| Aniline              | 184 |      | 0.62  | 4.40                 | 1.586 |     |
| Diethylamine         | 56  |      | 0.63  | 0.33                 | 1.386 | 275 |
| Nitromethane         | 101 |      | 0.64  | 0.67                 | 1.394 | 380 |
| Acetonitrile         | 82  | 0.50 | 0.65  | 0.37                 | 1.344 | 190 |
| Pyridine             | 115 |      | 0.71  | 0.97                 | 1.510 | 330 |
| 2-Butoxyethanol      | 170 |      | 0.74  | 3.15 <sup>25°C</sup> | 1.420 | 220 |
| 1-Propanol           | 97  |      | 0.82  | 2.25                 | 1.386 | 210 |
| 2-Propanol           | 82  |      | 0.82  | 2.50                 | 1.377 | 210 |
| Ethanol              | 78  |      | 0.88  | 1.20                 | 1.361 | 210 |
| Methanol             | 65  |      | 0.95  | 0.59                 | 1.328 | 210 |
| Ethylene glycol      | 198 |      | 1.11  | 21.8                 | 1.432 | 210 |
| Acetic acid          | 118 |      | large | 1.23                 | 1.372 | 260 |
| Water                | 100 |      | large | 1.00                 | 1.333 | 191 |

**TABLE 11.12** Solvents Having the Same Refractive Index and the Same Density at 25°C

| Solvent 1                | Solvent 2                | Refractive index |       | Density, g/mL |       |
|--------------------------|--------------------------|------------------|-------|---------------|-------|
|                          |                          | 1                | 2     | 1             | 2     |
| Acetone                  | Ethanol                  | 1.357            | 1.359 | 0.788         | 0.786 |
| Ethyl formate            | Methyl acetate           | 1.358            | 1.360 | 0.916         | 0.935 |
| Ethanol                  | Propionitrile            | 1.359            | 1.363 | 0.786         | 0.777 |
| 2,2-Dimethylbutane       | 2-Methylpentane          | 1.366            | 1.369 | 0.644         | 0.649 |
| 2-Methylpentane          | Hexane                   | 1.369            | 1.372 | 0.649         | 0.655 |
| Isopropyl acetate        | 2-Chloropropane          | 1.375            | 1.376 | 0.868         | 0.865 |
| 3-Butanone               | Butyraldehyde            | 1.377            | 1.378 | 0.801         | 0.799 |
| Butyraldehyde            | Butyronitrile            | 1.378            | 1.382 | 0.799         | 0.786 |
| Dipropyl ether           | Butyl ethyl ether        | 1.379            | 1.380 | 0.753         | 0.746 |
| Propyl acetate           | Ethyl propionate         | 1.382            | 1.382 | 0.883         | 0.888 |
| Propyl acetate           | 1-Chloropropane          | 1.382            | 1.386 | 0.883         | 0.890 |
| Butyronitrile            | 2-Methyl-2-propanol      | 1.382            | 1.385 | 0.786         | 0.781 |
| Ethyl propionate         | 1-Chloropropane          | 1.382            | 1.386 | 0.888         | 0.890 |
| 1-Propanol               | 2-Pentanone              | 1.383            | 1.387 | 0.806         | 0.804 |
| Isobutyl formate         | 1-Chloropropane          | 1.383            | 1.386 | 0.881         | 0.890 |
| 1-Chloropropane          | Butyl formate            | 1.386            | 1.387 | 0.890         | 0.888 |
| Butyl formate            | Methyl butyrate          | 1.387            | 1.391 | 0.888         | 0.875 |
| Methyl butyrate          | 2-Chlorobutane           | 1.392            | 1.395 | 0.875         | 0.868 |
| Butyl acetate            | 2-Chlorobutane           | 1.392            | 1.395 | 0.877         | 0.868 |
| 4-Methyl-2-pentanone     | Pentanonitrile           | 1.394            | 1.395 | 0.797         | 0.795 |
| 4-Methyl-2-pentanone     | 1-Butanol                | 1.394            | 1.397 | 0.797         | 0.812 |
| 2-Methyl-1-propanol      | Pentanonitrile           | 1.394            | 1.395 | 0.798         | 0.795 |
| 2-Methyl-1-propanol      | 2-Hexanone               | 1.394            | 1.395 | 0.798         | 0.810 |
| 2-Butanol                | 2,4-Dimethyl-3-pentanone | 1.395            | 1.399 | 0.803         | 0.805 |
| 2-Hexanone               | 1-Butanol                | 1.395            | 1.397 | 0.810         | 0.812 |
| Pentanonitrile           | 2,4-Dimethyl-3-pentanone | 1.395            | 1.399 | 0.795         | 0.805 |
| 2-Chlorobutane           | Isobutyl butyrate        | 1.395            | 1.399 | 0.868         | 0.860 |
| Butyric acid             | 2-Methoxyethanol         | 1.396            | 1.400 | 0.955         | 0.960 |
| 1-Butanol                | 3-Methyl-2-pentanone     | 1.397            | 1.398 | 0.812         | 0.808 |
| 1-Chloro-2-methylpropane | Isobutyl butyrate        | 1.397            | 1.399 | 0.872         | 0.860 |
| 1-Chloro-2-methylpropane | Pentyl acetate           | 1.397            | 1.400 | 0.872         | 0.871 |
| Methyl methacrylate      | 3-Methyl-2-pentanone     | 1.398            | 1.398 | 0.795         | 0.808 |
| Triethylamine            | 2,2,3-Trimethylpentane   | 1.399            | 1.401 | 0.723         | 0.712 |
| Butylamine               | Dodecane                 | 1.399            | 1.400 | 0.736         | 0.746 |
| Isobutyl butyrate        | 1-Chlorobutane           | 1.399            | 1.401 | 0.860         | 0.875 |
| 1-Nitropropane           | Propionic anhydride      | 1.399            | 1.400 | 0.995         | 1.007 |
| Pentyl acetate           | 1-Chlorobutane           | 1.400            | 1.400 | 0.871         | 0.881 |
| Pentyl acetate           | Tetrahydrofuran          | 1.400            | 1.404 | 0.871         | 0.885 |
| Dodecane                 | Dipropylamine            | 1.400            | 1.400 | 0.746         | 0.736 |
| 1-Chlorobutane           | Tetrahydrofuran          | 1.401            | 1.404 | 0.871         | 0.885 |
| Isopentanoic acid        | 2-Ethoxyethanol          | 1.402            | 1.405 | 0.923         | 0.926 |
| Dipropylamine            | Cyclopentane             | 1.403            | 1.404 | 0.736         | 0.740 |
| 2-Pentanol               | 4-Heptanone              | 1.404            | 1.405 | 0.804         | 0.813 |
| 3-Methyl-1-butanol       | Hexanonitrile            | 1.404            | 1.405 | 0.805         | 0.801 |
| 3-Methyl-1-butanol       | 4-Heptanone              | 1.404            | 1.405 | 0.805         | 0.813 |
| Hexanonitrile            | 4-Heptanone              | 1.405            | 1.405 | 0.801         | 0.813 |
| Hexanonitrile            | 1-Pentanol               | 1.405            | 1.408 | 0.801         | 0.810 |
| Hexanonitrile            | 2-Methyl-1-butanol       | 1.405            | 1.409 | 0.801         | 0.815 |
| 4-Heptanone              | 1-Pentanol               | 1.405            | 1.408 | 0.813         | 0.810 |

**TABLE 11.12** Solvents Having the Same Refractive Index and the Same Density at 25°C (*Continued*)

| Solvent 1                          | Solvent 2                           | Refractive index |       | Density, g/mL |       |
|------------------------------------|-------------------------------------|------------------|-------|---------------|-------|
|                                    |                                     | 1                | 2     | 1             | 2     |
| 2-Ethoxyethanol                    | Pentanoic acid                      | 1.405            | 1.406 | 0.926         | 0.936 |
| 2-Heptanone                        | 1-Pentanol                          | 1.406            | 1.408 | 0.811         | 0.810 |
| 2-Heptanone                        | 2-Methyl-1-butanol                  | 1.406            | 1.409 | 0.811         | 0.815 |
| 2-Heptanone                        | Dipentyl ether                      | 1.406            | 1.410 | 0.811         | 0.799 |
| 2-Pentanol                         | 3-Isopropyl-2-pentanone             | 1.407            | 1.409 | 0.804         | 0.808 |
| 1-Pentanol                         | Dipentyl ether                      | 1.408            | 1.410 | 0.810         | 0.799 |
| 2-Methyl-1-butanol                 | Dipentyl ether                      | 1.409            | 1.410 | 0.815         | 0.799 |
| Isopentyl isopentanoate            | Allyl alcohol                       | 1.410            | 1.411 | 0.853         | 0.847 |
| Dipentyl ether                     | 2-Octanone                          | 1.410            | 1.414 | 0.799         | 0.814 |
| 2,4-Dimethyldioxane                | 3-Chloropentene                     | 1.412            | 1.413 | 0.935         | 0.932 |
| 2,4-Dimethyldioxane                | Hexanoic acid                       | 1.412            | 1.415 | 0.935         | 0.923 |
| Diethyl malonate                   | Ethyl cyanoacetate                  | 1.412            | 1.415 | 1.051         | 1.056 |
| 3-Chloropentene                    | Octanoic acid                       | 1.413            | 1.415 | 0.932         | 0.923 |
| 2-Octanone                         | 1-Hexanol                           | 1.414            | 1.416 | 0.814         | 0.814 |
| 2-Octanone                         | Octanonitrile                       | 1.414            | 1.418 | 0.814         | 0.810 |
| 3-Octanone                         | 3-Methyl-2-heptanone                | 1.414            | 1.416 | 0.830         | 0.818 |
| 3-Methyl-2-heptanone               | 1-Hexanol                           | 1.415            | 1.416 | 0.818         | 0.814 |
| 3-Methyl-2-heptanone               | Octanonitrile                       | 1.415            | 1.418 | 0.818         | 0.810 |
| 1-Hexanol                          | Octanonitrile                       | 1.416            | 1.418 | 0.814         | 0.810 |
| Dibutylamine                       | Allylamine                          | 1.416            | 1.419 | 0.756         | 0.758 |
| Allylamine                         | Methylcyclohexane                   | 1.419            | 1.421 | 0.758         | 0.765 |
| Butyrolactone                      | 1,3-Propanediol                     | 1.434            | 1.438 | 1.051         | 1.049 |
| Butyrolactone                      | Diethyl maleate                     | 1.434            | 1.438 | 1.051         | 1.064 |
| 2-Chloromethyl-2-propanol          | Diethyl maleate                     | 1.436            | 1.438 | 1.059         | 1.064 |
| <i>N</i> -Methylmorpholine         | Dibutyl decanedioate                | 1.436            | 1.440 | 0.924         | 0.932 |
| 1,3-Propanediol                    | Diethyl maleate                     | 1.438            | 1.438 | 1.049         | 1.064 |
| Methyl salicylate                  | Diethyl sulfide                     | 1.438            | 1.442 | 0.836         | 0.831 |
| Methyl salicylate                  | 1-Butanethiol                       | 1.438            | 1.442 | 0.836         | 0.837 |
| 1-Chlorodecane                     | Mesityl oxide                       | 1.441            | 1.442 | 0.862         | 0.850 |
| Diethylene glycol                  | Formamide                           | 1.445            | 1.446 | 1.128         | 1.129 |
| Diethylene glycol                  | Ethylene glycol diglycidyl ether    | 1.445            | 1.447 | 1.128         | 1.134 |
| Formamide                          | Ethylene glycol diglycidyl ether    | 1.446            | 1.447 | 1.129         | 1.134 |
| 2-Methylmorpholine                 | Cyclohexanone                       | 1.446            | 1.448 | 0.951         | 0.943 |
| 2-Methylmorpholine                 | 1-Amino-2-propanol                  | 1.446            | 1.448 | 0.951         | 0.961 |
| Dipropylene glycol monoethyl ether | Tetrahydrofurfuryl alcohol          | 1.446            | 1.450 | 1.043         | 1.050 |
| 1-Amino-2-methyl-2-pentanol        | 2-Butylcyclohexanone                | 1.449            | 1.453 | 0.904         | 0.901 |
| 2-Propylcyclohexanone              | 4-Methylcyclohexanol                | 1.452            | 1.454 | 0.923         | 0.908 |
| Carbon tetrachloride               | 4,5-Dichloro-1,3-dioxolane-2-one    | 1.459            | 1.461 | 1.584         | 1.591 |
| <i>N</i> -Butyldiethanolamine      | Cyclohexanol                        | 1.461            | 1.465 | 0.965         | 0.968 |
| <b>D</b> - $\alpha$ -Pinene        | <i>trans</i> -Decahydro-naphthalene | 1.464            | 1.468 | 0.855         | 0.867 |
| Propylbenzene                      | <i>p</i> -Xylene                    | 1.490            | 1.493 | 0.858         | 0.857 |
| Propylbenzene                      | Toluene                             | 1.490            | 1.494 | 0.858         | 0.860 |

**TABLE 11.12** Solvents Having the Same Refractive Index and the Same Density at 25°C (*Continued*)

| Solvent 1                    | Solvent 2                  | Refractive index |       | Density, g/mL |       |
|------------------------------|----------------------------|------------------|-------|---------------|-------|
|                              |                            | 1                | 2     | 1             | 2     |
| Phenyl 1-hydroxyphenyl ether | 1,3-Dimorpholyl-2-propanol | 1.491            | 1.493 | 1.081         | 1.094 |
| Phenetole                    | Pyridine                   | 1.505            | 1.507 | 0.961         | 0.978 |
| 2-Furanmethanol              | Thiophene                  | 1.524            | 1.526 | 1.057         | 1.059 |
| <i>m</i> -Cresol             | Benzaldehyde               | 1.542            | 1.544 | 1.037         | 1.041 |

**TABLE 11.13** McReynolds' Constants for Stationary Phases in Gas Chromatography

| Stationary phase   | Chemical type                           | Similar stationary phases     | Temp., °C |     | McReynolds' constants |           |           |           |           |     | USP code |
|--|---|-------------------------------|-----------|-----|-----------------------|-----------|-----------|-----------|-----------|-----|----------|
|  |   |                               | Min       | Max | <i>x'</i>             | <i>y'</i> | <i>z'</i> | <i>u'</i> | <i>s'</i> | Σ   |          |
| Boiling-point separation of broad molecular weight range of compounds; nonpolar phases |   |                               |           |     |                       |           |           |           |           |     |          |
| Squalane   | 2,6,10,15,19,23-Hexamethyltetracosane   |                               | 20        | 150 | 0                     | 0         | 0         | 0         | 0         | 0   |          |
| Paraffin oil   |   |                               |           |     | 9                     | 5         | 2         | 6         | 11        | 33  |          |
| Apiezon® L   |   |                               | 50        | 300 | 32                    | 22        | 15        | 32        | 42        | 143 |          |
| SPB-1  | Poly(dimethylsiloxane)                  | SA-1, DB-1                    | −60       | 320 | 4                     | 58        | 43        | 56        | 38        | 199 |          |
| SP™-2100   | Poly(dimethylsiloxane)                  | DC-200, SE 30, UC W98, DC 200 | 0         | 350 | 17                    | 57        | 45        | 67        | 43        | 229 | G 9      |
| OV-1   | Methylsiloxane gum                      |                               | 100       | 350 | 16                    | 55        | 44        | 65        | 42        | 227 | G 2      |
| OV-101   | Methylsiloxane fluid                    |                               | 20        | 350 | 17                    | 57        | 45        | 67        | 43        | 234 | G 1      |
| SPB-5  | 1% Vinyl, 5% phenyl methyl polysiloxane | SA-5, DB-5                    | −60       | 320 | 19                    | 74        | 64        | 93        | 62        | 312 |          |
| SE-54  | 1% Vinyl, 5% phenyl methyl polysiloxane | PTE-5                         | 50        | 300 | 19                    | 74        | 64        | 93        | 62        | 312 | G 36     |
| SE-52  | 5% Phenyl methyl polysiloxane           |                               | 50        | 300 | 32                    | 72        | 65        | 98        | 67        | 334 | G 27     |
| OV-73  | 5.5% Phenyl methyl polysiloxane         | SP-400                        | 0         | 325 | 40                    | 86        | 76        | 114       | 85        | 401 | G 27     |
| OV-3   | Poly(dimethyldiphenylsiloxane); 90%:10% |                               | 0         | 350 | 44                    | 86        | 81        | 124       | 88        | 423 |          |
| Dexsil® 300  | Carborane—methyl silicone               |                               | 50        | 450 | 47                    | 80        | 103       | 148       | 96        | 474 | G 33     |
| Dexsil® 400  | Carborane—methyl-phenyl silicone        |                               | 50        | 400 | 72                    | 108       | 118       | 166       | 123       | 587 |          |

**TABLE 11.13** McReynolds' Constants for Stationary Phases in Gas Chromatography (*Continued*)

| Stationary phase  | Chemical type                          | Similar stationary phases  | Temp., °C |     | McReynolds' constants |           |           |           |           |      | USP code |
|---|--|----------------------------|-----------|-----|-----------------------|-----------|-----------|-----------|-----------|------|----------|
|   |  |                            | Min       | Max | <i>x'</i>             | <i>y'</i> | <i>z'</i> | <i>u'</i> | <i>s'</i> | Σ    |          |
| Boiling-point separation of broad molecular weight range of compounds; nonpolar phases ( <i>continued</i> ) |  |                            |           |     |                       |           |           |           |           |      |          |
| OV-7  | 20% Phenyl methyl polysiloxane         | DC 550                     | 0         | 350 | 69                    | 113       | 111       | 171       | 128       | 592  |          |
| SPB-20  | 20% Phenyl methyl polysiloxane         | SPB-35, SPB-1701, DB-1301  | <20       | 300 | 67                    | 116       | 117       | 174       | 131       | 605  |          |
| Di-(2-ethylhexyl)-sebacate  |  |                            | −20       | 125 | 72                    | 168       | 108       | 180       | 125       | 653  | G 11     |
| DC 550  | 25% Phenyl methyl polysiloxane         |                            | 20        | 225 | 81                    | 124       | 124       | 189       | 145       | 663  | G 28     |
| Unsaturated hydrocarbons and other compounds of intermediate polarity                                       |  |                            |           |     |                       |           |           |           |           |      |          |
| Diisodecyl phthalate  |  |                            | 20        | 150 | 84                    | 173       | 137       | 218       | 155       | 767  | G 24     |
| OV-11   | 35% Phenyl methyl polysiloxane         |                            | 0         | 350 | 102                   | 142       | 145       | 219       | 178       | 786  |          |
| OV-1701   | Vinyl methyl polysiloxane              | SPB-1701, SA-1701, DB-1701 | 0         | 250 | 67                    | 170       | 152       | 228       | 171       | 789  |          |
| Poly-I 110  |  |                            |           | 275 | 115                   | 194       | 122       | 204       | 202       | 837  | G 37     |
| SP-2250   | Poly(phenylmethylsiloxane); 50% phenyl | OV-17, DB-17               | 0         | 375 | 119                   | 158       | 162       | 243       | 202       | 884  | G 3      |
| Dexsil® 410   | Carborane—methylcyanoethyl silicone    |                            | 50        | 400 | 72                    | 286       | 174       | 249       | 171       | 952  |          |
| UCON® LB-550-X  | Polyalkylene glycol                    |                            | 20        | 200 | 118                   | 271       | 158       | 243       | 206       | 996  |          |
| UCON LB-1880-X  | Polyalkylene glycol                    |                            |           | 200 | 123                   | 275       | 161       | 249       | 212       | 1020 | G 18     |
| Poly-A 103  |  |                            |           | 275 | 115                   | 331       | 144       | 263       | 214       | 1072 | G 10     |

|                            |  |                 |     |     |     |     |     |     |     |      |      |
|----------------------------|--|-----------------|-----|-----|-----|-----|-----|-----|-----|------|------|
| OV-22                      | Poly(diphenyldimethylsiloxane); 65%:35%        |                 | 0   | 350 | 160 | 188 | 191 | 283 | 253 | 1075 |      |
| Di(2-ethylhexyl) phthalate |  |                 |     | 150 | 135 | 254 | 213 | 320 | 235 | 1157 | G 22 |
| OV-25                      | Poly(diphenyldimethylsiloxane); 75%:25%        |                 | 0   | 350 | 178 | 204 | 208 | 305 | 280 | 1175 | G 17 |
| Moderately polar compounds |  |                 |     |     |     |     |     |     |     |      |      |
| DC QF-1                    |  |                 | 0   | 250 | 144 | 233 | 355 | 463 | 305 | 1500 |      |
| OV-210                     | 50% Trifluoropropylmethylpolysiloxane          | SP-2401, DB-210 | 0   | 275 | 146 | 238 | 358 | 468 | 310 | 1520 | G 6  |
| OV-215                     | Poly(trifluoropropylmethylsiloxane)            |                 | 0   | 275 | 149 | 240 | 363 | 478 | 315 | 1545 |      |
| UCON-50-HB-2000            | Polyalkylene glycol                            |                 | 0   | 200 | 202 | 394 | 253 | 392 | 341 | 1582 |      |
| Triton® X-100              | Octylphenoxy polyethoxy ethanol                |                 | 0   | 190 | 203 | 399 | 268 | 402 | 362 | 1634 |      |
| UCON 50-HB-5100            | Polyglycol                                     |                 | 0   | 200 | 214 | 418 | 278 | 421 | 375 | 1706 |      |
| XE-60                      | Poly(cyanoethylphenylmethylsiloxane)           |                 | 0   | 250 | 204 | 381 | 340 | 493 | 367 | 1785 | G 26 |
| OV-225                     | 25% Cyanopropyl 25% phenyl methyl polysiloxane | DB-225, DB-23   | 0   | 265 | 228 | 369 | 338 | 492 | 386 | 1813 | G 19 |
| Ipegal CO-880              | Nonylphenoxy poly(ethyleneoxy)ethanol          |                 | 100 | 200 | 259 | 461 | 311 | 482 | 426 | 1939 | G 31 |
| Triton® X-305              | Octylphenoxy polyethoxy ethanol                |                 | 200 | 250 | 262 | 467 | 314 | 488 | 430 | 1961 |      |

**TABLE 11.13** McReynolds' Constants for Stationary Phases in Gas Chromatography (*Continued*)

| Stationary phase | Chemical type                               | Similar stationary phases | Temp., °C |     | McReynolds' constants |      |      |      |      |          | USP code |
|------------------|---|---------------------------|-----------|-----|-----------------------|------|------|------|------|----------|----------|
|                  |   |                           | Min       | Max | $x'$                  | $y'$ | $z'$ | $u'$ | $s'$ | $\Sigma$ |          |
| Polar compounds  |   |                           |           |     |                       |      |      |      |      |          |          |
| Hi-EFF-3BP       | Neopentylglycol succinate                   |                           | 50        | 230 | 272                   | 469  | 366  | 539  | 474  | 2120     | G 21     |
| Carbowax 20M-TPA | Polyethyleneglycol + terephthalic acid      |                           | 60        | 250 | 321                   | 367  | 368  | 573  | 520  | 2149     | G 25     |
| Supelcowax™ 10   | Polyethyleneglycol + terephthalic acid      | DB-WAX, SA-WAX            | 50        | 280 | 305                   | 551  | 360  | 562  | 484  | 2262     |          |
| SP-1000          | Polyethyleneglycol + terephthalic acid      |                           | 60        | 220 | 304                   | 552  | 359  | 549  | 498  | 2262     |          |
| Carbowax 20M     | Polyethyleneglycol                          | SP-2300                   | 25        | 275 | 322                   | 536  | 368  | 572  | 510  | 2308     | G 16     |
| Nukol™           |   | SP-1000, FFAP, OV-351     |           |     | 311                   | 572  | 374  | 572  | 520  | 2349     |          |
| Carbowax 3350    |   | Formerly Carbowax 4000    | 60        | 200 | 325                   | 551  | 375  | 582  | 520  | 2353     | G 15     |
| OV-351           | Polyethyleneglycol + nitroterephthalic acid | SP-1000                   | 50        | 270 | 335                   | 552  | 382  | 583  | 540  | 2392     |          |
| SP-2300          | 36% Cyanopropyl                             |                           | 25        | 275 | 316                   | 495  | 446  | 637  | 530  | 2424     |          |
| Silar 5 CP       | 50% Cyanopropyl phenyl silicone             | SP-2300                   | 0         | 250 | 319                   | 495  | 446  | 637  | 531  | 2428     | G 7      |

|  |                                |                |      |     |     |     |     |     |     |      |      |
|--|--------------------------------|----------------|------|-----|-----|-----|-----|-----|-----|------|------|
| FFAP   |                                |                | 50   | 250 | 340 | 580 | 397 | 602 | 627 | 2546 | G 35 |
| Hi-EFF-10BP  | Phenyldiethanolamine succinate |                | 20   | 230 | 386 | 555 | 472 | 674 | 656 | 2744 | G 21 |
| Carbowax 1450  |                                | Formerly 1540  | 50   | 175 | 371 | 639 | 453 | 666 | 641 | 2770 | G 14 |
| SP-2380  |                                |                |      |     | 402 | 629 | 520 | 744 | 623 | 2918 |      |
| SP-2310  | 55% Cyanopropyl                | Silar 7 CP     | 25   | 275 | 440 | 637 | 605 | 840 | 670 | 3192 |      |
| SP-2330  | 68% Cyanopropyl                | SP-2331, SH-60 | 25   | 275 | 490 | 725 | 630 | 913 | 778 | 3536 |      |
| Silar 9 CP   | 90% Cyanopropyl phenyl         |                | 50   | 250 | 489 | 725 | 631 | 913 | 778 | 3536 | G 8  |
| Hi-EFF-1BP   | Diethyleneglycol succinate     |                | 20   | 200 | 499 | 751 | 593 | 840 | 860 | 3543 | G 4  |
| SP-2340  | 75% Cyanopropyl phenyl         | OV-275, SH-80  | < 25 | 275 | 520 | 757 | 659 | 942 | 800 | 3678 |      |
| Silar 10 CP  | 100% Cyanopropyl silicone      | SP-2340        | 25   | 275 | 523 | 757 | 659 | 942 | 801 | 3682 | G 5  |
| THEED  | Amino alcohol                  |                | 0    | 125 | 463 | 942 | 626 | 801 | 893 | 3725 |      |
| OV-275   | Dicyanoallylsilicone           |                | 25   | 250 | 629 | 872 | 763 | 110 | 849 | 4219 |      |
| Absolute index values on squalane for reference compounds: |                                |                |      | 653 | 590 | 627 | 652 |     | 699 |      |      |

**Note:** USP code is the United States Pharmacopeia designation.

### 11.4.1 McReynolds' Constants

The *Kovats retention indices* (R.I.) indicate where compounds will appear on a chromatogram with respect to unbranched alkanes injected with the sample. By definition, the R.I. for pentane is 500, for hexane is 600, for heptane is 700, and so on, regardless of the column used or the operating conditions, although the exact conditions and column must be specified, such as liquid loading, particular support used, and any pretreatment. For example, suppose that on a 20% squalane column at 100°C, the retention times for hexane, benzene, and octane are found to be 15, 16, and 25 min, respectively. On a graph of  $\ln t'_R$  (napierian logarithm of the adjusted retention time) of the alkanes versus their retention indices, a R.I. of 653 for benzene is read off the graph. The number 653 for benzene (see last line of Table 11.13 in the column headed "1" under "Reference compounds") means that it elutes halfway between hexane and heptane on a logarithmic time scale. If the experiment is repeated with a dinonyl phthalate column, the R.I. for benzene is found to be 736 (lying between heptane and octane), which implies that dinonyl phthalate will retard benzene slightly more than squalane will; that is, dinonyl phthalate is slightly more polar than squalane by  $\Delta I = 83$  units (the entry in Table 11.13 for dinonyl phthalate in the column headed "1" under "Reference compounds"). The difference gives a measure of solute-solvent interaction due to all intermolecular forces other than London dispersion forces. The latter are the principal solute-solvent effects with squalane.

**TABLE 11.14** Characteristics of Selected Supercritical Fluids

| Fluid                  | Critical<br>temperature, K (°C) | Critical<br>pressure, atm (psi) |
|------------------------|---------------------------------|---------------------------------|
| Ammonia                | 406 (133)                       | 111.3 (1636)                    |
| Argon                  | 151 (−122)                      | 48.1 (707)                      |
| Benzene                | 562 (289)                       | 48.3 (710)                      |
| Butane                 | 425 (125)                       | 37.5 (551)                      |
| Carbon dioxide         | 304 (31)                        | 72.8 (1070)                     |
| Carbon disulfide       | 552 (279)                       | 78.0 (1147)                     |
| Chlorotrifluoromethane | 379 (106)                       | 40 (588)                        |
| 2,2-Dimethylpropane    | 434 (161)                       | 31.6 (464)                      |
| Ethane                 | 305 (32)                        | 48.2 (706)                      |
| Fluoromethane          | 318 (45)                        | 58.0 (853)                      |
| Heptane                | 540 (267)                       | 27.0 (397)                      |
| Hexane                 | 507 (234)                       | 29.3 (431)                      |
| Hydrogen sulfide       | 373 (100)                       | 88.2 (1296)                     |
| Krypton                | 209 (−64)                       | 54.3 (798)                      |
| Methane                | 191 (−82)                       | 45.4 (667)                      |
| Methanol               | 513 (240)                       | 79.9 (1175)                     |
| 2-Methylpropane        | 408 (65)                        | 36.0 (529)                      |
| Nitrogen               | 126 (−147)                      | 33.5 (492)                      |
| Nitrogen(I) oxide      | 310 (37)                        | 71.5 (1051)                     |
| Pentane                | 470 (197)                       | 33.3 (490)                      |
| Propane                | 470 (197)                       | 41.9 (616)                      |
| Sulfur dioxide         | 431 (158)                       | 77.8 (1144)                     |
| Sulfur hexafluoride    | 319 (46)                        | 37.1 (545)                      |
| Trichloromethane       | 536 (263)                       | 54.9 (807)                      |
| Trifluoromethane       | 299 (26)                        | 47.7 (701)                      |
| Water                  | 647 (374)                       | 217.6 (3199)                    |
| Xenon                  | 290 (17)                        | 57.6 (847)                      |

Now the overall effects due to hydrogen bonding, dipole moment, acid-base properties, and molecular configuration can be expressed as

$$\sum \Delta I = ax' + by' + cz' + du' + es'$$

where  $x' = \Delta I$  for benzene (the column headed "1" in Table 11.13, intermolecular forces typical of aromatics and olefins),  $y' = \Delta I$  for 1-butanol (the column headed "2" in Table 11.13, electron attraction typical of alcohols, nitriles, acids, and nitro and alkyl monochlorides, dichlorides and trichlorides),  $z' = \Delta I$  for 2-pentanone (the column headed "3" in Table 11.13, electron repulsion typical of ketones, ethers, aldehydes, esters, epoxides, and dimethylamino derivatives),  $u' = \Delta I$  for 1-nitropropane (the column headed "4" in Table 11.13, typical of nitro and nitrile derivatives), and  $s' = \Delta I$  for pyridine (or dioxane) (the column headed "5" in Table 11.13).

## 11.4.2 Chromatographic Behavior of Solutes

**11.4.2.1 Retention Behavior.** On a chromatogram the distance on the time axis from the point of sample injection to the peak of an eluted component is called the *uncorrected retention time*  $t_R$ . The corresponding retention volume is the product of retention time and flow rate, expressed as volume of mobile phase per unit time:

$$V_R = t_R F_c$$

The *average linear velocity*  $u$  of the mobile phase in terms of the column length  $L$  and the average linear velocity of eluent  $t_M$  (which is measured by the transit time of a nonretained solute) is

$$u = \frac{L}{t_M}$$

The *adjusted retention time*  $t'_R$  is given by

$$t'_R = t_R - t_M$$

When the mobile phase is a gas, a *compressibility factor*  $j$  must be applied to the adjusted retention volume to give the *net retention volume*:

$$V_N = jV'_R$$

The compressibility factor is expressed by

$$j = \frac{3[(P_i/P_o)^2 - 1]}{2[(P_i/P_o)^3 - 1]}$$

where  $P_i$  is the carrier gas pressure at the column inlet and  $P_o$  that at the outlet.

**11.4.2.2 Partition Ratio.** The partition ratio is the additional time a solute band takes to elute, as compared with an unretained solute (for which  $k' = 0$ ), divided by the elution time of an unretained band:

$$k' = \frac{t_R - t_M}{t_M} = \frac{V_R - V_M}{V_M}$$

Retention time may be expressed as

$$t_R = t_M(1 + k') = \frac{L}{u}(1 + k')$$

**11.4.2.3 Relative Retention.** The relative retention  $\alpha$  of two solutes, where solute 1 elutes before solute 2, is given variously by

$$\alpha = \frac{k'_2}{k'_1} = \frac{V'_{R,2}}{V'_{R,1}} = \frac{t'_{R,2}}{t'_{R,1}}$$

The relative retention is dependent on (1) the nature of the stationary and mobile phases and (2) the column operating temperature.

**11.4.2.4 Column Efficiency.** Under ideal conditions the profile of a solute band resembles that given by a Gaussian distribution curve (Fig. 11.1). The efficiency of a chromatographic system is expressed by the effective plate number  $N_{\text{eff}}$ , defined from the chromatogram of a single band,

$$N_{\text{eff}} = \frac{L}{H} = 16 \left( \frac{t'_R}{W_b} \right)^2 = 5.54 \left( \frac{t'_R}{W_{1/2}} \right)^2$$

where  $L$  is the column length,  $H$  is the plate height,  $t'_R$  is the adjusted time for elution of the band center,  $W_b$  is the width at the base of the peak ( $W_b = 4\sigma$ ) as determined from the intersections of tangents to the inflection points with the baseline, and  $W_{1/2}$  is the width at half the peak height. Column efficiency, when expressed as the number of theoretical plates  $N_{\text{theor}}$  uses the uncorrected retention time in the foregoing expression. The two column efficiencies are related by

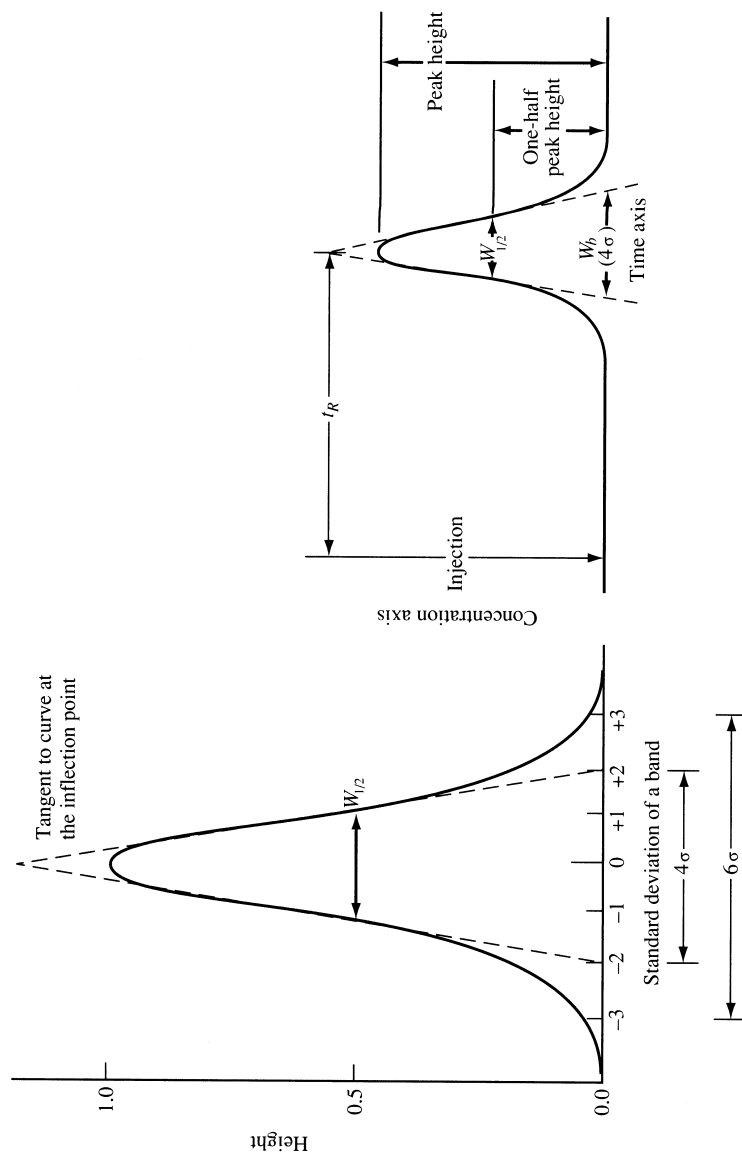
$$N_{\text{eff}} = N_{\text{theor}} \left( \frac{k'}{k' + 1} \right)^2$$

**11.4.2.5 Band Asymmetry.** The peak asymmetry factor  $AF$  is often defined as the ratio of peak half-widths at 10% of peak height, that is, the ratio  $b/a$ , as shown in Fig. 11.2. When the asymmetry ratio lies outside the range 0.95–1.15 for a peak of  $k' = 2$ , the effective plate number should be calculated from the expression

$$N = \frac{41.7(t'_R/W_{0.1})}{(a/b) + 1.25}$$

**11.4.2.6 Resolution.** The degree of separation or resolution,  $R_s$ , of two adjacent peaks is defined as the distance between band peaks (or centers) divided by the average bandwidth using  $W_b$ , as shown in Fig. 11.3.

$$R_s = \frac{t_{R,2} - t_{R,1}}{0.5(W_2 + W_1)}$$



**FIGURE 11.1** Profile of a solute band.

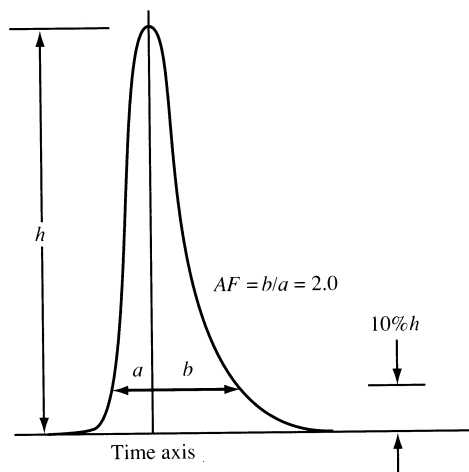


FIGURE 11.2 Band asymmetry.

For reasonable quantitative accuracy, peak maxima must be at least  $4\sigma$  apart. If so, then  $R_s = 1.0$ , which corresponds approximately to a 3% overlap of peak areas. A value of  $R_s = 1.5$  (for  $6\sigma$ ) represents essentially complete resolution with only 0.2% overlap of peak areas. These criteria pertain to roughly equal solute concentrations.

The fundamental resolution equation incorporates the terms involving the thermodynamics and kinetics of the chromatographic system:

$$R_s = \frac{1}{4} \left( \frac{\alpha - 1}{\alpha} \right) \left( \frac{k'}{1 + k'} \right) \left( \frac{L}{H} \right)^{1/2}$$

Three separate factors affect resolution: (1) a column selectivity factor that varies with  $\alpha$ , (2) a capacity factor that varies with  $k'$  (taken usually as  $k_2$ ), and (3) an efficiency factor that depends on the theoretical plate number.

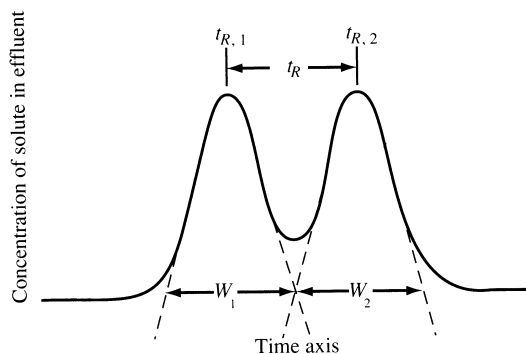


FIGURE 11.3 Definition of resolution.

**11.4.2.7 Time of Analysis.** The retention time required to perform a separation is given by

$$t_R = 16Rs^2 \left( \frac{\alpha}{\alpha - 1} \right)^2 \left[ \frac{(1 + k')^3}{(k')^2} \right] \left( \frac{H}{u} \right)$$

Now  $t_R$  is a minimum when  $k' = 2$ , that is, when  $t_R = 3t_M$ . There is little increase in analysis time when  $k'$  lies between 1 and 10. A twofold increase in the mobile-phase velocity roughly halves the analysis time (actually it is the ratio  $H/u$  which influences the analysis time). The ratio  $H/u$  can be obtained from the experimental plate height/velocity graph.

**11.4.2.8 High-Performance Liquid Chromatography.** Typical performances for various experimental conditions are given in Table 11.15. The data assume these reduced parameters:  $h = 3$ ,  $v = 4.5$ . The *reduced plate height* is

$$h = \frac{H}{d_p} = \frac{L}{Nd_p}$$

The *reduced velocity* of the eluent is

$$v = \frac{ud_p}{D_M} = \frac{Ld_p}{t_M D_M}$$

In these expressions,  $d_p$  is the particle diameter of the stationary phase that constitutes one plate height.  $D_M$  is the diffusion coefficient of the solute in the mobile phase.

**TABLE 11.15** Typical Performances in HPLC for Various Conditions

| Performances |           | Column parameters |                       |                 |
|--------------|-----------|-------------------|-----------------------|-----------------|
| $N$          | $t_M$ , s | $L$ , cm          | $d_p$ , $\mu\text{m}$ | $P$ , atm (psi) |
| 2 500        | 30        | 2.3               | 3                     | 18.4 (270)      |
| 2 500        | 30        | 3.7               | 5                     | 18.4 (270)      |
| 2 500        | 30        | 7.5               | 10                    | 18.4 (270)      |
| 5 000        | 30        | 4.5               | 3                     | 74 (1088)       |
| 5 000        | 30        | 7.5               | 5                     | 74 (1088)       |
| 5 000        | 30        | 15.0              | 10                    | 74 (1088)       |
| 10 000       | 30        | 9.0               | 3                     | 300 (4410)      |
| 10 000       | 30        | 15.0              | 5                     | 300 (4410)      |
| 10 000       | 30        | 30.0              | 10                    | 300 (4410)      |
| 10 000       | 30        | 9.0               | 3                     | 300 (4410)      |
| 10 000       | 60        | 9.0               | 3                     | 150 (2200)      |
| 10 000       | 90        | 9.0               | 3                     | 100 (1470)      |
| 15 000       | 90        | 2.3               | 3                     | 223 (3275)      |
| 15 000       | 120       | 2.3               | 3                     | 167 (2459)      |
| 11 100       | 30        | 10.0              | 3                     | 369 (5420)      |
| 11 100       | 37        | 10.0              | 3                     | 300 (4410)      |
| 11 100       | 101       | 10.0              | 3                     | 100 (1470)      |
| 27 800       | 231       | 25.0              | 3                     | 300 (4410)      |

Assumed reduced parameters:  $h = 3$ ,  $v = 4.5$ . These are optimum values from a graph of reduced plate height versus reduced linear velocity of the mobile phase.

### 11.4.3 Ion-Exchange (Normal Pressure, Columnar)

Ion-exchange methods are based essentially on a reversible exchange of ions between an external liquid phase and an ionic solid phase. The solid phase consists of a polymeric matrix, insoluble, but permeable, which contains fixed charge groups and mobile counter ions of opposite charge. These counter ions can be exchanged for other ions in the external liquid phase. Enrichment of one or several of the components is obtained if selective exchange forces are operative. The method is limited to substances at least partially in ionized form.

**11.4.3.1 Chemical Structure of Ion-Exchange Resins.** An ion-exchange resin usually consists of polystyrene copolymerized with divinylbenzene to build up an inert three-dimensional, cross-linked matrix of hydrocarbon chains. Protruding from the polymer chains are the ion-exchange sites distributed statistically throughout the entire resin particle. The ionic sites are balanced by an equivalent number of mobile counter ions. The type and strength of the exchanger is determined by these active groups. Ion-exchangers are designated anionic or cationic, according to whether they have an affinity for negative or positive counter ions. Each main group is further subdivided into strongly or weakly ionized groups. A selection of commercially available ion-exchange resins is given in Table 11.16.

The cross-linking of a polystyrene resin is expressed as the proportion by weight percent of divinylbenzene in the reaction mixture; for example, "×8" for 8 percent cross-linking. As the percentage is increased, the ionic groups come into effectively closer proximity, resulting in increased selectivity. Intermediate cross-linking, in the range of 4 to 8 percent, is usually used. An increase in cross-linking decreases the diffusion rate in the resin particles; the diffusion rate is the rate-controlling step in column operations. Decreasing the particle size reduces the time required for attaining equilibrium, but at the same time decreases the flow rate until it is prohibitively slow unless pressure is applied.

In most inorganic chromatography, resins of 100 to 200 mesh size are suitable; difficult separations may require 200 to 400 mesh resins. A flow rate of  $1 \text{ mL} \cdot \text{cm}^{-2} \cdot \text{min}^{-1}$  is often satisfactory. With HPLC columns, the flow rate in long columns of fine adsorbent can be increased by applying pressure.

**11.4.3.1.1 Macroreticular Resins.** Macroreticular resins are an agglomerate of randomly packed microspheres which extend through the agglomerate in a continuous non-gel pore structure. The channels throughout the rigid pore structure render the bead centers accessible even in non-aqueous solvents, in which microreticular resins do not swell sufficiently. Because of their high porosity and large pore diameters, these resins can handle large organic molecules.

**11.4.3.1.2 Microreticular Resins.** Microreticular resins, by contrast, are elastic gels that, in the dry state, avidly absorb water and other polar solvents in which they are immersed. While taking up solvent, the gel structure expands until the retractile stresses of the distended polymer network balance the osmotic effect. In nonpolar solvents, little or no swelling occurs and diffusion is impaired.

**11.4.3.1.3 Ion-Exchange Membranes.** Ion-exchange membranes are extremely flexible, strong membranes, composed of analytical grade ion-exchange resin beads (90%) permanently enmeshed in a poly(tetrafluoroethylene) membrane (10%). The membranes offer an alternative to column and batch methods, and can be used in many of the same applications as traditional ion exchange resins. Three ion-exchange resin types have been incorporated into membranes: AG 1-X8, AG 50W-X8, and Chelex 100.

#### 11.4.3.2 Functional Groups

*Sulfonate exchangers* contain the group  $\text{—SO}_3^-$ , which is strongly acidic and completely dissociated whether in the H form or the cation form. These exchangers are used for cation exchange.

**TABLE 11.16** Guide to Ion-Exchange Resins

Dowex is the trade name of Dow resins; X (followed by a numeral) is percent cross-linked. Mesh size (dry) are available in the range 50 to 100, 100 to 200, 200 to 400, and sometimes minus 400.

S-DVB is the acronym for styrene-divinylbenzene.

MP is the acronym for macroporous resin. Mesh size (dry) is available in the range 20 to 50, 100 to 200, and 200 to 400.

Bio-Rex is the trade name for certain resins sold by Bio-Rad Laboratories.

Amberlite and Duolite are trade names of Rohm & Haas resins.

| Resin type and nominal percent cross-linkage                                    | Minimum wet capacity, mequiv · mL <sup>-1</sup> | Density (nominal), g · mL <sup>-1</sup> | Comments  |
|---|---|---|---|
| Anion exchange resins—gel type—strongly basic—quaternary ammonium functionality |   |   |   |
| Dowex 1-X2  | 0.6   | 0.65                                    | Strongly basic anion exchanger with S-DVB matrix for separation of small peptides, nucleotides, and large metal complexes. Molecular weight exclusion is <2700.                             |
| Dowex 1-X4  | 1.0   | 0.70                                    | Strongly basic anion exchanger with S-DVB matrix for separation of organic acids, nucleotides, phosphoinositides, and other anions. Molecular weight exclusion is <1400.                    |
| Dowex 1-X8  | 1.2   | 0.75                                    | Strongly basic anion exchanger with S-DVB matrix for separation of inorganic and organic anions with molecular weight exclusion <1000. 100–200 mesh is standard for analytical separations. |
| Dowex 2-X8  | 1.2   | 0.75                                    | Strongly basic (but less basic than Dowex 1 type) anion exchanger with S-DVB matrix for deionization of carbohydrates and separation of sugars, sugar alcohols, and glycosides.             |
| Amberlite IRA-400   | 1.4   | 1.11                                    | 8% cross-linkage. Used for systems essentially free of organic materials.   |
| Amberlite IRA-402   | 1.3   | 1.07                                    | Lower cross-linkage than IRA-400; better diffusion rate with large organic molecules.   |
| Amberlite IRA-410   | 1.4   | 1.12                                    | Dimethylethanolamine functionality and slightly lower basicity than IRA-400.  |
| Amberlite IRA-458   | 1.2   | 1.08                                    | Has an acrylic structure rather than S-DVB; hence more hydrophilic and resistant to organic fouling.  |
| Anion exchange resin—gel type—intermediate basicity                             |   |   |   |
| Bio-Rex 5   | 2.8   | 0.70                                    | Intermediate basic anion exchanger with primarily tertiary amines on a polyalkyle-neamine matrix for separation of organic acids.   |

**TABLE 11.16** Guide to Ion-Exchange Resins (*Continued*)

| Resin type and nominal percent cross-linkage                                | Minimum wet capacity, mequiv · mL <sup>-1</sup> | Density (nominal), g · mL <sup>-1</sup> | Comments  |
|---|---|---|---|
| Anion exchange resins—gel type—weakly basic—polyamine functionality         |   |   |   |
| Dowex 4-X4  | 1.6   | 0.70                                    | Weakly basic anion exchanger with tertiary amines on an acrylic matrix for the deionization of carbohydrates. Use at pH <7.   |
| Amberlite IRA-68  | 1.6   | 1.06                                    | Acrylic-DVB with unusually high capacity for large organic molecules.   |
| Cation exchange resins—gel type—strongly acidic—sulfonic acid functionality |   |   |   |
| Dowex 50W-X2  | 0.6   | 0.70                                    | Strongly acidic cation exchanger with S-DVB matrix for separation of peptides, nucleotides, and cations. Molecular weight exclusion <2700.  |
| Dowex 50W-X4  | 1.1   | 0.80                                    | Strongly acidic cation exchanger with S-DVB matrix for separation of amino acids, nucleosides and cations. Molecular weight exclusion is <1400.   |
| Dowex 50W-X8  | 1.7   | 0.80                                    | Strongly acidic cation exchanger with S-DVB matrix for separation of amino acids, metal cations, and cations. Molecular weight exclusion is <1000. 100–200 mesh is standard for analytical applications.  |
| Dowex 50W-X12   | 2.1   | 0.85                                    | Strongly acidic cation exchanger with S-DVB matrix used primarily for metal separations.  |
| Dowex 50W-X16   | 2.4   | 0.85                                    | Strongly acidic cation exchanger with S-DVB matrix and high cross linkage.  |
| Amberlite IR-120  | 1.9   | 1.26                                    | 8% styrene-DVB type; high physical stability.   |
| Amberlite IR-122  | 2.1   | 1.32                                    | 10% styrene-DVB type; high physical stability and high capacity.  |
| Weakly acidic cation exchangers—gel type—carboxylic acid functionality      |   |   |   |
| Duolite C-433   | 4.5   | 1.19                                    | Acrylic-DVB type; very high capacity. Used for metals removal and neutralization of alkaline solutions.   |
| Bio-Rex 70  | 2.4   | 0.70                                    | Weakly acidic cation exchanger with carboxylate groups on a macroreticular acrylic matrix for separation and fractionation of proteins, peptides, enzymes, and amines, particularly high molecular weight solutes. Does not denature proteins as do styrene-based resins. |

**TABLE 11.16** Guide to Ion-Exchange Resins (*Continued*)

| Resin type and nominal percent cross-linkage   | Minimum wet capacity, mequiv · mL <sup>-1</sup> | Density (nominal), g · mL <sup>-1</sup> | Comments   |
|--|---|---|--|
| Selective ion exchange resins  |   |   |  |
| Duolite GT-73  | 1.3   | 1.30                                    | Removal of Ag, Cd, Cu, Hg, and Pb.   |
| Amberlite IRA-743A   | 0.6   | 1.05                                    | Boron specific ion exchange resin.   |
| Amberlite IRC-718  | 1.0   | 1.14                                    | Removal of transition metals.  |
| Chelex® 100  | 0.4   | 0.65                                    | Weakly acidic chelating resin with S-DVB matrix for heavy metal concentration.   |
| Anion exchanger—macroreticular type—strongly basic—quaternary ammonium functionality |   |   |  |
| Amberlite IRA-910  | 1.1   | 1.09                                    | Dimethylethanolamine styrene-DVB type which offers slightly less silica removal than Amberlite IRA resin, but offers improved regeneration efficiency. |
| Amberlite IRA-938  | 0.5   | 1.20                                    | Pore size distribution between 2500 and 23 000 nm; suitable for removal of high molecular weight organic materials.                                    |
| Amberlite IRA-958  | 0.8   |   | Acrylic-DVB; resistant to organic fouling.   |
| AG MP-1  | 1.0   | 0.70                                    | Strongly basic macroporous anion exchanger with S-DVB matrix for separation of some enzymes, radioactive anions, and other applications.               |
| Cation exchange resin—macroreticular type—sulfonic acid functionality                |   |   |  |
| Amberlite 200  | 1.7   | 1.26                                    | Styrene-DVB with 20% DVB by weight; superior physical stability and greater resistance to oxidation by factor of three over comparable gel type resin. |
| AG MP-50   | 1.5   | 0.80                                    | Strongly acidic macroporous cation exchanger with S-DVB matrix for separation of radioactive cations and other applications.                           |
| Weak cation exchanger—macroreticular type—carboxylic acid or phenolic functionality  |   |   |  |
| Amberlite DP-1   | 2.5   | 1.17                                    | Methacrylic acid-DVB; high resin capacity. Use pH > 5.   |
| Amberlite IRC-50   | 3.5   | 1.25                                    | Methacrylic acid-DVB. Selectivity adsorbs organic gases such as antibiotics, alkaloids, peptides, and amino acids. Use pH > 5.                         |
| Duolite C-464  | 3.0   | 1.13                                    | Polyacrylic resin with high capacity and outstanding resistance to osmotic shock.  |

**TABLE 11.16** Guide to Ion-Exchange Resins (*Continued*)

| Resin type and nominal percent cross-linkage   | Minimum wet capacity, mequiv $\cdot$ mL <sup>-1</sup> | Density (nominal), g $\cdot$ mL <sup>-1</sup> | Comments   |
|--|---|---|--|
| Weak cation exchanger—macroreticular type—carboxylic acid or phenolic functionality ( <i>continued</i> ) |   |   |  |
| Duolite A-7  | 2.2   | 1.12  | Phenolic type resin. High porosity and hydrophilic matrix. pH range is 0 to 6.   |
| Duolite A-368  | 1.7   | 1.04  | Styrene-DVB; pH range is 0 to 9.   |
| Amberlite IRA-35   | 1.1   |   | Acrylic-DVB; pH range is 0 to 9.   |
| Amberlite IRA-93   | 1.3   | 1.04  | Styrene-DVB; pH range is 0 to 9. Excellent resistance to oxidation and organic fouling.  |
| Liquid amines  |   |   |  |
| Amberlite LA-1   |   |   | A secondary amine containing two highly branched aliphatic chains of M.W. 351 to 393. Solubility is 15 to 20 mg/mL in water. Used as 5 to 40% solutions in hydrocarbons.                     |
| Amberlite LA-2   |   |   | A secondary amine of M.W. 353 to 395. Insoluble in water.  |
| Microcrystalline exchanger   |   |   |  |
| AMP-1  | 4.0   |   | Microcrystalline ammonium molybdo-phosphate with cation exchange capacity of 1.2 mequiv/g. Selectively adsorbs larger alkali metal ions from smaller alkali metal ions, particularly cesium. |
| Ion retardation resin  |   |   |  |
| AG 11 A8   |   | 0.70  | Ion retardation resin containing paired anion (COO <sup>-</sup> ) and cation (CH <sub>3</sub> ) <sub>3</sub> N <sup>+</sup> sites. Selectively retards ionic substances.                     |

**Source:** J. A. Dean, ed., *Analytical Chemistry Handbook*, McGraw-Hill, New York, 1995.

*Carboxylate exchangers* contain —COOH groups which have weak acidic properties and will only function as cation exchangers when the pH is sufficiently high (pH > 6) to permit complete dissociation of the —COOH site. Outside this range the ion exchanger can be used only at the cost of reduced capacity.

*Quaternary ammonium exchangers* contain —R<sub>4</sub>N<sup>+</sup> groups which are strongly basic and completely dissociated in the OH form and the anion form.

*Tertiary amine exchangers* possess —R<sub>3</sub>NH<sub>2</sub> groups which have exchanging properties only in an acidic medium when a proton is bound to the nitrogen atom.

*Aminodiacetate exchangers* have the —N(CH<sub>2</sub>COOH)<sub>2</sub> group which has an unusually high preference for copper, iron, and other heavy metal cations and, to a lesser extent, for alkaline earth

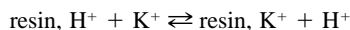
cations. The resin selectivity for divalent over monovalent ions is approximately 5000 to 1. The resin functions as a chelating resin at pH 4 and above. At very low pH, the resin acts as an anion exchanger. This exchanger is the column packing often used for ligand exchange.

**11.4.3.3 Ion-Exchange Equilibrium.** Retention differences among cations with an anion exchanger, or among anions with a cation exchanger, are governed by the physical properties of the solvated ions. The stationary phase will show these preferences:

1. The ion of higher charge.
2. The ion with the smaller solvated radius. Energy is needed to strip away the solvation shell surrounding ions with large hydrated radii, even though their crystallographic ionic radii may be less than the average pore opening in the resin matrix.
3. The ion that has the greater polarizability (which determines the Van der Waals' attraction).

To accomplish any separation of two cations (or two anions) of the same net charge, the stationary phase must show a preference for one more than the other. No variation in the eluant concentration will improve the separation. However, if the exchange involves ions of different net charges, the separation factor does depend on the eluant concentration. The more dilute the counterion concentration in the eluant, the more selective the exchange becomes for polyvalent ions.

In the case of an ionized resin, initially in the H-form and in contact with a solution containing  $K^+$  ions, an equilibrium exists:



which is characterized by the selectivity coefficient,  $k_{K/H}$ :

$$k_{K/H} = \frac{[K^+]_r [H^+]}{[H^+]_r [K^+]}$$

where the subscript  $r$  refers to the resin phase. Table 11.17 contains selectivity coefficients for cations and Table 11.18 for anions. Relative selectivities are of limited use for the prediction of the columnar

**TABLE 11.17** Relative Selectivity of Various Counter Cations

| Counterion                   | Relative selectivity for AG 50W-X8 resin | Counterion       | Relative selectivity for AG 50W-X8 resin |
|------------------------------|--|------------------|--|
| H <sup>+</sup>               | 1.0                                      | Zn <sup>2+</sup> | 2.7                                      |
| Li <sup>+</sup>              | 0.86                                     | Co <sup>2+</sup> | 2.8                                      |
| Na <sup>+</sup>              | 1.5                                      | Cu <sup>2+</sup> | 2.9                                      |
| NH <sub>4</sub> <sup>+</sup> | 1.95                                     | Cd <sup>2+</sup> | 2.95                                     |
| K <sup>+</sup>               | 2.5                                      | Ni <sup>2+</sup> | 3.0                                      |
| Rb <sup>+</sup>              | 2.6                                      | Ca <sup>2+</sup> | 3.9                                      |
| Cs <sup>+</sup>              | 2.7                                      | Sr <sup>2+</sup> | 4.95                                     |
| Cu <sup>+</sup>              | 5.3                                      | Hg <sup>2+</sup> | 7.2                                      |
| Ag <sup>+</sup>              | 7.6                                      | Pb <sup>2+</sup> | 7.5                                      |
| Tl <sup>+</sup>              | 10.7                                     | Ba <sup>2+</sup> | 8.7                                      |
| Mn <sup>2+</sup>             | 2.35                                     | Ce <sup>3+</sup> | 22                                       |
| Mg <sup>2+</sup>             | 2.5                                      | La <sup>3+</sup> | 22                                       |
| Fe <sup>2+</sup>             | 2.55                                     |                  |  |

**TABLE 11.18** Relative Selectivity of Various Counter Anions

| Counterion                                  | Relative selectivity for Dowex 1-X8 resin | Relative selectivity for Dowex 2-X8 resin |
|---|---|---|
| OH <sup>-</sup>                             | 1.0                                       | 1.0                                       |
| Benzenesulfonate <sup>-</sup>               | 500                                       | 75  |
| Salicylate <sup>-</sup>                     | 450                                       | 65  |
| Citrate                                     | 220                                       | 23  |
| I <sup>-</sup>                              | 175                                       | 17  |
| Phenate <sup>-</sup>                        | 110                                       | 27  |
| HSO <sub>4</sub> <sup>-</sup>               | 85  | 15  |
| ClO <sub>3</sub> <sup>-</sup>               | 74  | 12  |
| NO <sub>3</sub> <sup>-</sup>                | 65  | 8   |
| Br <sup>-</sup>                             | 50  | 6   |
| CN <sup>-</sup>                             | 28  | 3   |
| HSO <sub>3</sub> <sup>-</sup>               | 27  | 3   |
| BrO <sub>3</sub> <sup>-</sup>               | 27  | 3   |
| NO <sub>2</sub> <sup>-</sup>                | 24  | 3   |
| Cl <sup>-</sup>                             | 22  | 2.3                                       |
| ClO <sub>4</sub> <sup>-</sup>               | 20  |   |
| SCN <sup>-</sup>                            | 8.0                                       |   |
| HCO <sub>3</sub> <sup>-</sup>               | 6.0                                       | 1.2                                       |
| IO <sub>3</sub> <sup>-</sup>                | 5.5                                       | 0.5                                       |
| H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | 5.0                                       | 0.5                                       |
| Formate <sup>-</sup>                        | 4.6                                       | 0.5                                       |
| Acetate <sup>-</sup>                        | 3.2                                       | 0.5                                       |
| Propanoate <sup>-</sup>                     | 2.6                                       | 0.3                                       |
| F <sup>-</sup>                              | 1.6                                       | 0.3                                       |

exchange behavior of a cation because they do not take account of the influence of the aqueous phase. More specific information about the behavior to be expected from a cation in a column elution experiment is given by the equilibrium distribution coefficient  $K_d$ .

The partitioning of the potassium ion between the resin and solution phases is described by the concentration distribution ratio,  $D_c$ :

$$(D_c)_K = \frac{[K^+]_r}{[K^+]}$$

Combining the equations for the selectivity coefficient and for  $D_c$ :

$$(D_c)_K = k_{K/H} \frac{[H^+]_r}{[H^+]}$$

The foregoing equation reveals that essentially the concentration distribution ratio for trace concentrations of an exchanging ion is independent of the respective solution of that ion and that the uptake of each trace ion by the resin is directly proportional to its solution concentration. However, the

concentration distribution ratios are inversely proportional to the solution concentration of the resin counterion.

To accomplish any separation of two cations (or two anions), one of these ions must be taken up by the resin in distinct preference to the other. This preference is expressed by the separation factor (or relative retention),  $\alpha_{K/Na}$ , using  $K^+$  and  $Na^+$  as the example:

$$\alpha_{K/Na} = \frac{(D_c)_K}{(D_c)_{Na}} = \frac{k_{K/H}}{k_{Na/H}} = K_{K/Na}$$

The more  $\alpha$  deviates from unity for a given pair of ions, the easier it will be to separate them. If the selectivity coefficient is unfavorable for the separation of two ions of the same charge, no variation in the concentration of  $H^+$  (the eluant) will improve the separation.

The situation is entirely different if the exchange involves ions of different net charges. Now the separation factor does depend on the eluant concentration. For example, the more dilute the counterion concentration in the eluant, the more selective the exchange becomes for the ion of higher charge.

In practice, it is more convenient to predict the behavior of an ion, for any chosen set of conditions, by employing a much simpler distribution coefficient,  $D_g$ , which is defined as the concentration of a solute in the resin phase divided by its concentration in the liquid phase, or:

$$D_g = \frac{\text{concentration of solute, resin phase}}{\text{concentration of solute, liquid phase}}$$

$$D_g = \frac{\% \text{ solute within exchanger}}{\% \text{ solute within solution}} \times \frac{\text{volume of solution}}{\text{mass of exchanger}}$$

$D_g$  remains constant over a wide range of resin to liquid ratios. In a relatively short time, by simple equilibration of small known amounts of resin and solution followed by analysis of the phases, the distribution of solutes may be followed under many different sets of experimental conditions. Variables requiring investigation include the capacity and percent cross-linkage of resin, the type of resin itself, the temperature, and the concentration and pH of electrolyte in the equilibrating solution.

By comparing the ratio of the distribution coefficients for a pair of ions, a separation factor (or relative retention) is obtained for a specific experimental condition.

Instead of using  $D_g$ , separation data may be expressed in terms of a volume distribution coefficient  $D_v$ , which is defined as the amount of solution in the exchanger per cubic centimeter of resin bed divided by the amount per cubic centimeter in the liquid phase. The relation between  $D_g$  and  $D_v$  is given by:

$$D_v = D_g \rho$$

where  $\rho$  is the bed density of a column expressed in the units of mass of dry resin per cubic centimeter of column. The bed density can be determined by adding a known weight of dry resin to a graduated cylinder containing the eluting solution. After the resin has swelled to its maximum, a direct reading of the settled volume of resin is recorded.

Intelligent inspection of the relevant distribution coefficients will show whether a separation is feasible and what the most favorable eluant concentration is likely to be. In the columnar mode, an ion, even if not eluted, may move down the column a considerable distance and with the next eluant may appear in the eluate much earlier than indicated by the coefficient in the first eluant alone. A

distribution coefficient value of 12 or lower is required to elute an ion completely from a column containing about 10 g of dry resin using 250 to 300 mL of eluant. A larger volume of eluant is required only when exceptionally strong tailing occurs. Ions may be eluted completely by 300 to 400 mL of eluant from a column of 10 g of dry resin at  $D_g$  values of around 20. The first traces of an element will appear in the eluate at around 300 mL when its  $D_g$  value is about 50 to 60.

*Example* Shaking 50 mL of 0.001  $M$  cesium salt solution with 1.0 g of a strong cation exchanger in the H-form (with a capacity of 3.0 mequiv  $\cdot$  g $^{-1}$ ) removes the following amount of cesium. The selectivity coefficient,  $k_{Cs/H}$ , is 2.56, thus:

$$\frac{[Cs^+]_r[H^+]}{[Cs^+][H^+]_r} = 2.56$$

The maximum amount of cesium which can enter the resin is 50 mL  $\times$  0.001  $M$  = 0.050 equiv. The minimum value of  $[H^+]_r$  = 3.00 – 0.05 = 2.95 mequiv, and the maximum value, assuming complete exchange of cesium ion for hydrogen ion, is 0.001  $M$ . The minimum value of the distribution ratio is:

$$(D_e)_{Cs} = \frac{[Cs^+]_r}{[Cs^+]} = \frac{(2.56)(2.95)}{0.001} = 7550$$

$$\frac{\text{Amount of Cs, resin phase}}{\text{Amount of Cs, solution phase}} = \frac{(7550)(1.0 \text{ g})}{50 \text{ mL}} = 151$$

Thus, at equilibrium the 1.0 g of resin removed is:

$$\frac{100\% - x}{x} = 151$$

with all but 0.66% of cesium ions from solution. If the amount of resin were increased to 2.0 g, the amount of cesium remaining in solution would decrease to 0.33%, half the former value. However, if the depleted solution were decanted and placed in contact with 1 g of fresh resin, the amount of cesium remaining in solution would decrease to 0.004%. Two batch equilibrations would effectively remove the cesium from the solution.

## 11.5 GRAVIMETRIC ANALYSIS

**TABLE 11.19** Gravimetric Factors

In the following table the elements are arranged in alphabetical order.

*Example:* To convert a given weight of  $\text{Al}_2\text{O}_3$  to its equivalent of Al, multiply by the factor at the right, 0.52926; similarly to convert Al to  $\text{Al}_2\text{O}_3$ , multiply by the factor at the left, 1.8894.

| Factor                                     |  | Factor   |
|--|--|----------|
| <b>ALUMINUM</b>                            |  |          |
| <b>Al = 26.9815</b>                        |  |          |
| 0.74971                                    | $\text{Al} \leftrightarrow \text{Al}_4\text{C}_3$  | 1.3341   |
| 0.058728                                   | $\text{Al} \leftrightarrow \text{Al}(\text{C}_9\text{H}_6\text{ON})_3$ (oxinate)   | 17.027   |
| 0.65829                                    | $\text{Al} \leftrightarrow \text{AlN}$   | 1.5191   |
| 1.8894                                     | $\text{Al}_2\text{O}_3 \leftrightarrow \text{Al}$  | 0.52926  |
| 1.4165                                     | $\text{Al}_2\text{O}_3 \leftrightarrow \text{Al}_4\text{C}_3$  | 0.70596  |
| 0.38233                                    | $\text{Al}_2\text{O}_3 \leftrightarrow \text{AlCl}_3$  | 2.6155   |
| 0.41804                                    | $\text{Al}_2\text{O}_3 \leftrightarrow \text{AlPO}_4$  | 2.3921   |
| 0.29800                                    | $\text{Al}_2\text{O}_3 \leftrightarrow \text{Al}_2(\text{SO}_4)_3$   | 3.3557   |
| 0.15300                                    | $\text{Al}_2\text{O}_3 \leftrightarrow \text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$                                  | 6.5361   |
| 0.10746                                    | $\text{Al}_2\text{O}_3 \leftrightarrow \text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$      | 9.3055   |
| 0.11246                                    | $\text{Al}_2\text{O}_3 \leftrightarrow (\text{NH}_4)_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$ | 8.8922   |
| 4.5197                                     | $\text{AlPO}_4 \leftrightarrow \text{Al}$  | 0.22125  |
| 1.3946                                     | $\text{CaF}_2 \leftrightarrow \text{AlF}_3$  | 0.71704  |
| 0.58196                                    | $\text{P}_2\text{O}_5 \leftrightarrow \text{AlPO}_4$   | 1.7183   |
| <b>AMMONIUM</b>                            |  |          |
| <b><math>\text{NH}_4 = 18.03858</math></b> |  |          |
| 1.1013                                     | $\text{Ag} \leftrightarrow \text{NH}_4\text{Br}$   | 0.90802  |
| 2.0166                                     | $\text{Ag} \leftrightarrow \text{NH}_4\text{Cl}$   | 0.49590  |
| 0.74424                                    | $\text{Ag} \leftrightarrow \text{NH}_4\text{I}$  | 1.3437   |
| 1.9171                                     | $\text{AgBr} \leftrightarrow \text{NH}_4\text{Br}$   | 0.52161  |
| 2.6792                                     | $\text{AgCl} \leftrightarrow \text{NH}_4\text{Cl}$   | 0.37323  |
| 1.6198                                     | $\text{AgI} \leftrightarrow \text{NH}_4\text{I}$   | 0.61737  |
| 1.7663                                     | $\text{BaSO}_4 \leftrightarrow (\text{NH}_4)_2\text{SO}_4$   | 0.56615  |
| 0.81583                                    | $\text{Br} \leftrightarrow \text{NH}_4\text{Br}$   | 1.2257   |
| 1.9654                                     | $\text{Cl} \leftrightarrow \text{NH}_4$  | 0.50881  |
| 0.66277                                    | $\text{Cl} \leftrightarrow \text{NH}_4\text{Cl}$   | 1.5088   |
| 0.68162                                    | $\text{HCl} \leftrightarrow \text{NH}_4\text{Cl}$  | 1.4671   |
| 0.87553                                    | $\text{I} \leftrightarrow \text{NH}_4\text{I}$   | 1.1422   |
| 14.410                                     | $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \leftrightarrow \text{NH}_3$   | 0.069398 |
| 13.604                                     | $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \leftrightarrow \text{NH}_4$   | 0.073506 |
| 9.4249                                     | $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \leftrightarrow (\text{NH}_4)_2\text{O}$                                   | 0.10610  |
| 0.82244                                    | $\text{N} \leftrightarrow \text{NH}_3$   | 1.2159   |
| 0.77648                                    | $\text{N} \leftrightarrow \text{NH}_4$   | 1.2879   |
| 0.26185                                    | $\text{N} \leftrightarrow \text{NH}_4\text{Cl}$  | 3.8189   |
| 0.17499                                    | $\text{N} \leftrightarrow \text{NH}_4\text{NO}_3$  | 5.7145   |
| 0.53793                                    | $\text{N} \leftrightarrow (\text{NH}_4)_2\text{O}$   | 1.8590   |
| 0.21200                                    | $\text{N} \leftrightarrow (\text{NH}_4)_2\text{SO}_4$  | 4.7169   |
| 0.94412                                    | $\text{NH}_3 \leftrightarrow \text{NH}_4$  | 1.0592   |
| 0.35449                                    | $\text{NH}_3 \leftrightarrow (\text{NH}_4)_2\text{CO}_3$   | 2.8210   |
| 0.21543                                    | $\text{NH}_3 \leftrightarrow \text{NH}_4\text{HCO}_3$  | 4.6419   |
| 0.21277                                    | $\text{NH}_3 \leftrightarrow \text{NH}_4\text{NO}_3$   | 4.6998   |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                           |  | Factor   |
|----------------------------------|--|----------|
| <b>AMMONIUM (continued)</b>      |  |          |
| <b>NH<sub>4</sub> = 18.03858</b> |  |          |
| 0.65407                          | NH <sub>3</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> O  | 1.5289   |
| 0.48596                          | NH <sub>3</sub> ↔ NH <sub>4</sub> OH   | 2.0578   |
| 0.25777                          | NH <sub>3</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>  | 3.8794   |
| 3.1409                           | NH <sub>4</sub> Cl ↔ NH <sub>3</sub>   | 0.31838  |
| 2.9654                           | NH <sub>4</sub> Cl ↔ NH <sub>4</sub>   | 0.33723  |
| 2.0543                           | NH <sub>4</sub> Cl ↔ (NH <sub>4</sub> ) <sub>2</sub> O   | 0.48677  |
| 1.5263                           | NH <sub>4</sub> Cl ↔ NH <sub>4</sub> OH  | 0.65516  |
| 2.5020                           | NH <sub>4</sub> OH ↔ N   | 0.39967  |
| 1.9428                           | NH <sub>4</sub> OH ↔ NH <sub>4</sub>   | 0.51472  |
| 13.032                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ NH <sub>3</sub>                                      | 0.076737 |
| 12.303                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ NH <sub>4</sub>                                      | 0.081279 |
| 4.1490                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ NH <sub>4</sub> Cl                                   | 0.24102  |
| 2.7728                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ NH <sub>4</sub> NO <sub>3</sub>                      | 0.36065  |
| 8.5235                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> O                    | 0.11732  |
| 6.3328                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ NH <sub>4</sub> OH                                   | 0.15791  |
| 3.3592                           | (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>      | 0.29769  |
| 1.3473                           | (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ↔ H <sub>2</sub> SO <sub>4</sub>                         | 0.74223  |
| 3.1710                           | N <sub>2</sub> O <sub>5</sub> ↔ NH <sub>3</sub>  | 0.31536  |
| 0.67470                          | N <sub>2</sub> O <sub>5</sub> ↔ NH <sub>4</sub> NO <sub>3</sub>  | 1.4821   |
| 2.0740                           | N <sub>2</sub> O <sub>5</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> O  | 0.48215  |
| 5.7275                           | Pt ↔ NH <sub>3</sub>   | 0.17460  |
| 5.4074                           | Pt ↔ NH <sub>4</sub>   | 0.18493  |
| 1.8235                           | Pt ↔ NH <sub>4</sub> Cl  | 0.54838  |
| 1.2187                           | Pt ↔ NH <sub>4</sub> NO <sub>3</sub>   | 0.82058  |
| 3.7462                           | Pt ↔ (NH <sub>4</sub> ) <sub>2</sub> O   | 0.26694  |
| 2.7833                           | Pt ↔ NH <sub>4</sub> OH  | 0.35928  |
| 1.4764                           | Pt ↔ (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>   | 0.67733  |
| 2.3505                           | SO <sub>3</sub> ↔ NH <sub>3</sub>  | 0.42545  |
| 0.60589                          | SO <sub>3</sub> ↔ (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>  | 1.6505   |
| <b>ANTIMONY</b>                  |  |          |
| <b>Sb = 121.760</b>              |  |          |
| 0.36460                          | Sb ↔ KSbO · C <sub>4</sub> H <sub>4</sub> O <sub>6</sub> · ½H <sub>2</sub> O                             | 2.7428   |
| 0.83535                          | Sb ↔ Sb <sub>2</sub> O <sub>4</sub>  | 1.1971   |
| 0.75271                          | Sb ↔ Sb <sub>2</sub> O <sub>5</sub>  | 1.3285   |
| 0.43646                          | Sb <sub>2</sub> O <sub>3</sub> ↔ KSbO · C <sub>4</sub> H <sub>4</sub> O <sub>6</sub> · ½H <sub>2</sub> O | 2.2912   |
| 0.90106                          | Sb <sub>2</sub> O <sub>3</sub> ↔ Sb <sub>2</sub> O <sub>5</sub>  | 1.1098   |
| 0.72184                          | Sb <sub>2</sub> O <sub>3</sub> ↔ Sb <sub>2</sub> S <sub>5</sub>  | 1.3853   |
| 0.46042                          | Sb <sub>2</sub> O <sub>4</sub> ↔ KSbO · C <sub>4</sub> H <sub>4</sub> O <sub>6</sub> · ½H <sub>2</sub> O | 2.1719   |
| 1.2628                           | Sb <sub>2</sub> O <sub>4</sub> ↔ Sb  | 0.79188  |
| 1.0549                           | Sb <sub>2</sub> O <sub>4</sub> ↔ Sb <sub>2</sub> O <sub>3</sub>  | 0.94796  |
| 0.95053                          | Sb <sub>2</sub> O <sub>4</sub> ↔ Sb <sub>2</sub> O <sub>5</sub>  | 1.0520   |
| 0.90523                          | Sb <sub>2</sub> O <sub>4</sub> ↔ Sb <sub>2</sub> S <sub>3</sub>  | 1.1047   |
| 0.76147                          | Sb <sub>2</sub> O <sub>4</sub> ↔ Sb <sub>2</sub> S <sub>5</sub>  | 1.3133   |
| 0.80110                          | Sb <sub>2</sub> O <sub>5</sub> ↔ Sb <sub>2</sub> S <sub>5</sub>  | 1.2483   |
| 0.50862                          | Sb <sub>2</sub> S <sub>3</sub> ↔ KSbO · C <sub>4</sub> H <sub>4</sub> O <sub>6</sub> · ½H <sub>2</sub> O | 1.9661   |
| 1.3950                           | Sb <sub>2</sub> S <sub>3</sub> ↔ Sb  | 0.71683  |
| 1.1653                           | Sb <sub>2</sub> S <sub>3</sub> ↔ Sb <sub>2</sub> O <sub>3</sub>  | 0.85812  |
| 1.0500                           | Sb <sub>2</sub> S <sub>3</sub> ↔ Sb <sub>2</sub> O <sub>5</sub>  | 0.95234  |
| 1.6584                           | Sb <sub>2</sub> S <sub>5</sub> ↔ Sb  | 0.60299  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor              |   | Factor  |
|---------------------|---|---------|
| <b>ARSENIC</b>      |   |         |
| <b>As = 74.9216</b> |   |         |
| 1.3203              | $\text{As}_2\text{O}_3 \leftrightarrow \text{As}$   | 0.75738 |
| 0.86079             | $\text{As}_2\text{O}_3 \leftrightarrow \text{As}_2\text{O}_5$   | 1.1617  |
| 1.5339              | $\text{As}_2\text{O}_5 \leftrightarrow \text{As}$   | 0.65195 |
| 1.6420              | $\text{As}_2\text{S}_3 \leftrightarrow \text{As}$   | 0.60903 |
| 1.2436              | $\text{As}_2\text{S}_3 \leftrightarrow \text{As}_2\text{O}_3$   | 0.80413 |
| 1.0705              | $\text{As}_2\text{S}_3 \leftrightarrow \text{As}_2\text{O}_5$   | 0.93418 |
| 0.79324             | $\text{As}_2\text{S}_3 \leftrightarrow \text{As}_2\text{S}_5$   | 1.2606  |
| 2.0699              | $\text{As}_2\text{S}_5 \leftrightarrow \text{As}$   | 0.48311 |
| 1.5678              | $\text{As}_2\text{S}_5 \leftrightarrow \text{As}_2\text{O}_3$   | 0.63787 |
| 1.3495              | $\text{As}_2\text{S}_5 \leftrightarrow \text{As}_2\text{O}_5$   | 0.74103 |
| 4.6729              | $\text{BaSO}_4 \leftrightarrow \text{As}$   | 0.21400 |
| 3.5392              | $\text{BaSO}_4 \leftrightarrow \text{As}_2\text{O}_3$   | 0.28255 |
| 3.0465              | $\text{BaSO}_4 \leftrightarrow \text{As}_2\text{O}_6$   | 0.32825 |
| 2.8482              | $\text{BaSO}_4 \leftrightarrow \text{AsO}_3$  | 0.35110 |
| 2.5202              | $\text{BaSO}_4 \leftrightarrow \text{AsO}_4$  | 0.39680 |
| 2.0719              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{As}$  | 0.48265 |
| 1.5692              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{As}_2\text{O}_3$                              | 0.63726 |
| 1.3509              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{As}_2\text{O}_5$                              | 0.74032 |
| 1.2629              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{AsO}_2$                                       | 0.79186 |
| 1.1174              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{AsO}_4$                                       | 0.89493 |
| 1.2619              | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{As}_2\text{S}_3$                              | 0.79249 |
| 2.5397              | $\text{MgNH}_4\text{AsO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{As}$             | 0.39374 |
| 1.9235              | $\text{MgNH}_4\text{AsO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{As}_2\text{O}_3$ | 0.51988 |
| 1.6558              | $\text{MgNH}_4\text{AsO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{As}_2\text{O}_5$ | 0.60395 |
| 1.5480              | $\text{MgNH}_4\text{AsO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{AsO}_3$          | 0.64600 |
| 1.3697              | $\text{MgNH}_4\text{AsO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{AsO}_4$          | 0.73008 |
| <b>BARIUM</b>       |   |         |
| <b>Ba = 137.34</b>  |   |         |
| 1.4369              | $\text{BaCO}_3 \leftrightarrow \text{Ba}$   | 0.69592 |
| 0.94766             | $\text{BaCO}_3 \leftrightarrow \text{BaCl}_2$   | 1.0552  |
| 0.76088             | $\text{BaCO}_3 \leftrightarrow \text{Ba}(\text{HCO}_3)_2$   | 1.3143  |
| 1.2871              | $\text{BaCO}_3 \leftrightarrow \text{BaO}$  | 0.77699 |
| 1.8446              | $\text{BaCrO}_4 \leftrightarrow \text{Ba}$  | 0.54214 |
| 1.2165              | $\text{BaCrO}_4 \leftrightarrow \text{BaCl}_2$  | 0.82205 |
| 1.2838              | $\text{BaCrO}_4 \leftrightarrow \text{BaCO}_3$  | 0.77902 |
| 1.6521              | $\text{BaCrO}_4 \leftrightarrow \text{BaO}$   | 0.60530 |
| 2.0345              | $\text{BaSiF}_6 \leftrightarrow \text{Ba}$  | 0.49152 |
| 1.5936              | $\text{BaSiF}_6 \leftrightarrow \text{BaF}_2$   | 0.62751 |
| 1.8222              | $\text{BaSiF}_6 \leftrightarrow \text{BaO}$   | 0.54878 |
| 1.6994              | $\text{BaSO}_4 \leftrightarrow \text{Ba}$   | 0.58843 |
| 1.1208              | $\text{BaSO}_4 \leftrightarrow \text{BaCl}_2$   | 0.89224 |
| 0.95546             | $\text{BaSO}_4 \leftrightarrow \text{BaCl}_2 \cdot 2\text{H}_2\text{O}$                               | 1.0466  |
| 1.1827              | $\text{BaSO}_4 \leftrightarrow \text{BaCO}_3$   | 0.84554 |
| 0.89308             | $\text{BaSO}_4 \leftrightarrow \text{Ba}(\text{NO}_3)_2$  | 1.1197  |
| 1.5221              | $\text{BaSO}_4 \leftrightarrow \text{BaO}$  | 0.65698 |
| 1.3783              | $\text{BaSO}_4 \leftrightarrow \text{BaO}_2$  | 0.72554 |
| 1.3778              | $\text{BaSO}_4 \leftrightarrow \text{BaS}$  | 0.72579 |
| 0.28701             | $\text{CO}_2 \leftrightarrow \text{BaO}$  | 3.4842  |
| 0.22300             | $\text{CO}_2 \leftrightarrow \text{BaCO}_3$   | 4.4842  |

**TABLE 11.19** Gravimetric Factors (*Continued*)

| Factor              |   | Factor   |
|---------------------|---|----------|
| <b>BERYLLIUM</b>    |   |          |
| <b>Be = 9.0122</b>  |   |          |
| 8.8678              | $\text{BeCl}_2 \leftrightarrow \text{Be}$   | 0.11277  |
| 2.7753              | $\text{BeO} \leftrightarrow \text{Be}$  | 0.36033  |
| 0.31296             | $\text{BeO} \leftrightarrow \text{BeCl}_2$  | 3.1953   |
| 0.14119             | $\text{BeO} \leftrightarrow \text{BeSO}_4 \cdot 4\text{H}_2\text{O}$                              | 7.0825   |
| <b>BISMUTH</b>      |   |          |
| <b>Bi = 208.980</b> |   |          |
| 0.89699             | $\text{Bi} \leftrightarrow \text{Bi}_2\text{O}_3$   | 1.1148   |
| 1.6648              | $\text{BiAsO}_4 \leftrightarrow \text{Bi}$  | 0.60069  |
| 1.4933              | $\text{BiAsO}_4 \leftrightarrow \text{Bi}_2\text{O}_4$  | 0.66968  |
| 0.48030             | $\text{Bi}_2\text{O}_3 \leftrightarrow \text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$        | 2.0820   |
| 0.81183             | $\text{Bi}_2\text{O}_3 \leftrightarrow \text{BiONO}_3$  | 1.2318   |
| 1.2462              | $\text{BiOCl} \leftrightarrow \text{Bi}$  | 0.80244  |
| 0.53689             | $\text{BiOCl} \leftrightarrow \text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$                 | 1.8626   |
| 1.1178              | $\text{BiOCl} \leftrightarrow \text{Bi}_2\text{O}_3$  | 0.89460  |
| 0.90748             | $\text{BiOCl} \leftrightarrow \text{BiONO}_3$   | 1.1019   |
| 1.2301              | $\text{Bi}_2\text{S}_3 \leftrightarrow \text{Bi}$   | 0.81291  |
| 1.1034              | $\text{Bi}_2\text{S}_3 \leftrightarrow \text{Bi}_2\text{O}_3$                                     | 0.90627  |
| <b>BORON</b>        |   |          |
| <b>B = 10.81</b>    |   |          |
| 3.2199              | $\text{B}_2\text{O}_3 \leftrightarrow \text{B}$   | 0.31057  |
| 0.81317             | $\text{B}_2\text{O}_3 \leftrightarrow \text{BO}_2$  | 1.2298   |
| 0.59193             | $\text{B}_2\text{O}_3 \leftrightarrow \text{BO}_3$  | 1.6894   |
| 0.89693             | $\text{B}_2\text{O}_3 \leftrightarrow \text{B}_4\text{O}_7$                                       | 1.1149   |
| 0.56298             | $\text{B}_2\text{O}_3 \leftrightarrow \text{H}_3\text{BO}_3$                                      | 1.7763   |
| 0.36510             | $\text{B}_2\text{O}_3 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ | 2.7389   |
| 6.4005              | $\text{B}_6\text{C} \leftrightarrow \text{C}$   | 0.15624  |
| 11.646              | $\text{KBF}_4 \leftrightarrow \text{B}$   | 0.085863 |
| 3.6171              | $\text{KBF}_4 \leftrightarrow \text{B}_2\text{O}_3$   | 0.27647  |
| 2.0363              | $\text{KBF}_4 \leftrightarrow \text{H}_3\text{BO}_3$  | 0.49108  |
| 1.3206              | $\text{KBF}_4 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$         | 0.75723  |
| <b>BROMINE</b>      |   |          |
| <b>Br = 79.90</b>   |   |          |
| 1.3499              | $\text{Ag} \leftrightarrow \text{Br}$   | 0.74079  |
| 0.84333             | $\text{Ag} \leftrightarrow \text{BrO}_3$  | 1.1858   |
| 1.3331              | $\text{Ag} \leftrightarrow \text{HBr}$  | 0.75013  |
| 2.3499              | $\text{AgBr} \leftrightarrow \text{Br}$   | 0.42555  |
| 1.4681              | $\text{AgBr} \leftrightarrow \text{BrO}_3$  | 0.68117  |
| 2.3206              | $\text{AgBr} \leftrightarrow \text{HBr}$  | 0.43091  |
| 0.55756             | $\text{Br} \leftrightarrow \text{AgCl}$   | 1.7935   |
| 9.9892              | $\text{Br} \leftrightarrow \text{O}$  | 0.10010  |
| 1.1858              | $\text{BrO}_3 \leftrightarrow \text{Ag}$  | 0.84333  |
| <b>CADMIUM</b>      |   |          |
| <b>Cd = 112.40</b>  |   |          |
| 0.61317             | $\text{Cd} \leftrightarrow \text{CdCl}_2$   | 1.6309   |
| 0.47545             | $\text{Cd} \leftrightarrow \text{Cd}(\text{NO}_3)_2$  | 2.1033   |
| 1.1423              | $\text{CdO} \leftrightarrow \text{Cd}$  | 0.87539  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                            |  | Factor   |
|-----------------------------------|--|----------|
| <b>CADMIUM (<i>continued</i>)</b> |  |          |
| <b>Cd = 112.40</b>                |  |          |
| 0.70045                           | $\text{CdO} \leftrightarrow \text{CdCl}_2$   | 1.4276   |
| 0.54312                           | $\text{CdO} \leftrightarrow \text{Cd}(\text{NO}_3)_2$  | 1.8412   |
| 1.2852                            | $\text{CdS} \leftrightarrow \text{Cd}$   | 0.77807  |
| 0.78806                           | $\text{CdS} \leftrightarrow \text{CdCl}_2$   | 1.2689   |
| 0.61106                           | $\text{CdS} \leftrightarrow \text{Cd}(\text{NO}_3)_2$  | 1.6365   |
| 1.1251                            | $\text{CdS} \leftrightarrow \text{CdO}$  | 0.88883  |
| 0.69298                           | $\text{CdS} \leftrightarrow \text{CdSO}_4$   | 1.4430   |
| 1.8546                            | $\text{CdSO}_4 \leftrightarrow \text{Cd}$  | 0.53919  |
| 1.1372                            | $\text{CdSO}_4 \leftrightarrow \text{CdCl}_2$  | 0.87935  |
| 0.88177                           | $\text{CdSO}_4 \leftrightarrow \text{Cd}(\text{NO}_3)_2$                                     | 1.1341   |
| 1.6235                            | $\text{CdSO}_4 \leftrightarrow \text{CdO}$   | 0.61595  |
| <b>CALCIUM</b>                    |  |          |
| <b>Ca = 40.08</b>                 |  |          |
| 3.2352                            | $\text{BaSO}_4 \leftrightarrow \text{CaS}$   | 0.30910  |
| 1.7144                            | $\text{BaSO}_4 \leftrightarrow \text{CaSO}_4$  | 0.58329  |
| 1.3556                            | $\text{BaSO}_4 \leftrightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$                      | 0.73766  |
| 0.36111                           | $\text{Ca} \leftrightarrow \text{CaCl}_2$  | 2.7692   |
| 0.51334                           | $\text{Ca} \leftrightarrow \text{CaF}_2$   | 1.9480   |
| 0.71471                           | $\text{Ca} \leftrightarrow \text{CaO}$   | 1.3992   |
| 2.4973                            | $\text{CaCO}_3 \leftrightarrow \text{Ca}$  | 0.40044  |
| 0.90179                           | $\text{CaCO}_3 \leftrightarrow \text{CaCl}_2$  | 1.1089   |
| 0.61742                           | $\text{CaCO}_3 \leftrightarrow \text{Ca}(\text{HCO}_3)_2$                                    | 1.6196   |
| 1.7848                            | $\text{CaCO}_3 \leftrightarrow \text{CaO}$   | 0.56029  |
| 0.73520                           | $\text{CaCO}_3 \leftrightarrow \text{CaSO}_4$  | 1.3602   |
| 0.58134                           | $\text{CaCO}_3 \leftrightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$                      | 1.7202   |
| 1.3726                            | $\text{CaCO}_3 \leftrightarrow \text{HCl}$   | 0.72856  |
| 0.50526                           | $\text{CaO} \leftrightarrow \text{CaCl}_2$   | 1.9792   |
| 0.71825                           | $\text{CaO} \leftrightarrow \text{CaF}_2$  | 1.3923   |
| 0.34593                           | $\text{CaO} \leftrightarrow \text{Ca}(\text{HCO}_3)_2$                                       | 2.8907   |
| 0.75685                           | $\text{CaO} \leftrightarrow \text{Ca}(\text{OH})_2$  | 1.3213   |
| 0.41192                           | $\text{CaO} \leftrightarrow \text{CaSO}_4$   | 2.4276   |
| 0.32572                           | $\text{CaO} \leftrightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$                         | 3.0701   |
| 2.5797                            | $\text{Ca}_3(\text{PO}_4)_2 \leftrightarrow \text{Ca}$                                       | 0.38765  |
| 1.8437                            | $\text{Ca}_3(\text{PO}_4)_2 \leftrightarrow \text{CaO}$                                      | 0.54239  |
| 0.75946                           | $\text{Ca}_3(\text{PO}_4)_2 \leftrightarrow \text{CaSO}_4$                                   | 1.3167   |
| 3.3967                            | $\text{CaSO}_4 \leftrightarrow \text{Ca}$  | 0.29440  |
| 1.2266                            | $\text{CaSO}_4 \leftrightarrow \text{CaCl}_2$  | 0.81526  |
| 1.3602                            | $\text{CaSO}_4 \leftrightarrow \text{CaCO}_3$  | 0.73520  |
| 1.7437                            | $\text{CaSO}_4 \leftrightarrow \text{CaF}_2$   | 0.57351  |
| 2.4276                            | $\text{CaSO}_4 \leftrightarrow \text{CaO}$   | 0.41192  |
| 1.7691                            | $\text{Cl} \leftrightarrow \text{Ca}$  | 0.56526  |
| 0.63885                           | $\text{Cl} \leftrightarrow \text{CaCl}_2$  | 1.5653   |
| 1.2644                            | $\text{Cl} \leftrightarrow \text{CaO}$   | 0.79089  |
| 0.78479                           | $\text{CO}_2 \leftrightarrow \text{CaO}$   | 1.2742   |
| 0.43970                           | $\text{CO}_2 \leftrightarrow \text{CaCO}_3$  | 2.2743   |
| 0.77989                           | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{Ca}_3(\text{AsO}_4)_2$               | 1.2822   |
| 0.71883                           | $\text{MgO} \leftrightarrow \text{CaO}$  | 1.3912   |
| 0.71755                           | $\text{Mg}_3\text{P}_2\text{O}_7 \leftrightarrow \text{Ca}_3(\text{PO}_4)_2$                 | 1.3936   |
| 12.098                            | $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 \leftrightarrow \text{Ca}_3(\text{PO}_4)_2$ | 0.082657 |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                     |   | Factor   |
|----------------------------|---|----------|
| <b>CALCIUM (continued)</b> |   |          |
| <b>Ca = 40.08</b>          |   |          |
| 0.65824                    | $\text{N}_2\text{O}_5 \leftrightarrow \text{Ca}(\text{NO}_3)_2$       | 1.5192   |
| 0.45761                    | $\text{P}_2\text{O}_3 \leftrightarrow \text{Ca}_3(\text{PO}_4)_2$     | 2.1853   |
| 1.4277                     | $\text{SO}_3 \leftrightarrow \text{CaO}$                              | 0.70044  |
| 0.58809                    | $\text{SO}_3 \leftrightarrow \text{CaSO}_4$                           | 1.7004   |
| 0.46502                    | $\text{SO}_3 \leftrightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ | 2.1505   |
| 0.80523                    | $\text{WO}_3 \leftrightarrow \text{CaWO}_4$                           | 1.2419   |
| <b>CARBON</b>              |   |          |
| <b>C = 12.011</b>          |   |          |
| 3.9913                     | $\text{Ag} \leftrightarrow \text{HCN}$                                | 0.25054  |
| 1.6565                     | $\text{Ag} \leftrightarrow \text{KCN}$                                | 0.60369  |
| 4.9541                     | $\text{AgCN} \leftrightarrow \text{HCN}$                              | 0.20185  |
| 2.0561                     | $\text{AgCN} \leftrightarrow \text{KCN}$                              | 0.48637  |
| 16.431                     | $\text{BaCO}_3 \leftrightarrow \text{C}$                              | 0.060861 |
| 4.4842                     | $\text{BaCO}_3 \leftrightarrow \text{CO}_2$                           | 0.22301  |
| 3.2887                     | $\text{BaCO}_3 \leftrightarrow \text{CO}_3$                           | 0.30407  |
| 3.4842                     | $\text{BaO} \leftrightarrow \text{CO}_2$                              | 0.28701  |
| 1.7421                     | $\text{BaO} \leftrightarrow \text{CO}_2$ , bicarbonate                | 0.57402  |
| 0.19432                    | $\text{CN} \leftrightarrow \text{AgCN}$                               | 5.1461   |
| 0.24120                    | $\text{CN} \leftrightarrow \text{Ag}$                                 | 4.1460   |
| 0.35000                    | $\text{SCN} \leftrightarrow \text{AgSCN}$                             | 2.8572   |
| 0.47757                    | $\text{SCN} \leftrightarrow \text{CuSCN}$                             | 2.0939   |
| 0.24885                    | $\text{SCN} \leftrightarrow \text{BaSO}_4$                            | 4.0185   |
| 1.2742                     | $\text{CaO} \leftrightarrow \text{CO}_2$                              | 0.78479  |
| 0.63712                    | $\text{CaO} \leftrightarrow \text{CO}_2$ , bicarbonate                | 1.5696   |
| 0.33936                    | $\text{CO}_2 \leftrightarrow \text{Ba}(\text{HCO}_3)_2$               | 2.9467   |
| 3.6641                     | $\text{CO}_2 \leftrightarrow \text{C}$                                | 0.27291  |
| 0.43970                    | $\text{CO}_2 \leftrightarrow \text{CaCO}_3$                           | 2.2743   |
| 0.54297                    | $\text{CO}_2 \leftrightarrow \text{Ca}(\text{HCO}_3)_2$               | 1.8417   |
| 0.73341                    | $\text{CO}_2 \leftrightarrow \text{CO}_3$                             | 1.3635   |
| 0.13507                    | $\text{CO}_2 \leftrightarrow \text{Cs}_2\text{CO}_3$                  | 7.4033   |
| 0.22695                    | $\text{CO}_2 \leftrightarrow \text{CsHCO}_3$                          | 4.4063   |
| 0.37986                    | $\text{CO}_2 \leftrightarrow \text{FeCO}_3$                           | 2.6326   |
| 0.49483                    | $\text{CO}_2 \leftrightarrow \text{Fe}(\text{HCO}_3)_2$               | 2.0209   |
| 0.31843                    | $\text{CO}_2 \leftrightarrow \text{K}_2\text{CO}_3$                   | 3.1404   |
| 0.43957                    | $\text{CO}_2 \leftrightarrow \text{KHCO}_3$                           | 2.2749   |
| 0.46718                    | $\text{CO}_2 \leftrightarrow \text{K}_2\text{O}$                      | 2.1405   |
| 0.59564                    | $\text{CO}_2 \leftrightarrow \text{Li}_2\text{CO}_3$                  | 1.6789   |
| 0.64762                    | $\text{CO}_2 \leftrightarrow \text{LiHCO}_3$                          | 1.5441   |
| 1.4730                     | $\text{CO}_2 \leftrightarrow \text{Li}_2\text{O}$                     | 0.67887  |
| 0.52193                    | $\text{CO}_2 \leftrightarrow \text{MgCO}_3$                           | 1.9159   |
| 0.60143                    | $\text{CO}_2 \leftrightarrow \text{Mg}(\text{HCO}_3)_2$               | 1.6627   |
| 1.0918                     | $\text{CO}_2 \leftrightarrow \text{MgO}$                              | 0.91595  |
| 0.38286                    | $\text{CO}_2 \leftrightarrow \text{MnCO}_3$                           | 2.6119   |
| 0.49737                    | $\text{CO}_2 \leftrightarrow \text{Mn}(\text{HCO}_3)_2$               | 2.0106   |
| 0.62041                    | $\text{CO}_2 \leftrightarrow \text{MnO}$                              | 1.6118   |
| 0.41523                    | $\text{CO}_2 \leftrightarrow \text{Na}_2\text{CO}_3$                  | 2.4083   |
| 0.52388                    | $\text{CO}_2 \leftrightarrow \text{NaHCO}_3$                          | 1.9088   |
| 0.71008                    | $\text{CO}_2 \leftrightarrow \text{Na}_2\text{O}$                     | 1.4083   |
| 0.45802                    | $\text{CO}_2 \leftrightarrow (\text{NH}_4)_2\text{CO}_3$              | 2.1833   |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                           |   | Factor  |
|----------------------------------|---|---------|
| <b>CARBON (<i>continued</i>)</b> |   |         |
| <b>C = 12.011</b>                |   |         |
| 0.55669                          | $\text{CO}_2 \leftrightarrow \text{NH}_4\text{HCO}_3$   | 1.7963  |
| 0.16471                          | $\text{CO}_2 \leftrightarrow \text{PbCO}_3$   | 6.0713  |
| 0.19055                          | $\text{CO}_2 \leftrightarrow \text{Rb}_2\text{CO}_3$  | 5.2477  |
| 0.30043                          | $\text{CO}_2 \leftrightarrow \text{RbHCO}_3$  | 3.3286  |
| 0.23542                          | $\text{CO}_2 \leftrightarrow \text{Rb}_2\text{O}$   | 4.2477  |
| 0.29811                          | $\text{CO}_2 \leftrightarrow \text{SrCO}_3$   | 3.3545  |
| 0.41984                          | $\text{CO}_2 \leftrightarrow \text{Sr}(\text{HCO}_3)_2$   | 2.3818  |
| 0.42474                          | $\text{CO}_2 \leftrightarrow \text{SrO}$  | 2.3545  |
| <b>CERIUM</b>                    |   |         |
| <b>Ce = 140.12</b>               |   |         |
| 0.36100                          | $\text{Ce} \leftrightarrow \text{Ce}(\text{NO}_3)_4$  | 2.7701  |
| 0.24746                          | $\text{Ce} \leftrightarrow \text{Ce}(\text{NO}_3)_4 \cdot 2\text{NH}_4\text{NO}_3 \cdot \text{H}_2\text{O}$             | 4.0411  |
| 0.81408                          | $\text{Ce} \leftrightarrow \text{CeO}_2$  | 1.2284  |
| 0.85377                          | $\text{Ce} \leftrightarrow \text{Ce}_2\text{O}_3$   | 1.1713  |
| 0.49302                          | $\text{Ce} \leftrightarrow \text{Ce}_2(\text{SO}_4)_3$  | 2.0283  |
| 1.0527                           | $\text{Ce}_2(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O} \leftrightarrow \text{Ce}_2(\text{SO}_4)_3$              | 0.94998 |
| 2.1351                           | $\text{Ce}_2(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O} \leftrightarrow \text{Ce}$                               | 0.46835 |
| 0.44345                          | $\text{CeO}_2 \leftrightarrow \text{Ce}(\text{NO}_3)_4$   | 2.2551  |
| 0.30397                          | $\text{CeO}_2 \leftrightarrow \text{Ce}(\text{NO}_3)_4 \cdot 2\text{NH}_4\text{NO}_3 \cdot \text{H}_2\text{O}$          | 3.2898  |
| 0.42284                          | $\text{Ce}_2\text{O}_3 \leftrightarrow \text{Ce}(\text{NO}_3)_4$  | 2.3650  |
| 0.28984                          | $\text{Ce}_2\text{O}_3 \leftrightarrow \text{Ce}(\text{NO}_3)_4 \cdot 2\text{NH}_4\text{NO}_3 \cdot \text{H}_2\text{O}$ | 3.4502  |
| 0.95352                          | $\text{Ce}_2\text{O}_3 \leftrightarrow \text{CeO}_2$  | 1.0487  |
| 0.57746                          | $\text{Ce}_2\text{O}_3 \leftrightarrow \text{Ce}_2(\text{SO}_4)_3$  | 1.7317  |
| <b>CESIUM</b>                    |   |         |
| <b>Cs = 137.905</b>              |   |         |
| 0.85127                          | $\text{AgCl} \leftrightarrow \text{CsCl}$   | 1.1747  |
| 0.26675                          | $\text{Cl} \leftrightarrow \text{Cs}$   | 3.7489  |
| 0.21058                          | $\text{Cl} \leftrightarrow \text{CsCl}$   | 4.7488  |
| 0.78944                          | $\text{Cs} \leftrightarrow \text{CsCl}$   | 1.2667  |
| 0.57200                          | $\text{Cs} \leftrightarrow \text{CsClO}_4$  | 1.7483  |
| 0.81585                          | $\text{Cs} \leftrightarrow \text{Cs}_2\text{CO}_3$  | 1.2257  |
| 0.94326                          | $\text{Cs} \leftrightarrow \text{Cs}_2\text{O}$   | 1.0602  |
| 0.83693                          | $\text{Cs}_2\text{O} \leftrightarrow \text{CsCl}$   | 1.1948  |
| 0.77876                          | $\text{Cs}_2\text{O} \leftrightarrow \text{Cs}_2\text{SO}_4$  | 1.2841  |
| 2.5341                           | $\text{Cs}_2\text{PtCl}_6 \leftrightarrow \text{Cs}$  | 0.39461 |
| 2.0005                           | $\text{Cs}_2\text{PtCl}_6 \leftrightarrow \text{CsCl}$  | 0.49987 |
| 2.0675                           | $\text{Cs}_2\text{PtCl}_6 \leftrightarrow \text{Cs}_2\text{CO}_3$   | 0.48369 |
| 2.3903                           | $\text{Cs}_2\text{PtCl}_6 \leftrightarrow \text{Cs}_2\text{O}$  | 0.41835 |
| 1.3613                           | $\text{Cs}_2\text{SO}_4 \leftrightarrow \text{Cs}$  | 0.73457 |
| 1.0747                           | $\text{Cs}_2\text{SO}_4 \leftrightarrow \text{CsCl}$  | 0.93050 |
| 1.1106                           | $\text{Cs}_2\text{SO}_4 \leftrightarrow \text{Cs}_2\text{CO}_3$   | 0.90038 |
| 0.28410                          | $\text{SO}_3 \leftrightarrow \text{Cs}_2\text{O}$   | 3.5199  |
| <b>CHLORINE</b>                  |   |         |
| <b>Cl = 35.453</b>               |   |         |
| 3.0426                           | $\text{Ag} \leftrightarrow \text{Cl}$   | 0.32866 |
| 2.9585                           | $\text{Ag} \leftrightarrow \text{HCl}$  | 0.33801 |
| 4.0425                           | $\text{AgCl} \leftrightarrow \text{Cl}$   | 0.24737 |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                      |  | Factor  |
|-----------------------------|--|---------|
| <b>CHLORINE (continued)</b> |  |         |
| <b>Cl = 35.453</b>          |  |         |
| 3.9308                      | $\text{AgCl} \leftrightarrow \text{HCl}$   | 0.25440 |
| 3.5728                      | $\text{BaCrO}_4 \leftrightarrow \text{Cl}$   | 0.27990 |
| 0.56526                     | $\text{Ca} \leftrightarrow \text{Cl}$  | 1.7691  |
| 0.97235                     | $\text{Cl} \leftrightarrow \text{HCl}$   | 1.0284  |
| 0.58227                     | $\text{ClO}_3 \leftrightarrow \text{AgCl}$   | 1.7174  |
| 1.1193                      | $\text{ClO}_3 \leftrightarrow \text{KCl}$  | 0.89340 |
| 1.4279                      | $\text{ClO}_3 \leftrightarrow \text{NaCl}$   | 0.70033 |
| 0.69391                     | $\text{ClO}_4 \leftrightarrow \text{AgCl}$   | 1.4411  |
| 1.3339                      | $\text{ClO}_4 \leftrightarrow \text{KCl}$  | 0.74967 |
| 1.7017                      | $\text{ClO}_4 \leftrightarrow \text{NaCl}$   | 0.58766 |
| 1.1029                      | $\text{K} \leftrightarrow \text{Cl}$   | 0.90668 |
| 2.1029                      | $\text{KCl} \leftrightarrow \text{Cl}$   | 0.47553 |
| 0.19572                     | $\text{Li} \leftrightarrow \text{Cl}$  | 5.1092  |
| 0.34288                     | $\text{Mg} \leftrightarrow \text{Cl}$  | 2.9165  |
| 1.3429                      | $\text{MgCl}_2 \leftrightarrow \text{Cl}$  | 0.74467 |
| 1.2261                      | $\text{MnO}_2 \leftrightarrow \text{Cl}$   | 0.81560 |
| 0.64846                     | $\text{Na} \leftrightarrow \text{Cl}$  | 1.5421  |
| 1.6485                      | $\text{NaCl} \leftrightarrow \text{Cl}$  | 0.60663 |
| 0.50881                     | $\text{NH}_4 \leftrightarrow \text{Cl}$  | 1.9654  |
| 1.4671                      | $\text{NH}_4\text{Cl} \leftrightarrow \text{HCl}$                                      | 0.68162 |
| 1.8121                      | $(\text{NH}_4)_2\text{SO}_4 \leftrightarrow \text{HCl}$                                | 0.55185 |
| 4.5580                      | $\text{PbCrO}_4 \leftrightarrow \text{Cl}$   | 0.21939 |
| <b>CHROMIUM</b>             |  |         |
| <b>Cr = 51.996</b>          |  |         |
| 4.8721                      | $\text{BaCrO}_4 \leftrightarrow \text{Cr}$   | 0.20525 |
| 3.3335                      | $\text{BaCrO}_4 \leftrightarrow \text{Cr}_2\text{O}_3$                                 | 0.29998 |
| 2.5335                      | $\text{BaCrO}_4 \leftrightarrow \text{CrO}_3$  | 0.39472 |
| 2.1841                      | $\text{BaCrO}_4 \leftrightarrow \text{CrO}_4$  | 0.45786 |
| 0.70718                     | $\text{BaCrO}_4 \leftrightarrow \text{Cr}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ | 1.4141  |
| 7.4935                      | $\text{Cr}_3\text{C}_2 \leftrightarrow \text{Cr}$                                      | 0.13345 |
| 1.9231                      | $\text{CrO}_3 \leftrightarrow \text{Cr}$   | 0.51999 |
| 1.4616                      | $\text{Cr}_2\text{O}_3 \leftrightarrow \text{Cr}$                                      | 0.68420 |
| 0.76000                     | $\text{Cr}_2\text{O}_3 \leftrightarrow \text{CrO}_3$                                   | 1.3158  |
| 0.65519                     | $\text{Cr}_2\text{O}_3 \leftrightarrow \text{CrO}_4$                                   | 1.5263  |
| 3.7349                      | $\text{K}_2\text{CrO}_4 \leftrightarrow \text{Cr}$                                     | 0.26774 |
| 1.9421                      | $\text{K}_2\text{CrO}_4 \leftrightarrow \text{CrO}_3$                                  | 0.51490 |
| 1.4710                      | $\text{K}_2\text{Cr}_2\text{O}_7 \leftrightarrow \text{CrO}_3$                         | 0.67979 |
| 6.2155                      | $\text{PbCrO}_4 \leftrightarrow \text{Cr}$   | 0.16089 |
| 4.2527                      | $\text{PbCrO}_4 \leftrightarrow \text{Cr}_2\text{O}_3$                                 | 0.23515 |
| 3.2320                      | $\text{PbCrO}_4 \leftrightarrow \text{CrO}_3$  | 0.30941 |
| 2.7863                      | $\text{PbCrO}_4 \leftrightarrow \text{CrO}_4$  | 0.35890 |
| 0.90217                     | $\text{PbCrO}_4 \leftrightarrow \text{Cr}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ | 1.1084  |
| 1.6642                      | $\text{PbCrO}_4 \leftrightarrow \text{K}_2\text{CrO}_4$                                | 0.60090 |
| 2.1971                      | $\text{PbCrO}_4 \leftrightarrow \text{K}_2\text{Cr}_2\text{O}_7$                       | 0.45515 |
| <b>COBALT</b>               |  |         |
| <b>Co = 58.9332</b>         |  |         |
| 0.20249                     | $\text{Co} \leftrightarrow \text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$         | 4.9385  |
| 0.78648                     | $\text{Co} \leftrightarrow \text{CoO}$   | 1.2715  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                             |  | Factor  |
|------------------------------------|--|---------|
| <b>COBALT</b> ( <i>continued</i> ) |  |         |
| <b>Co = 58.9332</b>                |  |         |
| 0.20965                            | $\text{Co} \leftrightarrow \text{CoSO}_4 \cdot 7\text{H}_2\text{O}$  | 4.7698  |
| 7.6743                             | $\text{K}_3[\text{Co}(\text{NO}_2)_6] \leftrightarrow \text{Co}$   | 0.13030 |
| 6.0357                             | $\text{K}_3[\text{Co}(\text{NO}_2)_6] \leftrightarrow \text{CoO}$  | 0.16568 |
| 1.3620                             | $\text{Co}_3\text{O}_4 \leftrightarrow \text{Co}$  | 0.73422 |
| 1.0712                             | $\text{Co}_3\text{O}_4 \leftrightarrow \text{CoO}$   | 0.93355 |
| 2.4758                             | $\text{Co}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Co}$  | 0.40391 |
| 1.9471                             | $\text{Co}_2\text{P}_2\text{O}_7 \leftrightarrow \text{CoO}$   | 0.51357 |
| 3.2233                             | $\text{CoNH}_4\text{PO}_4 \cdot \text{H}_2\text{O} \leftrightarrow \text{Co}$                                | 0.31024 |
| 2.5351                             | $\text{CoNH}_4\text{PO}_4 \cdot \text{H}_2\text{O} \leftrightarrow \text{CoO}$                               | 0.39447 |
| 2.6299                             | $\text{CoSO}_4 \leftrightarrow \text{Co}$  | 0.38024 |
| 2.0684                             | $\text{CoSO}_4 \leftrightarrow \text{CoO}$   | 0.48347 |
| 3.7514                             | $\text{CoSO}_4 \cdot 7\text{H}_2\text{O} \leftrightarrow \text{CoO}$   | 0.26657 |
| 7.0656                             | $(\text{CoSO}_4)_2 \cdot (\text{K}_2\text{SO}_4)_3 \leftrightarrow \text{Co}$                                | 0.14153 |
| 5.5569                             | $(\text{CoSO}_4)_2 \cdot (\text{K}_2\text{SO}_4)_3 \leftrightarrow \text{CoO}$                               | 0.17996 |
| <b>COPPER</b>                      |  |         |
| <b>Cu = 63.544</b>                 |  |         |
| 0.25071                            | $\text{Cu} \leftrightarrow \text{Cu}_2\text{C}_2\text{H}_3\text{O}_2 \cdot (\text{AsO}_2)_3$                 | 3.9887  |
| 0.79885                            | $\text{Cu} \leftrightarrow \text{CuO}$   | 1.2518  |
| 0.25449                            | $\text{Cu} \leftrightarrow \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  | 3.9295  |
| 1.9141                             | $\text{CuSCN} \leftrightarrow \text{Cu}$   | 0.52245 |
| 1.5291                             | $\text{CuSCN} \leftrightarrow \text{CuO}$  | 0.65400 |
| 0.31856                            | $\text{CuO} \leftrightarrow \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$   | 3.1391  |
| 1.1259                             | $\text{Cu}_2\text{O} \leftrightarrow \text{Cu}$  | 0.88817 |
| 1.2523                             | $\text{Cu}_2\text{S} \leftrightarrow \text{Cu}$  | 0.79854 |
| 1.0004                             | $\text{Cu}_2\text{S} \leftrightarrow \text{CuO}$   | 0.99961 |
| 1.1122                             | $\text{Cu}_2\text{S} \leftrightarrow \text{Cu}_2\text{O}$  | 0.89908 |
| 0.31869                            | $\text{Cu}_2\text{S} \leftrightarrow \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$                                | 3.1379  |
| 0.91872                            | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{Cu}_2\text{C}_2\text{H}_3\text{O}_2(\text{AsO}_2)_3$ | 1.0885  |
| <b>ERBIUM</b>                      |  |         |
| <b>Er = 167.26</b>                 |  |         |
| 1.1435                             | $\text{Er}_2\text{O}_3 \leftrightarrow \text{Er}$  | 0.87452 |
| <b>FLUORINE</b>                    |  |         |
| <b>F = 18.9984</b>                 |  |         |
| 1.5936                             | $\text{BaSiF}_6 \leftrightarrow \text{BaF}_2$  | 0.62751 |
| 2.4513                             | $\text{BaSiF}_6 \leftrightarrow \text{F}$  | 0.40795 |
| 2.3277                             | $\text{BaSiF}_6 \leftrightarrow 6\text{HF}$  | 0.42960 |
| 1.9392                             | $\text{BaSiF}_6 \leftrightarrow \text{H}_2\text{SiF}_6$  | 0.51568 |
| 2.6847                             | $\text{BaSiF}_6 \leftrightarrow \text{SiF}_4$  | 0.37249 |
| 1.9666                             | $\text{BaSiF}_6 \leftrightarrow \text{SiF}_6$  | 0.50848 |
| 1.6256                             | $\text{CaF}_2 \leftrightarrow \text{H}_2\text{SiF}_6$  | 0.61516 |
| 1.6486                             | $\text{CaF}_2 \leftrightarrow \text{SiF}_6$  | 0.60658 |
| 3.5829                             | $\text{CaSO}_4 \leftrightarrow \text{F}$   | 0.27910 |
| 2.4024                             | $\text{CaSO}_4 \leftrightarrow \text{HF}$  | 0.29391 |
| 0.48666                            | $\text{F} \leftrightarrow \text{CaF}_2$  | 2.0548  |
| 0.51248                            | $\text{HF} \leftrightarrow \text{CaF}_2$   | 1.9513  |
| 1.2641                             | $\text{H}_2\text{SiF}_6 \leftrightarrow \text{F}$  | 0.79109 |
| 3.6011                             | $\text{H}_2\text{SiF}_6 \leftrightarrow 2\text{HF}$  | 0.27769 |

**TABLE 11.19** Gravimetric Factors (*Continued*)

| Factor                             |  | Factor  |
|------------------------------------|--|---------|
| <b>FLUORINE (<i>continued</i>)</b> |  |         |
| <b>F = 18.9984</b>                 |  |         |
| 1.2004                             | $\text{H}_2\text{SiF}_6 \leftrightarrow 6\text{HF}$  | 0.83308 |
| 1.3844                             | $\text{H}_2\text{SiF}_6 \leftrightarrow \text{SiF}_4$  | 0.72233 |
| 1.0141                             | $\text{H}_2\text{SiF}_6 \leftrightarrow \text{SiF}_6$  | 0.98605 |
| 2.0556                             | $\text{KF} \cdot \text{HF} \leftrightarrow 2\text{F}$  | 0.48647 |
| 1.9520                             | $\text{KF} \cdot \text{HF} \leftrightarrow 2\text{HF}$                                       | 0.51228 |
| 0.67218                            | $\text{KF} \cdot \text{HF} \leftrightarrow 2\text{KF}$                                       | 1.4877  |
| 0.41489                            | $\text{KF} \cdot \text{HF} \leftrightarrow 2(\text{KF} \cdot 2\text{H}_2\text{O})$           | 2.4103  |
| 1.9325                             | $\text{K}_2\text{SiF}_6 \leftrightarrow \text{F}$  | 0.51748 |
| 1.8351                             | $\text{K}_2\text{SiF}_6 \leftrightarrow 6\text{HF}$  | 0.54494 |
| 1.5288                             | $\text{K}_2\text{SiF}_6 \leftrightarrow \text{H}_2\text{SiF}_6$                              | 0.65412 |
| 1.8957                             | $\text{K}_2\text{SiF}_6 \leftrightarrow 2\text{KF}$  | 0.52751 |
| 1.5504                             | $\text{K}_2\text{SiF}_6 \leftrightarrow \text{SiF}_6$  | 0.64500 |
| 1.9495                             | $\text{NH}_4\text{F} \leftrightarrow \text{F}$   | 0.51295 |
| 1.5013                             | $\text{NH}_4\text{F} \cdot \text{HF} \leftrightarrow 2\text{F}$                              | 0.66611 |
| 1.4256                             | $\text{NH}_4\text{F} \cdot \text{HF} \leftrightarrow 2\text{HF}$                             | 0.70145 |
| 0.49090                            | $\text{NH}_4\text{F} \cdot \text{HF} \leftrightarrow 2\text{KF}$                             | 2.0371  |
| 0.30300                            | $\text{NH}_4\text{F} \cdot \text{HF} \leftrightarrow 2(\text{KF} \cdot 2\text{H}_2\text{O})$ | 3.3003  |
| 1.5629                             | $(\text{NH}_4)_2\text{SiF}_6 \leftrightarrow \text{F}$                                       | 0.63985 |
| 1.4841                             | $(\text{NH}_4)_2\text{SiF}_6 \leftrightarrow 6\text{HF}$                                     | 0.67381 |
| 1.2364                             | $(\text{NH}_4)_2\text{SiF}_6 \leftrightarrow \text{H}_2\text{SiF}_6$                         | 0.80881 |
| 2.4050                             | $(\text{NH}_4)_2\text{SiF}_6 \leftrightarrow 2\text{NH}_4\text{F}$                           | 0.41580 |
| 1.2539                             | $(\text{NH}_4)_2\text{SiF}_6 \leftrightarrow \text{SiF}_6$                                   | 0.79753 |
| 2.2101                             | $\text{NaF} \leftrightarrow \text{F}$  | 0.45246 |
| 1.6498                             | $\text{Na}_2\text{SiF}_6 \leftrightarrow \text{F}$   | 0.60614 |
| 1.5666                             | $\text{Na}_2\text{SiF}_6 \leftrightarrow 6\text{HF}$   | 0.63831 |
| 1.3052                             | $\text{Na}_3\text{SiF}_6 \leftrightarrow \text{H}_2\text{SiF}_6$                             | 0.76619 |
| 2.2394                             | $\text{Na}_2\text{SiF}_6 \leftrightarrow 2\text{NaF}$  | 0.44654 |
| 1.3236                             | $\text{Na}_2\text{SiF}_6 \leftrightarrow \text{SiF}_6$                                       | 0.75550 |
| <b>GALLIUM</b>                     |  |         |
| <b>Ga = 69.72</b>                  |  |         |
| 1.3442                             | $\text{Ga}_2\text{O}_3 \leftrightarrow \text{Ga}$  | 0.74392 |
| 1.6898                             | $\text{Ga}_2\text{S}_3 \leftrightarrow \text{Ga}$  | 0.59178 |
| <b>GERMANIUM</b>                   |  |         |
| <b>Ge = 72.59</b>                  |  |         |
| 1.4408                             | $\text{GeO}_2 \leftrightarrow \text{Ge}$   | 0.69404 |
| 3.6476                             | $\text{K}_2\text{GeF}_6 \leftrightarrow \text{Ge}$   | 0.27415 |
| <b>GOLD</b>                        |  |         |
| <b>Au = 196.967</b>                |  |         |
| 0.64936                            | $\text{Au} \leftrightarrow \text{AuCl}_3$  | 1.5400  |
| 0.47826                            | $\text{Au} \leftrightarrow \text{HAuCl}_4 \cdot 4\text{H}_2\text{O}$                         | 2.0909  |
| 0.54995                            | $\text{Au} \leftrightarrow \text{KAu}(\text{CN})_4 \cdot \text{H}_2\text{O}$                 | 1.8183  |
| <b>HYDROGEN</b>                    |  |         |
| <b>H = 1.0079</b>                  |  |         |
| 8.9365                             | $\text{H}_2\text{O} \leftrightarrow \text{H}$  | 0.11190 |
| 7.9364                             | $\text{O} \leftrightarrow \text{H}$  | 0.12600 |
| 0.35607                            | $\text{HSCN} \leftrightarrow \text{AgSCN}$   | 2.8084  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                               |  | Factor  |
|--------------------------------------|--|---------|
| <b>HYDROGEN</b> ( <i>continued</i> ) |  |         |
| <b>H = 1.0079</b>                    |  |         |
| 0.48586                              | $\text{HSCN} \leftrightarrow \text{CuSCN}$   | 2.0582  |
| 0.25317                              | $\text{HSCN} \leftrightarrow \text{BaSO}_4$  | 3.9499  |
| <b>INDIUM</b>                        |  |         |
| <b>In = 114.82</b>                   |  |         |
| 1.2090                               | $\text{In}_2\text{O}_3 \leftrightarrow \text{In}$  | 0.82711 |
| 1.4189                               | $\text{In}_2\text{S}_3 \leftrightarrow \text{In}$  | 0.70476 |
| <b>IODINE</b>                        |  |         |
| <b>I = 126.904</b>                   |  |         |
| 0.84333                              | $\text{Ag} \leftrightarrow \text{HI}$  | 1.1858  |
| 0.85004                              | $\text{Ag} \leftrightarrow \text{I}$   | 1.1764  |
| 1.1294                               | $\text{AgCl} \leftrightarrow \text{I}$   | 0.88543 |
| 1.8354                               | $\text{AgI} \leftrightarrow \text{HI}$   | 0.54483 |
| 1.8500                               | $\text{AgI} \leftrightarrow \text{I}$  | 0.54053 |
| 1.3423                               | $\text{AgI} \leftrightarrow \text{IO}_3$   | 0.74498 |
| 1.2298                               | $\text{AgI} \leftrightarrow \text{IO}_4$   | 0.81314 |
| 1.4066                               | $\text{AgI} \leftrightarrow \text{I}_2\text{O}_5$  | 0.71091 |
| 1.2836                               | $\text{AgI} \leftrightarrow \text{I}_2\text{O}_7$  | 0.77904 |
| 0.41592                              | $\text{Pd} \leftrightarrow \text{HI}$  | 2.4043  |
| 0.41921                              | $\text{Pd} \leftrightarrow \text{I}$   | 2.3854  |
| 1.4081                               | $\text{PdI}_2 \leftrightarrow \text{HI}$   | 0.71020 |
| 1.4192                               | $\text{PdI}_2 \leftrightarrow \text{I}$  | 0.70462 |
| 1.0297                               | $\text{PdI}_2 \leftrightarrow \text{IO}_3$   | 0.97113 |
| 0.94343                              | $\text{PdI}_2 \leftrightarrow \text{IO}_4$   | 1.0600  |
| 1.0791                               | $\text{PdI}_2 \leftrightarrow \text{I}_2\text{O}_5$  | 0.92671 |
| 0.98472                              | $\text{PdI}_2 \leftrightarrow \text{I}_2\text{O}_7$  | 1.0155  |
| 2.5899                               | $\text{TlI} \leftrightarrow \text{HI}$   | 0.38612 |
| 2.6105                               | $\text{TlI} \leftrightarrow \text{I}$  | 0.38307 |
| 1.8941                               | $\text{TlI} \leftrightarrow \text{IO}_3$   | 0.52797 |
| 1.7353                               | $\text{TlI} \leftrightarrow \text{IO}_4$   | 0.57627 |
| 1.9848                               | $\text{TlI} \leftrightarrow \text{I}_2\text{O}_5$  | 0.50383 |
| 1.8112                               | $\text{TlI} \leftrightarrow \text{I}_2\text{O}_7$  | 0.55211 |
| <b>IRON</b>                          |  |         |
| <b>Fe = 55.845</b>                   |  |         |
| 2.2598                               | $\text{Ag} \leftrightarrow \text{Fe}_7(\text{CN})_{18}$ (Prussian blue)                              | 0.44252 |
| 0.54503                              | $\text{CN} \leftrightarrow \text{Fe}_7(\text{CN})_{18}$  | 1.8347  |
| 0.61256                              | $\text{CO}_2 \leftrightarrow \text{FeO}$   | 1.6325  |
| 0.37986                              | $\text{CO}_2 \leftrightarrow \text{FeCO}_3$  | 2.6326  |
| 0.49483                              | $\text{CO}_2 \leftrightarrow \text{Fe}(\text{HCO}_3)_2$  | 2.0209  |
| 0.31396                              | $\text{Fe} \leftrightarrow \text{Fe}(\text{HCO}_3)_2$  | 3.1851  |
| 0.44061                              | $\text{Fe} \leftrightarrow \text{FeCl}_2$  | 2.2696  |
| 0.77730                              | $\text{Fe} \leftrightarrow \text{FeO}$   | 1.2865  |
| 0.69943                              | $\text{Fe} \leftrightarrow \text{Fe}_2\text{O}_3$  | 1.4297  |
| 0.72359                              | $\text{Fe} \leftrightarrow \text{Fe}_3\text{O}_4$  | 1.3820  |
| 0.36763                              | $\text{Fe} \leftrightarrow \text{FeSO}_4$  | 2.7201  |
| 0.20087                              | $\text{Fe} \leftrightarrow \text{FeSO}_4 \cdot 7\text{H}_2\text{O}$                                  | 4.9782  |
| 0.14242                              | $\text{Fe} \leftrightarrow \text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ | 7.0217  |
| 0.62011                              | $\text{FeO} \leftrightarrow \text{FeCO}_3$   | 1.6126  |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                  |  | Factor  |
|-------------------------|--|---------|
| <b>IRON (continued)</b> |  |         |
| <b>Fe = 55.845</b>      |  |         |
| 0.40390                 | $\text{FeO} \leftrightarrow \text{Fe}(\text{HCO}_3)_2$   | 2.4759  |
| 0.89982                 | $\text{FeO} \leftrightarrow \text{Fe}_2\text{O}_3$   | 1.1113  |
| 0.49223                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeCl}_2$  | 2.0316  |
| 0.68915                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeCO}_3$  | 1.4511  |
| 0.44887                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{Fe}(\text{HCO}_3)_2$  | 2.2278  |
| 0.33422                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{Fe}(\text{HCO}_3)_3$  | 2.9920  |
| 1.1113                  | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeO}$   | 0.89982 |
| 1.0345                  | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{Fe}_3\text{O}_4$  | 0.96662 |
| 0.52941                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FePO}_4$  | 1.8889  |
| 0.52561                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeSO}_4$  | 1.9026  |
| 0.28719                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeSO}_4 \cdot 7\text{H}_2\text{O}$                                  | 3.4820  |
| 0.20361                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ | 4.9113  |
| 0.39934                 | $\text{Fe}_2\text{O}_3 \leftrightarrow \text{Fe}_2(\text{SO}_4)_3$   | 2.5041  |
| 2.7006                  | $\text{FePO}_4 \leftrightarrow \text{Fe}$  | 0.37029 |
| 2.0992                  | $\text{FePO}_4 \leftrightarrow \text{FeO}$   | 0.47637 |
| 1.5741                  | $\text{FeS} \leftrightarrow \text{Fe}$   | 0.63527 |
| 1.2236                  | $\text{FeS} \leftrightarrow \text{FeO}$  | 0.81726 |
| 1.1010                  | $\text{FeS} \leftrightarrow \text{Fe}_2\text{O}_3$   | 0.90825 |
| 0.79699                 | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{FeAsO}_4$  | 1.2547  |
| 1.1144                  | $\text{SO}_3 \leftrightarrow \text{FeO}$   | 0.89738 |
| 0.52704                 | $\text{SO}_3 \leftrightarrow \text{FeSO}_4$  | 1.8974  |
| <b>LANTHANUM</b>        |  |         |
| <b>La = 138.91</b>      |  |         |
| 1.1728                  | $\text{La}_2\text{O}_3 \leftrightarrow \text{La}$  | 0.85268 |
| <b>LEAD</b>             |  |         |
| <b>Pb = 207.2</b>       |  |         |
| 0.77541                 | $\text{Pb} \leftrightarrow \text{PbCO}_3$  | 1.2896  |
| 0.80141                 | $\text{Pb} \leftrightarrow (\text{PbCO}_3)_2 \cdot \text{Pb}(\text{OH})_2$                                       | 1.2478  |
| 0.85901                 | $\text{Pb} \leftrightarrow \text{Pb}(\text{OH})_2$   | 1.1641  |
| 0.92831                 | $\text{Pb} \leftrightarrow \text{PbO}$   | 1.0772  |
| 1.3422                  | $\text{PbCl}_2 \leftrightarrow \text{Pb}$  | 0.74502 |
| 1.2460                  | $\text{PbCl}_2 \leftrightarrow \text{PbO}$   | 0.80255 |
| 1.5598                  | $\text{PbCrO}_4 \leftrightarrow \text{Pb}$   | 0.64110 |
| 0.85198                 | $\text{PbCrO}_4 \leftrightarrow \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$           | 1.1737  |
| 1.2501                  | $\text{PbCrO}_4 \leftrightarrow (\text{PbCO}_3)_2 \cdot \text{Pb}(\text{OH})_2$                                  | 0.79997 |
| 1.4480                  | $\text{PbCrO}_4 \leftrightarrow \text{PbO}$  | 0.69061 |
| 1.4142                  | $\text{PbCrO}_4 \leftrightarrow \text{Pb}_3\text{O}_4$   | 0.70711 |
| 1.0657                  | $\text{PbCrO}_4 \leftrightarrow \text{PbSO}_4$   | 0.93833 |
| 0.83529                 | $\text{PbO} \leftrightarrow \text{PbCO}_3$   | 1.1972  |
| 0.67388                 | $\text{PbO} \leftrightarrow \text{Pb}(\text{NO}_3)_2$  | 1.4839  |
| 0.93311                 | $\text{PbO} \leftrightarrow \text{PbO}_2$  | 1.0717  |
| 1.1544                  | $\text{PbO}_2 \leftrightarrow \text{Pb}$   | 0.86622 |
| 0.72219                 | $\text{PbO}_2 \leftrightarrow \text{Pb}(\text{NO}_3)_2$  | 1.3847  |
| 1.1547                  | $\text{PbS} \leftrightarrow \text{Pb}$   | 0.86600 |
| 1.0720                  | $\text{PbS} \leftrightarrow \text{PbO}$  | 0.93287 |
| 0.78895                 | $\text{PbS} \leftrightarrow \text{PbSO}_4$   | 1.2675  |
| 1.2993                  | $\text{PbSO}_4 \leftrightarrow \text{BaSO}_4$  | 0.76966 |
| 1.4636                  | $\text{PbSO}_4 \leftrightarrow \text{Pb}$  | 0.68323 |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                  |   | Factor   |
|-------------------------|---|----------|
| <b>LEAD (continued)</b> |   |          |
| <b>Pb = 207.2</b>       |   |          |
| 0.79944                 | $\text{PbSO}_4 \leftrightarrow \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ | 1.2509   |
| 1.1349                  | $\text{PbSO}_4 \leftrightarrow \text{PbCO}_3$   | 0.88112  |
| 1.1730                  | $\text{PbSO}_4 \leftrightarrow (\text{PbCO}_3)_2 \cdot \text{Pb}(\text{OH})_2$                        | 0.85254  |
| 0.91561                 | $\text{PbSO}_4 \leftrightarrow \text{Pb}(\text{NO}_3)_2$  | 1.0922   |
| 1.3587                  | $\text{PbSO}_4 \leftrightarrow \text{PbO}$  | 0.73599  |
| 1.2678                  | $\text{PbSO}_4 \leftrightarrow \text{PbO}_2$  | 0.78875  |
| 1.3270                  | $\text{PbSO}_4 \leftrightarrow \text{Pb}_3\text{O}_4$   | 0.75358  |
| <b>LITHIUM</b>          |   |          |
| <b>Li = 6.941</b>       |   |          |
| 0.59562                 | $\text{CO}_2 \leftrightarrow \text{Li}_2\text{CO}_3$  | 1.6789   |
| 0.64759                 | $\text{CO}_2 \leftrightarrow \text{LiHCO}_3$  | 1.5442   |
| 1.4729                  | $\text{CO}_2 \leftrightarrow \text{Li}_2\text{O}$   | 0.67894  |
| 6.1086                  | $\text{LiCl} \leftrightarrow \text{Li}$   | 0.16369  |
| 2.8378                  | $\text{LiCl} \leftrightarrow \text{Li}_2\text{O}$   | 0.35239  |
| 5.3228                  | $\text{Li}_2\text{CO}_3 \leftrightarrow \text{Li}$  | 0.18787  |
| 0.87147                 | $\text{Li}_2\text{CO}_3 \leftrightarrow \text{LiCl}$  | 1.1475   |
| 0.54364                 | $\text{Li}_2\text{CO}_3 \leftrightarrow \text{LiHCO}_3$   | 1.8395   |
| 2.4730                  | $\text{Li}_2\text{CO}_3 \leftrightarrow \text{Li}_2\text{O}$  | 0.40436  |
| 4.5491                  | $\text{LiHCO}_3 \leftrightarrow \text{Li}_2\text{O}$  | 0.21983  |
| 3.7371                  | $\text{LiF} \leftrightarrow \text{Li}$  | 0.26759  |
| 2.1525                  | $\text{Li}_2\text{O} \leftrightarrow \text{Li}$   | 0.46457  |
| 0.27176                 | $\text{Li}_2\text{O} \leftrightarrow \text{Li}_2\text{SO}_4$  | 3.6798   |
| 5.5609                  | $\text{Li}_2\text{PO}_4 \leftrightarrow \text{Li}$  | 0.17983  |
| 0.91047                 | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{LiCl}$  | 1.0983   |
| 1.0447                  | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{Li}_2\text{CO}_3$                                       | 0.95717  |
| 0.56797                 | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{LiHCO}_3$   | 1.7607   |
| 2.5837                  | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{Li}_2\text{O}$  | 0.38704  |
| 0.70214                 | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{Li}_2\text{SO}_4$                                       | 1.4242   |
| 0.60331                 | $\text{Li}_3\text{PO}_4 \leftrightarrow \text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$              | 1.6575   |
| 7.9153                  | $\text{Li}_2\text{SO}_4 \leftrightarrow \text{Li}$  | 0.12634  |
| 1.2967                  | $\text{Li}_2\text{SO}_4 \leftrightarrow \text{LiCl}$  | 0.77118  |
| 2.6797                  | $\text{SO}_3 \leftrightarrow \text{Li}_2\text{O}$   | 0.37317  |
| 0.72823                 | $\text{SO}_3 \leftrightarrow \text{Li}_2\text{SO}_4$  | 1.3732   |
| <b>MAGNESIUM</b>        |   |          |
| <b>Mg = 24.305</b>      |   |          |
| 1.9390                  | $\text{BaSO}_4 \leftrightarrow \text{MgSO}_4$   | 0.51572  |
| 0.94693                 | $\text{BaSO}_4 \leftrightarrow \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$                               | 1.0560   |
| 6.5755                  | $\text{Br} \leftrightarrow \text{Mg}$   | 0.15208  |
| 0.86800                 | $\text{Br} \leftrightarrow \text{MgBr}_2$   | 1.1521   |
| 0.54691                 | $\text{Br} \leftrightarrow \text{MgBr}_2 \cdot 6\text{H}_2\text{O}$                                   | 1.8285   |
| 2.9173                  | $\text{Cl} \leftrightarrow \text{Mg}$   | 0.34278  |
| 0.74472                 | $\text{Cl} \leftrightarrow \text{MgCl}_2$   | 1.3429   |
| 0.25533                 | $\text{Mg} \leftrightarrow \text{MgCl}_2$   | 3.9165   |
| 0.28883                 | $\text{Mg} \leftrightarrow \text{MgCO}_3$   | 3.4683   |
| 10.4427                 | $\text{I} \leftrightarrow \text{Mg}$  | 0.095761 |
| 0.91261                 | $\text{I} \leftrightarrow \text{MgI}_2$   | 1.09576  |
| 0.34876                 | $\text{Cl} \leftrightarrow \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$                                   | 2.8673   |
| 0.52193                 | $\text{CO}_2 \leftrightarrow \text{MgCO}_3$   | 1.9160   |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                       |  | Factor   |
|------------------------------|--|----------|
| <b>MAGNESIUM (continued)</b> |  |          |
| <b>Mg = 24.305</b>           |  |          |
| 1.0918                       | $\text{CO}_2 \leftrightarrow \text{MgO}$   | 0.91595  |
| 0.57616                      | $\text{MgCO}_3 \leftrightarrow \text{Mg}(\text{HCO}_3)_2$  | 1.7356   |
| 10.094                       | $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \leftrightarrow \text{Mg}$                             | 0.099067 |
| 6.0879                       | $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \leftrightarrow \text{MgO}$                            | 0.16426  |
| 1.6581                       | $\text{MgO} \leftrightarrow \text{Mg}$   | 0.60311  |
| 0.47807                      | $\text{MgO} \leftrightarrow \text{MgCO}_3$   | 2.0918   |
| 0.27544                      | $\text{MgO} \leftrightarrow \text{Mg}(\text{HCO}_3)_2$   | 3.6305   |
| 0.33489                      | $\text{MgO} \leftrightarrow \text{MgSO}_4$   | 2.9860   |
| 4.5784                       | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Mg}$  | 0.21841  |
| 1.1687                       | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgCl}_2$  | 0.85562  |
| 0.54737                      | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$                  | 1.8269   |
| 0.40049                      | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgCl}_2 \cdot \text{KCl} \cdot 6\text{H}_2\text{O}$ | 2.4969   |
| 1.3198                       | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgCO}_3$  | 0.75770  |
| 0.76040                      | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Mg}(\text{HCO}_3)_2$                                | 1.3151   |
| 2.7607                       | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgO}$   | 0.36223  |
| 0.92452                      | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgSO}_4$  | 1.0816   |
| 0.45150                      | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$                  | 2.2149   |
| 4.9523                       | $\text{MgSO}_4 \leftrightarrow \text{Mg}$  | 0.20193  |
| 1.9864                       | $\text{SO}_3 \leftrightarrow \text{MgO}$   | 0.50343  |
| 0.6651                       | $\text{SO}_3 \leftrightarrow \text{MgSO}_4$  | 1.5034   |
| 0.38482                      | $\text{SO}_3 \leftrightarrow \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$                                      | 3.0786   |
| <b>MANGANESE</b>             |  |          |
| <b>Mn = 54.9380</b>          |  |          |
| 1.5457                       | $\text{BaSO}_4 \leftrightarrow \text{MnSO}_4$  | 0.64696  |
| 0.38286                      | $\text{CO}_2 \leftrightarrow \text{MnCO}_3$  | 2.6119   |
| 0.62041                      | $\text{CO}_2 \leftrightarrow \text{MnO}$   | 1.6118   |
| 0.47793                      | $\text{Mn} \leftrightarrow \text{MnCO}_3$  | 2.0924   |
| 0.77446                      | $\text{Mn} \leftrightarrow \text{MnO}$   | 1.2912   |
| 0.63193                      | $\text{Mn} \leftrightarrow \text{MnO}_2$   | 1.5825   |
| 0.69599                      | $\text{Mn} \leftrightarrow \text{Mn}_2\text{O}_3$  | 1.4368   |
| 0.76126                      | $\text{MnCO}_3 \leftrightarrow \text{MnSO}_4$  | 1.3136   |
| 1.5395                       | $\text{Mn}(\text{HCO}_3)_2 \leftrightarrow \text{MnCO}_3$  | 0.64955  |
| 0.61711                      | $\text{MnO} \leftrightarrow \text{MnCO}_3$   | 1.6205   |
| 0.40084                      | $\text{MnO} \leftrightarrow \text{Mn}(\text{HCO}_3)_2$   | 2.4947   |
| 0.89868                      | $\text{MnO} \leftrightarrow \text{Mn}_2\text{O}_3$   | 1.1127   |
| 0.46978                      | $\text{MnO} \leftrightarrow \text{MnSO}_4$   | 2.1286   |
| 1.3883                       | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{Mn}$  | 0.72031  |
| 0.66351                      | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{MnCO}_3$  | 1.5071   |
| 0.43098                      | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{Mn}(\text{HCO}_3)_2$  | 2.3203   |
| 1.0752                       | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{MnO}$   | 0.93008  |
| 0.96625                      | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{Mn}_2\text{O}_3$  | 1.0349   |
| 0.87731                      | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{MnO}_2$   | 1.1399   |
| 0.50510                      | $\text{Mn}_3\text{O}_4 \leftrightarrow \text{MnSO}_4$  | 1.9798   |
| 2.5831                       | $\text{Mn}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Mn}$  | 0.38713  |
| 1.2345                       | $\text{Mn}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MnCO}_3$  | 0.81002  |
| 2.0005                       | $\text{Mn}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MnO}$   | 0.49987  |
| 1.6324                       | $\text{Mn}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MnO}_2$   | 0.61261  |
| 0.93980                      | $\text{Mn}_2\text{P}_2\text{O}_7 \leftrightarrow \text{MnSO}_4$  | 1.0641   |
| 1.5836                       | $\text{MnS} \leftrightarrow \text{Mn}$   | 0.63146  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                              |   | Factor  |
|-------------------------------------|---|---------|
| <b>MANGANESE (<i>continued</i>)</b> |   |         |
| <b>Mn = 54.9380</b>                 |   |         |
| 0.75687                             | $\text{MnS} \leftrightarrow \text{MnCO}_3$  | 1.3212  |
| 1.2265                              | $\text{MnS} \leftrightarrow \text{MnO}$   | 0.81535 |
| 0.57617                             | $\text{MnS} \leftrightarrow \text{MnSO}_4$  | 1.7356  |
| 2.7486                              | $\text{MnSO}_4 \leftrightarrow \text{Mn}$   | 0.36383 |
| 1.1286                              | $\text{SO}_3 \leftrightarrow \text{MnO}$  | 0.88603 |
| 0.53021                             | $\text{SO}_3 \leftrightarrow \text{MnSO}_4$   | 1.8860  |
| <b>MERCURY</b>                      |   |         |
| <b>Hg = 200.59</b>                  |   |         |
| 0.73882                             | $\text{Hg} \leftrightarrow \text{HgCl}_2$   | 1.3535  |
| 0.92613                             | $\text{Hg} \leftrightarrow \text{HgO}$  | 1.0798  |
| 0.86220                             | $\text{Hg} \leftrightarrow \text{HgS}$  | 1.1598  |
| 1.1767                              | $\text{HgCl} \leftrightarrow \text{Hg}$   | 0.84981 |
| 0.86939                             | $\text{HgCl} \leftrightarrow \text{HgCl}_2$   | 1.1502  |
| 0.89889                             | $\text{HgCl} \leftrightarrow \text{HgNO}_3$   | 1.1125  |
| 1.1316                              | $\text{HgCl} \leftrightarrow \text{Hg}_2\text{O}$   | 0.88371 |
| 1.0898                              | $\text{HgCl} \leftrightarrow \text{HgO}$  | 0.91760 |
| 1.0146                              | $\text{HgCl} \leftrightarrow \text{HgS}$  | 0.98564 |
| 0.98564                             | $\text{HgS} \leftrightarrow \text{HgCl}$  | 1.0146  |
| 0.85691                             | $\text{HgS} \leftrightarrow \text{HgCl}_2$  | 1.1670  |
| 0.92091                             | $\text{HgS} \leftrightarrow \text{Hg}(\text{CN})_2$   | 1.0859  |
| 0.88598                             | $\text{HgS} \leftrightarrow \text{HgNO}_3$  | 1.1287  |
| 0.71673                             | $\text{HgS} \leftrightarrow \text{Hg}(\text{NO}_3)_2$   | 1.3952  |
| 0.67903                             | $\text{HgS} \leftrightarrow \text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$                | 1.4727  |
| 1.1153                              | $\text{HgS} \leftrightarrow \text{Hg}_2\text{O}$  | 0.89658 |
| 1.0741                              | $\text{HgS} \leftrightarrow \text{HgO}$   | 0.93097 |
| 0.78426                             | $\text{HgS} \leftrightarrow \text{HgSO}_4$  | 1.2751  |
| <b>MOLYBDENUM</b>                   |   |         |
| <b>Mo = 95.94</b>                   |   |         |
| 8.9876                              | $\text{MoC} \leftrightarrow \text{C}$   | 0.11126 |
| 1.5003                              | $\text{MoO}_3 \leftrightarrow \text{Mo}$  | 0.66653 |
| 0.73436                             | $\text{MoO}_3 \leftrightarrow (\text{NH}_4)_2\text{MoO}_4$                                    | 1.3617  |
| 2.0026                              | $\text{MoS}_3 \leftrightarrow \text{Mo}$  | 0.49935 |
| 1.3348                              | $\text{MoS}_4 \leftrightarrow \text{MoO}_3$   | 0.74918 |
| 0.98021                             | $\text{MoS}_3 \leftrightarrow (\text{NH}_4)_2\text{MoO}_4$                                    | 1.0202  |
| 1.0863                              | $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 \leftrightarrow \text{MoO}_3$                | 0.92058 |
| 0.79771                             | $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 \leftrightarrow (\text{NH}_4)_2\text{MoO}_4$ | 1.2536  |
| 3.8267                              | $\text{PbMoO}_4 \leftrightarrow \text{Mo}$  | 0.26132 |
| 2.5506                              | $\text{PbMoO}_4 \leftrightarrow \text{MoO}_3$   | 0.39207 |
| 1.8730                              | $\text{PbMoO}_4 \leftrightarrow (\text{NH}_4)_2\text{MoO}_4$                                  | 0.53390 |
| <b>NEODYMIUM</b>                    |   |         |
| <b>Nd = 144.24</b>                  |   |         |
| 1.1664                              | $\text{Nd}_2\text{O}_3 \leftrightarrow \text{Nd}$   | 0.85735 |
| <b>NICKEL</b>                       |   |         |
| <b>Ni = 58.71</b>                   |   |         |
| 0.20319                             | $\text{Ni} \leftrightarrow \text{Ni dimethylglyoxime}$  | 4.9215  |
| 0.20188                             | $\text{Ni} \leftrightarrow \text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$                | 4.9533  |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                    |  | Factor   |
|---------------------------|--|----------|
| <b>NICKEL (continued)</b> |  |          |
| <b>Ni = 58.71</b>         |  |          |
| 0.78585                   | $\text{Ni} \leftrightarrow \text{NiO}$   | 1.2725   |
| 0.20902                   | $\text{Ni} \leftrightarrow \text{NiSO}_4 \cdot 7\text{H}_2\text{O}$                | 4.7842   |
| 3.8675                    | $\text{Ni dimethylglyoxime} \leftrightarrow \text{NiO}$                            | 0.25856  |
| 0.25690                   | $\text{NiO} \leftrightarrow \text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$    | 3.8926   |
| 0.26598                   | $\text{NiO} \leftrightarrow \text{NiSO}_4 \cdot 7\text{H}_2\text{O}$               | 3.7597   |
| 2.6362                    | $\text{NiSO}_4 \leftrightarrow \text{Ni}$  | 0.37934  |
| 0.53220                   | $\text{NiSO}_4 \leftrightarrow \text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ | 1.8790   |
| 2.0716                    | $\text{NiSO}_4 \leftrightarrow \text{NiO}$   | 0.48271  |
| 0.55102                   | $\text{NiSO}_4 \leftrightarrow \text{NiSO}_4 \cdot 7\text{H}_2\text{O}$            | 1.8148   |
| <b>NIOBIUM</b>            |  |          |
| <b>Nb = 92.906</b>        |  |          |
| 7.7351                    | $\text{Nb} \leftrightarrow \text{C}$   | 0.12928  |
| 8.7353                    | $\text{NbC} \leftrightarrow \text{C}$  | 0.11448  |
| 11.065                    | $\text{Nb}_2\text{O}_5 \leftrightarrow 2\text{C}$                                  | 0.090373 |
| 1.4305                    | $\text{Nb}_2\text{O}_5 \leftrightarrow \text{Nb}$                                  | 0.69904  |
| <b>NITROGEN</b>           |  |          |
| <b>N = 14.0067</b>        |  |          |
| 3.2731                    | $\text{AgNO}_2 \leftrightarrow \text{HNO}_2$                                       | 0.30552  |
| 4.0488                    | $\text{AgNO}_2 \leftrightarrow \text{N}_2\text{O}_3$                               | 0.24698  |
| 1.8722                    | $\text{KNO}_3 \leftrightarrow \text{N}_2\text{O}_5$                                | 0.53412  |
| 0.22229                   | $\text{N} \leftrightarrow \text{HNO}_3$  | 4.4987   |
| 0.30446                   | $\text{N} \leftrightarrow \text{NO}_2$   | 3.2845   |
| 0.36855                   | $\text{N} \leftrightarrow \text{N}_2\text{O}_3$                                    | 2.7134   |
| 0.22590                   | $\text{N} \leftrightarrow \text{NO}_3$   | 4.4268   |
| 0.25936                   | $\text{N} \leftrightarrow \text{N}_2\text{O}_5$                                    | 3.8556   |
| 6.0680                    | $\text{NaNO}_3 \leftrightarrow \text{N}$   | 0.16480  |
| 1.5738                    | $\text{NaNO}_3 \leftrightarrow \text{N}_2\text{O}_5$                               | 0.63539  |
| 0.47619                   | $\text{NO} \leftrightarrow \text{HNO}_3$   | 2.1000   |
| 0.65222                   | $\text{NO} \leftrightarrow \text{NO}_2$  | 1.5332   |
| 0.78951                   | $\text{NO} \leftrightarrow \text{N}_2\text{O}_3$                                   | 1.2666   |
| 0.48393                   | $\text{NO} \leftrightarrow \text{NO}_3$  | 2.0664   |
| 0.55561                   | $\text{NO} \leftrightarrow \text{N}_2\text{O}_5$                                   | 1.7998   |
| 0.27028                   | $\text{NH}_3 \leftrightarrow \text{HNO}_3$   | 3.6999   |
| 1.2159                    | $\text{NH}_3 \leftrightarrow \text{N}$   | 0.82244  |
| 0.31536                   | $\text{NH}_3 \leftrightarrow \text{N}_2\text{O}_5$                                 | 3.1710   |
| 0.27467                   | $\text{NH}_3 \leftrightarrow \text{NO}_3$  | 3.6407   |
| 0.84890                   | $\text{NH}_4\text{Cl} \leftrightarrow \text{HNO}_3$                                | 1.1780   |
| 0.86270                   | $\text{NH}_4\text{Cl} \leftrightarrow \text{NO}_3$                                 | 1.1591   |
| 0.99050                   | $\text{NH}_4\text{Cl} \leftrightarrow \text{N}_2\text{O}_5$                        | 1.0096   |
| 3.8189                    | $\text{NH}_4\text{Cl} \leftrightarrow \text{N}$                                    | 0.26185  |
| 3.5221                    | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{HNO}_3$                        | 0.28393  |
| 15.845                    | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{N}$                            | 0.063112 |
| 4.1096                    | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{N}_2\text{O}_6$                | 0.24333  |
| 3.5794                    | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{NO}_3$                         | 0.27938  |
| 4.7169                    | $(\text{NH}_4)_2\text{SO}_4 \leftrightarrow \text{N}$                              | 0.21200  |
| 1.2234                    | $(\text{NH}_4)_2\text{SO}_4 \leftrightarrow \text{N}_2\text{O}_5$                  | 0.81739  |
| 1.5480                    | $\text{Pt} \leftrightarrow \text{HNO}_3$   | 0.64599  |
| 6.9640                    | $\text{Pt} \leftrightarrow \text{N}$   | 0.14360  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                             |  | Factor   |
|------------------------------------|--|----------|
| <b>NITROGEN (<i>continued</i>)</b> |  |          |
| <b>N = 14.0067</b>                 |  |          |
| 1.5732                             | Pt $\leftrightarrow$ NO <sub>3</sub>   | 0.63566  |
| 1.8062                             | Pt $\leftrightarrow$ N <sub>2</sub> O <sub>5</sub>   | 0.55364  |
| 0.63528                            | SO <sub>3</sub> $\leftrightarrow$ HNO <sub>3</sub>   | 1.5741   |
| 2.8579                             | SO <sub>3</sub> $\leftrightarrow$ N  | 0.34990  |
| 0.74125                            | SO <sub>3</sub> $\leftrightarrow$ N <sub>2</sub> O <sub>5</sub>  | 1.3491   |
| <b>OSMIUM</b>                      |  |          |
| <b>Os = 190.2</b>                  |  |          |
| 1.3365                             | OsO <sub>4</sub> $\leftrightarrow$ Os  | 0.74823  |
| <b>PALLADIUM</b>                   |  |          |
| <b>Pd = 106.4</b>                  |  |          |
| 0.49873                            | Pd $\leftrightarrow$ PdCl <sub>2</sub> · 2H <sub>2</sub> O   | 2.0051   |
| 0.46179                            | Pd $\leftrightarrow$ Pd(NO <sub>3</sub> ) <sub>2</sub>   | 2.1655   |
| 3.3854                             | PdI <sub>2</sub> $\leftrightarrow$ Pd  | 0.29538  |
| 3.7342                             | K <sub>2</sub> PdCl <sub>6</sub> $\leftrightarrow$ Pd  | 0.26779  |
| 1.8624                             | K <sub>2</sub> PdCl <sub>6</sub> $\leftrightarrow$ PdCl <sub>2</sub> · 2H <sub>2</sub> O                               | 0.53695  |
| <b>PHOSPHORUS</b>                  |  |          |
| <b>P = 30.9738</b>                 |  |          |
| 13.514                             | Ag <sub>3</sub> PO <sub>4</sub> $\leftrightarrow$ P  | 0.073998 |
| 4.4075                             | Ag <sub>3</sub> PO <sub>4</sub> $\leftrightarrow$ PO <sub>4</sub>  | 0.22689  |
| 5.8980                             | Ag <sub>3</sub> PO <sub>4</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>  | 0.16955  |
| 9.7730                             | Ag <sub>4</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ P  | 0.10232  |
| 3.1874                             | Ag <sub>4</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ PO <sub>4</sub>  | 0.31374  |
| 4.2653                             | Ag <sub>4</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>                          | 0.23445  |
| 0.71833                            | Al <sub>2</sub> O <sub>3</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>   | 1.3921   |
| 1.2841                             | AlPO <sub>4</sub> $\leftrightarrow$ PO <sub>4</sub>  | 0.77877  |
| 1.7183                             | AlPO <sub>4</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>  | 0.58196  |
| 2.1853                             | Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>                        | 0.45761  |
| 1.5881                             | FePO <sub>4</sub> $\leftrightarrow$ PO <sub>4</sub>  | 0.62970  |
| 2.1251                             | FePO <sub>4</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>  | 0.47056  |
| 0.78392                            | Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ Na <sub>2</sub> HPO <sub>4</sub>                       | 1.2756   |
| 0.31073                            | Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ Na <sub>2</sub> HPO <sub>4</sub> · 12H <sub>2</sub> O  | 3.2182   |
| 0.53229                            | Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ NaNH <sub>4</sub> HPO <sub>4</sub> · 4H <sub>2</sub> O | 1.8787   |
| 3.5929                             | Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ P  | 0.27833  |
| 1.1718                             | Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ PO <sub>4</sub>  | 0.85340  |
| 1.5681                             | Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>                          | 0.63773  |
| 60.577                             | (NH <sub>4</sub> ) <sub>3</sub> PO <sub>4</sub> · 12MoO <sub>3</sub> $\leftrightarrow$ P                               | 0.016508 |
| 19.757                             | (NH <sub>4</sub> ) <sub>3</sub> PO <sub>4</sub> · 12MoO <sub>3</sub> $\leftrightarrow$ PO <sub>4</sub>                 | 0.050616 |
| 26.438                             | (NH <sub>4</sub> ) <sub>3</sub> PO <sub>4</sub> · 12MoO <sub>3</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>   | 0.037824 |
| 0.63773                            | P <sub>2</sub> O <sub>5</sub> $\leftrightarrow$ Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>                          | 1.5681   |
| 0.49993                            | P <sub>2</sub> O <sub>5</sub> $\leftrightarrow$ Na <sub>2</sub> HPO <sub>4</sub>                                       | 2.0003   |
| 0.19816                            | P <sub>2</sub> O <sub>5</sub> $\leftrightarrow$ Na <sub>2</sub> HPO <sub>4</sub> · 12H <sub>2</sub> O                  | 5.0464   |
| 0.33946                            | P <sub>2</sub> O <sub>5</sub> $\leftrightarrow$ NaNH <sub>4</sub> HPO <sub>4</sub> · 4H <sub>2</sub> O                 | 2.9459   |
| 2.2913                             | P <sub>2</sub> O <sub>5</sub> $\leftrightarrow$ P  | 0.43644  |
| 58.057                             | P <sub>2</sub> O <sub>5</sub> · 24MoO <sub>3</sub> $\leftrightarrow$ P   | 0.017225 |
| 18.935                             | P <sub>2</sub> O <sub>5</sub> · 24MoO <sub>3</sub> $\leftrightarrow$ PO <sub>4</sub>                                   | 0.052813 |
| 25.338                             | P <sub>2</sub> O <sub>5</sub> · 24MoO <sub>3</sub> $\leftrightarrow$ P <sub>2</sub> O <sub>5</sub>                     | 0.039466 |
| 11.526                             | U <sub>2</sub> P <sub>2</sub> O <sub>11</sub> $\leftrightarrow$ P  | 0.086762 |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                        |   | Factor  |
|-------------------------------|---|---------|
| <b>PHOSPHORUS (continued)</b> |   |         |
| <b>P = 30.9738</b>            |   |         |
| 3.7590                        | $\text{U}_2\text{P}_2\text{O}_{11} \leftrightarrow \text{PO}_4$                             | 0.26603 |
| 5.0303                        | $\text{U}_2\text{P}_2\text{O}_{11} \leftrightarrow \text{P}_2\text{O}_5$                    | 0.19880 |
| <b>PLATINUM</b>               |   |         |
| <b>Pt = 195.09</b>            |   |         |
| 0.93839                       | $\text{K}_2\text{PtCl}_6 \leftrightarrow \text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ | 1.0657  |
| 2.4912                        | $\text{K}_2\text{PtCl}_6 \leftrightarrow \text{Pt}$   | 0.40141 |
| 1.4426                        | $\text{K}_2\text{PtCl}_6 \leftrightarrow \text{PtCl}_4$                                     | 0.69320 |
| 1.1383                        | $\text{K}_2\text{PtCl}_6 \leftrightarrow \text{PtCl}_4 \cdot 5\text{H}_2\text{O}$           | 0.87854 |
| 2.2753                        | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{Pt}$                                    | 0.43950 |
| 1.3176                        | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{PtCl}_4$                                | 0.75897 |
| 1.0885                        | $(\text{NH}_4)_2\text{PtCl}_6 \leftrightarrow \text{PtCl}_6$                                | 0.91872 |
| 0.37668                       | $\text{Pt} \leftrightarrow \text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$               | 2.6548  |
| 0.57907                       | $\text{Pt} \leftrightarrow \text{PtCl}_4$   | 1.7269  |
| 0.45691                       | $\text{Pt} \leftrightarrow \text{PtCl}_4 \cdot 5\text{H}_2\text{O}$                         | 2.1886  |
| <b>POTASSIUM</b>              |   |         |
| <b>K = 39.098</b>             |   |         |
| 0.90639                       | $\text{Ag} \leftrightarrow \text{KBr}$  | 1.1033  |
| 1.4469                        | $\text{Ag} \leftrightarrow \text{KCl}$  | 0.69116 |
| 0.88021                       | $\text{Ag} \leftrightarrow \text{KClO}_3$   | 1.1361  |
| 0.77856                       | $\text{Ag} \leftrightarrow \text{KClO}_4$   | 1.2844  |
| 1.6565                        | $\text{Ag} \leftrightarrow \text{KCN}$  | 0.60369 |
| 0.64978                       | $\text{Ag} \leftrightarrow \text{KI}$   | 1.5390  |
| 1.5779                        | $\text{AgBr} \leftrightarrow \text{KBr}$  | 0.63377 |
| 1.1244                        | $\text{AgBr} \leftrightarrow \text{KBrO}_3$   | 0.88939 |
| 1.9223                        | $\text{AgCl} \leftrightarrow \text{KCl}$  | 0.52020 |
| 1.1695                        | $\text{AgCl} \leftrightarrow \text{KClO}_3$   | 0.85508 |
| 1.0344                        | $\text{AgCl} \leftrightarrow \text{KClO}_4$   | 0.96672 |
| 2.0561                        | $\text{AgCN} \leftrightarrow \text{KCN}$  | 0.48637 |
| 1.4142                        | $\text{AgI} \leftrightarrow \text{KI}$  | 0.70712 |
| 1.0971                        | $\text{AgI} \leftrightarrow \text{KIO}_3$   | 0.91153 |
| 1.3045                        | $\text{BaCrO}_4 \leftrightarrow \text{K}_2\text{CrO}_4$                                     | 0.76659 |
| 1.7222                        | $\text{BaCrO}_4 \leftrightarrow \text{K}_2\text{Cr}_2\text{O}_7$                            | 0.58065 |
| 1.7140                        | $\text{BaSO}_4 \leftrightarrow \text{KHSO}_4$   | 0.58342 |
| 2.1166                        | $\text{BaSO}_4 \leftrightarrow \text{K}_2\text{S}$  | 0.47245 |
| 1.3393                        | $\text{BaSO}_4 \leftrightarrow \text{K}_2\text{SO}_4$                                       | 0.74666 |
| 2.0436                        | $\text{Br} \leftrightarrow \text{K}$  | 0.48933 |
| 0.67145                       | $\text{Br} \leftrightarrow \text{KBr}$  | 1.4893  |
| 0.41473                       | $\text{CaF}_2 \leftrightarrow \text{KF} \cdot 2\text{H}_2\text{O}$                          | 2.4112  |
| 0.72315                       | $\text{CaSO}_4 \leftrightarrow \text{KF} \cdot 2\text{H}_2\text{O}$                         | 1.3828  |
| 0.90668                       | $\text{Cl} \leftrightarrow \text{K}$  | 1.1029  |
| 0.47553                       | $\text{Cl} \leftrightarrow \text{KCl}$  | 2.1029  |
| 0.28929                       | $\text{Cl} \leftrightarrow \text{KClO}_3$   | 3.4567  |
| 0.25589                       | $\text{Cl} \leftrightarrow \text{KClO}_4$   | 3.9080  |
| 0.75269                       | $\text{Cl} \leftrightarrow \text{K}_2\text{O}$  | 1.3286  |
| 0.46718                       | $\text{CO}_2 \leftrightarrow \text{K}_2\text{O}$  | 2.1405  |
| 0.31843                       | $\text{CO}_2 \leftrightarrow \text{K}_2\text{CO}_3$   | 3.1404  |
| 0.76441                       | $\text{I} \leftrightarrow \text{KI}$  | 1.3082  |
| 0.59299                       | $\text{I} \leftrightarrow \text{KIO}_3$   | 1.6864  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                              |   | Factor  |
|-------------------------------------|---|---------|
| <b>POTASSIUM (<i>continued</i>)</b> |   |         |
| <b>K = 39.098</b>                   |   |         |
| 0.31907                             | $K \leftrightarrow KClO_3$  | 3.1341  |
| 0.83016                             | $K \leftrightarrow K_2O$  | 1.2046  |
| 0.38673                             | $K \leftrightarrow KNO_3$   | 2.5858  |
| 3.0436                              | $KBr \leftrightarrow K$   | 0.32856 |
| 2.5267                              | $KBr \leftrightarrow K_2O$  | 0.39578 |
| 1.9067                              | $KCl \leftrightarrow K$   | 0.52447 |
| 1.0789                              | $KCl \leftrightarrow K_2CO_3$                                       | 0.92690 |
| 0.50685                             | $KCl \leftrightarrow K_2Cr_2O_7$                                    | 1.9730  |
| 0.74466                             | $KCl \leftrightarrow KHCO_3$  | 1.3429  |
| 0.73737                             | $KCl \leftrightarrow KNO_3$   | 1.3562  |
| 1.5829                              | $KCl \leftrightarrow K_2O$  | 0.63177 |
| 0.85563                             | $KCl \leftrightarrow K_2SO_4$                                       | 1.1687  |
| 1.6437                              | $KClO_3 \leftrightarrow KCl$  | 0.60836 |
| 3.5433                              | $KClO_4 \leftrightarrow K$  | 0.28222 |
| 1.8584                              | $KClO_4 \leftrightarrow KCl$  | 0.53811 |
| 2.9415                              | $KClO_4 \leftrightarrow K_2O$                                       | 0.33996 |
| 4.2456                              | $KI \leftrightarrow K$  | 0.23554 |
| 3.5245                              | $KI \leftrightarrow K_2O$   | 0.28373 |
| 0.38435                             | $K_2O \leftrightarrow KClO_3$                                       | 2.6018  |
| 0.68159                             | $K_2O \leftrightarrow K_2CO_3$                                      | 1.4672  |
| 0.32021                             | $K_2O \leftrightarrow K_2Cr_2O_7$                                   | 3.1229  |
| 0.47045                             | $K_2O \leftrightarrow KHCO_3$                                       | 2.1256  |
| 0.46584                             | $K_2O \leftrightarrow KNO_3$  | 2.1466  |
| 0.81194                             | $KOH \leftrightarrow K_2CO_3$                                       | 1.2316  |
| 1.1912                              | $KOH \leftrightarrow K_2O$  | 0.83946 |
| 6.2146                              | $K_2PtCl_6 \leftrightarrow K$                                       | 0.16091 |
| 3.5165                              | $K_2PtCl_6 \leftrightarrow K_2CO_3$                                 | 0.28438 |
| 3.2594                              | $K_2PtCl_6 \leftrightarrow KCl$                                     | 0.30680 |
| 2.4271                              | $K_2PtCl_6 \leftrightarrow KHCO_3$                                  | 0.41201 |
| 2.4034                              | $K_2PtCl_6 \leftrightarrow KNO_3$                                   | 0.41608 |
| 5.1592                              | $K_2PtCl_6 \leftrightarrow K_2O$                                    | 0.19383 |
| 2.7888                              | $K_2PtCl_6 \leftrightarrow K_2SO_4$                                 | 0.35857 |
| 0.51224                             | $K_2PtCl_6 \leftrightarrow K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$ | 1.9522  |
| 0.48659                             | $K_2PtCl_6 \leftrightarrow K_2SO_4 \cdot Cr_2(SO_4)_3 \cdot 24H_2O$ | 2.0551  |
| 1.2609                              | $K_2SO_4 \leftrightarrow K_2CO_3$                                   | 0.79308 |
| 0.87031                             | $K_2SO_4 \leftrightarrow KHCO_3$                                    | 1.1490  |
| 0.63990                             | $K_2SO_4 \leftrightarrow KHSO_4$                                    | 1.5627  |
| 1.0238                              | $K_2SO_4 \leftrightarrow KNO_2$                                     | 0.97674 |
| 0.86179                             | $K_2SO_4 \leftrightarrow KNO_3$                                     | 1.1604  |
| 2.2285                              | $K_2SO_4 \leftrightarrow K$   | 0.44875 |
| 1.8499                              | $K_2SO_4 \leftrightarrow K_2O$                                      | 0.54056 |
| 1.5804                              | $K_2SO_4 \leftrightarrow K_2S$                                      | 0.63275 |
| 0.60582                             | $Mg_2As_2O_7 \leftrightarrow K_3AsO_4$                              | 1.6506  |
| 0.71164                             | $Mg_2As_2O_7 \leftrightarrow K_2HASO_4$                             | 1.4052  |
| 0.40040                             | $Mn_2O_3 \leftrightarrow K_2MnO_4$                                  | 2.4975  |
| 0.49946                             | $Mn_2O_3 \leftrightarrow KMnO_4$                                    | 2.0022  |
| 0.44132                             | $MnS \leftrightarrow K_2MnO_4$                                      | 2.2659  |
| 0.55051                             | $MnS \leftrightarrow KMnO_4$  | 1.8165  |
| 0.13853                             | $N \leftrightarrow KNO_3$   | 7.2185  |
| 0.16844                             | $NH_3 \leftrightarrow KNO_3$  | 5.9368  |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                       |   | Factor  |
|------------------------------|---|---------|
| <b>POTASSIUM (continued)</b> |   |         |
| <b>K = 39.098</b>            |   |         |
| 0.29677                      | $\text{NO} \leftrightarrow \text{KNO}_3$                          | 3.3697  |
| 0.44656                      | $\text{N}_2\text{O}_3 \leftrightarrow \text{KNO}_2$               | 2.2393  |
| 1.1466                       | $\text{N}_2\text{O}_5 \leftrightarrow \text{K}_2\text{O}$         | 0.87217 |
| 0.53412                      | $\text{N}_2\text{O}_5 \leftrightarrow \text{KNO}_3$               | 1.8722  |
| 2.4946                       | $\text{Pt} \leftrightarrow \text{K}$                              | 0.40086 |
| 1.3084                       | $\text{Pt} \leftrightarrow \text{KCl}$                            | 0.76431 |
| 2.0710                       | $\text{Pt} \leftrightarrow \text{K}_2\text{O}$                    | 0.48287 |
| 0.38943                      | $\text{SiO}_2 \leftrightarrow \text{K}_2\text{SiO}_3$             | 2.5679  |
| 0.45941                      | $\text{SO}_3 \leftrightarrow \text{K}_2\text{SO}_4$               | 2.1767  |
| <b>PRASEODYMIUM</b>          |   |         |
| <b>Pr = 140.908</b>          |   |         |
| 1.1703                       | $\text{Pr}_2\text{O}_3 \leftrightarrow \text{Pr}$                 | 0.85449 |
| <b>RHODIUM</b>               |   |         |
| <b>Rh = 102.905</b>          |   |         |
| 0.26758                      | $\text{Rh} \leftrightarrow \text{Na}_3\text{RhCl}_6$              | 3.7372  |
| 0.49178                      | $\text{Rh} \leftrightarrow \text{RhCl}_3$                         | 2.0334  |
| <b>RUBIDIUM</b>              |   |         |
| <b>Rb = 85.468</b>           |   |         |
| 1.6768                       | $\text{AgCl} \leftrightarrow \text{Rb}$                           | 0.59636 |
| 1.1852                       | $\text{AgCl} \leftrightarrow \text{RbCl}$                         | 0.84371 |
| 0.41480                      | $\text{Cl} \leftrightarrow \text{Rb}$                             | 2.4108  |
| 0.29319                      | $\text{Cl} \leftrightarrow \text{RbCl}$                           | 3.4107  |
| 0.70683                      | $\text{Rb} \leftrightarrow \text{RbCl}$                           | 1.4148  |
| 0.74016                      | $\text{Rb} \leftrightarrow \text{Rb}_2\text{CO}_3$                | 1.3511  |
| 0.91441                      | $\text{Rb} \leftrightarrow \text{Rb}_2\text{O}$                   | 1.0936  |
| 0.64023                      | $\text{Rb} \leftrightarrow \text{Rb}_2\text{SO}_4$                | 1.5620  |
| 1.0472                       | $\text{RbCl} \leftrightarrow \text{Rb}_2\text{CO}_3$              | 0.95497 |
| 0.90577                      | $\text{RbCl} \leftrightarrow \text{Rb}_2\text{SO}_4$              | 1.1040  |
| 2.1636                       | $\text{RbClO}_4 \leftrightarrow \text{Rb}$                        | 0.46220 |
| 0.78828                      | $\text{Rb}_2\text{CO}_3 \leftrightarrow \text{RbHCO}_3$           | 1.2686  |
| 0.77299                      | $\text{Rb}_2\text{O} \leftrightarrow \text{RbCl}$                 | 1.2937  |
| 0.70015                      | $\text{Rb}_2\text{O} \leftrightarrow \text{Rb}_2\text{SO}_4$      | 1.4283  |
| 3.3857                       | $\text{Rb}_2\text{PtCl}_6 \leftrightarrow \text{Rb}$              | 0.29536 |
| 2.3931                       | $\text{Rb}_2\text{PtCl}_6 \leftrightarrow \text{RbCl}$            | 0.41787 |
| 2.5060                       | $\text{Rb}_2\text{PtCl}_6 \leftrightarrow \text{Rb}_2\text{CO}_3$ | 0.39905 |
| 1.9754                       | $\text{Rb}_2\text{PtCl}_6 \leftrightarrow \text{RbHCO}_3$         | 0.50623 |
| 3.0959                       | $\text{Rb}_2\text{PtCl}_6 \leftrightarrow \text{Rb}_2\text{O}$    | 0.32301 |
| 1.1561                       | $\text{Rb}_2\text{SO}_4 \leftrightarrow \text{Rb}_2\text{CO}_3$   | 0.86498 |
| 0.91133                      | $\text{Rb}_2\text{SO}_4 \leftrightarrow \text{RbHCO}_3$           | 1.0973  |
| <b>SELENIUM</b>              |   |         |
| <b>Se = 78.96</b>            |   |         |
| 0.61224                      | $\text{Se} \leftrightarrow \text{H}_2\text{SeO}_3$                | 1.6334  |
| 0.54466                      | $\text{Se} \leftrightarrow \text{H}_2\text{SeO}_4$                | 1.8360  |
| 0.71161                      | $\text{Se} \leftrightarrow \text{SeO}_2$                          | 1.4053  |
| 0.62193                      | $\text{Se} \leftrightarrow \text{SeO}_3$                          | 1.6079  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor              |   | Factor  |
|---------------------|---|---------|
| <b>SILICON</b>      |   |         |
| <b>Si = 28.086</b>  |   |         |
| 2.6847              | $\text{BaSiF}_6 \leftrightarrow \text{SiF}_4$                                     | 0.37249 |
| 4.6504              | $\text{BaSiF}_6 \leftrightarrow \text{SiO}_2$                                     | 0.21503 |
| 2.1163              | $\text{K}_2\text{SiF}_6 \leftrightarrow \text{SiF}_4$                             | 0.47249 |
| 3.6661              | $\text{K}_2\text{SiF}_6 \leftrightarrow \text{SiO}_2$                             | 0.27277 |
| 3.3384              | $\text{SiC} \leftrightarrow \text{C}$   | 0.29954 |
| 0.91111             | $\text{SiC} \leftrightarrow \text{CO}_2$  | 1.0976  |
| 0.76933             | $\text{SiO}_2 \leftrightarrow \text{H}_2\text{SiO}_3$                             | 1.2998  |
| 2.1393              | $\text{SiO}_2 \leftrightarrow \text{Si}$  | 0.46744 |
| 0.57730             | $\text{SiO}_2 \leftrightarrow \text{SiF}_4$                                       | 1.7322  |
| 0.78972             | $\text{SiO}_2 \leftrightarrow \text{SiO}_3$                                       | 1.2663  |
| 0.65250             | $\text{SiO}_2 \leftrightarrow \text{SiO}_4$                                       | 1.5326  |
| 1.6651              | $\text{SiO}_2 \leftrightarrow \text{Si}_2\text{O}$                                | 0.60057 |
| 0.62514             | $\text{SiO}_2 \leftrightarrow \text{Si}(\text{OH})_4$                             | 1.5997  |
| <b>SILVER</b>       |   |         |
| <b>Ag = 107.868</b> |   |         |
| 0.63501             | $\text{Ag} \leftrightarrow \text{AgNO}_3$   | 1.5748  |
| 0.93096             | $\text{Ag} \leftrightarrow \text{Ag}_2\text{O}$                                   | 1.0742  |
| 1.7408              | $\text{AgBr} \leftrightarrow \text{Ag}$   | 0.57445 |
| 1.3286              | $\text{AgCl} \leftrightarrow \text{Ag}$   | 0.75265 |
| 0.84371             | $\text{AgCl} \leftrightarrow \text{AgNO}_3$                                       | 1.1852  |
| 1.2369              | $\text{AgCl} \leftrightarrow \text{Ag}_2\text{O}$                                 | 0.80847 |
| 1.7935              | $\text{AgCl} \leftrightarrow \text{Br}$   | 0.55756 |
| 1.2412              | $\text{AgCN} \leftrightarrow \text{Ag}$   | 0.80566 |
| 2.1764              | $\text{AgI} \leftrightarrow \text{Ag}$  | 0.45947 |
| 1.2935              | $\text{Ag}_3\text{PO}_4 \leftrightarrow \text{Ag}$                                | 0.77311 |
| 1.4031              | $\text{Ag}_4\text{P}_2\text{O}_7 \leftrightarrow \text{Ag}$                       | 0.71269 |
| 0.74079             | $\text{Br} \leftrightarrow \text{Ag}$   | 1.3499  |
| 0.42555             | $\text{Br} \leftrightarrow \text{AgBr}$   | 2.3499  |
| 0.32866             | $\text{Cl} \leftrightarrow \text{Ag}$   | 3.0426  |
| 0.24737             | $\text{Cl} \leftrightarrow \text{AgCl}$   | 4.0425  |
| 1.1764              | $\text{I} \leftrightarrow \text{Ag}$  | 0.85004 |
| 0.54053             | $\text{I} \leftrightarrow \text{AgI}$   | 1.8500  |
| <b>SODIUM</b>       |   |         |
| <b>Na = 22.9898</b> |   |         |
| 1.0483              | $\text{Ag} \leftrightarrow \text{NaBr}$   | 0.95393 |
| 1.8457              | $\text{Ag} \leftrightarrow \text{NaCl}$   | 0.54179 |
| 0.71966             | $\text{Ag} \leftrightarrow \text{NaI}$  | 1.3895  |
| 1.8249              | $\text{AgBr} \leftrightarrow \text{NaBr}$   | 0.54798 |
| 2.4523              | $\text{AgCl} \leftrightarrow \text{NaCl}$   | 0.40778 |
| 1.5663              | $\text{AgI} \leftrightarrow \text{NaI}$   | 0.63845 |
| 1.9440              | $\text{BaSO}_4 \leftrightarrow \text{NaHSO}_4$                                    | 0.51440 |
| 1.6905              | $\text{BaSO}_4 \leftrightarrow \text{NaHSO}_4 \cdot \text{H}_2\text{O}$           | 0.59156 |
| 2.9906              | $\text{BaSO}_4 \leftrightarrow \text{Na}_2\text{S}$                               | 0.33438 |
| 1.8518              | $\text{BaSO}_4 \leftrightarrow \text{Na}_2\text{SO}_3$                            | 0.54002 |
| 0.92564             | $\text{BaSO}_4 \leftrightarrow \text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$  | 1.0803  |
| 1.6432              | $\text{BaSO}_4 \leftrightarrow \text{Na}_2\text{SO}_4$                            | 0.60857 |
| 0.72442             | $\text{BaSO}_4 \leftrightarrow \text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ | 1.3804  |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                    |   | Factor   |
|---------------------------|---|----------|
| <b>SODIUM (continued)</b> |   |          |
| <b>Na = 22.9898</b>       |   |          |
| 0.69198                   | $\text{B}_2\text{O}_3 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7$  | 1.4451   |
| 0.36510                   | $\text{B}_2\text{O}_3 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$   | 2.7389   |
| 3.4758                    | $\text{Br} \leftrightarrow \text{Na}$   | 0.28770  |
| 0.77657                   | $\text{Br} \leftrightarrow \text{NaBr}$   | 1.2877   |
| 2.5786                    | $\text{Br} \leftrightarrow \text{Na}_2\text{O}$   | 0.38781  |
| 0.94956                   | $\text{CaCl}_2 \leftrightarrow \text{NaCl}$   | 1.0531   |
| 0.94433                   | $\text{CaCO}_3 \leftrightarrow \text{Na}_2\text{CO}_3$  | 1.0590   |
| 0.92975                   | $\text{CaF}_2 \leftrightarrow \text{NaF}$   | 1.0756   |
| 0.52910                   | $\text{CaO} \leftrightarrow \text{Na}_2\text{CO}_3$   | 1.8900   |
| 1.2845                    | $\text{CaSO}_4 \leftrightarrow \text{Na}_2\text{CO}_3$  | 0.77854  |
| 1.5421                    | $\text{Cl} \leftrightarrow \text{Na}$   | 0.64846  |
| 0.60663                   | $\text{Cl} \leftrightarrow \text{NaCl}$   | 1.6485   |
| 1.1442                    | $\text{Cl} \leftrightarrow \text{Na}_2\text{O}$   | 0.87410  |
| 0.41520                   | $\text{CO}_2 \leftrightarrow \text{Na}_2\text{CO}_3$  | 2.4083   |
| 0.71008                   | $\text{CO}_2 \leftrightarrow \text{Na}_2\text{O}$   | 1.4083   |
| 1.2292                    | $\text{H}_3\text{BO}_3 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7$   | 0.81357  |
| 0.64853                   | $\text{H}_3\text{BO}_3 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  | 1.5419   |
| 5.5198                    | $\text{I} \leftrightarrow \text{Na}$  | 0.18117  |
| 0.84662                   | $\text{I} \leftrightarrow \text{NaI}$   | 1.1812   |
| 4.0949                    | $\text{I} \leftrightarrow \text{Na}_2\text{O}$  | 0.24420  |
| 2.5029                    | $\text{KBF}_4 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7$  | 0.39954  |
| 1.3206                    | $\text{KBF}_4 \leftrightarrow \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$   | 0.75724  |
| 0.91360                   | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{Na}_2\text{HAsO}_3$   | 1.0946   |
| 0.83497                   | $\text{Mg}_2\text{As}_2\text{O}_7 \leftrightarrow \text{Na}_2\text{HAsO}_4$   | 1.1976   |
| 0.81462                   | $\text{MgCl}_2 \leftrightarrow \text{NaCl}$   | 1.2276   |
| 0.67882                   | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Na}_3\text{PO}_4$  | 1.4731   |
| 0.78392                   | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Na}_2\text{HPO}_4$   | 1.2757   |
| 0.31073                   | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{NaHPO}_4 \cdot 12\text{H}_2\text{O}$   | 3.2182   |
| 0.53229                   | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{NaNH}_4 \cdot \text{HPO}_4 \cdot 4\text{H}_2\text{O}$                                | 1.8787   |
| 0.49897                   | $\text{Mg}_2\text{P}_2\text{O}_7 \leftrightarrow \text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$                                | 2.0041   |
| 4.4759                    | $\text{NaBr} \leftrightarrow \text{Na}$   | 0.22342  |
| 3.3205                    | $\text{NaBr} \leftrightarrow \text{Na}_2\text{O}$   | 0.30116  |
| 65.502                    | $\text{NaOAc} \cdot \text{Mg}(\text{OAc})_2 \cdot \text{UO}_2(\text{OAc})_2 \cdot 6\frac{1}{2}\text{H}_2\text{O} \leftrightarrow \text{Na}$ | 0.015267 |
| 14.635                    | Triple $\text{MgOAc} \leftrightarrow \text{NaBr}$   | 0.066331 |
| 28.416                    | Triple $\text{MgOAc} \leftrightarrow \text{Na}_2\text{CO}_3$  | 0.035192 |
| 25.768                    | Triple $\text{MgOAc} \leftrightarrow \text{NaCl}$   | 0.038809 |
| 17.926                    | Triple $\text{MgOAc} \leftrightarrow \text{NaHCO}_3$  | 0.055785 |
| 10.047                    | Triple $\text{MgOAc} \leftrightarrow \text{NaI}$  | 0.099535 |
| 37.650                    | Triple $\text{MgOAc} \leftrightarrow \text{NaOH}$   | 0.026560 |
| 48.594                    | Triple $\text{MgOAc} \leftrightarrow \text{Na}_2\text{O}$   | 0.020579 |
| 21.204                    | Triple $\text{MgOAc} \leftrightarrow \text{Na}_2\text{SO}_4$  | 0.047161 |
| 66.894                    | $\text{NaOAc} \cdot \text{Zn}(\text{OAc})_2 \cdot \text{UO}_2(\text{OAc})_2 \cdot 6\text{H}_2\text{O} \leftrightarrow \text{Na}$            | 0.014949 |
| 14.946                    | Triple $\text{ZnOAc} \leftrightarrow \text{NaBr}$   | 0.066909 |
| 29.020                    | Triple $\text{ZnOAc} \leftrightarrow \text{Na}_2\text{CO}_3$  | 0.034459 |
| 26.315                    | Triple $\text{ZnOAc} \leftrightarrow \text{NaCl}$   | 0.038002 |
| 18.307                    | Triple $\text{ZnOAc} \leftrightarrow \text{NaHCO}_3$  | 0.054624 |
| 10.260                    | Triple $\text{ZnOAc} \leftrightarrow \text{NaI}$  | 0.097464 |
| 38.451                    | Triple $\text{ZnOAc} \leftrightarrow \text{NaOH}$   | 0.026008 |
| 49.626                    | Triple $\text{ZnOAc} \leftrightarrow \text{Na}_2\text{O}$   | 0.020151 |
| 21.654                    | Triple $\text{ZnOAc} \leftrightarrow \text{Na}_2\text{SO}_4$  | 0.046180 |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                             |  | Factor  |
|------------------------------------|--|---------|
| <b>SODIUM</b> ( <i>continued</i> ) |  |         |
| <b>Na = 22.9898</b>                |  |         |
| 2.5421                             | $\text{NaCl} \leftrightarrow \text{Na}$  | 0.39337 |
| 1.1028                             | $\text{NaCl} \leftrightarrow \text{Na}_2\text{CO}_3$   | 0.90678 |
| 0.69569                            | $\text{NaCl} \leftrightarrow \text{NaHCO}_3$   | 1.4374  |
| 0.82337                            | $\text{NaCl} \leftrightarrow \text{Na}_2\text{HPO}_4$  | 1.2145  |
| 1.8859                             | $\text{NaCl} \leftrightarrow \text{Na}_2\text{O}$  | 0.53025 |
| 0.82291                            | $\text{NaCl} \leftrightarrow \text{Na}_2\text{SO}_4$   | 1.2152  |
| 0.74267                            | $\text{NaClO}_3 \leftrightarrow \text{AgCl}$   | 1.3465  |
| 1.8213                             | $\text{NaClO}_3 \leftrightarrow \text{NaCl}$   | 0.54907 |
| 0.85432                            | $\text{NaClO}_4 \leftrightarrow \text{AgCl}$   | 1.1705  |
| 2.0950                             | $\text{NaClO}_4 \leftrightarrow \text{NaCl}$   | 0.47732 |
| 2.3051                             | $\text{Na}_2\text{CO}_3 \leftrightarrow \text{Na}$   | 0.43381 |
| 0.63084                            | $\text{Na}_2\text{CO}_3 \leftrightarrow \text{NaHCO}_3$  | 1.5852  |
| 1.7101                             | $\text{Na}_2\text{CO}_3 \leftrightarrow \text{Na}_2\text{O}$   | 0.58476 |
| 1.3250                             | $\text{Na}_2\text{CO}_3 \leftrightarrow \text{NaOH}$   | 0.75473 |
| 3.6541                             | $\text{NaHCO}_3 \leftrightarrow \text{Na}$   | 0.27367 |
| 2.7108                             | $\text{NaHCO}_3 \leftrightarrow \text{Na}_2\text{O}$   | 0.36889 |
| 6.5198                             | $\text{NaI} \leftrightarrow \text{Na}$   | 0.15338 |
| 4.8368                             | $\text{NaI} \leftrightarrow \text{Na}_2\text{O}$   | 0.20675 |
| 1.3480                             | $\text{Na}_2\text{O} \leftrightarrow \text{Na}$  | 0.74186 |
| 0.43659                            | $\text{Na}_2\text{O} \leftrightarrow \text{Na}_2\text{HPO}_4$  | 2.2905  |
| 0.36460                            | $\text{Na}_2\text{O} \leftrightarrow \text{NaNO}_3$  | 2.7427  |
| 0.77480                            | $\text{Na}_2\text{O} \leftrightarrow \text{NaOH}$  | 1.2907  |
| 0.93653                            | $\text{Na}_4\text{P}_2\text{O}_7 \leftrightarrow \text{Na}_2\text{HPO}_4$                            | 1.0678  |
| 0.37122                            | $\text{Na}_4\text{P}_2\text{O}_7 \leftrightarrow \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ | 2.6938  |
| 3.0892                             | $\text{Na}_2\text{SO}_4 \leftrightarrow \text{Na}$   | 0.32371 |
| 1.3401                             | $\text{Na}_2\text{SO}_4 \leftrightarrow \text{Na}_2\text{CO}_3$                                      | 0.74620 |
| 0.49640                            | $\text{Na}_2\text{SO}_4 \leftrightarrow \text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$           | 2.0145  |
| 2.2917                             | $\text{Na}_2\text{SO}_4 \leftrightarrow \text{Na}_2\text{O}$   | 0.43635 |
| 0.16480                            | $\text{N} \leftrightarrow \text{NaNO}_3$   | 6.0680  |
| 0.20038                            | $\text{NH}_3 \leftrightarrow \text{NaNO}_3$  | 4.9906  |
| 0.081461                           | $\text{NH}_3 \leftrightarrow \text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$                    | 12.276  |
| 0.35303                            | $\text{NO} \leftrightarrow \text{NaNO}_3$  | 2.8326  |
| 0.63539                            | $\text{N}_2\text{O}_5 \leftrightarrow \text{NaNO}_3$   | 1.5738  |
| 1.7427                             | $\text{N}_2\text{O}_5 \leftrightarrow \text{Na}_2\text{O}$   | 0.57383 |
| 0.49993                            | $\text{P}_2\text{O}_5 \leftrightarrow \text{Na}_2\text{HPO}_4$                                       | 2.0003  |
| 0.19816                            | $\text{P}_2\text{O}_5 \leftrightarrow \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$            | 5.0464  |
| 0.33946                            | $\text{P}_2\text{O}_5 \leftrightarrow \text{NaNH}_4\text{HPO}_4 \cdot \text{H}_2\text{O}$            | 2.9459  |
| 0.61564                            | $\text{SO}_2 \leftrightarrow \text{NaHSO}_3$   | 1.6243  |
| 0.50828                            | $\text{SO}_2 \leftrightarrow \text{Na}_2\text{SO}_3$   | 1.9674  |
| 0.25407                            | $\text{SO}_2 \leftrightarrow \text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$                       | 3.9360  |
| 1.2918                             | $\text{SO}_2 \leftrightarrow \text{Na}_2\text{O}$  | 0.77414 |
| 0.56366                            | $\text{SO}_2 \leftrightarrow \text{Na}_2\text{SO}_4$   | 1.7741  |
| <b>STRONTIUM</b>                   |  |         |
| <b>Sr = 87.62</b>                  |  |         |
| 0.29811                            | $\text{CO}_2 \leftrightarrow \text{SrCO}_3$  | 3.3545  |
| 0.77265                            | $\text{SO}_3 \leftrightarrow \text{SrO}$   | 1.2942  |
| 0.43588                            | $\text{SO}_3 \leftrightarrow \text{SrSO}_4$  | 2.2942  |
| 0.41402                            | $\text{Sr} \leftrightarrow \text{Sr}(\text{NO}_3)_2$   | 2.4153  |
| 1.6849                             | $\text{SrCO}_3 \leftrightarrow \text{Sr}$  | 0.59351 |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor                       |  | Factor   |
|------------------------------|--|----------|
| <b>STRONTIUM (continued)</b> |  |          |
| <b>Sr = 87.62</b>            |  |          |
| 0.93124                      | $\text{SrCO}_3 \leftrightarrow \text{SrCl}_2$                                | 1.0738   |
| 0.70424                      | $\text{SrCO}_3 \leftrightarrow \text{Sr}(\text{HCO}_3)_2$                    | 1.4200   |
| 0.69759                      | $\text{SrCO}_3 \leftrightarrow \text{Sr}(\text{NO}_3)_2$                     | 1.4335   |
| 1.1826                       | $\text{SrO} \leftrightarrow \text{Sr}$                                       | 0.84559  |
| 0.65363                      | $\text{SrO} \leftrightarrow \text{SrCl}_2$                                   | 1.5299   |
| 0.70189                      | $\text{SrO} \leftrightarrow \text{SrCO}_3$                                   | 1.4247   |
| 0.49430                      | $\text{SrO} \leftrightarrow \text{Sr}(\text{HCO}_3)_2$                       | 2.0231   |
| 0.48963                      | $\text{SrO} \leftrightarrow \text{Sr}(\text{NO}_3)_2$                        | 2.0424   |
| 2.0963                       | $\text{SrSO}_4 \leftrightarrow \text{Sr}$                                    | 0.47703  |
| 1.1586                       | $\text{SrSO}_4 \leftrightarrow \text{SrCl}_2$                                | 0.86308  |
| 1.2442                       | $\text{SrSO}_4 \leftrightarrow \text{SrCO}_3$                                | 0.80373  |
| 0.86793                      | $\text{SrSO}_4 \leftrightarrow \text{Sr}(\text{NO}_3)_2$                     | 1.1522   |
| 1.7726                       | $\text{SrSO}_4 \leftrightarrow \text{SrO}$                                   | 0.56413  |
| <b>SULFUR</b>                |  |          |
| <b>S = 32.06</b>             |  |          |
| 2.4064                       | $\text{As}_2\text{S}_3 \leftrightarrow \text{H}_2\text{S}$                   | 0.41556  |
| 2.5577                       | $\text{As}_2\text{S}_3 \leftrightarrow \text{S}$                             | 0.39097  |
| 3.8906                       | $\text{BaSO}_4 \leftrightarrow \text{FeS}_2$                                 | 0.25703  |
| 6.8486                       | $\text{BaSO}_4 \leftrightarrow \text{H}_2\text{S}$                           | 0.14602  |
| 2.8436                       | $\text{BaSO}_4 \leftrightarrow \text{H}_2\text{SO}_3$                        | 0.35166  |
| 2.3797                       | $\text{BaSO}_4 \leftrightarrow \text{H}_2\text{SO}_4$                        | 0.42022  |
| 7.2792                       | $\text{BaSO}_4 \leftrightarrow \text{S}$                                     | 0.13738  |
| 3.6433                       | $\text{BaSO}_4 \leftrightarrow \text{SO}_2$                                  | 0.27448  |
| 2.9152                       | $\text{BaSO}_4 \leftrightarrow \text{SO}_3$                                  | 0.34302  |
| 2.4297                       | $\text{BaSO}_4 \leftrightarrow \text{SO}_4$                                  | 0.41158  |
| 4.2388                       | $\text{CdS} \leftrightarrow \text{H}_2\text{S}$                              | 0.23591  |
| 4.5054                       | $\text{CdS} \leftrightarrow \text{S}$  | 0.22196  |
| 1.2250                       | $\text{H}_2\text{SO}_4 \leftrightarrow \text{SO}_3$                          | 0.81631  |
| 1.6505                       | $(\text{NH}_4)_2\text{SO}_4 \leftrightarrow \text{SO}_3$                     | 0.60589  |
| 1.3473                       | $(\text{NH}_4)_2\text{SO}_4 \leftrightarrow \text{H}_2\text{SO}_4$           | 0.74223  |
| 2.3492                       | $\text{SO}_3 \leftrightarrow \text{H}_2\text{S}$                             | 0.42567  |
| <b>TANTALUM</b>              |  |          |
| <b>Ta = 180.948</b>          |  |          |
| 0.81898                      | $\text{Ta} \leftrightarrow \text{Ta}_2\text{O}_5$                            | 1.2210   |
| 0.50515                      | $\text{Ta} \leftrightarrow \text{TaCl}_5$                                    | 1.9796   |
| 16.065                       | $\text{TaC} \leftrightarrow \text{C}$  | 0.062246 |
| 1.0664                       | $\text{TaC} \leftrightarrow \text{Ta}$                                       | 0.93776  |
| 0.61680                      | $\text{Ta}_2\text{O}_5 \leftrightarrow \text{TaCl}_5$                        | 1.6213   |
| 1.0376                       | $\text{Ta}_2\text{O}_5 \leftrightarrow \text{Ta}_2\text{O}_4$                | 0.96379  |
| <b>TELLURIUM</b>             |  |          |
| <b>Te = 127.60</b>           |  |          |
| 0.65906                      | $\text{Te} \leftrightarrow \text{H}_2\text{TeO}_4$                           | 1.5173   |
| 0.55565                      | $\text{Te} \leftrightarrow \text{H}_2\text{TeO}_4 \cdot 2\text{H}_2\text{O}$ | 1.7997   |
| 0.79950                      | $\text{Te} \leftrightarrow \text{TeO}_2$                                     | 1.2508   |
| 0.72665                      | $\text{Te} \leftrightarrow \text{TeO}_3$                                     | 1.3762   |
| 1.5645                       | $(\text{TeO}_2)_2\text{SO}_3 \leftrightarrow \text{Te}$                      | 0.63918  |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor              |   | Factor  |
|---------------------|---|---------|
| <b>THALLIUM</b>     |   |         |
| <b>Tl = 204.37</b>  |   |         |
| 0.87198             | $\text{Tl} \leftrightarrow \text{Tl}_2\text{CO}_3$                              | 1.1468  |
| 0.85218             | $\text{Tl} \leftrightarrow \text{TlCl}$   | 1.1735  |
| 0.61693             | $\text{Tl} \leftrightarrow \text{TlI}$  | 1.6209  |
| 0.76724             | $\text{Tl} \leftrightarrow \text{TlNO}_3$                                       | 1.3034  |
| 0.96232             | $\text{Tl} \leftrightarrow \text{Tl}_2\text{O}$                                 | 1.0391  |
| 1.2838              | $\text{Tl}_2\text{CrO}_4 \leftrightarrow \text{Tl}$                             | 0.77895 |
| 1.4750              | $\text{TiHSO}_4 \leftrightarrow \text{Tl}$                                      | 0.67798 |
| 1.9977              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{Tl}$                            | 0.50057 |
| 1.7024              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{TlCl}$                          | 0.58740 |
| 1.7420              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{Tl}_2\text{CO}_3$               | 0.57406 |
| 1.2325              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{TlI}$                           | 0.81139 |
| 1.5327              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{TlNO}_3$                        | 0.65243 |
| 1.9225              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{Tl}_2\text{O}$                  | 0.52017 |
| 1.6176              | $\text{Tl}_2\text{PtCl}_6 \leftrightarrow \text{Tl}_2\text{SO}_4$               | 0.61821 |
| 1.2350              | $\text{Tl}_2\text{SO}_4 \leftrightarrow \text{Tl}$                              | 0.80971 |
| <b>THORIUM</b>      |   |         |
| <b>Th = 232.038</b> |   |         |
| 1.1379              | $\text{ThO}_2 \leftrightarrow \text{Th}$  | 0.87881 |
| 0.70627             | $\text{ThO}_2 \leftrightarrow \text{ThCl}_4$                                    | 1.4159  |
| 0.44893             | $\text{ThO}_2 \leftrightarrow \text{Th(NO}_3)_4 \cdot 6\text{H}_2\text{O}$      | 2.2275  |
| <b>TIN</b>          |   |         |
| <b>Sn = 118.69</b>  |   |         |
| 0.62600             | $\text{Sn} \leftrightarrow \text{SnCl}_2$                                       | 1.5974  |
| 0.52604             | $\text{Sn} \leftrightarrow \text{SnCl}_2 \cdot 2\text{H}_2\text{O}$             | 1.9010  |
| 0.45562             | $\text{Sn} \leftrightarrow \text{SnCl}_4$                                       | 2.1948  |
| 0.32297             | $\text{Sn} \leftrightarrow \text{SnCl}_4 \cdot (\text{NH}_4\text{Cl})_2$        | 3.0962  |
| 0.88121             | $\text{Sn} \leftrightarrow \text{SnO}$  | 1.1348  |
| 0.78764             | $\text{Sn} \leftrightarrow \text{SnO}_2$  | 1.2696  |
| 0.79478             | $\text{SnO}_2 \leftrightarrow \text{SnCl}_2$                                    | 1.2582  |
| 0.66786             | $\text{SnO}_2 \leftrightarrow \text{SnCl}_2 \cdot 2\text{H}_2\text{O}$          | 1.4973  |
| 0.57846             | $\text{SnO}_2 \leftrightarrow \text{SnCl}_4$                                    | 1.7287  |
| 0.41005             | $\text{SnO}_2 \leftrightarrow \text{SnCl}_4 \cdot (\text{NH}_4\text{Cl})_2$     | 2.4387  |
| 1.1188              | $\text{SnO}_2 \leftrightarrow \text{SnO}$                                       | 0.89382 |
| <b>TITANIUM</b>     |   |         |
| <b>Ti = 47.867</b>  |   |         |
| 2.1059              | $\text{K}_2\text{TiF}_6 \leftrightarrow \text{F}$                               | 0.47485 |
| 3.0699              | $\text{K}_2\text{TiF}_6 \leftrightarrow \text{K}$                               | 0.32574 |
| 2.0660              | $\text{K}_2\text{TiF}_6 \leftrightarrow 2\text{KF}$                             | 0.48403 |
| 1.2752              | $\text{K}_2\text{TiF}_6 \leftrightarrow 2(\text{KF} \cdot 2\text{H}_2\text{O})$ | 0.78421 |
| 5.0150              | $\text{K}_2\text{TiF}_6 \leftrightarrow \text{Ti}$                              | 0.19940 |
| 3.0057              | $\text{K}_2\text{TiF}_6 \leftrightarrow \text{TiO}_2$                           | 0.33270 |
| 3.9853              | $\text{Ti} \leftrightarrow \text{C}$  | 0.25092 |
| 4.9853              | $\text{TiC} \leftrightarrow \text{C}$   | 0.20059 |
| 1.2509              | $\text{TiC} \leftrightarrow \text{Ti}$  | 0.79940 |
| 1.6299              | $\text{TiF}_4 \leftrightarrow \text{F}$   | 0.61354 |
| 1.6685              | $\text{TiO}_2 \leftrightarrow \text{Ti}$  | 0.59934 |

TABLE 11.19 Gravimetric Factors (Continued)

| Factor             |   | Factor   |
|--------------------|---|----------|
| <b>TUNGSTEN</b>    |   |          |
| <b>W = 183.85</b>  |   |          |
| 3.9348             | $\text{FeWO}_4 \leftrightarrow \text{Fe}_3\text{O}_4$                                       | 0.25414  |
| 1.3099             | $\text{FeWO}_4 \leftrightarrow \text{WO}_3$   | 0.76344  |
| 6.7515             | $\text{MgWO}_4 \leftrightarrow \text{MgO}$  | 0.14812  |
| 1.1739             | $\text{MgWO}_4 \leftrightarrow \text{WO}_3$   | 0.85189  |
| 4.2684             | $\text{MnWO}_4 \leftrightarrow \text{MnO}$  | 0.23428  |
| 1.3060             | $\text{MnWO}_4 \leftrightarrow \text{WO}_3$   | 0.76571  |
| 2.0387             | $\text{PbWO}_4 \leftrightarrow \text{PbO}$  | 0.49051  |
| 2.4751             | $\text{PbWO}_4 \leftrightarrow \text{W}$  | 0.40403  |
| 1.9626             | $\text{PbWO}_4 \leftrightarrow \text{WO}_3$   | 0.50952  |
| 15.307             | $\text{W} \leftrightarrow \text{C}$   | 0.065330 |
| 0.96837            | $\text{W} \leftrightarrow \text{W}_2\text{C}$   | 1.0327   |
| 0.93868            | $\text{W} \leftrightarrow \text{WC}$  | 1.0653   |
| 31.614             | $\text{W}_2\text{C} \leftrightarrow \text{C}$   | 0.031632 |
| 16.307             | $\text{WC} \leftrightarrow \text{C}$  | 0.061324 |
| 1.1741             | $\text{WO}_2 \leftrightarrow \text{W}$  | 0.85175  |
| 4.1515             | $\text{WO}_3 \leftrightarrow \text{Fe}$   | 0.24088  |
| 1.2611             | $\text{WO}_3 \leftrightarrow \text{W}$  | 0.79297  |
| <b>URANIUM</b>     |   |          |
| <b>U = 238.03</b>  |   |          |
| 1.1344             | $\text{UO}_2 \leftrightarrow \text{U}$  | 0.88149  |
| 1.1792             | $\text{U}_3\text{O}_8 \leftrightarrow \text{U}$   | 0.84800  |
| 1.0395             | $\text{U}_3\text{O}_8 \leftrightarrow \text{UO}_2$  | 0.96200  |
| 0.55901            | $\text{U}_3\text{O}_8 \leftrightarrow \text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ | 1.7889   |
| 1.4998             | $\text{U}_2\text{P}_2\text{O}_{11} \leftrightarrow \text{U}$                                | 0.66675  |
| 1.3221             | $\text{U}_2\text{P}_2\text{O}_{11} \leftrightarrow \text{UO}_2$                             | 0.75639  |
| <b>VANADIUM</b>    |   |          |
| <b>V = 50.941</b>  |   |          |
| 5.2413             | $\text{VC} \leftrightarrow \text{C}$  | 0.19079  |
| 1.7852             | $\text{V}_2\text{O}_5 \leftrightarrow \text{V}$   | 0.56017  |
| 0.79120            | $\text{V}_2\text{O}_5 \leftrightarrow \text{VO}_4$  | 1.2639   |
| <b>YTTERBIUM</b>   |   |          |
| <b>Yb = 173.04</b> |   |          |
| 1.1387             | $\text{Yb}_2\text{O}_3 \leftrightarrow \text{Yb}$   | 0.87820  |
| <b>ZINC</b>        |   |          |
| <b>Zn = 65.38</b>  |   |          |
| 2.3955             | $\text{BaSO}_4 \leftrightarrow \text{ZnS}$  | 0.41745  |
| 0.81171            | $\text{BaSO}_4 \leftrightarrow \text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$                     | 1.2320   |
| 0.80338            | $\text{Zn} \leftrightarrow \text{ZnO}$  | 1.2447   |
| 2.7288             | $\text{ZnNH}_4\text{PO}_4 \leftrightarrow \text{Zn}$  | 0.36646  |
| 2.1922             | $\text{ZnNH}_4\text{PO}_4 \leftrightarrow \text{ZnO}$                                       | 0.45616  |
| 0.59707            | $\text{ZnO} \leftrightarrow \text{ZnCl}_2$  | 1.6748   |
| 0.64898            | $\text{ZnO} \leftrightarrow \text{ZnCO}_3$  | 1.5409   |
| 0.28298            | $\text{ZnO} \leftrightarrow \text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$                        | 3.5338   |
| 2.3304             | $\text{Zn}_3\text{P}_2\text{O}_7 \leftrightarrow \text{Zn}$                                 | 0.42911  |
| 1.8722             | $\text{Zn}_3\text{P}_2\text{O}_7 \leftrightarrow \text{ZnO}$                                | 0.53413  |
| 1.4905             | $\text{ZnS} \leftrightarrow \text{Zn}$  | 0.67091  |
| 1.1974             | $\text{ZnS} \leftrightarrow \text{ZnO}$   | 0.83512  |
| 0.33885            | $\text{ZnS} \leftrightarrow \text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$                        | 2.9511   |

TABLE 11.19 Gravimetric Factors (*Continued*)

| Factor                                |  | Factor  |
|---------------------------------------|--|---------|
| <b>ZIRCONIUM</b><br><b>Zr = 91.22</b> |  |         |
| 2.4864                                | $K_2ZrF_6 \leftrightarrow F$                 | 0.40219 |
| 2.4390                                | $K_2ZrF_6 \leftrightarrow 2KF$               | 0.41001 |
| 1.5054                                | $K_2ZrF_6 \leftrightarrow 2(KF \cdot 2H_2O)$ | 0.66427 |
| 3.1069                                | $K_2ZrF_6 \leftrightarrow Zr$                | 0.32187 |
| 2.3000                                | $K_2ZrF_6 \leftrightarrow ZrO_2$             | 0.43478 |
| 8.5946                                | $ZrC \leftrightarrow C$                      | 0.11635 |
| 2.2004                                | $ZrF_4 \leftrightarrow F$                    | 0.45447 |
| 1.3508                                | $ZrO_2 \leftrightarrow Zr$                   | 0.74030 |
| 0.46470                               | $ZrO_2 \leftrightarrow ZrP_2O_7$             | 2.1519  |

TABLE 11.20 Elements Precipitated by General Analytical Reagents

This table includes the more common reagents used in gravimetric determinations. The lists of elements precipitated are not in all cases exhaustive. The usual solvent for a precipitating agent is indicated in parentheses after its name or formula. When the symbol of an element or radical is italicized, the element may be quantitatively determined by the use of the reagent in question.

| Reagent   | Conditions  | Substances precipitated  |
|---|---|--|
| Ammonia, $NH_3$ (aqueous)   | After removal of acid sulfide group.  | <i>Al</i> , <i>Au</i> , <i>Be</i> , <i>Co</i> , <i>Cr</i> , <i>Cu</i> , <i>Fe</i> , <i>Ga</i> , <i>In</i> , <i>Ir</i> , <i>La</i> , <i>Nb</i> , <i>Ni</i> , <i>Os</i> , <i>P</i> , <i>Pb</i> , <i>rare earths</i> , <i>Sc</i> , <i>Si</i> , <i>Sn</i> , <i>Ta</i> , <i>Th</i> , <i>Ti</i> , <i>U</i> , <i>V</i> , <i>Y</i> , <i>Zn</i> , <i>Zr</i> |
| Ammonium polysulfide, $(NH_4)_2S_x$ (aqueous)                         | After removal of acid sulfide and $(NH_4)_2S$ groups.   | <i>Co</i> , <i>Mn</i> , <i>Ni</i> , <i>Si</i> , <i>Tl</i> , <i>V</i> , <i>W</i> , <i>Zn</i>  |
| Anthranilic acid, $NH_2C_6H_4COOH$ (aqueous)                          | 1% aqueous solution (pH 6); Cu separated from others at pH 2.9.   | <i>Ag</i> , <i>Cd</i> , <i>Co</i> , <i>Cu</i> , <i>Fe</i> , <i>Hg</i> , <i>Mn</i> , <i>Ni</i> , <i>Pb</i> , <i>Zn</i>  |
| $\alpha$ -Benzoin oxime, $C_6H_5CHOHC(=NOH)C_6H_5$ (1–2% alcohol)     | (a) Strongly acid medium.   | (a) <i>Cr(VI)</i> , <i>Mo(VI)</i> , <i>Nb</i> , <i>Pd(II)</i> , <i>Ta(V)</i> , <i>V(V)</i> , <i>W(VI)</i>  |
| Benzidine, $H_2NC_6H_4C_6H_4NH_2$ (alcohol), 0.1 <i>M</i> HCl         | (b) Ammoniacal tartrate medium.   | (b) Above list   |
| <i>N</i> -Benzoylphenylhydroxylamine, $C_6H_5CO(C_6H_5)NOH$ (aqueous) | Similar to cupferron ( <i>q.v.</i> ). <i>Cu</i> , <i>Fe(III)</i> , and <i>Al</i> complexes can be weighed as such; <i>Ti</i> compound must be ignited to the oxide. | <i>Cd</i> , <i>Fe(III)</i> , $IO_3^-$ , $PO_4^{3-}$ , $SO_4^{2-}$ , <i>W(VI)</i><br>See Cupferron  |
| Cinchonine, $C_{19}H_{21}N_2OH$ , 6 <i>M</i> HCl                      |   | <i>Ir</i> , <i>Mo</i> , <i>Pt</i> , <i>W</i>   |
| Cupferron, $C_6H_5N(NO)ONH_4$ (aqueous)                               | Group precipitant for several higher-charged metal ions from strongly acid solution. Precipitate ignited to metal oxide.  | <i>Al</i> , <i>Bi</i> , <i>Cu</i> , <i>Fe</i> , <i>Ga</i> , <i>La</i> , <i>Mo</i> , <i>Nb</i> , <i>Pd</i> , <i>rare earths</i> , <i>Sb</i> , <i>Sn</i> , <i>Ta</i> , <i>Th</i> , <i>Ti</i> , <i>Tl</i> , <i>U</i> , <i>V</i> , <i>W</i> , <i>Zr</i>  |
| 1,2-Cyclohexanedionedioxime   | More water soluble than dimethylglyoxime; less subject to coprecipitation with metal chelate.   | See Dimethylglyoxime   |

**TABLE 11.20** Elements Precipitated by General Analytical Reagents (*Continued*)

| Reagent  | Conditions  | Substances precipitated  |
|--|---|--|
| Diammonium hydrogen phosphate, $(\text{NH}_4)_2\text{HPO}_4$ (aqueous)   | (a) Acid medium.<br>(b) Ammoniacal medium containing citrate or tartrate.   | (a) <i>Bi, Co, Hf, In, Ti, Zn, Zr</i><br>(b) <i>Au, Ba, Be, Ca, Hg, In, La, Mg, Mn, Pb, rare earths, Sr, Th, U, Zr</i>   |
| Dimethylglyoxime, $[\text{CH}_3\text{C}(\text{NOH})_2]$ (alcohol)  | (a) Dilute $\text{HCl}$ or $\text{H}_2\text{SO}_4$ medium.<br>(b) Ammoniacal tartrate medium about pH 8. Weighed as such. | (a) <i>Au, Pd, Se</i><br>(b) <i>Ni</i> (and <i>Co, Fe</i> if present in large amounts)   |
| Hydrazine, $\text{N}_2\text{H}_4$ (aqueous)  |   | <i>Ag, Au, Cu, Hg, Ir, Os, Pd, Pt, Rh, Ru, Se, Te</i>  |
| Hydrogen sulfide, $\text{H}_2\text{S}$   | (a) $0.2\text{--}0.5M \text{ H}^+$ .  | (a) <i>Ag, As, Au, Bi, Cd, Cu, Ge, Hg, In, Ir, Mo, Os, Pb, Pd, Pt, Re, Rh, Ru, Sb, Se, Sn, Te, Tl, V, W, Zn</i>  |
| 4-Hydroxyphenylarsonic acid, $\text{C}_6\text{H}_4(\text{OH})\text{AsO}(\text{OH})_2$ (aqueous)                            | (b) Ammoniacal solution after removal of acid sulfide group.<br>Dilute acid solution.                                     | (b) <i>Co, Fe, Ga, In, Mn, Ni, Tl, U, V, Zn</i><br><i>Ce, Fe, Sn, Th, Ti, Zr</i>   |
| 8-Hydroxyquinoline (oxine), $\text{C}_9\text{H}_6\text{NOH}$ , (alcohol)   | (a) $\text{HOAc}\text{--}\text{OAc}^-$ buffer.<br>(b) Ammoniacal solution.  | (a) <i>Ag, Al, Bi, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, La, Mn, Mo, Nb, Ni, Pb, Pd, rare earths, Sb, Ta, Th, Ti, V, W, Zn, Zr</i><br>(b) Same as in (a) except for <i>Ag</i> ; in addition, <i>Ba, Be, Ca, Mg, Sn, Sr</i> |
| 2-Mercaptobenzothiazole, $\text{C}_6\text{H}_4(\text{SCN})\text{SH}$ (acetic acid solution)                                | Ammoniacal solution, except for <i>Cu</i> , when a dilute acid solution is used.  | <i>Ag, Au, Bi, Cd, Cu, Hg, Ir, Pb, Pt, Rh, Tl</i>  |
| Nitron (diphenylenedianilohydrotriazole), $\text{C}_{20}\text{H}_{16}\text{N}_4$ , (5% acetic acid)                        | Dilute $\text{H}_2\text{SO}_4$ medium.  | <i>B, ClO_3^-, ClO_4^-, NO_3^-, ReO_4^-, W</i>   |
| 1-Nitroso-2-naphthol, $\text{C}_{10}\text{H}_6(\text{NO})\text{OH}$ (very dilute alkali)                                   | Selective for <i>Co</i> ; acid solution. Precipitate ignited to $\text{Co}_3\text{O}_4$ .                                 | <i>Ag, Au, B, Co, Cr, Cu, Fe, Mo, Pd, Ti, V, W, Zr</i>   |
| Oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$ , (aqueous)  | Dilute acid solution.   | <i>Ag, Au, Cu, Hg, La, Ni, Pb, rare earths, Sc, Th, U(IV), W, Zr</i>   |
| Phenylarsonic acid, $\text{C}_6\text{H}_5\text{AsO}(\text{OH})_2$ , (aqueous)  | Selective precipitants for quadrivalent metals in acid solution. Metals weighed as dioxides.                              | <i>Bi, Ce(IV), Fe, Hf, Mg, Sn, Ta, Th, Ti, U(IV), W, Zr</i>  |
| Phenylthiohydantoic acid, $\text{C}_6\text{H}_5\text{N}=\text{C}(\text{NH}_2)\text{SCH}_2\text{COOH}$ (aqueous or alcohol) |   | <i>Bi, Cd, Co, Cu, Fe, Hg, Ni, Pb, Sb</i>  |
| Picrolonic acid, $\text{C}_{10}\text{H}_7\text{O}_5\text{N}_4\text{H}$ (aqueous)   | Neutral solution.   | <i>Ca, Mg, Pb, Th</i>  |
| Propylarsonic acid, $\text{C}_3\text{H}_9\text{AsO}(\text{OH})_2$ (aqueous)  | Preferred for <i>W</i> ; see Phenylarsonic acid.  |  |
| Pyridine plus thiocyanate  | Dilute acid solution.   | <i>Ag, Cd, Cu, Mn, Ni</i>  |
| Quinaldic acid, $\text{C}_9\text{H}_6\text{NCOOH}$ (aqueous)   | Dilute acid solution.   | <i>Ag, Cd, Co, Cu, Fe, Hg, Mo, Ni, Pb, Pd, Pt(II), U, W, Zn</i>  |
| Salicylaldoxime, $\text{C}_7\text{H}_5(\text{OH})\text{NOH}$ (alcohol)   | Dilute acid solution.   | <i>Ag, Bi, Cd, Co, Cu, Fe, Hg, Mg, Mn, Ni, Pb, Pd, V, Zn</i>   |
| Silver nitrate, $\text{AgNO}_3$ (aqueous)  | (a) Dilute $\text{HNO}_3$ solution.<br>(b) Acetate buffer, pH 5–7.  | (a) $\text{Br}^-$ , $\text{Cl}^-$ , $\text{I}^-$ , $\text{SCN}^-$<br>(b) <i>As(V), CN^-, OCN^-, IO_3^-, Mo(VI), N_3^-, S^{2-}, V(V)</i>  |

**TABLE 11.20** Elements Precipitated by General Analytical Reagents (*Continued*)

| Reagent   | Conditions  | Substances precipitated   |
|---|---|---|
| Sodium tetraphenylborate,<br>$\text{NaB}(\text{C}_6\text{H}_5)_4$ (aqueous)   | Specific for K group of alkali metals from dilute $\text{HNO}_3$ or $\text{HOAc}$ solution (pH 2), or pH 6.5 in presence of EDTA.   | <i>Cs, K, NH<sub>4</sub><sup>+</sup>, Rb</i>  |
| Tannic acid (tannin), $\text{C}_{14}\text{H}_{10}\text{O}_9$ (aqueous)  | Acts as negative colloid that is a flocculent for positively charged hydrous oxide sols. Noteworthy for W in acid solution, and for Ta (from Nb in acidic oxalate medium).                | <i>Al, Be, Cr, Ga, Ge, Nb, Sb, Sn, Ta, Th, Ti, U, V, W, Zr</i>  |
| Tartaric acid,<br>$\text{HOOC}(\text{CHOH})_2\text{COOH}$ (aqueous)   |   | <i>Ca, K, Mg, Sc, Sr, Ta</i>  |
| Tetraphenylarsonium chloride,<br>$(\text{C}_6\text{H}_5)_4\text{AsCl}$ (aqueous)                                    | $(\text{C}_6\text{H}_5)_4\text{AsTiCl}_4$ and $(\text{C}_6\text{H}_5)_4\text{AsReO}_4$ weighed as such.   | <i>Re, Ti</i>   |
| Thioglycolic- $\beta$ -aminonaphthalide, thionalide,<br>$\text{C}_{10}\text{H}_7\text{NHCOCH}_2\text{SH}$ (alcohol) | (a) Acid solution.<br>(b) Carbonate medium containing tartrate.<br>(c) Carbonate medium containing tartrate and cyanide.<br>(d) Strongly alkaline medium containing tartrate and cyanide. | (a) <i>Ag, As, Au, Bi, Cu, Hg, Os, Pb, Pd, Rh, Ru, Sb, Sn, Tl</i><br>(b) <i>Au, Cd, Cu, Hg(II), Tl(I)</i><br>(c) <i>Au, Bi, Pb, Sb, Sn, Tl</i><br>(d) <i>Tl</i> |

*Source:* J. A. Dean, ed., *Analytical Chemistry Handbook*, McGraw-Hill, New York, 1995.

**TABLE 11.21** Cleaning Solutions for Fritted Glassware

| Material                         | Cleaning solution   |
|----------------------------------|---|
| Fatty materials                  | Carbon tetrachloride.   |
| Organic matter                   | Hot concentrated sulfuric acid plus a few drops of sodium or potassium nitrate solution.  |
| Albumen                          | Hot aqueous ammonia or hot hydrochloric acid.   |
| Glucose                          | Hot mixed acid (sulfuric plus nitric acids).  |
| Copper or iron oxides            | Hot hydrochloric acid plus potassium chlorate.  |
| Mercury residue                  | Not nitric acid.  |
| Silver chloride                  | Aqueous ammonia or sodium thiosulfate.  |
| Aluminous and siliceous residues | A 2% hydrofluoric acid solution followed by concentrated sulfuric acid; rinse immediately with distilled water followed by a few milliliters of acetone. Repeat rinsing until all trace of acid is removed. |

**TABLE 11.22** Common Fluxes

| Flux   | Melting point, °C | Types of crucible used for fusion                   | Type of substances decomposed   |
|--|-------------------|---|---|
| $\text{Na}_2\text{CO}_3$   | 851               | Pt  | For silicates, and silica-containing samples; alumina-containing samples; insoluble phosphates and sulfates |
| $\text{Na}_2\text{CO}_3$ plus an oxidizing agent such as $\text{KNO}_3$ , $\text{KClO}_3$ , or $\text{Na}_2\text{O}_2$ |                   | Pt (do not use with $\text{Na}_2\text{O}_2$ ) or Ni | For samples needing an oxidizing agent  |
| $\text{NaOH}$ or $\text{KOH}$  | 320–380           | Au, Ag, Ni  | For silicates, silicon carbide, certain minerals  |
| $\text{Na}_2\text{O}_2$  | Decomposes        | Fe, Ni  | For sulfides, acid-insoluble alloys of Fe, Ni, Cr, Mo, W, and Li; Pt alloys; Cr, Sn, Zn minerals            |
| $\text{K}_2\text{S}_2\text{O}_7$   | 300               | Pt or porcelain                                     | Acid flux for insoluble oxides and oxide-containing samples   |
| $\text{B}_2\text{O}_3$   | 577               | Pt  | For silicates and oxides when alkalis are to be determined  |
| $\text{CaCO}_3$ plus $\text{NH}_4\text{Cl}$  |                   | Ni  | For decomposing silicates in the determination of alkali element  |

**TABLE 11.23** Membrane Filters

| Filter pore size, $\mu\text{m}$ | Maximum rigid particle to penetrate, $\mu\text{m}$ | Filter pore size, $\mu\text{m}$ | Maximum rigid particle to penetrate, $\mu\text{m}$ |
|---------------------------------|--|---------------------------------|--|
| 14                              | 17   | 0.65                            | 0.68   |
| 10                              | 12   | 0.60                            | 0.65   |
| 8                               | 9.4  | 0.45                            | 0.47   |
| 7                               | 9.0  | 0.30                            | 0.32   |
| 5                               | 6.2  | 0.22                            | 0.24   |
| 3                               | 3.9  | 0.20                            | 0.25   |
| 2                               | 2.5  | 0.10                            | 0.108  |
| 1.2                             | 1.5  | 0.05                            | 0.053  |
| 1.0                             | 1.1  | 0.025                           | 0.028  |
| 0.8                             | 0.95   |                                 |  |

**TABLE 11.24** Porosities of Fritted Glassware

| Porosity     | Nominal maximum pore size, $\mu\text{m}$ | Principal uses   |
|--------------|--|--|
| Extra coarse | 170–220                                  | Filtration of very coarse materials. Gas dispersion, gas washing, and extractor beds. Support of other filter materials.   |
| Coarse       | 40–60                                    | Filtration of coarse materials. Gas dispersion, gas washing, gas absorption. Mercury filtration. For extraction apparatus. |
| Medium       | 10–15                                    | Filtration of crystalline precipitates. Removal of “floaters” from distilled water.  |
| Fine         | 4–5.5                                    | Filtration of fine precipitates. As a mercury valve. In extraction apparatus.  |
| Very fine    | 2–2.5                                    | General bacteria filtrations.  |
| Ultra fine   | 0.9–1.4                                  | General bacteria filtrations.  |

**TABLE 11.25** Tolerances for Analytical Weights

By Alan D. Westland with Fred E. Beamish.

This table gives the individual and group tolerances established by the National Bureau of Standards (Washington, D.C.) for classes M, S, S-1, and P weights. Individual tolerances are “acceptance tolerances” for new weights. Group tolerances are defined by the National Bureau of Standards as follows: “The corrections of individual weights shall be such that no combination of weights that is intended to be used in a weighing shall differ from the sum of the nominal values by more than the amount listed under the group tolerances.”

For class S-1 weights, two-thirds of the weights in a set must be within one-half of the individual tolerances given below. No group tolerances have been specified for class P weights. See *Natl. Bur. Standards Circ.* 547, sec. 1 (1954).

| Denomination | Class M                  |                     | Class S                  |                     | Class S-1, individual tolerance, mg | Class P, individual tolerance, mg |
|--------------|--------------------------|---------------------|--------------------------|---------------------|-------------------------------------|-----------------------------------|
|              | Individual tolerance, mg | Group tolerance, mg | Individual tolerance, mg | Group tolerance, mg |                                     |                                   |
| 100 g        | 0.50                     | None specified      | 0.25                     | None specified      | 1.0                                 | 2.0                               |
| 50 g         | 0.25                     |                     | 0.12                     | 0.154               | 0.60                                | 1.2                               |
| 30 g         | 0.15                     |                     | 0.074                    |                     | 0.45                                | 0.90                              |
| 20 g         | 0.10                     |                     | 0.074                    |                     | 0.35                                | 0.70                              |
| 10 g         | 0.050                    |                     | 0.074                    |                     | 0.25                                | 0.50                              |
| 5 g          | 0.034                    | 0.065               | 0.054                    | 0.105               | 0.18                                | 0.36                              |
| 3 g          | 0.034                    |                     | 0.054                    |                     | 0.15                                | 0.14                              |
| 2 g          | 0.034                    |                     | 0.054                    |                     | 0.13                                | 0.26                              |
| 1 g          | 0.034                    |                     | 0.054                    |                     | 0.10                                | 0.20                              |
| 500 mg       | 0.0054                   | 0.0105              | 0.025                    | 0.055               | 0.080                               | 0.16                              |
| 300 mg       | 0.0054                   |                     | 0.025                    |                     | 0.070                               | 0.14                              |
| 200 mg       | 0.0054                   |                     | 0.025                    |                     | 0.060                               | 0.12                              |
| 100 mg       | 0.0054                   |                     | 0.025                    |                     | 0.050                               | 0.10                              |
| 50 mg        | 0.0054                   |                     | 0.014                    | 0.034               | 0.042                               | 0.085                             |
| 30 mg        | 0.0054                   | 0.0105              | 0.014                    |                     | 0.038                               | 0.076                             |
| 20 mg        | 0.0054                   |                     | 0.014                    |                     | 0.035                               | 0.070                             |

**TABLE 11.25** Tolerances for Analytical Weights (*Continued*)

| Denomination | Class M                        |                           | Class S                        |                           | Class S-1,<br>individual<br>tolerance,<br>mg | Class P,<br>individual<br>tolerance,<br>mg |
|--------------|--------------------------------|---------------------------|--------------------------------|---------------------------|--|--|
|              | Individual<br>tolerance,<br>mg | Group<br>tolerance,<br>mg | Individual<br>tolerance,<br>mg | Group<br>tolerance,<br>mg |  |  |
| 10 mg        | 0.0054                         | 0.0105                    | 0.014                          | 0.034                     | 0.030  | 0.060                                      |
| 5 mg         | 0.0054                         |                           | 0.014                          |                           | 0.028  | 0.055                                      |
| 3 mg         | 0.0054                         |                           | 0.014                          |                           | 0.026  | 0.052                                      |
| 2 mg         | 0.0054                         |                           | 0.014                          |                           | 0.025  | 0.050                                      |
| 1 mg         | 0.0054                         |                           | 0.014                          |                           | 0.025  | 0.050                                      |
| ½ mg         | 0.0054                         |                           | 0.014                          |                           | 0.025  | .....                                      |

**TABLE 11.26** Heating Temperatures, Composition of Weighing Forms, and Gravimetric Factors

The minimum temperature required for heating a pure precipitate to constant weight is frequently lower than that commonly recommended in gravimetric procedures. However, the higher temperature is very often still to be preferred in order to ensure that contaminating substances are expelled. The thermal stability ranges of various precipitates as deduced from thermograms are also tabulated. Where a stronger ignition is advisable, the safe upper limit can be ascertained.

Gravimetric factors are based on the 1993 International Atomic Weights. The factor Ag: 0.7526 given in the first line of the table indicates that the weight of precipitate obtained (AgCl) is to be multiplied by 0.7526 to calculate the corresponding weight of silver.

| Element | Thermal stability<br>range, °C | Final heating<br>temperature, °C | Composition of<br>weighing form                                  | Gravimetric factors                                 |
|---------|--------------------------------|----------------------------------|--|---|
| Ag      | 70–600                         | 130–150                          | AgCl   | Ag: 0.7526  |
| Al      | >475                           | 1200                             | Al <sub>2</sub> O <sub>3</sub>                                   | Al: 0.5293  |
|         | >743                           | >743                             | AlPO <sub>4</sub>  | Al: 0.2212; Al <sub>2</sub> O <sub>3</sub> : 0.4180 |
| As      | 102–220                        | 110                              | Al(C <sub>9</sub> H <sub>6</sub> NO) <sub>3</sub>                | Al: 0.0587; Al <sub>2</sub> O <sub>3</sub> : 0.1110 |
|         | 200–275                        | 105–110                          | Al <sub>2</sub> S <sub>3</sub>                                   | As: 0.6090; As <sub>2</sub> O <sub>3</sub> : 0.8041 |
|         |                                | 850                              | Mg <sub>2</sub> As <sub>2</sub> O <sub>7</sub>                   | As: 0.4827; As <sub>2</sub> O <sub>3</sub> : 0.6373 |
|         |                                | vacuum at 25                     | MgNH <sub>4</sub> AsO <sub>4</sub> · 6H <sub>2</sub> O           | As: 0.2589  |
| Au      | 20–957                         | 1060                             | Au   |   |
| Ba      | 780–1100                       | 780                              | BaSO <sub>4</sub>  | Ba: 0.5884; BaO: 0.6570                             |
|         | <60                            | <60                              | BaCrO <sub>4</sub>   | Ba: 0.5421; BaO: 0.6053                             |
| Be      | >900                           | 1000                             | BeO  | Be: 0.3603  |
| Bi      | 379–961                        | 100                              | BiOCl  | Bi: 0.8024; Bi <sub>2</sub> O <sub>3</sub> : 0.8946 |
|         |                                | 100                              | Bi(C <sub>12</sub> H <sub>10</sub> NOS) <sub>3</sub>             | Bi: 0.2387  |
|         |                                | 800                              | BiPO <sub>4</sub>  | Bi: 0.6875; Bi <sub>2</sub> O <sub>3</sub> : 0.7665 |
| Br      | 70–946                         | 130–150                          | AgBr   | Br: 0.4256  |
| Ca      | 478–635                        | 475–525                          | CaCO <sub>3</sub>  | Ca: 0.4004; CaO: 0.5601                             |
|         | 838–1025                       | 950–1000                         | CaO  | Ca: 0.7147  |
|         |                                | air-dried                        | Ca(picrolonate) <sub>2</sub> · 8H <sub>2</sub> O                 | Ca: 0.05642   |
| Cd      | 218–420                        | >320                             | CdSO <sub>4</sub>  | Cd: 0.5392; CdO: 0.6159                             |
|         |                                | 125                              | Cd(C <sub>10</sub> H <sub>6</sub> NO <sub>2</sub> ) <sub>2</sub> | Cd: 0.2462  |
|         |                                |                                  | CdS  | Cd: 0.7781; CdO: 0.8888                             |
| Ce      | >360                           | 500–600                          | CeO <sub>2</sub>   | Ce: 0.8141  |
| Cl      | 70–600                         | 130–150                          | AgCl   | Cl: 0.2474  |

**TABLE 11.26** Heating Temperatures, Composition of Weighing Forms, and Gravimetric Factors (*Continued*)

| Element                              | Thermal stability range, °C | Final heating temperature, °C | Composition of weighing form  | Gravimetric factors   |
|--------------------------------------|-----------------------------|-------------------------------|---|---|
| Co                                   | 285–946                     | 750–850                       | Co <sub>3</sub> O <sub>4</sub>  | Co: 0.7342  |
|                                      |                             | 130                           | Co(C <sub>10</sub> H <sub>6</sub> NO <sub>2</sub> ) <sub>3</sub> · 2H <sub>2</sub> O                                    | Co: 0.09639; CoO: 0.1226                                      |
|                                      |                             | 450–500                       | CoSO <sub>4</sub>   | Co: 0.3802  |
| Cr                                   |                             | 120                           | PbCrO <sub>4</sub>  | Cr: 0.1609  |
| Cu                                   |                             | 105–120                       | CuSCN   | Cu: 0.5225; CuO: 0.6540                                       |
|                                      | < 115                       | 100–105                       | Cu(C <sub>7</sub> H <sub>5</sub> NO <sub>2</sub> ) <sub>2</sub>   | Cu: 0.1891  |
|                                      |                             | 105–115                       | Cu(C <sub>13</sub> H <sub>11</sub> NO <sub>2</sub> )  | Cu: 0.2201  |
|                                      |                             | 110–115                       | Cu(C <sub>10</sub> H <sub>6</sub> NO <sub>2</sub> ) · H <sub>2</sub> O  | Cu: 0.1494  |
|                                      |                             | 105                           | Cu(C <sub>12</sub> H <sub>10</sub> NOS) <sub>2</sub> · H <sub>2</sub> O   | Cu: 0.1237  |
| F                                    | 66–538                      | 130–140                       | PbClF   | F: 0.07261  |
| Fe                                   | 470–946                     | 900                           | Fe <sub>2</sub> O <sub>3</sub>  | Fe: 0.6994  |
| Ga                                   | 408–946                     | 900                           | Ga <sub>2</sub> O <sub>3</sub>  | Ga: 0.7439  |
| Hg                                   |                             | 105                           | Hg(C <sub>12</sub> H <sub>10</sub> NOS) <sub>2</sub>  | Hg: 0.3169  |
| I                                    | 60–900                      | 130–150                       | AgI   | I: 0.5405   |
| In                                   | 345–1200                    | 1200                          | In <sub>2</sub> O <sub>3</sub>  | In: 0.8271  |
| Ir                                   |                             |                               | IrO <sub>2</sub>  | Ir: 0.8573  |
| K                                    | 73–653                      | < 653                         | KClO <sub>4</sub>   | K: 0.2822; K <sub>2</sub> O: 0.3399                           |
|                                      |                             | < 270                         | K <sub>2</sub> PtCl <sub>6</sub>  | K: 0.1609; K <sub>2</sub> O: 0.1938                           |
|                                      |                             |                               | KIO <sub>4</sub>  | K: 0.1700   |
|                                      |                             | 120                           | KB(C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub>   | K: 0.1091   |
| Li                                   |                             | 200                           | Li <sub>2</sub> SO <sub>4</sub>   | Li: 0.1263; Li <sub>2</sub> O: 0.2718                         |
| Mg                                   |                             | 1050–1100                     | Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>   | Mg: 0.2184; MgO: 0.3622                                       |
|                                      | 88–300                      | 155–160                       | Mg(C <sub>9</sub> H <sub>6</sub> NO) <sub>2</sub>   | Mg: 0.07775; MgO: 0.1289                                      |
| Mn                                   | > 946                       | 1000                          | Mn <sub>3</sub> O <sub>4</sub>  | Mn: 0.7203  |
|                                      |                             | 1000                          | Mn <sub>2</sub> P <sub>2</sub> O <sub>7</sub>   | Mn: 0.3871; MnO: 0.4998                                       |
| Mo                                   |                             | > 505                         | PbMoO <sub>4</sub>  | Mo: 0.2613; MoO <sub>3</sub> : 0.3291                         |
|                                      |                             | 500–525                       | MoO <sub>3</sub>  | Mo: 0.6666  |
| N (as NO <sub>3</sub> <sup>-</sup> ) | 20–242                      | 105                           | Nitron nitrate  | N: 0.3732; NO <sub>3</sub> : 0.1652                           |
| Na                                   | 360–674                     | 125                           | NaMg(UO <sub>2</sub> ) <sub>3</sub> (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>9</sub> · 6.5 H <sub>2</sub> O | Na: 0.01527; Na <sub>2</sub> O: 0.02058                       |
| Nb                                   | 650–950                     | 900                           | Nb <sub>2</sub> O <sub>3</sub>  | Nb: 0.6990  |
| Ni                                   | 79–172                      | 110–120                       | Ni(C <sub>4</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub>   | Ni: 0.2032; NiO: 0.2586                                       |
| Os                                   |                             | 800 (in H <sub>2</sub> )      | Os metal  |   |
| P                                    |                             | > 477                         | Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>   | P: 0.2783; PO <sub>4</sub> : 0.8536                           |
|                                      | 160–415                     | 110                           | (NH <sub>4</sub> ) <sub>3</sub> [P(Mo <sub>3</sub> O <sub>10</sub> ) <sub>4</sub> ]                                     | P: 0.0165; P <sub>2</sub> O <sub>5</sub> : 0.0378             |
| Pb                                   | 271–959                     | 500–600                       | PbSO <sub>4</sub>   | Pb: 0.6832; PbO: 0.7359                                       |
|                                      |                             | 600                           | PbMoO <sub>4</sub>  | Pb: 0.5643; PbO: 0.6078                                       |
|                                      |                             | 120                           | PbCrO <sub>4</sub>  | Pb: 0.6411  |
|                                      | 271–959                     | 600–800                       | PbSO <sub>4</sub>   | Pb: 0.6832; PbO: 0.7359                                       |
|                                      |                             | 105                           | Pb(C <sub>12</sub> H <sub>10</sub> NOS) <sub>2</sub>  | Pb: 0.3240  |
| Pd                                   | 45–171                      | 110                           | Pd(C <sub>4</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub>   | Pd: 0.3162  |
| Rb                                   | 70–674                      | < 674                         | Rb <sub>2</sub> PtCl <sub>6</sub>   | Rb: 0.2954; Rb <sub>2</sub> O: 0.3230                         |
| Re                                   |                             | 130                           | (C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub> AsReO <sub>4</sub>  | Re: 0.2939  |
|                                      |                             | 110                           | Nitron perrhenate   | Re: 0.3306  |
| S                                    |                             | > 780                         | BaSO <sub>4</sub>   | S: 0.1374; SO <sub>3</sub> : 0.3430; SO <sub>4</sub> : 0.4116 |
| Sb                                   |                             | 100                           | Sb(C <sub>12</sub> H <sub>10</sub> NOS) <sub>3</sub>  | Sb: 0.1581  |
| SCN <sup>-</sup>                     |                             | 130                           | AgSCN   | SCN: 0.3500   |
|                                      |                             | 110–120                       | CuSCN   | SCN: 0.4775   |
| Se                                   |                             | 120–130                       | Se metal  | SeO <sub>2</sub> : 1.4052                                     |
| Si                                   | 358–946                     | > 358                         | SiO <sub>2</sub>  | Si: 0.4675  |

**TABLE 11.26** Heating Temperatures, Composition of Weighing Forms, and Gravimetric Factors (*Continued*)

| Element | Thermal stability range, °C | Final heating temperature, °C | Composition of weighing form  | Gravimetric factors                   |
|---------|-----------------------------|-------------------------------|---|---------------------------------------|
| Sn      | > 834                       | 900                           | SnO <sub>2</sub>  | Sn: 0.7877                            |
| Sr      |                             | 130–140                       | Sr(NO <sub>3</sub> ) <sub>2</sub>   | Sr: 0.4140                            |
|         | 100–300                     | 100–300                       | SrSO <sub>4</sub>   | Sr: 0.4770; SrO: 0.5641               |
| Te      |                             | 105                           | Te metal  |                                       |
| Th      | 610–946                     | 700–800                       | ThO <sub>2</sub>  | Th: 0.8788                            |
|         |                             | 900                           | ThP <sub>2</sub> O <sub>7</sub>   | Th: 0.5863                            |
| Ti      | 350–946                     | 900                           | TiO <sub>2</sub>  | Ti: 0.5992                            |
| Tl(III) |                             | 100                           | Tl(C <sub>12</sub> H <sub>10</sub> NOS)   | Tl: 0.4860                            |
| U       |                             | 1000                          | U <sub>3</sub> O <sub>8</sub>   | U: 0.8480; UO <sub>2</sub> : 0.9620   |
| V       | 581–946                     | 700–800                       | V <sub>2</sub> O <sub>5</sub>   | V: 0.5602                             |
| W       | > 674                       | 800–900                       | WO <sub>3</sub>   | W: 0.7930                             |
| Zn      | > 1000                      | 950–1000                      | ZnO   | Zn: 0.8034                            |
|         |                             | 1000                          | Zn <sub>3</sub> P <sub>2</sub> O <sub>7</sub>                                       | Zn: 0.4292; ZnO: 0.5342               |
|         |                             | 125                           | Zn(C <sub>10</sub> H <sub>6</sub> NO <sub>2</sub> ) <sub>2</sub> · H <sub>2</sub> O | Zn: 0.1529                            |
| Zr      |                             | > 850                         | ZrP <sub>2</sub> O <sub>7</sub>   | Zr: 0.3440; ZrO <sub>2</sub> : 0.4647 |
|         |                             | 1200                          | ZrO <sub>2</sub>  | Zr: 0.7403                            |

*Source:* J. A. Dean, ed., *Analytical Chemistry Handbook*, McGraw-Hill, New York, 1995.

## 11.6 VOLUMETRIC ANALYSIS

### 11.6.1 Acid-Base Titrations in Aqueous Media

**TABLE 11.27** Primary Standards for Aqueous Acid-Base Titrations

| Standard   | Formula weight | Preparation  |
|--|----------------|--|
| Basic substances for standardizing acidic solutions                |                |  |
| (HOCH <sub>2</sub> ) <sub>3</sub> CNHH <sub>2</sub>                | 121.137        | Tris(hydroxymethyl)aminomethane is available commercially as a primary standard. Dry at 100–103°C (<110°C). In titrations with a strong acid the equivalence point is at about pH 4.5–5. Equivalent weight is the formula weight. [J. H. Fossum, P. C. Markunas, and J. A. Riddick, <i>Anal. Chem.</i> , <b>23</b> :491 (1951).]   |
| HgO  | 216.59         | Dissolve 100 g pure HgCl <sub>2</sub> in 1 L H <sub>2</sub> O, and add with stirring to 650 mL 1.5 M NaOH. Filter and wash with H <sub>2</sub> O until washings are neutral to phenolphthalein. Dry to constant weight at or below 40°C, and store in a dark bottle. To 0.4 g HgO (≡ 40 mL 0.1N acid) add 10–15 g KBr plus 20–25 mL H <sub>2</sub> O. Stir, excluding CO <sub>2</sub> , until solution is complete. Titrate with acid to pH 5–8. Equivalent weight is one-half formula weight. |
| Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> · 10H <sub>2</sub> O | 381.372        | Recrystallize reagent-grade salt twice from water at temperatures below 55°C. Wash the crystals with H <sub>2</sub> O, twice with ethanol, and twice with diethyl ether. Let stand in a hygostat oversaturated NaBr · 2H <sub>2</sub> O or saturated NaCl-sucrose solution. Use methyl red indicator. Equivalent weight is one-half the formula weight.  |

**TABLE 11.27** Primary Standards for Aqueous Acid-Base Titrations (*Continued*)

| Standard   | Formula weight | Preparation  |
|--|----------------|--|
| Basic substances for standardizing acidic solutions ( <i>continued</i> ) |                |  |
| Na <sub>2</sub> CO <sub>3</sub>  | 105.989        | Heat reagent-grade material for 1 hr at 255–265°C. Cool in an efficient desiccator. Titrate sample with acid to pH 4–5 (first green tint of bromocresol green), boil the solution to eliminate the carbon dioxide, cool, and again titrate to pH 4–5. Equivalent weight is one-half the formula weight.  |
| NaCl   | 58.45          | Accurately weigh about 6 g NaCl and dissolve in distilled water. Pass the solution through a well-rinsed cation exchange column (Dowex 50W) in the hydrogen form. The equivalent amount of HCl is washed from the column (in 10 column volumes) into a volumetric flask and made up to volume. Equivalent weight is the formula weight.  |
| Acidic substances for standardizing basic solutions                      |                |  |
| C <sub>6</sub> H <sub>5</sub> COOH                                       | 122.125        | Pure benzoic acid is available from NIST (National Institute for Science and Technology). Dissolve 0.5 g in 20 mL of neutral ethanol (run a blank), excluding CO <sub>2</sub> , add 20–50 mL, and titrate using phenolphthalein as indicator.  |
| <i>o</i> -C <sub>6</sub> H <sub>4</sub> (COOK)(COOH)                     | 204.22         | Potassium hydrogen <i>o</i> -phthalate is available commercially as primary standard, also from NIST. Dry at <135°C. Dissolve in water, excluding CO <sub>2</sub> , and titrate with phenolphthalein as indicator. For Ba(OH) <sub>2</sub> solution, perform the titration at an elevated temperature to prevent precipitation of Ba phthalate.  |
| KH(IO <sub>3</sub> ) <sub>2</sub>  | 389.915        | Potassium hydrogen bis(iodate) is available commercially in a primary standard grade. Dry at 110°C. Dissolve a weighed amount of the salt in water, excluding CO <sub>2</sub> , and titrate to pH 5–8. [I. M. Kolthoff and L. H. van Berk, <i>J. Am. Chem. Soc.</i> , <b>48</b> :2800 (1926)].   |
| NH <sub>2</sub> SO <sub>3</sub> H  | 97.09          | Hydrogen amidosulfate (sulfamic acid) acts as a strong acid. Primary standard grade is available commercially. Since it does undergo slow hydrolysis, an acid end point (pH 4 to 6.5) should be chosen unless fresh reagent is available, then the end point can be in the range pH 4 to 9. [W. F. Wagner, J. A. Wuellner, and C. E. Feiler, <i>Anal. Chem.</i> , <b>24</b> :1491 (1952). M. J. Butler, G. F. Smith, and L. F. Audrieth, <i>Ind. Eng. Chem., Anal. Ed.</i> , <b>10</b> :690 (1938)]. |

**TABLE 11.28** Titrimetric (Volumetric) Factors

| Acids                          |  |          |
|--------------------------------|--|----------|
| Substance                      | Formula  | Grams    |
| Ammonia                        | NH <sub>3</sub>  | 0.017031 |
| Ammonium                       | NH <sub>4</sub>  | 0.018039 |
| Ammonium chloride              | NH <sub>4</sub> Cl   | 0.053492 |
| Ammonium hydroxide             | NH <sub>4</sub> OH   | 0.035046 |
| Ammonium oleate                | C <sub>17</sub> H <sub>33</sub> CO <sub>2</sub> NH <sub>4</sub>    | 0.29950  |
| Ammonium oxide                 | (NH <sub>4</sub> ) <sub>2</sub> O                                  | 0.026038 |
| Amyl acetate                   | CH <sub>3</sub> CO <sub>2</sub> C <sub>5</sub> H <sub>11</sub>     | 0.13019  |
| Barium carbonate (MO)          | BaCO <sub>3</sub>  | 0.09867  |
| Barium hydroxide               | Ba(OH) <sub>2</sub>  | 0.085677 |
| Barium oxide                   | BaO  | 0.07667  |
| Bornyl acetate                 | CH <sub>3</sub> CO <sub>2</sub> C <sub>10</sub> H <sub>17</sub>    | 0.19629  |
| Calcium carbonate (MO)         | CaCO <sub>3</sub>  | 0.05004  |
| Calcium hydroxide              | Ca(OH) <sub>2</sub>  | 0.037047 |
| Calcium oleate                 | (C <sub>17</sub> H <sub>33</sub> CO <sub>2</sub> ) <sub>2</sub> Ca | 0.30150  |
| Calcium oxide                  | CaO  | 0.02804  |
| Calcium stearate               | (C <sub>17</sub> H <sub>35</sub> CO <sub>2</sub> ) <sub>2</sub> Ca | 0.30352  |
| Casein (N 6.38)                | .....  | 0.089371 |
| Ethyl acetate                  | CH <sub>3</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>      | 0.088107 |
| Glue (N 5.60)                  | .....  | 0.078445 |
| Hydrochloric acid              | HCl  | 0.036461 |
| Magnesium carbonate (MO)       | MgCO <sub>3</sub>  | 0.04216  |
| Magnesium oxide                | MgO  | 0.02016  |
| Menthyl acetate                | CH <sub>3</sub> CO <sub>2</sub> C <sub>10</sub> H <sub>19</sub>    | 0.19831  |
| Methyl acetate                 | CH <sub>3</sub> CO <sub>2</sub> CH <sub>3</sub>                    | 0.074080 |
| Nicotine                       | C <sub>10</sub> H <sub>14</sub> N <sub>2</sub>                     | 0.16224  |
| Nitrogen                       | N  | 0.014007 |
| Potassium carbonate (MO)       | K <sub>2</sub> CO <sub>3</sub>                                     | 0.06911  |
| Potassium carbonate, acid (MO) | KHCO <sub>3</sub>  | 0.10012  |
| Potassium nitrate              | KNO <sub>3</sub>   | 0.10111  |
| Potassium oleate               | C <sub>17</sub> H <sub>33</sub> CO <sub>2</sub> K                  | 0.32057  |
| Potassium oxide                | K <sub>2</sub> O   | 0.04710  |
| Potassium stearate             | C <sub>17</sub> H <sub>35</sub> CO <sub>2</sub> K                  | 0.32258  |
| Protein (N 5.70)               | .....  | 0.079846 |
| Protein (N 6.25)               | .....  | 0.087550 |
| Sodium acetate                 | CH <sub>3</sub> CO <sub>2</sub> Na                                 | 0.082035 |
| Sodium acetate                 | CH <sub>3</sub> CO <sub>2</sub> Na · 3H <sub>2</sub> O             | 0.13608  |
| Sodium borate, tetra- (MO)     | Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>                      | 0.10061  |
| Sodium borate, tetra- (MO)     | Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> · 10H <sub>2</sub> O | 0.19069  |
| Sodium carbonate (MO)          | Na <sub>2</sub> CO <sub>3</sub>                                    | 0.052994 |
| Sodium carbonate (MO)          | Na <sub>2</sub> CO <sub>3</sub> · H <sub>2</sub> O                 | 0.062002 |
| Sodium carbonate (MO)          | Na <sub>2</sub> CO <sub>3</sub> · 10H <sub>2</sub> O               | 0.14307  |
| Sodium carbonate, acid (MO)    | NaHCO <sub>3</sub>   | 0.084007 |
| Sodium hydroxide               | NaOH   | 0.39997  |
| Sodium oleate                  | C <sub>17</sub> H <sub>33</sub> CO <sub>2</sub> Na                 | 0.30445  |

The following factors are the equivalent of 1 mL of *normal acid*. Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed.

The equivalents of the esters are based on the results of saponification.

The indicators methyl orange and phenolphthalein are indicated by the abbreviations MO and pH, respectively.

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Acids ( <i>continued</i> ) |  |          |
|----------------------------|--|----------|
| Substance                  | Formula  | Grams    |
| Sodium oxalate             | $\text{Na}_2\text{C}_2\text{O}_4$                    | 0.067000 |
| Sodium oxide               | $\text{Na}_2\text{O}$                                | 0.030990 |
| Sodium phosphate (MO)      | $\text{Na}_2\text{HPO}_4$                            | 0.14196  |
| Sodium phosphate (MO)      | $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ | 0.35814  |
| Sodium phosphate (MO)      | $\text{Na}_3\text{PO}_4$                             | 0.081970 |
| Sodium phosphate (PH)      | $\text{Na}_3\text{PO}_4$                             | 0.16394  |
| Sodium silicate            | $\text{Na}_2\text{Si}_4\text{O}_9$                   | 0.15111  |
| Sodium stearate            | $\text{C}_{17}\text{H}_{35}\text{CO}_2\text{Na}$     | 0.30647  |
| Sodium sulfide (MO)        | $\text{Na}_2\text{S}$                                | 0.039022 |
| Alkali                     |  |          |

The following factors are the equivalent of the milliliter of *normal alkali*. Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed.

The equivalents of the esters are based on the results of saponification.

The indicators methyl orange and phenolphthalein are indicated by the abbreviations MO and PH, respectively.

| Substance                 | Formula   | Grams    |
|---------------------------|---|----------|
| Abietic acid (PH)         | $\text{HC}_{20}\text{H}_{29}\text{O}_2$                             | 0.30246  |
| Acetic acid (PH)          | $\text{CH}_3\text{CO}_2\text{H}$                                    | 0.06005  |
| Acetic anhydride (PH)     | $(\text{CH}_3\text{CO})_2\text{O}$                                  | 0.051045 |
| Aluminum sulfate          | $\text{Al}_2(\text{SO}_4)_3$  | 0.05702  |
| Amyl acetate              | $\text{CH}_3\text{CO}_2\text{C}_5\text{H}_{11}$                     | 0.13019  |
| Benzoic acid (PH)         | $\text{C}_6\text{H}_5\text{CO}_2\text{H}$                           | 0.12212  |
| Borate tetra- (PH)        | $\text{B}_4\text{O}_7$  | 0.03881  |
| Boric acid (PH)           | $\text{H}_3\text{BO}_3$   | 0.061833 |
| Boric anhydride (PH)      | $\text{B}_2\text{O}_3$  | 0.03486  |
| Bornyl acetate            | $\text{CH}_3\text{CO}_2\text{C}_{10}\text{H}_{17}$                  | 0.19629  |
| Butyric acid (PH)         | $\text{C}_3\text{H}_7\text{CO}_2\text{H}$                           | 0.088107 |
| Calcium acetate           | $(\text{CH}_3\text{CO}_2)_2\text{Ca}$                               | 0.079085 |
| Calcium oleate            | $(\text{C}_{17}\text{H}_{33}\text{CO}_2)_2\text{Ca}$                | 0.30150  |
| Calcium stearate          | $(\text{C}_{17}\text{H}_{35}\text{CO}_2)_2\text{Ca}$                | 0.30352  |
| Carbon dioxide (PH)       | $\text{CO}_2$   | 0.022005 |
| Chlorine                  | $\text{Cl}$   | 0.035453 |
| Citric acid (PH)          | $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 \cdot \text{H}_2\text{O}$ | 0.070047 |
| Ethyl acetate             | $\text{CH}_3\text{CO}_2\text{C}_2\text{H}_5$                        | 0.088107 |
| Formaldehyde              | $\text{HCHO}$   | 0.030026 |
| Formic acid (PH)          | $\text{HCO}_2\text{H}$  | 0.046026 |
| Glycerol (sap. of acetyl) | $\text{C}_3\text{H}_5(\text{OH})_3$                                 | 0.030698 |
| Hydriodic acid            | $\text{HI}$   | 0.12791  |
| Hydrobromic acid          | $\text{HBr}$  | 0.080917 |
| Hydrochloric acid         | $\text{HCl}$  | 0.036461 |
| Lactic acid (PH)          | $\text{HC}_3\text{H}_5\text{O}_3$                                   | 0.090079 |
| Lead acetate              | $(\text{CH}_3\text{CO}_2)_2\text{Pb} \cdot 3\text{H}_2\text{O}$     | 0.18966  |
| Maleic acid (PH)          | $(\text{CHCO}_2\text{H})_2$   | 0.058037 |
| Malic acid (PH)           | $\text{H}_2\text{C}_4\text{H}_4\text{O}_5$                          | 0.067045 |
| Menthol (sap. of acetyl)  | $\text{C}_{10}\text{H}_{19}\text{OH}$                               | 0.15627  |

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Alkali ( <i>continued</i> )    |   |          |
|--------------------------------|---|----------|
| Substance                      | Formula   | Grams    |
| Menthyl acetate                | $\text{CH}_3\text{CO}_2\text{C}_{10}\text{H}_{19}$                  | 0.19831  |
| Methyl acetate                 | $\text{CH}_3\text{CO}_2\text{CH}_3$                                 | 0.074080 |
| Nitrate                        | $\text{NO}_3$   | 0.062005 |
| Nitric acid                    | $\text{HNO}_3$  | 0.063013 |
| Nitrogen                       | N   | 0.014007 |
| Nitrogen pentoxide             | $\text{N}_2\text{O}_5$  | 0.054005 |
| Oleic acid (PH)                | $\text{C}_{17}\text{H}_{33}\text{CO}_2\text{H}$                     | 0.28247  |
| Oxalic acid (PH)               | $(\text{CO}_2\text{H})_2$   | 0.045018 |
| Oxalic acid (PH)               | $(\text{CO}_2\text{H})_2 \cdot 2\text{H}_2\text{O}$                 | 0.063033 |
| Phosphoric acid (MO)           | $\text{H}_3\text{PO}_4$   | 0.097995 |
| Phosphoric acid (PH)           | $\text{H}_3\text{PO}_4$   | 0.048998 |
| Potassium carbonate, acid (MO) | $\text{KHCO}_3$   | 0.10012  |
| Potassium oleate               | $\text{C}_{17}\text{K}_{33}\text{CO}_2\text{K}$                     | 0.32056  |
| Potassium oxalate, acid (PH)   | $\text{KHC}_2\text{O}_4$  | 0.12813  |
| Potassium phthalate, acid (PH) | $\text{HC}_8\text{H}_4\text{O}_4\text{K}$                           | 0.20423  |
| Potassium stearate             | $\text{C}_{17}\text{H}_{33}\text{CO}_2\text{K}$                     | 0.32258  |
| Sodium benzoate                | $\text{C}_6\text{H}_5\text{CO}_2\text{Na}$                          | 0.14411  |
| Sodium borate, tetra- (PH)     | $\text{Na}_2\text{B}_4\text{O}_7$                                   | 0.050305 |
| Sodium borate, tetra- (PH)     | $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$        | 0.095343 |
| Sodium carbonate, acid (MO)    | $\text{NaHCO}_3$  | 0.084007 |
| Sodium oleate                  | $\text{C}_{17}\text{H}_{33}\text{CO}_2\text{Na}$                    | 0.30445  |
| Sodium salicylate              | $\text{C}_6\text{H}_5\text{OCO}_2\text{Na}$                         | 0.16011  |
| Stearic acid (PH)              | $\text{C}_{17}\text{H}_{33}\text{CO}_2\text{H}$                     | 0.28449  |
| Succinic acid (PH)             | $(\text{CH}_2\text{CO}_2\text{H})_2$                                | 0.059045 |
| Sulfate                        | $\text{SO}_4$   | 0.048031 |
| Sulfur dioxide (PH)            | $\text{SO}_2$   | 0.032031 |
| Sulfur trioxide                | $\text{SO}_3$   | 0.040031 |
| Sulfuric acid                  | $\text{H}_2\text{SO}_4$   | 0.049039 |
| Sulfurous acid (PH)            | $\text{H}_2\text{SO}_3$   | 0.041039 |
| Tartaric acid (PH)             | $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$                          | 0.075044 |
| Tartaric acid (PH)             | $\text{H}_2\text{C}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O}$ | 0.084052 |
| Iodine                         |   |          |

The following factors are the equivalent of 1 mL of *normal iodine*. Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed.

| Substance         | Formula                       | Grams     |
|-------------------|-------------------------------|-----------|
| Acetone           | $(\text{CH}_3)_2\text{CO}$    | 0.0096801 |
| Ammonium chromate | $(\text{NH}_4)_2\text{CrO}_4$ | 0.050690  |
| Antimony          | Sb                            | 0.06088   |
| Antimony trioxide | $\text{Sb}_2\text{O}_3$       | 0.07287   |
| Arsenic           | As                            | 0.037461  |
| Arsenic pentoxide | $\text{As}_2\text{O}_5$       | 0.057460  |
| Arsenic trioxide  | $\text{As}_2\text{O}_3$       | 0.049460  |
| Arsenite          | $\text{AsO}_3$                | 0.061460  |
| Bleaching powder  | $\text{CaOCl}_2$              | 0.063493  |
| Bromine           | Br                            | 0.079909  |
| Chlorine          | Cl                            | 0.035453  |
| Chromic oxide     | $\text{Cr}_2\text{O}_3$       | 0.02533   |

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Iodine ( <i>continued</i> ) |  |           |
|-----------------------------|--|-----------|
| Substance                   | Formula  | Grams     |
| Chromium trioxide           | $\text{CrO}_3$   | 0.033331  |
| Copper                      | $\text{Cu}$  | 0.06354   |
| Copper oxide                | $\text{CuO}$   | 0.07954   |
| Copper sulfate              | $\text{CuSO}_4$  | 0.15960   |
| Copper sulfate              | $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$                    | 0.24968   |
| Ferric iron                 | $\text{Fe}^{3+}$   | 0.05585   |
| Ferric oxide                | $\text{Fe}_2\text{O}_3$                                      | 0.07985   |
| Hydrogen sulfide            | $\text{H}_2\text{S}$   | 0.017040  |
| Iodine                      | $\text{I}$   | 0.126904  |
| Lead chromate               | $\text{PbCrO}_4$   | 0.10773   |
| Lead dioxide                | $\text{PbO}_2$   | 0.11959   |
| Nitrous acid                | $\text{HNO}_2$   | 0.023507  |
| Oxygen                      | $\text{O}$   | 0.0079997 |
| Potassium chlorate          | $\text{KClO}_3$  | 0.020426  |
| Potassium chromate          | $\text{K}_2\text{CrO}_4$                                     | 0.064733  |
| Potassium dichromate        | $\text{K}_2\text{Cr}_2\text{O}_7$                            | 0.049032  |
| Potassium nitrite           | $\text{KNO}_2$   | 0.042554  |
| Potassium permanganate      | $\text{KMnO}_4$  | 0.031608  |
| Red lead                    | $\text{Pb}_3\text{O}_4$                                      | 0.34278   |
| Sodium chromate             | $\text{Na}_2\text{CrO}_4$                                    | 0.053991  |
| Sodium dichromate           | $\text{Na}_2\text{Cr}_2\text{O}_7$                           | 0.043661  |
| Sodium dichromate           | $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ | 0.049666  |
| Sodium nitrite              | $\text{NaNO}_2$  | 0.034498  |
| Sodium sulfide              | $\text{Na}_2\text{S}$  | 0.039022  |
| Sodium sulfide              | $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$              | 0.12009   |
| Sodium sulfite              | $\text{Na}_2\text{SO}_3$                                     | 0.063021  |
| Sodium sulfite              | $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$           | 0.12607   |
| Sodium thiosulfate          | $\text{Na}_2\text{S}_2\text{O}_3$                            | 0.15811   |
| Sulfur                      | $\text{S}$   | 0.016032  |
| Sulfur dioxide              | $\text{SO}_2$  | 0.032031  |
| Sulfurous acid              | $\text{H}_2\text{SO}_3$                                      | 0.041039  |
| Tin                         | $\text{Sn}$  | 0.059345  |

## Potassium dichromate

The following factors are the equivalent of 1 mL of *normal potassium dichromate*. Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed.

| Substance            | Formula                                   | Grams     |
|----------------------|---|-----------|
| Chromic oxide        | $\text{Cr}_2\text{O}_3$                   | 0.025332  |
| Chromium trioxide    | $\text{CrO}_3$                            | 0.033331  |
| Ferrous iron         | $\text{Fe}^{2+}$                          | 0.055847  |
| Ferrous oxide        | $\text{FeO}$                              | 0.071846  |
| Ferroso-ferric oxide | $\text{Fe}_3\text{O}_4$                   | 0.077180  |
| Ferrous sulfate      | $\text{FeSO}_4$                           | 0.15191   |
| Ferrous sulfate      | $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ | 0.27802   |
| Glycerol             | $\text{C}_3\text{H}_5(\text{OH})_3$       | 0.0065782 |
| Lead chromate        | $\text{PbCrO}_4$                          | 0.10773   |
| Zinc                 | $\text{Zn}$                               | 0.032685  |

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Potassium permanganate  |   |           |
|---|---|-----------|
| Substance   | Formula   | Grams     |
| Ammonium oxalate  | $(\text{NH}_4)_2\text{C}_2\text{O}_4$                               | 0.062049  |
| Ammonium oxalate  | $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$      | 0.071056  |
| Ammonium peroxydisulfate  | $(\text{NH}_4)_2\text{S}_2\text{O}_8$                               | 0.11410   |
| Antimony  | Sb  | 0.060875  |
| Barium peroxide   | $\text{BaO}_2$  | 0.084669  |
| Barium peroxide   | $\text{BaO}_2 \cdot 8\text{H}_2\text{O}$                            | 0.15673   |
| Calcium carbonate   | $\text{CaCO}_3$   | 0.050045  |
| Calcium oxide   | CaO   | 0.02804   |
| Calcium peroxide  | $\text{CaO}_2$  | 0.036039  |
| Calcium sulfate   | $\text{CaSO}_4$   | 0.068071  |
| Calcium sulfate   | $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$                           | 0.086086  |
| Ferric oxide  | $\text{Fe}_2\text{O}_3$   | 0.079846  |
| Ferroso-ferric oxide  | $\text{Fe}_3\text{O}_4$   | 0.077180  |
| Ferrous ammonium sulfate  | $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ | 0.39214   |
| Ferrous oxide   | FeO   | 0.071846  |
| Ferrous sulfate   | $\text{FeSO}_4$   | 0.15191   |
| Ferrous sulfate   | $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$                           | 0.27802   |
| Formic acid   | $\text{HCO}_2\text{H}$  | 0.023013  |
| Hydrogen peroxide   | $\text{H}_2\text{O}_2$  | 0.017007  |
| Iodine  | I   | 0.126904  |
| Iron  | Fe  | 0.055847  |
| Manganese   | Mn  | 0.010988  |
| Manganese dioxide   | $\text{MnO}_2$  | 0.043468  |
| Manganous oxide (Volhard)   | MnO   | 0.035469  |
| Molybdenum trioxide titration from yellow ppt.<br>after reduction     | $\text{MoO}_3$  | 0.047979  |
| Oxalic acid   | $(\text{CO}_2\text{H})_2$   | 0.045018  |
| Oxalic acid   | $(\text{CO}_2\text{H})_2 \cdot 2\text{H}_2\text{O}$                 | 0.063033  |
| Phosphorus titration from yellow ppt. after reduction                 | P   | 0.0008604 |
| Phosphorus pentoxide to titration from yellow ppt.<br>after reduction | $\text{P}_2\text{O}_5$  | 0.0019715 |
| Potassium dichromate  | $\text{K}_2\text{Cr}_2\text{O}_7$                                   | 0.049032  |
| Potassium nitrite   | $\text{KNO}_2$  | 0.042552  |
| Potassium persulfate  | $\text{K}_2\text{S}_2\text{O}_8$                                    | 0.13516   |
| Sodium nitrite  | $\text{NaNO}_2$   | 0.034498  |
| Sodium oxalate  | $\text{Na}_2\text{C}_2\text{O}_4$                                   | 0.067000  |
| Sodium persulfate   | $\text{Na}_2\text{S}_2\text{O}_8$                                   | 0.11905   |
| Tin   | Sn  | 0.059345  |

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Silver nitrate        |                                       |          |
|-----------------------|---------------------------------------|----------|
| Substance             | Formula                               | Grams    |
| Ammonium bromide      | NH <sub>4</sub> Br                    | 0.097948 |
| Ammonium chloride     | NH <sub>4</sub> Cl                    | 0.053492 |
| Ammonium iodide       | NH <sub>4</sub> I                     | 0.14494  |
| Ammonium thiocyanate  | NH <sub>4</sub> SCN                   | 0.076120 |
| Barium chloride       | BaCl <sub>2</sub>                     | 0.10412  |
| Barium chloride       | BaCl <sub>2</sub> · 2H <sub>2</sub> O | 0.12214  |
| Bromine               | Br                                    | 0.079909 |
| Cadmium chloride      | CdCl <sub>2</sub>                     | 0.091653 |
| Cadmium iodide        | CdI <sub>2</sub>                      | 0.18310  |
| Calcium chloride      | CaCl <sub>2</sub>                     | 0.055493 |
| Chlorine              | Cl                                    | 0.035453 |
| Ferric chloride       | FeCl <sub>3</sub>                     | 0.054069 |
| Ferrous chloride      | FeCl <sub>2</sub>                     | 0.063377 |
| Hydriodic acid        | HI                                    | 0.12791  |
| Hydrobromic acid      | HBr                                   | 0.080917 |
| Hydrochloric acid     | HCl                                   | 0.036461 |
| Iodine                | I                                     | 0.126904 |
| Lithium chloride      | LiCl                                  | 0.042392 |
| Lead chloride         | PbCl <sub>2</sub>                     | 0.13905  |
| Magnesium chloride    | MgCl <sub>2</sub>                     | 0.047609 |
| Magnesium chloride    | MgCl <sub>2</sub> · 6H <sub>2</sub> O | 0.10166  |
| Potassium bromide     | KBr                                   | 0.11901  |
| Potassium chloride    | KCl                                   | 0.074555 |
| Potassium iodide      | KI                                    | 0.16601  |
| Potassium oxide       | K <sub>2</sub> O                      | 0.047102 |
| Potassium thiocyanate | KSCN                                  | 0.097184 |
| Silver                | Ag                                    | 0.10787  |
| Silver iodide         | AgI                                   | 0.23477  |
| Silver nitrate        | AgNO <sub>3</sub>                     | 0.16987  |
| Sodium bromide        | NaBr                                  | 0.10290  |
| Sodium bromide        | NaBr · 2H <sub>2</sub> O              | 0.13893  |
| Sodium chloride       | NaCl                                  | 0.058443 |
| Sodium iodide         | NaI                                   | 0.14989  |
| Sodium iodide         | NaI · 2H <sub>2</sub> O               | 0.18592  |
| Sodium oxide          | Na <sub>2</sub> O                     | 0.030990 |
| Strontium chloride    | SrCl <sub>2</sub>                     | 0.079263 |
| Strontium chloride    | SrCl <sub>2</sub> · 6H <sub>2</sub> O | 0.13331  |
| Zinc chloride         | ZnCl <sub>2</sub>                     | 0.068138 |

The following factors are the equivalent of 1 mL of *normal silver nitrate*. Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed.

**TABLE 11.28** Titrimetric (Volumetric) Factors (*Continued*)

| Sodium thiosulfate   |  |           |
|--|--|-----------|
| The following factors are the equivalent of 1 mL of <i>normal sodium thiosulfate</i> . Where the normality of the solution being used is other than normal, multiply the factors given in the table below by the normality of the solution employed. |  |           |
| Substance  | Formula  | Grams     |
| Acetone  | $(\text{CH}_3)_2\text{CO}$                                   | 0.0096801 |
| Ammonium chromate  | $(\text{NH}_4)_2\text{CrO}_4$                                | 0.050690  |
| Antimony   | Sb   | 0.06088   |
| Antimony trioxide  | $\text{Sb}_2\text{O}_3$                                      | 0.07287   |
| Bleaching powder   | $\text{CaOCl}_2$   | 0.063493  |
| Bromine  | Br   | 0.079909  |
| Chlorine   | Cl   | 0.035453  |
| Chromic oxide  | $\text{Cr}_2\text{O}_3$                                      | 0.02533   |
| Chromium trioxide  | $\text{CrO}_3$   | 0.033331  |
| Copper   | Cu   | 0.06354   |
| Copper oxide   | $\text{CuO}$   | 0.07954   |
| Copper sulfate   | $\text{CuSO}_4$  | 0.15960   |
| Copper sulfate   | $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$                    | 0.24968   |
| Iodine   | I  | 0.126904  |
| Lead chromate  | $\text{PbCrO}_4$   | 0.10773   |
| Lead dioxide   | $\text{PbO}_2$   | 0.11959   |
| Nitrous acid   | $\text{HNO}_2$   | 0.023507  |
| Potassium chromate   | $\text{K}_2\text{CrO}_4$                                     | 0.064733  |
| Potassium dichromate   | $\text{K}_2\text{Cr}_2\text{O}_7$                            | 0.049032  |
| Red lead   | $\text{Pb}_3\text{O}_4$                                      | 0.34278   |
| Sodium chromate  | $\text{Na}_2\text{CrO}_4$                                    | 0.053991  |
| Sodium dichromate  | $\text{Na}_2\text{Cr}_2\text{O}_7$                           | 0.043661  |
| Sodium dichromate  | $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ | 0.049666  |
| Sodium nitrite   | $\text{NaNO}_2$  | 0.034498  |
| Sodium thiosulfate   | $\text{Na}_2\text{S}_2\text{O}_3$                            | 0.15811   |
| Sodium thiosulfate   | $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  | 0.24818   |
| Sulfur   | S  | 0.016032  |
| Sulfur dioxide   | $\text{SO}_2$  | 0.032031  |
| Tin  | Sn   | 0.059345  |

### 11.6.2 Titrimetric (Volumetric) Factors for Acid-Base Titrations

Titrimetric (volumetric) factors for acids and bases are given in Table 11.28. Suitable indicators for acid-base titrations may be found in Tables 8.23 and 8.24.

### 11.6.3 Standard Volumetric (Titrimetric) Redox Solutions

**Alkaline arsenite**, 0.1N As(III) to As(V). Dissolve 4.9460 g of primary standard grade  $\text{As}_2\text{O}_3$  in 40 mL of 30% NaOH solution. Dilute with 200 mL of water. Acidify the solution with 6N HCl to the acid color of methyl red indicator. Add to this solution 40 g of  $\text{NaHCO}_3$  and dilute to 1 L.

**Ceric sulfate**, 0.1N Ce(IV) to Ce(III). Dissolve 63.26 g of cerium(IV) ammonium sulfate dihydrate in 500 mL of 2N sulfuric acid. Dilute the solution to 1 L and standardize against the

alkaline arsenite solution as follows: measure, accurately, 30 to 40 mL of arsenite solution into an Erlenmeyer flask and dilute to 150 mL. Add slowly, to prevent excessive frothing, 20 mL of 4*N* sulfuric acid, 2 drops of 0.01*M* osmium tetroxide solution, and 4 drops of 1,10-phenanthroline iron(II) complex indicator. Titrate with the ceric sulfate solution to a faint blue endpoint. Compute the normality of the ceric solution from the normality of the arsenite solution.

**Iron(II) ammonium sulfate hexahydrate**, 0.1*N* Fe(II) to Fe(III). Dissolve 39.2139 g of  $\text{FeSO}_4 \cdot 2(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  in 500 mL of 1*N* sulfuric acid and dilute to 1 L. If desired, check against standard dichromate or permanganate solution.

**Iodine**, 0.1*N* (0 to 1–). Dissolve 12.690 g of resublimed iodine in 25 mL of a solution containing 15 g of KI which is free from iodate. After all the solid has dissolved, dilute to 1 L. If desired, check against a standard arsenite or standard thiosulfate solution.

**Potassium bromate**, 0.1*N* (5+ to 1–). Weigh out 2.7833 g of  $\text{KBrO}_3$ , dissolve in water, and dilute to 1 L.

**Potassium dichromate**, 0.1*N* Cr(VI) to Cr(III). Weigh out 4.9030 g of  $\text{K}_2\text{Cr}_2\text{O}_7$  that has been dried at 120°C, dissolve in water, and dilute to 1 L.

**Potassium iodate**, 0.1*N* (5+ to 1–). Weigh out exactly 3.5667 g of  $\text{KIO}_3$  (free from iodide), dried at 120°C, and dissolve in water containing about 15 g of KI, and dilute to 1 L.

**Potassium permanganate**, 0.1*N* (7+ to 2+). Dissolve about 3.3 g in a liter of distilled water. Allow this to stand for 2 or 3 days, then siphon it carefully through clean glass tubes or filter it through a Gooch crucible into the glass container in which it is to be kept, discarding the first 25 mL and allowing the last inch of liquid to remain in the bottle. In this way any dust or reducing substance in the water is oxidized, and the  $\text{MnO}_2$  formed is removed. Permanganate solutions should never be allowed to come into contact with rubber, filter paper, or any other organic matter, and should be stored away from light. To standardize the  $\text{KMnO}_4$ , weigh accurately samples of about 0.3 g of primary standard grade  $\text{Na}_2\text{C}_2\text{O}_4$  into Erlenmeyer flasks, add 150 mL of distilled water and 4 mL of concentrated  $\text{H}_2\text{SO}_4$ , and heat to 70°C and maintain at this temperature throughout the titration with the permanganate solution. The end point is a faint, permanent pink color throughout the solution. Equivalent weight of  $\text{Na}_2\text{C}_2\text{O}_4/2$  is 67.000 g.

**Sodium thiosulfate**, 0.1*N*. Weigh 24.818 g of fresh crystals of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ , dissolve in distilled water. Add 0.5 g of  $\text{Na}_2\text{CO}_3$  and 0.5 mL of chloroform as preservative. Dilute to 1 L.

Equations for the principal methods for the redox determinations of the elements are given in Table 11.29. Volumetric factors in redox titrations for the common titrants are given in Table 11.28.

#### 11.6.4 Indicators for Redox Titrations

A selected list of redox indicators will be found in Table 8.26. A redox indicator should be selected so that its  $E^0$  is approximately equal to the electrode potential at the equivalent point, or so that the color change will occur at an appropriate part of the titration curve. If  $n$  is the number of electrons involved in the transition from the reduced to the oxidized form of the indicator, the range in which the color change occurs is approximately given by  $E^0 \pm 0.06/n$  volt (V) for a two-color indicator whose forms are equally intensely colored. Since hydrogen ions are involved in the redox equilibria of many indicators, it must be recognized that the color change interval of such an indicator will vary with pH.

In Table 8.26,  $E^0$  represents the redox potential at which the color change of the indicator would normally be perceived in a solution containing approximately 1*M*  $\text{H}^+$ . For a one-color indicator this is the potential at which the concentration of the colored form is just large enough to impart a visible color to the solution and depends on the total concentration of indicator added to the solution. If it is the reduced form of the indicator that is colorless, the potential at which the first visible color

appears becomes less positive as the total concentration of indicator increases. For a two-color indicator, the potential at which the middle tint appears is independent of the total indicator concentration, but may differ from the potentiometrically determined formal potential of the indicator in either direction, depending on which of the two forms is more intensely colored. If the reduced form is the more intense color, the middle tint will appear at a potential more positive than the potentiometrically measured formal potential, which is the potential at which the two forms are present at equal concentrations.

In addition to those indicators listed in Table 8.26, there are indicators for bromometric and iodometric titrations:

*Specific reagents for titrations with bromine or bromate*

|                             |   |
|-----------------------------|---|
| Methyl orange or methyl red | Use acid-base indicator solutions. Oxidation causes bleaching of indicator to colorless           |
| Bordeaux acid red 17        | Dissolve 2 g dye in 1 L water. The red solution is oxidized to pale yellowish green or colorless. |
| Naphthol blue black         | Dissolve 2 g dye in 1 L water. The blue solution is oxidized to pale red.                         |

*Specific reagents for iodometric titrations*

|   |  |
|---|--|
| Organic solvents such as CCl <sub>4</sub> , CHCl <sub>3</sub> | Up to 5 mL solvent is usually added per titration. Near the end point the mixture is shaken vigorously after each addition of titrant, and the appearance or disappearance of the I <sub>2</sub> color in the organic layer is observed. |
| Starch  | Suspend 5 g of soluble starch in 50 mL of saturated NaCl solution, and stir slowly into 500 mL of boiling saturated NaCl solution. Cool and bottle. Free iodine produces a blue-black color.   |

**TABLE 11.29** Equations for the Redox Determinations of the Elements with Equivalent Weights

|                 |  |
|-----------------|--|
| Al              | $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3 + 3 \text{HCl} = \text{AlCl}_3 + 3 \text{C}_9\text{H}_7\text{NO} \text{ (8-hydroxyquinoline)}$ $3 \text{C}_9\text{H}_7\text{NO} + 6 \text{Br}_2 = 3 \text{C}_9\text{H}_5\text{Br}_2\text{NO} + 6 \text{HBr}$ $\text{Al}/12 = 2.2485; \text{Al}_2\text{O}_3/24 = 4.2483$  |
| As <sup>0</sup> | $\text{As} + 5 \text{Ce(IV)} + 4 \text{H}_2\text{O} = \text{H}_3\text{AsO}_4 + 5 \text{Ce(III)} + 5 \text{H}^+$ $\text{As}/5 = 14.9843$  |
| As(III)         | $5 \text{H}_3\text{AsO}_3 + 2 \text{KMnO}_4 + 6 \text{HCl} = 5 \text{H}_3\text{AsO}_4 + 2 \text{MnCl}_2 + 3 \text{H}_2\text{O}$ $\text{H}_3\text{AsO}_3 + 2 \text{Ce}(\text{SO}_4)_2 + \text{H}_2\text{O} = \text{H}_3\text{AsO}_4 + \text{Ce}_2(\text{SO}_4)_3 + \text{H}_2\text{SO}_4$ $\text{As}/2 = 37.4608; \text{As}_2\text{O}_3/4 = 49.460$ $3 \text{H}_3\text{AsO}_3 + \text{KBrO}_3 (+ \text{HCl}) = 3 \text{H}_3\text{AsO}_4 + \text{KBr}$ $\text{H}_3\text{AsO}_3 + \text{I}_2 + 2 \text{H}_2\text{O} = \text{H}_3\text{AsO}_4 + 2 \text{I}^- + 2 \text{H}^+$ $\text{As}/2 = 37.4608; \text{As}_2\text{O}_3/4 = 49.460$ |
| As(V)           | $\text{H}_3\text{AsO}_4 + 2 \text{KI} (\text{excess}) + 2 \text{HCl} = \text{H}_3\text{AsO}_3 + \text{I}_2 + 2 \text{KCl} + \text{H}_2\text{O}$ $\text{I}_2 + 2 \text{Na}_2\text{S}_2\text{O}_3 = 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6$ $\text{As}/2 = 37.4608; \text{As}_2\text{O}_3/4 = 49.460$  |

**TABLE 11.29** Equations for the Redox Determinations of the Elements with Equivalent Weights (*Continued*)

|                             |   |
|-----------------------------|---|
| Ba                          | $\text{BaCrO}_4 + 6 \text{KI (excess)} + 16 \text{HCl} = 2 \text{BaCl}_2 + 3 \text{I}_2 + 6 \text{KCl} + 2 \text{CrCl}_3 + 8 \text{H}_2\text{O}$ $\text{I}_2 + 2 \text{Na}_2\text{S}_2\text{O}_3 = 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6 \quad \text{Ba/3} = 45.78$ $\text{BaCrO}_4 + 3 \text{Fe}^{2+} \text{ (excess)} + 8 \text{H}^+ = \text{Ba}^{2+} + \text{Cr}^{3+} + 3 \text{Fe}^{3+} + 4 \text{H}_2\text{O}$ Titrate excess $\text{Fe}^{2+}$ with permanganate or dichromate; $\text{Ba/3} = 45.78$   |
| $\text{Br}_2$               | $\text{Br}_2 + 2 \text{KI (excess)} = 2 \text{KBr} + \text{I}_2$ $\text{I}_2 + 2 \text{Na}_2\text{S}_2\text{O}_3 \rightarrow 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6 \quad \text{Br}_2/2 = 79.904$   |
| $\text{Br}^-$               | $\text{Br}^- + 3 \text{HClO} = \text{BrO}_3^- + 3 \text{Cl}^- + 3 \text{H}^+$ $\text{Br/6} = 13.317$  |
| $\text{BrO}_3^-$            | $\text{BrO}_3^- + 6 \text{I}^- \text{ (excess)} + 6 \text{H}^+ = \text{Br}^- + 3 \text{I}_2 + 3 \text{H}_2\text{O}$ $\text{I}_2 + 2 \text{Na}_2\text{S}_2\text{O}_3 = 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6$ $\text{KBrO}_3/6 = 27.835$  |
| CO                          | $5 \text{CO} + \text{I}_2\text{O}_5 = 5 \text{CO}_2 + \text{I}_2 \text{ (at } 125^\circ\text{C; adsorbed and measured colorimetrically)}$ $5/2 \text{CO} = 70.02$   |
| $\text{C}_2\text{O}_4^{2-}$ | Titrate as for $\text{CaC}_2\text{O}_4$   |
| $\text{C}_2\text{O}_6^{2-}$ | Acidify and titrate as for $\text{H}_2\text{O}_2$ ; $\text{C}_2\text{O}_6^{2-} + 2 \text{H}^+ = \text{H}_2\text{O}_2 + \text{CO}_2$<br>$\text{K}_2\text{C}_2\text{O}_6/2 = 99.11$   |
| Ca                          | $5 \text{CaC}_2\text{O}_4 + 2 \text{KMnO}_4 + 8 \text{H}_2\text{SO}_4 = 5 \text{CaSO}_4 + 10 \text{CO}_2 + \text{K}_2\text{SO}_4 + 2 \text{MnSO}_4 + 8 \text{H}_2\text{O}$ $\text{Ca/2} = 20.039; \text{CaO/2} = 28.04$   |
| Cd                          | $\text{Cd(anthranilate)}_2 + 4 \text{Br}_2 = 2 \text{NH}_2\text{C}_6\text{H}_2\text{Br}_2\text{COOH} + 4 \text{Br}^-$ Titrate with $\text{KBrO}_3$ — $\text{KBr}$ until color of indigo changes to yellow.<br>Add KI and back-titrate iodine liberated with thiosulfate. $\text{Cd/8} = 14.05$  |
| Ce                          | Oxidize $\text{Ce(III)}$ to $\text{Ce(IV)}$ with $(\text{NH}_4)_2\text{S}_2\text{O}_8$ plus $\text{Ag}^+$ ; destroy excess by boiling.<br>$2 \text{Ce(SO}_4)_2 + 2 \text{FeSO}_4 = \text{Ce}_2(\text{SO}_4)_3 + \text{Fe}_2(\text{SO}_4)_3$ $\text{Ce/1} = 140.12; \text{Ce}_2\text{O}_3/2 = 164.12$  |
| $\text{Cl}_2$               | Same as for $\text{Br}_2$ ; $\text{Cl}_2/2 = 35.453$  |
| $\text{ClO}^-$              | $\text{ClO}^- + 2 \text{I}^- + 2 \text{H} = \text{Cl}^- + \text{I}_2 + \text{H}_2\text{O}$ Titrate liberated $\text{I}_2$ with thiosulfate; $\text{HClO/2} = 26.230$  |
| $\text{ClO}_2^-$            | $\text{ClO}_2^- + 4 \text{I}^- + 4 \text{H}^+ = \text{Cl}^- + 2 \text{I}_2 + 2 \text{H}_2\text{O}$ Titrate liberated $\text{I}_2$ with thiosulfate; $\text{HClO}_2/2 = 26.230$  |
| $\text{ClO}_3^-$            | $\text{ClO}_3^- + 6 \text{I}^- + 6 \text{H}_2\text{O} = \text{Cl}^- + 3 \text{I}_2 + 3 \text{H}_2\text{O}$ Titrated liberated $\text{I}_2$ with thiosulfate; $\text{HClO}_2/4 = 17.115$<br>$\text{ClO}_3^- + 3 \text{H}_3\text{AsO}_3 \text{ (excess; boil with strong HCl)} = \text{Cl}^- + 3 \text{H}_3\text{AsO}_4$ Titrate excess $\text{H}_3\text{AsO}_3$ with bromate; $\text{HClO}_3/6 = 14.077$   |
| Co                          | $\text{Co(NH}_3)_6^{2+} + \text{Fe(CN)}_6^{3-} \text{ [Citrate-NH}_3 \text{ buffer]} = \text{Co(NH}_3)_6^{3+} + \text{Fe(CN)}_6^{4-}$ $\text{Co/1} = 58.9332$ Precipitate Co anthranilate and treat as for cadmium; $\text{Co/8} = 7.3667$  |
| Cr                          | $\text{Cr}_2\text{O}_7^{2-} + 6 \text{Fe}^{2+} + 14 \text{H}^+ = 2 \text{Cr}^{3+} + 6 \text{Fe}^{3+} + 7 \text{H}_2\text{O}$ $\text{Cr/3} = 17.332; \text{Cr}_2\text{O}_3/6 = 25.337$   |
| Cu                          | $2 \text{Cu}^{2+} + 2 \text{I}^- + 2 \text{SCN}^- = 2 \text{CuSCN} + \text{I}_2$ Titrate the liberated iodine with thiosulfate; $\text{Cu/1} = 63.546$<br>$4 \text{CuSCN} + 7 \text{IO}_3^- + 14 \text{H}^+ + 7 \text{Cl}^- = 4 \text{Cu}^{2+} + 4 \text{SO}_4^{2-} + 7 \text{ICl} + 4 \text{HCN} + 5 \text{H}_2\text{O}$ Precipitate and wash $\text{CuSCN}$ . Titrate with standard $\text{KIO}_3$ solution with 5 mL $\text{CHCl}_3$ until a definite $\text{I}_2$ color appears in the organic layer. Back-titrate the excess $\text{I}_2$ with standard thiosulfate solution. $\text{Cu/7} = 9.078; \text{KIO}_3/4 = 53.505$ |
| Fe(II)                      | $5 \text{Fe}^{2+} + \text{MnO}_4^- + 8 \text{H}^+ = 5 \text{Fe}^{3+} + \text{Mn}^{2+} + 4 \text{H}_2\text{O}$ $\text{Fe}^{2+} + \text{Ce(IV)} = \text{Fe}^{3+} + \text{Ce(III)}; \text{ use 1,10-phenanthroline iron(II) indicator.}$ $6 \text{Fe}^{2+} + \text{Cr}_2\text{O}_7^{2-} + 14 \text{H}^+ = 6 \text{Fe}^{3+} + 2 \text{Cr}^{3+} + 7 \text{H}_2\text{O}; \text{ use diphenylamine sulfonate indicator.}$ $\text{Fe/1} = 55.847; \text{Fe}_2\text{O}_3/2 = 79.845$   |

**TABLE 11.29** Equations for the Redox Determinations of the Elements with Equivalent Weights (*Continued*)

|                        |  |
|------------------------|--|
| Fe(III)                | $\text{Fe}^{3+} + 4 \text{SCN}^- = \text{Fe}(\text{SCN})_4^-; \text{Fe}(\text{SCN})_4^- + \text{Ti(III)} = \text{Fe}^{2+} + \text{Ti(IV)} + 4 \text{SCN}^-$ $\text{Fe/1} = 55.847; \text{Fe}_2\text{O}_3/2 = 79.845$ $2 \text{Fe}^{3+} + \text{Zn} = 2 \text{Fe}^{2+} + \text{Zn}^{2+}; \text{then proceed by a method under Fe(II).}$ $\text{Fe}^{3+} + \text{Ag} + \text{Cl}^- = \text{Fe}^{2+} + \text{AgCl}; \text{then proceed by a method under Fe(II).}$ $2 \text{Fe}^{3+} + \text{SnCl}_2(\text{slight excess}) + 4 \text{Cl}^- = 2 \text{Fe}^{2+} + \text{SnCl}_6^{2-}$ $2 \text{HgCl}_2 + \text{SnCl}_2 + 2 \text{Cl}^- = \text{Hg}_2\text{Cl}_2 + \text{SnCl}_6^{2-}$ <p>Pour above mixture into an <math>\text{H}_3\text{PO}_4</math> plus <math>\text{MnSO}_4</math> solution and titrate with <math>\text{KMnO}_4</math> as under Fe(II).</p> $\text{Fe/1} = 55.847; \text{Fe}_2\text{O}_3/2 = 79.845$ $2 \text{Fe}^{3+} + 2 \text{I}^- = \text{Fe}^{2+} + \text{I}_2$ <p>Titrate liberated iodine with thiosulfate; <math>\text{Fe/1} = 55.847; \text{Fe}_2\text{O}_3/2 = 79.845</math></p>   |
| $\text{I}_2$           | $\text{I}_2 + 2 \text{S}_2\text{O}_3^{2-} = 2 \text{I}^- + \text{S}_4\text{O}_6^{2-} \text{ [titrate solution (pH } \circ 7.0) \text{ with thiosulfate until color is pale yellow. Add KI and starch and continue titration to disappearance of blue color. } \text{I}_2/2 = 126.9045$ $\text{I}_2 + \text{H}_3\text{AsO}_3 + \text{H}_2\text{O} = 2 \text{I}^- + \text{H}_3\text{AsO}_4 + 2 \text{H}^+; \text{use starch and KI as indicator. } \text{I}_2/2 = 126.9045$  |
| $\text{I}^-$           | $2 \text{I}^- + \text{Br}_2(\text{excess}) = \text{I}_2 + 2\text{Br}^-$ <p>Remove excess <math>\text{Br}_2</math> formic acid and titrate <math>\text{I}_2</math> with thiosulfate. <math>\text{I}_2/2 = 126.9045</math></p>   |
| $\text{IO}_3^-$        | $\text{IO}_3^- + 5 \text{I}^- (\text{excess}) + 6 \text{H}^+ = 3 \text{I}_2 + 3 \text{H}_2\text{O}; \text{titrate } \text{I}_2 \text{ with thiosulfate. } \text{KIO}_3/6 = 35.67$  |
| $\text{IO}_4^-$        | $\text{IO}_4^- + 7 \text{I}^- (\text{excess}) + 8 \text{H}^+ = 4 \text{I}_2 + 4 \text{H}_2\text{O}; \text{use a neutral buffered solution. Titrate } \text{I}_2 \text{ with thiosulfate. } \text{KIO}_4/2 = 115.00$  |
| K                      | $\text{K}_2\text{Na}[\text{Co}(\text{NO}_2)_6]; \text{dissolve in } \text{H}_2\text{SO}_4 \text{ and titrate with either } \text{KMnO}_4 \text{ or } \text{Ce(IV)}. \text{ ca. } \text{K}/5.5 \text{ but use an empirical factor.}$  |
| Mg                     | $\text{Mg}(\text{oxine})_2; \text{dissolve precipitate and use procedure for } \text{Al}(8\text{-hydroxyquinoline})_3. \text{ Mg}/8 = 3.0381$  |
| Mn(II)                 | $2 \text{Mn}^{2+} + 5 \text{BiO}_3^- + 14 \text{H}^+ = 2 \text{MnO}_4^- + 5 \text{Bi}^{3+} + 7 \text{H}_2\text{O}$ $2 \text{MnO}_4^- + 5 \text{AsO}_3^{3-} + 6 \text{H}^+ = 2 \text{Mn}^{2+} + 5 \text{AsO}_4^{3-} + 3 \text{H}_2\text{O}; \text{Mn}/5 = 10.9876$ $2 \text{Mn}^{2+} + 5 \text{S}_2\text{O}_8^{2-} + 8 \text{H}_2\text{O} (\text{Ag}^+ \text{ catalyst}) = 2 \text{MnO}_4^- + 10 \text{SO}_4^{2-} + 16 \text{H}^+$ <p>Titrate the permanganate formed with iron(II) as under iron(II); <math>\text{Mn}/5 = 10.9876</math></p> $2 \text{Mn}^{2+} + 5 \text{IO}_4^- + 3 \text{H}_2\text{O} = 2 \text{MnO}_4^- + 5 \text{IO}_3^- + 6 \text{H}^+$ <p>Slowly precipitate excess <math>\text{KIO}_4</math> with <math>\text{Hg}(\text{NO}_3)_2</math>. Filter, add excess <math>\text{Fe}^{2+}</math> and titrate excess with standard <math>\text{KMnO}_4</math> solution; <math>\text{Mn}/5 = 10.9876</math></p> $\text{MnO}_4^- + 4 \text{Mn}^{2+} + 15 \text{H}_2\text{P}_2\text{O}_7^- [\text{pH range 4 to 7}] = 5 \text{Mn}(\text{H}_2\text{P}_2\text{O}_7)_3^- + 4 \text{H}_2\text{O}$ <p>Use Pt—SCE indicator system; <math>\text{Mn}/1 = 54.9380</math></p> |
| Mn(IV)                 | $\text{MnO}_2 + 2 \text{Fe}^{2+} (\text{excess standard}) + 4 \text{H}^+ = \text{Mn}^{2+} + 2 \text{Fe}^{3+} + 2 \text{H}_2\text{O} (\text{use } \text{CO}_2 \text{ atmosphere})$ $\text{MnO}_2 + \text{H}_2\text{C}_2\text{O}_4 (\text{excess standard}) + 2 \text{H}^+ = \text{Mn}^{2+} + 2 \text{CO}_2 + 2 \text{H}_2\text{O} (\text{use } \text{CO}_2 \text{ atmosphere})$ <p>In either of the above, titrate excess with <math>\text{KMnO}_4</math>. <math>\text{Mn}/2 = 27.469</math>; <math>\text{MnO}_2/2 = 43.47</math></p>   |
| Mn(VI)                 | $\text{MnO}_3^{2-} + 2 \text{H}_2\text{C}_2\text{O}_4 + 4 \text{H}^+ = \text{Mn}^{2+} + 4 \text{CO}_2 + 4 \text{H}_2\text{O}$ <p>Add excess oxalate and back-titrate with permanganate. <math>\text{Mn}/4 = 13.7345</math></p>   |
| Mn(VII)                | $2 \text{MnO}_4^- + 5 \text{H}_2\text{C}_2\text{O}_4 + 6 \text{H}^+ = 2 \text{Mn}^{2+} + 10 \text{CO}_2 + 3 \text{H}_2\text{O}; \text{Mn}/5 = 10.9876$   |
| Mo                     | $\text{Mo(VI)} + \text{Zn} = \text{Mo(III)} + \text{Zn}^{2+}; \text{catch eluate in excess } \text{Fe}_2(\text{SO}_4)_3 \text{ solution}$ $\text{Mo(III)} + 3 \text{Fe}^{3+} + 4 \text{H}_2\text{O} = \text{MoO}_4^{2-} + 3 \text{Fe}^{2+} + 8 \text{H}^+; \text{titrate Fe(II) with } \text{KMnO}_4$ $\text{Mo}/3 = 31.98$ $\text{Mo(VI)} + \text{Ag} + \text{Cl}^- = \text{Mo(V)} + \text{AgCl}; \text{pass through Ag reductor at } 60\text{--}80^\circ\text{C.}$ $\text{Mo(V)} + \text{Ce(IV)} = \text{Mo(VI)} + \text{Ce(III)}; \text{Mo}/1 = 95.94$  |
| $\text{N}_2\text{H}_4$ | $3 \text{N}_2\text{H}_4 + 2 \text{BrO}_3^- (\text{excess}) = 3 \text{N}_2 + 2 \text{Br}^- + 6 \text{H}_2\text{O}; \text{add excess KI and titrate } \text{I}_2 \text{ with thio-sulfate. } \text{N}_2\text{H}_4/4 = 8.01$  |
| $\text{NH}_2\text{OH}$ | $\text{NH}_2\text{OH} + \text{BrO}_3^- = \text{NO}_3^- + \text{Br}^- + \text{H}^+ + \text{H}_2\text{O}; \text{proceed as above for } \text{N}_2\text{H}_4. \text{NH}_2\text{OH}/6 = 5.505$   |

**TABLE 11.29** Equations for the Redox Determinations of the Elements with Equivalent Weights (*Continued*)

|                                 |   |
|---------------------------------|---|
| $\text{HN}_3$                   | $2 \text{HN}_3 + 2 \text{Ce(IV)(excess)} = 3 \text{N}_2 + 2 \text{Ce(III)} + 2 \text{H}^+$ ; done under inert atmosphere.<br>Add excess KI and titrate with thiosulfate. $\text{HN}_3/1 = 43.03$  |
| $\text{NO}_2^-$                 | $5 \text{NO}_2^- + 2 \text{MnO}_4^- (\text{excess}) + 6 \text{H}^+ = 5 \text{NO}_3^- + 2 \text{Mn}^{2+} + 3 \text{H}_2\text{O}$ ; determine excess $\text{KMnO}_4$ standard $\text{Na}_2\text{C}_2\text{O}_4$ solution. $\text{NaNO}_2/1 = 69.00$<br>$\text{NO}_2^- + 2 \text{Ce(IV)(excess)} + \text{H}_2\text{O} = \text{NO}_3^- + 2 \text{Ce(III)} + 2 \text{H}^+$ ; warmed to $50^\circ\text{C}$ . Add excess standard $\text{Fe(II)}$ solution and back-titrate with standard $\text{Ce(IV)}$ using erioglaucine indicator.<br>$\text{NaNO}_2/1 = 69.00$   |
| $\text{NO}_3^-$                 | $\text{NO}_3^- + \text{excess Fe}^{2+} (\text{Mo catalyst}) + 4 \text{H}^+ = \text{NO} + \text{Fe}^{3+}$ . Add $\text{H}_3\text{PO}_4$ and back-titrate excess $\text{Fe(II)}$ with $\text{K}_2\text{Cr}_2\text{O}_7$ . $\text{NaNO}_3/3 = 28.34$   |
| $\text{Nb(V)}$                  | $\text{Nb(V)} + \text{Zn} = \text{Nb(III)} + \text{Zn}^{2+}$ ; catch reduced solution under excess $\text{Fe(III)}$ .<br>$\text{Nb(III)} + 2 \text{Fe}^{3+} = \text{Nb(V)} + 2 \text{Fe}^{2+}$ ; titrate $\text{Fe(II)}$ with $\text{MnO}_4$ solution using 1,10-phenanthroline as indicator. $\text{Nb}/2 = 46.453$ ; $\text{Nb}_2\text{O}_5 = 66.455$   |
| $\text{Ni}$                     | Precipitate $\text{Ni(anthranilate)}_2$ and proceed as under $\text{Cd}$ . $\text{Ni}/8 = 7.336$  |
| $\text{O}_2$                    | $\text{O}_2 + 2 \text{Mn}^{2+} + 2 \text{OH}^- = 2 \text{MnO}_2 + 2 \text{H}^+$ ; stoppered flask plus KI<br>$\text{MnO}_2 + 2 \text{I}^- + 4 \text{H}^+ = \text{Mn}^{2+} + \text{I}_2 + 2 \text{H}_2\text{O}$ ; titrate $\text{I}_2$ released with thiosulfate. $\text{O}_2/4 = 7.007$   |
| $\text{O}_3$                    | $\text{O}_3 + 2 \text{I}^- + \text{H}_2\text{O} = \text{O}_2 + \text{I}_2 + 2 \text{OH}^-$ ; acidify and titrate with thiosulfate. $\text{O}_3/2 = 24.00$   |
| $\text{H}_2\text{O}_2$          | $5 \text{H}_2\text{O}_2 + 2 \text{MnO}_4^- + 6 \text{H}^+ = 5 \text{O}_2 + 2 \text{Mn}^{2+} + 8 \text{H}_2\text{O}$ ; $\text{H}_2\text{O}_2/2 = 17.01$<br>$\text{H}_2\text{O}_2 + 2 \text{Ce(IV)} + 2 \text{H}^+ = 2 \text{Ce(III)} + 2 \text{H}_2\text{O}$ ; use 1,10-phenanthroline indicator<br>$\text{H}_2\text{O}_2/1 = 34.02$<br>$\text{H}_2\text{O}_2 + 2 \text{I}^- + 2 \text{H}^+ = \text{I}_2 + 2 \text{H}_2\text{O}$ ; titrate $\text{I}_2$ with thiosulfate. $\text{H}_2\text{O}_2/2 = 17.01$<br>$\text{H}_2\text{O}_2 + 2 \text{Ti(III)} + 2 \text{H}^+ = 2 \text{Ti(IV)} + 2 \text{H}_2\text{O}$ ; end point is disappearance of the yellow color of peroxotitanic acid. $\text{H}_2\text{O}_2/2 = 17.01$ |
| $\text{P}$                      | The yellow precipitate of $(\text{NH}_4)_3[\text{P}(\text{Mo}_3\text{O}_{10})_4]$ is dissolved in $\text{NH}_4\text{OH}$ , then solution is strongly acidified with $\text{H}_2\text{SO}_4$ . See molybdenum; 12 moles $\text{Mo}$ per $\text{P}$ . $\text{P}/36 = 0.86038$   |
| $\text{HPH}_2\text{O}_2$        | $\text{HPH}_2\text{O}_2 + 2 \text{I}_2 (\text{excess}) + 2 \text{H}_2\text{O} = \text{H}_3\text{PO}_4 + 4 \text{I}^- + 4 \text{H}^+$ (let stand 10 h)<br>Make solution alkaline with $\text{NaHCO}_3$ and titrate excess $\text{I}_2$ with standard arsenite solution.<br>$\text{HPH}_2\text{O}_2/4 = 16.499$   |
| $\text{H}_3\text{PO}_3$         | $\text{H}_3\text{PO}_3 + \text{I}_2 (\text{excess}) + \text{H}_2\text{O} = \text{H}_3\text{PO}_4 + 2 \text{I}^- + 2 \text{H}^+$ (use $\text{CO}_2/\text{NaHCO}_3$ buffer; let stand 40–60 min in stoppered flask). Titrate excess $\text{I}_2$ with standard arsenite solution. $\text{H}_3\text{PO}_3/2 = 41.00$   |
| $\text{Pb}$                     | Isolate $\text{Pb}$ as $\text{PbSO}_4$ , dissolve it in $\text{NaOAc}$ and precipitate with $\text{K}_2\text{Cr}_2\text{O}_7$ . Dissolve $\text{K}_2\text{CrO}_4$ in $\text{NaCl-HCl}$ solution, add $\text{KI}$ , and titrate $\text{I}_2$ with thiosulfate solution.<br>$2 \text{PbCrO}_4 + 6 \text{I}^- + 16 \text{H}^+ = 2 \text{Pb}^{2+} + 2 \text{Cr}^{3+} + 3 \text{I}_2 + 8 \text{H}_2\text{O}$ $\text{Pb}/3 = 69.1$ ; $\text{PbO}/3 = 74.4$  |
| $\text{S}^{2-}$                 | $\text{H}_2\text{S} + \text{I}_2 (\text{excess}) = \text{S} + 2 \text{I}^- + 2 \text{H}^+$ Back-titrate excess $\text{I}_2$ with standard thiosulfate solution.<br>$\text{S}/2 = 16.03$ ; $\text{H}_2\text{S}/2 = 17.04$<br>$\text{H}_2\text{S} + 4 \text{Br}_2 + 4 \text{H}_2\text{O} = \text{SO}_4^{2-} + 8 \text{Br}^- + 10 \text{H}^+$ Use excess $\text{KBr}$ and standard $\text{KBrO}_3$ solution. Let stand until clear, add excess $\text{KI}$ , and titrate with standard thiosulfate solution.<br>$\text{H}_2\text{S}/8 = 4.260$ ; $\text{SO}_2/2 = 32.03$ ; $\text{SCN}/6 = 9.681$  |
| $\text{SO}_2, \text{SO}_3^{2-}$ | $\text{SO}_2 + \text{I}_2 + 2 \text{H}_2\text{O} = \text{SO}_4^{2-} + 2 \text{I}^- + 4 \text{H}^+$ (Titrate excess $\text{I}_2$ with standard thiosulfate)<br>$\text{SO}_2/2 = 32.03$<br>$\text{SO}_2 + 4 \text{Br}_2 + 2 \text{H}_2\text{O} = \text{SO}_4^{2-} + 2 \text{Br}^- + 4 \text{H}^+$ (Titrate with standard $\text{KBrO}_3$ — $\text{KBr}$ solution until methyl orange is bleached.) $\text{SO}_2/2 = 32.03$  |
| $\text{S}_2\text{O}_3^{2-}$     | $2 \text{S}_2\text{O}_3^{2-} + \text{I}_2 = \text{S}_4\text{O}_6^{2-} + 2 \text{I}^-$ (Use starch indicator) $\text{Na}_2\text{S}_2\text{O}_3/1 = 158.11$   |
| $\text{H}_2\text{SO}_5$         | $\text{SO}_3^{2-} + \text{H}_3\text{AsO}_3 = \text{SO}_4^{2-} + \text{H}_3\text{AsO}_4$ $\text{H}_2\text{SO}_5/2 = 57.04$   |
| $\text{S}_2\text{O}_8^{2-}$     | $\text{S}_2\text{O}_8^{2-} + \text{H}_3\text{AsO}_3 + \text{H}_2\text{O} = 2 \text{SO}_4^{2-} + \text{H}_3\text{AsO}_4 + 2 \text{H}^+$ $\text{H}_2\text{S}_2\text{O}_8/2 = 97.07$<br>$\text{S}_2\text{O}_8^{2-} + 2 \text{Fe}^{2+} = 2 \text{SO}_4^{2-} + 2 \text{Fe}^{3+}$ $\text{H}_2\text{S}_2\text{O}_8/2 = 97.07$  |

**TABLE 11.29** Equations for the Redox Determinations of the Elements with Equivalent Weights (*Continued*)

|                     |   |
|---------------------|---|
| Sb                  | $5 \text{ Sb(III)} + 2 \text{ MnO}_4^- + 16 \text{ H}^+ = 5 \text{ Sb(V)} + 2 \text{ Mn}^{2+} + 8 \text{ H}_2\text{O}$<br>$3 \text{ Sb(III)} + \text{BrO}_3^- + 6 \text{ H}^+ = 3 \text{ Sb(V)} + \text{Br}^- + 3 \text{ H}_2\text{O}$<br>$\text{Sb(III)} + \text{I}_2 [\text{tartrate buffer, pH} > 7] = \text{Sb(V)} + 2 \text{ I}^-$<br>$\text{Sb(III)} + 2 \text{ Ce(IV)} = \text{Sb(V)} + 2 \text{ Ce(III)}$<br>For all four methods: $\text{Sb}/2 = 60.88$ ; $\text{Sb}_2\text{O}_3/4 = 72.88$  |
| $\text{SeO}_3^{2-}$ | $5 \text{ H}_2\text{SeO}_3 + 2 \text{ MnO}_4^- + 6 \text{ H}^+ = 5 \text{ H}_2\text{SeO}_4 + 2 \text{ Mn}^{2+} + 3 \text{ H}_2\text{O} \quad \text{Na}_2\text{SeO}_3/2 = 86.47$<br>$\text{H}_2\text{SeO}_3 + 4 \text{ I}^- + 4 \text{ H}^+ = \text{Se} + 2 \text{ I}_2 + 3 \text{ H}_2\text{O}$ (titrate $\text{I}_2$ with standard thiosulfate solution)<br>$\text{Na}_2\text{SeO}_3/2 = 86.47$<br>$\text{H}_2\text{SeO}_3 + 4 \text{ S}_2\text{O}_3^{2-} + 4 \text{ H}^+ = \text{SeS}_4\text{O}_6^{2-} + \text{S}_4\text{O}_6^{2-} + 3 \text{ H}_2\text{O}$ (add small excess of thiosulfate and back-titrate with standard iodine solution) $\text{Na}_2\text{SeO}_3/4 = 47.23$  |
| $\text{SeO}_4^{2-}$ | $\text{SeO}_4^{2-} + 2 \text{ H}^+ + 2 \text{ Cl}^- = \text{SeO}_3^{2-} + \text{Cl}_2 + \text{H}_2\text{O}$ (absorb $\text{Cl}_2$ in KI solution)<br>$\text{Cl}_2 + 2 \text{ I}^- = 2 \text{ Cl}^- + \text{I}_2$ (titrate $\text{I}_2$ with standard thiosulfate) $\text{Na}_2\text{SeO}_4/2 = 94.47$   |
| Sn(IV)              | $\text{SnCl}_6^{2-} + \text{Pb} = \text{Sn}^{2+} + \text{Pb}^{2+} + 6 \text{ Cl}^-$ (in $\text{CO}_2$ atmosphere boil 40 min)<br>$\text{Sn}^{2+} + \text{I}_2 + 6 \text{ Cl}^- = \text{SnCl}_6^{2-} + 2 \text{ I}^-$ (at $0-3^\circ\text{C}$ ) $\text{Sn}/2 = 59.35$ ; $\text{SnO}_2/2 = 67.35$   |
| Sn(II)              | $\text{Sn(II)} + 2 \text{ Ce(IV)} = \text{Sn(IV)} + 2 \text{ Ce(III)} \quad \text{Sn}/2 = 59.35$  |
| Te(IV)              | $3 \text{ H}_2\text{TeO}_3 + \text{Cr}_2\text{O}_7^{2-} + 8 \text{ H}^+ = 3 \text{ H}_2\text{TeO}_4 + 2 \text{ Cr}^{3+} + 4 \text{ H}_2\text{O} \quad \text{Te}/2 = 63.80$  |
| Te(VI)              | $\text{H}_2\text{TeO}_4 + 2 \text{ Cl}^- + 2 \text{ H}^+ = \text{H}_2\text{TeO}_3 + \text{Cl}_2 + \text{H}_2\text{O}$ (see $\text{SeO}_4^{2-}$ ) $\text{Te}/2 = 63.80$  |
| Ti                  | $2 \text{ Ti(IV)} + \text{Zn(reductor)} = 2 \text{ Ti(III)} + \text{Zn(II)}$<br>$\text{Ti(III)} + \text{Fe}^{3+} = \text{Ti(IV)} + \text{Fe}^{2+}$ (in $\text{CO}_2$ atmosphere; use KSCN as indicator) $\text{Ti}/1 = 47.88$<br>or<br>$\text{Ti(III)} + \text{Methylene blue} = \text{Ti(IV)} + \text{colorless leuco base}$ (in $\text{CO}_2$ atmosphere) $\text{Ti}/1 = 47.88$   |
| Tl                  | $2 \text{ Tl}^+ + \text{MnO}_4^- + 8 \text{ H}^+ = 2 \text{ Tl}^{3+} + \text{Mn}^{2+} + 4 \text{ H}_2\text{O} \quad \text{Tl}/2 = 102.19$<br>$\text{Tl}^+ + 2 \text{ Ce}^{3+} = \text{Tl}^{3+} + 2 \text{ Ce}^{3+}$ (to a yellow color or use 1,10-phenanthroline) $\text{Tl}/2 = 102.19$   |
| U                   | $\text{U(VI)} + \text{Zn} = \text{U(III)} + \text{U(IV)} + \text{Zn(II)}$ [pass air through solution to oxidize U(III) to U(IV)]<br>$5 \text{ U}^{4+} + 2 \text{ MnO}_4^- + 2 \text{ H}_2\text{O} = 5 \text{ UO}_2^{2+} + 2 \text{ Mn}^{2+} + 4 \text{ H}^+ \quad \text{U}/2 = 119.01$ ; $\text{U}_3\text{O}_8/6 = 140.35$  |
| V                   | Oxidize V(IV) to V(V) with permanganate. Destroy excess with sodium azide and boiling.<br>$\text{VO}_2^+ + \text{Fe}^{2+} + 2 \text{ H}^+ = \text{VO}^{2+} + \text{Fe}^{3+} + \text{H}_2\text{O}$ (diphenyaminesulfonic acid indicator)<br>$\text{V}/1 = 50.94$<br>Reduce V(V) with $\text{SO}_2$ and bubble $\text{CO}_2$ through boiling solution to remove excess $\text{SO}_2$ .<br>$5 \text{ VO}^{2+} + \text{MnO}_4^- + \text{H}_2\text{O} = 5 \text{ VO}_2^+ + \text{Mn}^{2+} + 2 \text{ H}^+ \quad \text{V}/1 = 50.94$<br>Reduce V(V) to V(II) with Zn; catch eluate in excess $\text{Fe}^{3+}$ .<br>$\text{V}^{2+} + 2 \text{ Fe}^{3+} + \text{H}_2\text{O} = \text{VO}^{2+} + 2 \text{ Fe}^{2+} + 2 \text{ H}^+$<br>Titrate $\text{VO}^{2+}-\text{Fe}^{2+}$ mixture with permanganate to $\text{VO}_2^+-\text{Fe}^{3+} \quad \text{V}/3 = 16.98$ ; $\text{V}_2\text{O}_5/6 = 30.32$ |
| Zn                  | Dissolve precipitate of $\text{Zn}[\text{Hg}(\text{SCN})_4]$ in 4M HCl in stoppered flask, add $\text{CHCl}_3$ .<br>$2 \text{ SCN}^- + 3 \text{ IO}_3^- + 2 \text{ H}^+ + \text{CN}^- = 2 \text{ SO}_4^{2-} + 3 \text{ ICN} + \text{H}_2\text{O} \quad \text{Zn}/24 = 2.725$<br>$2 \text{ Fe(CN)}_6^{3-} + 2 \text{ I}^- + 3 \text{ Zn}^{2+} + 2 \text{ K}^+ = \text{K}_2\text{Zn}_3[\text{Fe(CN)}_6]_2 + \text{I}_2$<br>Remove $\text{I}_2$ as formed by standard thiosulfate solution.<br>$3 \text{ Zn}/2 = 98.07$ but empirical value of 99.07 is recommended.<br>Precipitate $\text{Zn}(\text{anthranilate})_2$ ; proceed as with Cd. $\text{Zn}/8 = 8.174$   |

**Note:** Additional procedural information plus interferences and general remarks will be found in J. A. Dean, ed., *Analytical Chemistry Handbook*, McGraw-Hill, New York, 1995.

### 11.6.5 Precipitation Titrations

Many precipitation reactions that are useful as separation techniques for gravimetric analysis fail to meet one or both of two requirements for titrimetry:

1. The reaction rate must be sufficiently rapid, particularly in the titration of dilute solutions and in the immediate vicinity of the end point. To increase the precipitation rate, it is sometimes beneficial to change solvents or to raise the temperature. By adding an excess of reagent and back-titrating, it may be possible to take advantage of a more rapid precipitation in the reverse direction. By choosing an end-point detection method that does not require equilibrium to be reached in the immediate vicinity of the end point, advantage may be taken of a faster reaction rate at points removed from the end point. Examples are: amperometric titrations, conductometric titrations, and photometric titrations.
2. The stoichiometry must be exact. Coprecipitation by solid-solution formation, foreign ion entrapment, and adsorption are possible sources of error.

Table 11.30 lists standard solutions for precipitation titrations and Table 11.31 lists specific reagents as indicators, adsorption indicators, and protective colloids for precipitation titrations.

### 11.6.6 Complexometric Titrations

A complexometric titration is based on the essentially stoichiometric reaction of a complexing agent (*chelon*) with another species to form a complex species (*chelonate*) that is only slightly dissociated and is soluble in the titration medium. In such a titration, either the chelon or the chelonate may serve as the limiting reagent (that is, as the titrant). The end point is detected by measuring or observing some property that reflects the change, in the vicinity of the equivalence point, in the concentration of the chelon or the chelonate. Examples of the application of metal-ion indicators are listed in Table 11.32. For a metal indicator to be useful, a proper sequence of effective stabilities must be met. On the one hand, the metal-indicator complex must be sufficiently stable to maintain itself in extremely dilute solution; otherwise the end-point color change will be spread over a broad interval of the titration, owing to the extended dissociation. On the other hand, the metal-indicator complex must be less stable than the metal chelonate; otherwise a sluggish end point, a late end point, or no end point at all will be obtained. Furthermore, the metal-indicator complex must react rapidly with the chelon. Only a limited number of the numerous chromogenic agents for metals allow this sequence and have useful indicator properties in chelometric titrations.

Among the complexing agents that find use as titrating agents, ethylenediamine-*N,N,N',N'*-tetraacetic acid (acronym EDTA, and equation abbreviation,  $H_4Y$ ) is by far the more important, and it is used in the vast majority of complexometric titrations. The successive acid  $pK_a$  values of  $H_4Y$  are  $pK_1 = 2.0$ ,  $pK_2 = 2.67$ ,  $pK_3 = 6.16$ ,  $pK_4 = 10.26$  at 20°C and an ionic strength of 0.1. The fraction  $\alpha_4$  present as the tetravalent anion is of particular importance in equilibrium calculations. Its magnitude at various pH values is given in Table 11.33.

The formation constants of EDTA complexes are gathered in Table 11.34. Based on their stability, the EDTA complexes of the most common metal ions may be roughly divided into three groups:

|                     |   |
|---------------------|---|
| $\log K > 20$       | Tri- and tetravalent cations including Bi, Fe(III), Ga, Hg(II), In, Sc, Th, U(IV), V(III), and Zr |
| $\log K = 15$ to 18 | Divalent transition metals, rare earths, and Al   |
| $\log K = 8$ to 11  | Alkaline earths and Mg  |

The more stable the metal complex, the lower the pH at which it can be quantitatively formed. Elements in the first group may be titrated with EDTA at pH 1 to 3 without interference from cations of the last two groups, while cations of the second group may be titrated at pH 4 to 5 without interference from the alkaline earths.

In practice, an auxiliary complexing (masking) agent is usually added during EDTA titrations to prevent the precipitation of heavy metals as hydroxides or basic salts. The concentration of auxiliary complexing agents is generally high compared with the metal-ion concentration, and the solution is sufficiently well buffered so that the hydrogen ions produced during complexing of a metal ion by  $H_4Y$  do not cause an appreciable change in pH. Many EDTA titrations are carried out in ammonia–ammonium chloride buffers, which serve also to provide ammonia as an auxiliary complexing agent. The cumulative formation constants of ammine complexes are listed in Table 11.35.

**11.6.6.1 Types of Chelometric Titrations.** Chelometric titrations may be classified according to their manner of performance: direct titrations, back titrations, substitution titrations, redox titrations, or indirect methods.

**11.6.6.1.1 Direct Titrations.** The most convenient and simplest manner is the measured addition of a standard chelon solution to the sample solution (brought to the proper conditions of pH, buffer, etc.) until the metal ion is stoichiometrically chelated. Auxiliary complexing agents such as citrate, tartrate, or triethanolamine are added, if necessary, to prevent the precipitation of metal hydroxides or basic salts at the optimum pH for titration. For example, tartrate is added in the direct titration of lead. If a pH range of 9 to 10 is suitable, a buffer of ammonia and ammonium chloride is often added in relatively concentrated form, both to adjust the pH and to supply ammonia as an auxiliary complexing agent for those metal ions which form ammine complexes. A few metals, notably iron(III), bismuth, and thorium, are titrated in acid solution.

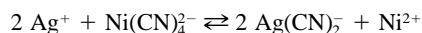
Direct titrations are commonly carried out using disodium dihydrogen ethylenediaminetetraacetate,  $Na_2H_2Y$ , which is available in pure form. The reaction of the chelon with the indicator must be rapid for a practical, direct titration. Where it is slow, heating of the titration medium is often expedient, or another indicator is employed.

**11.6.6.1.2 Back Titrations.** In the performance of a back titration, a known, but excess quantity of EDTA or other chelon is added, the pH is now properly adjusted, and the excess of the chelon is titrated with a suitable standard metal salt solution. Back titration procedures are especially useful when the metal ion to be determined cannot be kept in solution under the titration conditions or where the reaction of the metal ion with the chelon occurs too slowly to permit a direct titration, as in the titration of chromium(III) with EDTA. Back titration procedures sometimes permit a metal ion to be determined by the use of a metal indicator that is blocked by that ion in a direct titration. For example, nickel, cobalt, or aluminum form such stable complexes with Eriochrome Black T that the direct titration would fail. However, if an excess of EDTA is added before the indicator, no blocking occurs in the back titration with a magnesium or zinc salt solution. These metal ion titrants are chosen because they form EDTA complexes of relatively low stability, thereby avoiding the possible titration of EDTA bound by the sample metal ion.

In a back titration, a slight excess of the metal salt solution must sometimes be added to yield the color of the metal-indicator complex. Where metal ions are easily hydrolyzed, the complexing agent is best added at a suitable, low pH and only when the metal is fully complexed is the pH adjusted upward to the value required for the back titration. In back titrations, solutions of the following metal ions are commonly employed: Cu(II), Mg, Mn(II), Pb(II), Th(IV), and Zn. These solutions are usually prepared in the approximate strength desired from their nitrate salts (or the solution of the metal or its oxide or carbonate in nitric acid), and a minimum amount of acid is added to repress hydrolysis of the metal ion. The solutions are then standardized against an EDTA solution (or other chelon solution) of known strength.

**11.6.6.1.3 Substitution Titrations.** Upon the introduction of a substantial or equivalent amount of the chelonate of a metal that is less stable than that of the metal being determined, a substitution occurs, and the metal ion displaced can be titrated by the chelon in the same solution. This is a direct titration with regard to its performance, but in terms of the mechanism it can be considered as a substitution titration (or replacement titration).

In principle any ion can be used if it forms a weaker EDTA complex than the metal ion being determined. Still weaker metal-EDTA complexes would not interfere. Exchange reactions are also possible with other metal complexes to permit application of the chelometric titration to non-titrable cations and anions. The exchange reagent can be added and the titration performed in the sample solution without prior removal of the excess reagent. A most important example is the exchange of silver ion with an excess of the tetracyanonickelate ion according to the equation:

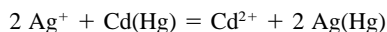


The nickel ion freed may then be determined by an EDTA titration. Note that two moles of silver are equivalent to one mole of nickel and thus to one mole of EDTA.

**11.6.6.1.4 Redox Titrations.** Redox titrations can be carried out in the presence of excess EDTA. Here EDTA acts to change the oxidation potential by forming a more stable complex with one oxidation state than with the other. Generally the oxidized form of the metal forms a more stable complex than the reduced form, and the couple becomes a stronger reducing agent in the presence of excess EDTA. For example, the Co(III)–Co(II) couple is shifted about 1.2 volts, so that Co(II) can be titrated with Ce(IV). Alternatively, Co(III) can be titrated to Co(II), with Cr(II) as a reducing agent.

Manganese(II) can be titrated directly to Mn(III) using hexacyanoferrate(III) as the oxidant. Alternatively, Mn(III), prepared by oxidation of the Mn(II)–EDTA complex with lead dioxide, can be determined by titration with standard iron(II) sulfate.

**11.6.6.1.5 Indirect Procedures.** Numerous inorganic anions that do not form complexes with a complexing agent are accessible to a chelometric titration by indirect procedures. Frequently the anion can be precipitated as a compound containing a stoichiometric amount of a titrable cation. Another indirect approach employing replacement mechanism is the reduction of a species with the liquid amalgam of a metal that can be determined by a chelometric titration after removal of excess amalgam. For example:



The equivalent amount of cadmium ion exchanged for the silver ion can readily be determined by EDTA titration procedures.

### **11.6.6.2 Preparation of Standard Solutions**

**11.6.6.2.1 Standard EDTA Solutions.** Disodium dihydrogen ethylenediaminetetraacetate dihydrate is available commercially of analytical reagent purity. After drying at 80°C for at least 24 hr, its composition agrees exactly with the dihydrate formula (molecular weight 372.25). It may be weighed directly. If an additional check on the concentration is required, it may be standardized by titration with nearly neutralized zinc chloride or zinc sulfate solution.

**11.6.6.2.2 Standard Magnesium Solution.** Dissolve 24.647 g of magnesium sulfate heptahydrate in water and dilute to 1 L for 0.1M solution.

**11.6.6.2.3 Standard Manganese(II) Solution.** Dissolve exactly 16.901 g ACS reagent grade manganese(II) sulfate hydrate in water and dilute to 1 L.

**11.6.6.2.4 Standard Zinc Solution.** Dissolve exactly 13.629 g of zinc chloride, ACS reagent grade, or 28.754 g of zinc sulfate heptahydrate, and dilute to 1 L for 0.1000M solution.

*11.6.6.2.5 Buffer Solution, pH 10.* Add 142 mL of concentrated ammonia solution (sp. grav. 0.88–0.90) to 17.5 g of analytical reagent ammonium chloride, and dilute to 250 mL.

*11.6.6.2.6 Water.* Distilled water must be (a) redistilled in an all-Pyrex glass apparatus or (b) purified by passage through a column of cation exchange resin in the sodium form. For storage, polyethylene bottles are most satisfactory, particularly for very dilute (0.001*M*) EDTA solutions.

*11.6.6.2.7 Murexide Indicator.* Suspend 0.5 g of powdered murexide in water, shake thoroughly, and allow the undissolved solid to settle. Use 5–6 drops of the supernatant liquid for each titration. Decant the old supernatant liquid daily and treat the residue with water to provide a fresh solution of the indicator.

Alternatively, grind 0.1 g of murexide with 10 g of ACS reagent grade sodium chloride; use about 50 mg of the mixture for each titration.

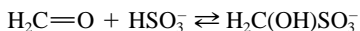
*11.6.6.2.8 Pyrocatechol Violet Indicator Solution.* Dissolve 0.1 g of the solid dyestuff in 100 mL of water.

## 11.6.7 Masking Agents

Masking (and demasking) techniques are widely used in analytical chemistry because they frequently provide convenient and elegant methods by which to avoid the effects of unwanted components of a system without having to resort to physical separation. The best molecules or ligands to use as masking agents are those that are chemically stable and nontoxic and react rapidly to form strong, colorless complexes with the ions to be masked, but form only relatively weak complexes with other ions that are present. Tables 11.36 and 11.37 are intended as qualitative guides to the types of masking agents likely to be suitable for particular analytical problems.

Masking must not be identified solely with complex formation. There are numerous complex compounds in which solutions show no masking effects. On the other hand, examples can be cited in which the product of soluble principal valence compounds may lead to masking. This latter category includes the annulment of the base action of  $\text{NH}_2$ — groups in carboxylic acids by the addition of formaldehyde, the masking of the iodometric oxidation of sulfites by formaldehyde, as well as the masking of almost all reactions of molybdenum(VI), tungsten(VI), and vanadium(V) by hydrogen peroxide or fluoride ion. Sometimes the masking agent changes the valence state of the metal ion. Examples include the reduction of Fe(III) to Fe(II) with hydrazine, hydroxylamine hydrochloride, or tin(II) chloride. Hydroxylamine also reduces Ce(IV) to Ce(III), Cu(II) to Cu(I), and Hg(II) to free Hg. Ascorbic acid reduces Cu(II) to Cu(I) in the presence of the chloride ion.

The reaction of the hydrogen sulfite ion in an alkaline solution with ketones and aldehydes is:



The carbon-oxygen double bond of the carbonyl group is opened, and the hydrogen sulfite radical is added. An increase in temperature reverses the reaction more easily for ketones than for aldehydes.

Certain organic substances have no charge at any pH but form complexes with substances that do have a charge. The sugars and polyalcohols form such complexes in the pH range between 9 and 10 with a number of anions; including borate, molybdate, and arsenite. Elegant ion exchange methods have been devised for the sugars.

Probably the most extensively applied masking agent is cyanide ion. In alkaline solution, cyanide forms strong cyano complexes with the following ions and masks their action toward EDTA: Ag, Cd, Co(II), Cu(II), Fe(II), Hg(II), Ni, Pd(II), Pt(II), Tl(III), and Zn. The alkaline earths, Mn(II), Pb, and the rare earths are virtually unaffected; hence, these latter ions may be titrated with EDTA with the former ions masked by cyanide. Iron(III) is also masked by cyanide. However, as the hexacyanoferrate(III) ion oxidizes many indicators, ascorbic acid is added to form hexacyanoferrate(II) ion. Moreover, since the addition of cyanide to an acidic solution results in the formation of deadly

hydrogen cyanide, the solution must first be made alkaline, with hydrous oxide formation prevented by the addition of tartrate. Zinc and cadmium may be demasked from their cyanide complexes by the action of formaldehyde.

Masking by oxidation or reduction of a metal ion to a state which does not react with EDTA is occasionally of value. For example, Fe(III) ( $\log K_{MY} = 24.23$ ) in acidic media may be reduced to Fe(II) ( $\log K_{MY} = 14.33$ ) by ascorbic acid; in this state iron does not interfere in the titration of some trivalent and tetravalent ions in strong acidic medium (pH 0 to 2). Similarly, Hg(II) can be reduced to the metal. In favorable conditions, Cr(III) may be oxidized by alkaline peroxide to chromate which does not complex with EDTA.

In resolving complex metal-ion mixtures, more than one masking or demasking process may be utilized with various aliquots of the sample solution, or applied simultaneously or stepwise with a single aliquot. In favorable cases, even four or five metals can be determined in a mixture by the application of direct and indirect masking processes. Of course, not all components of the mixture need be determined by chelometric titrations. For example, redox titrimetry may be applied to the determination of one or more of the metals present.

### 11.6.8 Demasking

For the major part, masking reactions that occur in solutions and lead to soluble compounds are equilibrium reactions. They usually require the use of an excess of the masking agent and can be reversed again by removal of the masking agent. The freeing of previously masked ionic or molecular species has been called *demasking*. This merits consideration in regard to its use in analysis. Masking never completely removes certain ionic or molecular species, but only reduces their concentrations. The extent of this lowering determines which color or precipitation reactions can be prevented. A system masked against a certain reagent is not necessarily masked against another but more aggressive reagent. It is therefore easy to see that masked reaction systems can also function as reagents at times (e.g., Fehling's solution, Nessler's reagent).

The methods used in demasking are varied. One approach is to change drastically the hydrogen ion concentration of the solution. The conditional stability constants of most metal complexes depend greatly on pH, so that simply raising or lowering the pH is frequently sufficient for selective demasking. In most cases a strong mineral acid is added, and the ligand is removed from the coordination sphere of the complex through the formation of a slightly ionized acid, as with the polyprotic (citric, tartaric, EDTA, and nitriloacetic) acids.

Another type of demasking involves formation of new complexes or other compounds that are more stable than the masked species. For example, boric acid is used to demask fluoride complexes of tin(IV) and molybdenum(VI). Formaldehyde is often used to remove the masking action of cyanide ions by converting the masking agent to a nonreacting species through the reaction:



which forms glycollic nitrile. Pertinent instances are the demasking of  $\text{Ni}(\text{CN})_4^{2-}$  ions to  $\text{Ni}^{2+}$  ions by formaldehyde and the demasking of dimethylglyoxime (dmg) from  $\text{Pd}(\text{dmg})_2^{2-}$  ions by cyanide. Selectivity is evident in that  $\text{Zn}(\text{CN})_4^{2-}$  is demasked whereas  $\text{Cu}(\text{CN})_3^{2-}$  is not.

Destruction of the masking ligand by chemical reaction may be possible, as in the oxidation of EDTA in acid solutions by permanganate or another strong oxidizing agent. Hydrogen peroxide and Cu(II) ion destroy the tartrate complex of aluminum.

Demasking methods for a number of masking agents are enumerated in Table 11.38.

**TABLE 11.30** Standard Solutions for Precipitation Titrations

The list given below includes the substances that are most used and most useful for the standardization of solutions for precipitation titrations. Primary standard solutions are denoted by the letter (P) in Column 1.

| Standard   | Formula weight | Preparation   |
|--|----------------|---|
| AgNO <sub>3</sub> (P)                                    | 169.89         | Weigh the desired amount of ACS reagent grade* AgNO <sub>3</sub> , dried at 105°C for 2 hr, and dissolve in double distilled water. Store in amber container and away from light. Check against NaCl.   |
| BaCl <sub>2</sub> · 2H <sub>2</sub> O                    | 244.28         | Dissolve clear crystals of the salt in distilled water. Standardize against K <sub>2</sub> SO <sub>4</sub> or Na <sub>2</sub> SO <sub>4</sub> .   |
| Hg(NO <sub>3</sub> ) <sub>2</sub> · H <sub>2</sub> O     | 342.62         | Dissolve the reagent grade salt in distilled water and dilute to desired volume. Standardize against NaCl.  |
| KBr  | 119.01         | The commercial reagent (ACS) may contain 0.2% chloride. Prepare an aqueous solution of approximately the desired concentration and standardize it against AgNO <sub>3</sub> .   |
| K <sub>4</sub> [Fe(CN)] <sub>6</sub> · 3H <sub>2</sub> O | 422.41         | Dissolve the high-purity commercial salt in distilled water containing 0.2 g/L of Na <sub>2</sub> CO <sub>3</sub> . Kept in an amber container and away from direct sunlight, solutions are stable for a month or more. Standardize against zinc metal. |
| KSCN   | 97.18          | Prepare aqueous solutions having the concentration desired. Standardize against AgNO <sub>3</sub> solution. Protect from direct sunlight.   |
| K <sub>2</sub> SO <sub>4</sub> (P)                       | 174.26         | Dissolve about 17.43 g, previously dried at 150°C and accurately weighed, in distilled water and dilute exactly to 1 L.   |
| NaCl (P)   | 58.44          | Dry at 130–150°C and weigh accurately, from a closed container, 5.844 g, dissolve in water, and dilute exactly to 1 L.  |
| NaF (P)  | 41.99          | Dry at 110°C and weigh the appropriate amount of ACS reagent. Dissolve in water and dilute exactly to 1 L.  |
| Na <sub>2</sub> SO <sub>4</sub> (P)                      | 142.04         | Weigh accurately 14.204 g, dried at 150°C, and dissolve in distilled water. Dilute to exactly 1 L.  |
| Th(NO <sub>3</sub> ) <sub>4</sub> · 4H <sub>2</sub> O    | 552.12         | Weigh the appropriate amount of crystals and dissolve in water. Standardize against NaF.  |

\* Meets standards of purity (and impurity) set by the American Chemical Society.

**TABLE 11.31** Indicators for Precipitation Titrations

| Indicator  | Preparation and use  |
|--|--|
| Specific reagents  |  |
| $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ | Use reagent (ACS)* grade salt, low in chloride. Dissolve 175 g in 100 mL 6 M $\text{HNO}_3$ which has been gently boiled for 10 min to expel nitrogen oxides. Dilute with 500 mL water. Use 2 mL per 100 mL of end-point volume. |
| $\text{K}_2\text{CrO}_4$   | Use reagent (ACS)* grade salt, low in chloride. Prepare 0.1M aqueous solution (19.421 g/L). Use 2.5 mL per 100 mL of end-point volume.   |
| Tetrahydroxy-1,4-benzoquinone (THQ)                              | Prepare fresh as required by dissolving 15 mg in 5 mL of water. Use 10 drops for each titration.   |
| Adsorption indicators  |  |
| Bromophenol blue   | Dissolve 0.1 g of the acid in 200 mL 95% ethanol.  |
| 2',7'-Dichlorofluorescein  | Dissolve 0.1 g of the acid in 100 mL 70% ethanol. Use 1 mL for 100 mL of initial solution.   |
| Eosin, tetrabromofluorescein                                     | See Dichlorofluorescein.   |
| Fluorescein  | Dissolve 0.4 g of the acid in 200 mL 70% ethanol. Use 10 drops.  |
| Potassium rhodizonate, $\text{C}_4\text{O}_4(\text{OK})_2$       | Prepare fresh as required by dissolving 15 mg in 5 mL of water. Use 10 drops for each titration.   |
| Rhodamine 6G   | Dissolve 0.1 g in 200 mL 70% ethanol.  |
| Sodium 3-alizarinsulfonate                                       | Prepare a 0.2% aqueous solution. Use 5 drops per 120 mL end-point volume.  |
| Thorin   | Prepare a 0.025% aqueous solution. Use 5 drops.  |
| Protective colloids  |  |
| Dextrin  | Use 5 mL of 2% aqueous solution of chloride-free dextrin per 25 mL of 0.1M halide solution.  |
| Polyethylene glycol 400  | Prepare a 50% (v/v) aqueous solution of the surfactant. Use 5 drops per 100 mL end-point volume.   |

\*Meets standards as set forth in *Reagent Chemicals*, American Chemical Society, Washington, D.C.; revised periodically.

TABLE 11.32 Properties and Applications of Selected Metal Ion Indicators

| Indicator   | Chemical name  | Dissociation constants and colors of free indicator species  | Colors of metal-indicator complexes                                     | Applications  |
|---|--|--|---|---|
| Calmagite<br>0.05 g/100 mL water; stable 1 year                           | 1-(6-Hydroxy- <i>m</i> -tolylazo)-2-naphthol-4-sulfonic acid   | $H_2In^-$ (red); $pK_2 = 8.1$<br>$HIn^{2-}$ (blue); $pK_3 = 12.4$<br>$In^{3-}$ (orange)  | Wine-red  | Titration performed with Eriochrome Black T as indicator may be carried out equally well with Calmagite   |
| Eriochrome Black T<br>0.1 g/100 mL water; prepare fresh daily             | 1-(2-Hydroxy-1-naphthyl-azo)-6-nitro-2-naphthol-4-sulfonic acid  | $H_2In^-$ (red); $pK_2 = 6.3$<br>$HIn^{2-}$ (blue); $pK_3 = 11.5$<br>$In^{3-}$ (yellow-orange)   | Wine-red  | <i>Direct titration:</i> Ba, Ca, Cd, In, Mg, Mn, Pb, Sc, Sr, Tl, Zn, and lanthanides<br><i>Back titration:</i> Al, Ba, Bi, Ca, Co, Cr, Fe, Ga, Hg, Mn, Ni, Pb, Pd, Sc, Tl, V<br><i>Substitution titration:</i> Au, Ba, Ca, Cu, Hg, Pb, Pd, Sr |
| Murexide<br>Suspend 0.5 g in water; use fresh supernatant liquid each day | 5-[(Hexahydro-2,4,6-trioxo-5-pyrimidinyl)imino]-2,4,6-(1 <i>H</i> ,3 <i>H</i> ,5 <i>H</i> )-pyrimidinetrione monoammonium salt | $H_4In^-$ (red-violet); $pK_2 = 9.2$<br>$H_3In^{2-}$ (violet); $pK_3 = 10.9$<br>$H_2In^{3-}$ (blue)  | Red with $Ca^{2+}$<br>Yellow with $Co^{2+}$ , $Ni^{2+}$ , and $Cu^{2+}$ | <i>Direct titration:</i> Ca, Co, Cu, Ni<br><i>Back titration:</i> Ca, Cr, Ga<br><i>Substitution titration:</i> Ag, Au, Pd   |
| PAN   | 1-(2-Pyridylazo)-2-naphthol  | $HIn$ (orange-red); $pK_1 = 12.3$<br>$In^-$ (pink)   | Red   | <i>Direct titration:</i> Cd, Cu, In, Sc, Tl, Zn<br><i>Back titration:</i> Cu, Fe, Ga, Ni, Pb, Sc, Sn, Zn<br><i>Substitution titration:</i> Al, Ca, Co, Fe, Ga, Hg, In, Mg, Mn, Ni, Pb, V, Zn  |
| Pyrocatechol Violet<br>0.1 g/100 mL; stable several weeks                 | Pyrocatecholsulfonephthalein   | $H_4In$ (red); $pK_1 = 0.2$<br>$H_3In^-$ (yellow); $pK_2 = 7.8$<br>$H_2In^{2-}$ (violet); $pK_3 = 9.8$<br>$HIn^{3-}$ (red-purple); $pK_4 = 11.7$ | Blue, except red with Th(IV)  | <i>Direct titration:</i> Al, Bi, Cd, Co, Fe, Ga, Mg, Mn, Ni, Pb, Th, Zn<br><i>Back titration:</i> Al, Bi, Fe, Ga, In, Ni, Pd, Sn, Th, Ti  |
| Salicylic acid  | 2-Hydroxybenzoic acid  | $H_2In$ ; $pK_1 = 2.98$<br>$HIn^-$ ; $pK_2 = 12.38$  | $FeSCN^{2+}$ at pH 3 is reddish-brown                                   | Typical uses: Fe(III) titrated with EDTA to colorless iron-EDTA complex   |
| Xylenol orange  | 3,3'-Bis[ <i>N,N</i> -di(carboxyethyl)aminomethyl]- <i>o</i> -cresolsulfonephthalein   | —COOH groups:<br>$pK_3 = 0.76$ ; $pK_4 = 1.15$ ;<br>$pK_5 = 2.58$ ; $pK_6 = 3.23$  |   | Typical uses: Bi, Pb, Th  |

Source: J. A. Dean, ed., *Analytical Chemistry Handbook*, McGraw-Hill, New York, 1995.

**TABLE 11.33** Variation of  $\alpha_4$  with pH

| pH  | $-\log \alpha_4$ | pH   | $-\log \alpha_4$ |
|-----|------------------|------|------------------|
| 2.0 | 13.44            | 7.0  | 3.33             |
| 2.5 | 11.86            | 8.0  | 2.29             |
| 3.0 | 10.60            | 9.0  | 1.29             |
| 4.0 | 8.48             | 10.0 | 0.46             |
| 5.0 | 6.45             | 11.0 | 0.07             |
| 6.0 | 4.66             | 12.0 | 0.00             |

**TABLE 11.34** Formation Constants of EDTA Complexes at 25°C, Ionic Strength Approaching Zero

| Metal ion | $\log K_{MY}$ | Metal ion | $\log K_{MY}$ |
|-----------|---------------|-----------|---------------|
| Co(III)   | 36            | V(IV)     | 18.0          |
| V(III)    | 25.9          | U(IV)     | 17.5          |
| In        | 24.95         | Ti(IV)    | 17.3          |
| Fe(III)   | 24.23         | Ce(III)   | 16.80         |
| Th        | 23.2          | Zn        | 16.4          |
| Sc        | 23.1          | Cd        | 16.4          |
| Cr(III)   | 23            | Co(II)    | 16.31         |
| Bi        | 22.8          | Al        | 16.13         |
| Tl(III)   | 22.5          | La        | 16.34         |
| Sn(II)    | 22.1          | Fe(II)    | 14.33         |
| Ti(III)   | 21.3          | Mn(II)    | 13.8          |
| Hg(II)    | 21.80         | Cr(II)    | 13.6          |
| Ga        | 20.25         | V(II)     | 12.7          |
| Zr        | 19.40         | Ca        | 11.0          |
| Cu(II)    | 18.7          | Be        | 9.3           |
| Ni        | 18.56         | Mg        | 8.64          |
| Pd(II)    | 18.5          | Sr        | 8.80          |
| Pb(II)    | 18.3          | Ba        | 7.78          |
| V(V)      | 18.05         | Ag        | 7.32          |

**TABLE 11.35** Cumulative Formation Constants of Ammine Complexes at 20°C, Ionic Strength 0.1

| Cation        | $\log K_1$ | $\log K_2$ | $\log K_3$ | $\log K_4$ | $\log K_5$ | $\log K_6$ |
|---------------|------------|------------|------------|------------|------------|------------|
| Cadmium       | 2.65       | 4.75       | 6.19       | 7.12       | 6.80       | 5.14       |
| Cobalt(II)    | 2.11       | 3.74       | 4.79       | 5.55       | 5.73       | 5.11       |
| Cobalt(III)   | 6.7        | 14.0       | 20.1       | 25.7       | 30.8       | 35.2       |
| Copper(I)     | 5.93       | 10.86      |            |            |            |            |
| Copper(II)    | 4.31       | 7.98       | 11.02      | 13.32      | 12.66      |            |
| Iron(II)      | 1.4        | 2.2        |            |            |            |            |
| Manganese(II) | 0.8        | 1.3        |            |            |            |            |
| Mercury(II)   | 8.8        | 17.5       | 18.5       | 19.28      |            |            |
| Nickel        | 2.80       | 5.04       | 6.77       | 7.96       | 8.71       | 8.74       |
| Platinum(II)  |            |            |            |            |            | 35.3       |
| Silver(I)     | 3.24       | 7.05       |            |            |            |            |
| Zinc          | 2.37       | 4.81       | 7.31       | 9.46       |            |            |

**TABLE 11.36** Masking Agents for Various Elements

| Element | Masking agent   |
|---------|---|
| Ag      | $\text{Br}^-$ , citrate, $\text{Cl}^-$ , $\text{CN}^-$ , $\text{I}^-$ , $\text{NH}_3$ , $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , thiourea, thioglycolic acid, diethyldithiocarbamate, thiosemicarbazide, bis(2-hydroxyethyl)dithiocarbamate   |
| Al      | Acetate, acetylacetone, $\text{BF}_4^-$ , citrate, $\text{C}_2\text{O}_4^{2-}$ , EDTA, $\text{F}^-$ , formate, 8-hydroxyquinoline-5-sulfonic acid, mannitol, 2,3-mercaptopropanol, $\text{OH}^-$ , salicylate, sulfosalicylate, tartrate, triethanolamine, tiron  |
| As      | Citrate, 2,3-dimercaptopropanol, $\text{NH}_2\text{OH} \cdot \text{HCl}$ , $\text{OH}^-$ , $\text{S}_2^{2-}$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate   |
| Au      | $\text{Br}^-$ , $\text{CN}^-$ , $\text{NH}_3$ , $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , thiourea   |
| Ba      | Citrate, cyclohexanediaminetetraacetic acid, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , $\text{SO}_4^{2-}$ , tartrate  |
| Be      | Acetylacetone, citrate, EDTA, $\text{F}^-$ , sulfosalicylate, tartrate  |
| Bi      | $\text{Br}^-$ , citrate, $\text{Cl}^-$ , 2,3-dimercaptopropanol, dithizone, EDTA, $\text{I}^-$ , $\text{OH}^-$ , $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{SCN}^-$ , tartrate, thiosulfate, thiourea, triethanolamine   |
| Ca      | $\text{BF}_4^-$ , citrate, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , polyphosphates, tartrate   |
| Cd      | Citrate, $\text{CN}^-$ , 2,3-dimercaptopropanol, dimercaptosuccinic acid, dithizone, EDTA, glycine, $\text{I}^-$ , malonate, $\text{NH}_3$ , 1,10-phenanthroline, $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate   |
| Ce      | Citrate, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , $\text{PO}_4^{3-}$ , reducing agents (ascorbic acid), tartrate, tiron  |
| Co      | Citrate, $\text{CN}^-$ , diethyldithiocarbamate, 2,3-dimercaptopropanol, dimethylglyoxime, ethylenediamine, EDTA, $\text{F}^-$ , glycine, $\text{H}_2\text{O}_2$ , $\text{NH}_3$ , $\text{NO}_2^-$ , 1,10-phenanthroline, $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate  |
| Cr      | Acetate, (reduction with) ascorbic acid + KI, citrate, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , formate, $\text{NaOH} + \text{H}_2\text{O}_2$ , oxidation to $\text{CrO}_4^{2-}$ , $\text{Na}_5\text{P}_3\text{O}_{10}$ , sulfosalicylate, tartrate, triethylamine, tiron  |
| Cu      | Ascorbic acid + KI, citrate, $\text{CN}^-$ , diethyldithiocarbamate, 2,3-dimercaptopropanol, ethylenediamine, EDTA, glycine, hexacyanocobalt(III)(3-), hydrazine, $\text{I}^-$ , $\text{NaH}_2\text{PO}_2$ , $\text{NH}_2\text{OH} \cdot \text{HCl}$ , $\text{NH}_3$ , $\text{NO}_2^-$ , 1,10-phenanthroline, $\text{S}^{2-}$ , $\text{SCN}^- + \text{SO}_3^{2-}$ , $\text{S}_2\text{O}_3^{2-}$ , sulfosalicylate, tartrate, thioglycolic acid, thiosemicarbazide, thiocarbohydrazide, thiourea                                 |
| Fe      | Acetylacetone, (reduction with) ascorbic acid, $\text{C}_2\text{O}_4^{2-}$ , citrate, $\text{CN}^-$ , 2,3-dimercaptopropanol, EDTA, $\text{F}^-$ , $\text{NH}_3$ , $\text{NH}_2\text{OH} \cdot \text{HCl}$ , $\text{OH}^-$ , oxine, 1,10-phenanthroline, 2,2'-bipyridyl, $\text{PO}_4^{3-}$ , $\text{P}_2\text{O}_7^{4-}$ , $\text{S}^{2-}$ , $\text{SCN}^-$ , $\text{SnCl}_2$ , $\text{S}_2\text{O}_3^{2-}$ , sulfamic acid, sulfosalicylate, tartrate, thioglycolic acid, thiourea, tiron, triethanolamine, triethiocarbonate |
| Ga      | Citrate, $\text{Cl}^-$ , EDTA, $\text{OH}^-$ , oxalate, sulfosalicylate, tartrate   |
| Ge      | $\text{F}^-$ , oxalate, tartrate  |
| Hf      | See Zr  |
| Hg      | Acetone, (reduction with) ascorbic acid, citrate, $\text{Cl}^-$ , $\text{CN}^-$ , 2,3-dimercaptopropan-1-ol, EDTA, formate, $\text{I}^-$ , $\text{SCN}^-$ , $\text{SO}_3^{2-}$ , tartrate, thiosemicarbazide, thiourea, triethanolamine   |
| In      | $\text{Cl}^-$ , EDTA, $\text{F}^-$ , $\text{SCN}^-$ , tartrate, thiourea, triethanolamine   |
| Ir      | Citrate, $\text{CN}^-$ , $\text{SCN}^-$ , tartrate, thiourea  |
| La      | Citrate, EDTA, $\text{F}^-$ , oxalate, tartrate, tiron  |
| Mg      | Citrate, $\text{C}_2\text{O}_4^{2-}$ , cyclohexane-1,2-diaminetetraacetic acid, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , glycol, hexametaphosphate, $\text{OH}^-$ , $\text{P}_2\text{O}_7^{4-}$ , triethanolamine  |
| Mn      | Citrate, $\text{CN}^-$ , $\text{C}_2\text{O}_4^{2-}$ , 2,3-dimercaptopropanol, EDTA, $\text{F}^-$ , $\text{Na}_5\text{P}_3\text{O}_{10}$ , oxidation to $\text{MnO}_4^-$ , $\text{P}_2\text{O}_7^{4-}$ , reduction to Mn(II) with $\text{NH}_2\text{OH} \cdot \text{HCl}$ or hydrazine, sulfosalicylate, tartrate, triethanolamine, triphosphate, tiron   |
| Mo      | Acetylacetone, ascorbic acid, citrate, $\text{C}_2\text{O}_4^{2-}$ , EDTA, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , hydrazine, mannitol, $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{NH}_2\text{OH} \cdot \text{HCl}$ , oxidation to molybdate, $\text{SCN}^-$ , tartrate, tiron, triphosphate   |

TABLE 11.36 Masking Agents for Various Elements (*Continued*)

| Element         | Masking agent  |
|-----------------|--|
| Nb              | Citrate, $\text{C}_2\text{O}_4^{2-}$ , $\text{F}^-$ , $\text{H}_2\text{O}_2$ , $\text{OH}^-$ , tartrate  |
| Nd              | EDTA   |
| $\text{NH}_4^+$ | HCHO   |
| Ni              | Citrate, $\text{CN}^-$ , <i>N,N</i> -dihydroxyethylglycine, dimethylglyoxime, EDTA, $\text{F}^-$ , glycine, malonate, $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{NH}_3$ , 1,10-phenanthroline, $\text{SCN}^-$ , sulfosalicylate, thioglycolic acid, triethanolamine, tartrate   |
| Np              | $\text{F}^-$   |
| Os              | $\text{CN}^-$ , $\text{SCN}^-$ , thiourea  |
| Pa              | $\text{H}_2\text{O}_2$   |
| Pb              | Acetate, $(\text{C}_6\text{H}_5)_4\text{AsCl}$ , citrate, 2,3-dimercaptopropanol, EDTA, $\text{I}^-$ , $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{SO}_4^{2-}$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate, tiron, tetraphenylarsonium chloride, triethanolamine, thioglycolic acid   |
| Pd              | Acetylacetone, citrate, $\text{CN}^-$ , EDTA, $\text{I}^-$ , $\text{NH}_3$ , $\text{NO}_2^-$ , $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate, triethanol amine   |
| Pt              | Citrate, $\text{CN}^-$ , EDTA, $\text{I}^-$ , $\text{NH}_3$ , $\text{NO}_2^-$ , $\text{SCN}^-$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate, urea  |
| Pu              | Reduction to Pu(IV) with sulfamic acid   |
| Rare earths     | $\text{C}_2\text{O}_4^{2-}$ , citrate, EDTA, $\text{F}^-$ , tartrate   |
| Re              | Oxidation to perrhenate  |
| Rh              | Citrate, tartrate, thiourea  |
| Ru              | $\text{CN}^-$ , thiourea   |
| Sb              | Citrate, 2,3-dimercaptopropanol, EDTA, $\text{F}^-$ , $\text{I}^-$ , $\text{OH}^-$ , oxalate, $\text{S}^{2-}$ , $\text{S}_2^{2-}$ , $\text{S}_2\text{O}_3^{2-}$ , tartrate, triethanolamine  |
| Sc              | Cyclohexane-1,2-diaminetetraacetic acid, $\text{F}^-$ , tartrate   |
| Se              | Citrate, $\text{F}^-$ , $\text{I}^-$ , reducing agents, $\text{S}^{2-}$ , $\text{SO}_3^{2-}$ , tartrate  |
| Sn              | Citrate, $\text{C}_2\text{O}_3^{2-}$ , 2,3-dimercaptopropanol, EDTA, $\text{F}^-$ , $\text{I}^-$ , $\text{OH}^-$ , oxidation with bromine water, phosphate(3-), tartrate, triethanolamine, thioglycolic acid   |
| Sr              | Citrate, <i>N,N</i> -dihydroxyethylglycine, EDTA, $\text{F}^-$ , $\text{SO}_4^{2-}$ , tartrate   |
| Ta              | Citrate, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , $\text{OH}^-$ , oxalate, tartrate   |
| Te              | Citrate, $\text{F}^-$ , $\text{I}^-$ , reducing agents, $\text{S}^{2-}$ , sulfite, tartrate  |
| Th              | Acetate, acetylacetone, citrate, EDTA, $\text{F}^-$ , $\text{SO}_4^{2-}$ , 4-sulfobenzenearsonic acid, sulfosalicylic acid, tartrate, triethanolamine  |
| Ti              | Ascorbic acid, citrate, $\text{F}^-$ , gluconate, $\text{H}_2\text{O}_2$ , mannitol, $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{OH}^-$ , $\text{SO}_4^{2-}$ , sulfosalicylic acid, tartrate, triethanolamine, tiron   |
| Tl              | Citrate, $\text{Cl}^-$ , $\text{CN}^-$ , EDTA, HCHO, hydrazine, $\text{NH}_2\text{OH} \cdot \text{HCl}$ , oxalate, tartrate, triethanolamine   |
| U               | Citrate, $(\text{NH}_4)_2\text{CO}_3$ , $\text{C}_2\text{O}_4^{2-}$ , EDTA, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , hydrazine + triethanolamine, phosphate(3-), tartrate   |
| V               | (Reduction with) ascorbic acid, hydrazine, or $\text{NH}_2\text{OH} \cdot \text{HCl}$ , $\text{CN}^-$ , EDTA, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , mannitol, oxidation to vanadate, triethanolamine, tiron  |
| W               | Citrate, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , hydrazine, $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{NH}_2\text{OH} \cdot \text{HCl}$ , oxalate, $\text{SCN}^-$ , tartrate, tiron, triphosphate, oxidation to tungstate(VI)   |
| Y               | Cyclohexane-1,2-diaminetetraacetic acid, $\text{F}^-$  |
| Zn              | Citrate, $\text{CN}^-$ , <i>N,N</i> -dihydroxyethylglycine, 2,3-dimercaptopropanol, dithizone, EDTA, $\text{F}^-$ , glycerol, glycol, hexacyanoferrate(II)(4-), $\text{Na}_5\text{P}_3\text{O}_{10}$ , $\text{NH}_3$ , $\text{OH}^-$ , $\text{SCN}^-$ , tartrate, triethanolamine  |
| Zr              | Arsenazo, carbonate, citrate, $\text{C}_2\text{O}_4^{2-}$ , cyclohexane-1,2-diaminetetraacetic acid, EDTA, $\text{F}^-$ , $\text{H}_2\text{O}_2$ , $\text{PO}_4^{3-}$ , $\text{P}_2\text{O}_7^{4-}$ , pyrogallol, quinalizarinesulfonic acid, salicylate, $\text{SO}_4^{2-}$ + $\text{H}_2\text{O}_2$ , sulfosalicylate, tartrate, triethanolamine |

**TABLE 11.37** Masking Agents for Anions and Neutral Molecules

| Anion or neutral molecule         | Masking agent  |
|-----------------------------------|--|
| Boric acid                        | F <sup>-</sup> , glycol, mannitol, tartrate, and other hydroxy acids   |
| Br <sup>-</sup>                   | Hg(II)   |
| Br <sub>2</sub>                   | Phenol, sulfosalicylic acid  |
| BrO <sub>3</sub> <sup>-</sup>     | Reduction with arsenate(III), hydrazine, sulfite, or thiosulfate   |
| Chromate(VI)                      | Reduction with arsenate(III), ascorbic acid, hydrazine, hydroxylamine, sulfite, or thiosulfate                     |
| Citrate                           | Ca(II)   |
| Cl <sup>-</sup>                   | Hg(II), Sb(III)  |
| Cl <sub>2</sub>                   | Sulfite  |
| ClO <sub>3</sub> <sup>-</sup>     | Thiosulfate  |
| ClO <sub>4</sub> <sup>-</sup>     | Hydrazine, sulfite   |
| CN <sup>-</sup>                   | HCHO, Hg(II), transition metal ions  |
| EDTA                              | Cu(II)   |
| F <sup>-</sup>                    | Al(III), Be(II), boric acid, Fe(III), Th(IV), Ti(IV), Zr(IV)   |
| Fe(CN) <sub>6</sub> <sup>3-</sup> | Arsenate(III), ascorbic acid, hydrazine, hydroxylamine, thiosulfate  |
| Germanic acid                     | Glucose, glycerol, mannitol  |
| I <sup>-</sup>                    | Hg(II)   |
| I <sub>2</sub>                    | Thiosulfate  |
| IO <sub>3</sub> <sup>-</sup>      | Hydrazine, sulfite, thiosulfate  |
| IO <sub>4</sub> <sup>-</sup>      | Arsenate(III), hydrazine, molybdate(VI), sulfite, thiosulfate  |
| MnO <sub>4</sub> <sup>-</sup>     | Reduction with arsenate(III), ascorbic acid, azide, hydrazine, hydroxylamine, oxalic acid, sulfite, or thiosulfate |
| MoO <sub>4</sub> <sup>2-</sup>    | Citrate, F <sup>-</sup> , H <sub>2</sub> O <sub>2</sub> , oxalate, thiocyanate + Sn(II)                            |
| NO <sub>2</sub> <sup>-</sup>      | Co(II), sulfamic acid, sulfanilic acid, urea   |
| Oxalate                           | Molybdate(VI), permanganate  |
| Phosphate                         | Fe(III), tartrate  |
| S                                 | CN <sup>-</sup> , S <sup>2-</sup> , sulfite  |
| S <sup>2-</sup>                   | Permanganate + sulfuric acid, sulfur   |
| Sulfate                           | Cr(III) + heat   |
| Sulfite                           | HCHO, Hg(II), permanganate + sulfuric acid   |
| SO <sub>3</sub> <sup>2-</sup>     | Ascorbic acid, hydroxylamine, thiosulfate  |
| Se and its anions                 | Diaminobenzidine, sulfide, sulfite   |
| Te                                | I <sup>-</sup>   |
| Tungstate                         | Citrate, tartrate  |
| Vanadate                          | Tartrate   |

**TABLE 11.38** Common Demasking Agents

Abbreviations: DPC, diphenylcarbazide; HDMG, dimethylglyoxime; PAN, 1-(2-pyridylazo)-2-naphthol.

| Complexing agent | Ion demasked  | Demasking agent        | Application                                  |
|------------------|---|------------------------|--|
| CN <sup>-</sup>  | Ag <sup>+</sup><br>Cd <sup>2+</sup>   | H <sup>+</sup>         | Precipitation of Ag                          |
|                  |   | H <sup>+</sup>         | Free Cd <sup>2+</sup>                        |
|                  |   | HCHO + OH <sup>-</sup> | Detection of Cd (with DPC) in presence of Cu |
|                  | Cu <sup>+</sup><br>Cu <sup>2+</sup><br>Fe <sup>2+</sup><br>Fe <sup>3+</sup> | H <sup>+</sup>         | Precipitation of Cu                          |
|                  |   | HgO                    | Determination of Cu                          |
|                  |   | Hg <sup>2+</sup>       | Free Fe <sup>2+</sup>                        |
|                  |   | HgO                    | Determination of Fe                          |

TABLE 11.38 Common Demasking Agents (*Continued*)

| Complexing agent   | Ion demasked  | Demasking agent                                       | Application  |
|--|---|---|--|
| CN <sup>-</sup> ( <i>continued</i> )   | HDMG<br>Hg <sup>2+</sup><br>Ni <sup>2+</sup>        | Pd <sup>2+</sup>                                      | Detection of CN <sup>-</sup> (with Ni <sup>2+</sup> )  |
|  |   | Pd <sup>2+</sup>                                      | Detection of Pd (with DPC)   |
|  |   | HCHO  | Detection of Ni (with HDMG)  |
|  |   | H <sup>+</sup>  | Free Ni <sup>2+</sup>  |
|  |   | HgO   | Determination of Ni  |
|  |   | Ag <sup>+</sup>                                       | Detection and determination of Ni (with HDMG) in presence of Co                                      |
|  | Pd <sup>2+</sup>                                    | Ag <sup>+</sup> , Hg <sup>2+</sup> , Pb <sup>2+</sup> | Detection of Ag, Hg, Pb (with HDMG)  |
|  |   | H <sup>+</sup>  | Precipitation of Pd  |
|  | Zn <sup>2+</sup>                                    | HgO   | Determination of Pd  |
|  |   | Cl <sub>3</sub> CCHO · H <sub>2</sub> O               | Titration of Zn with EDTA  |
| CO <sub>3</sub> <sup>2-</sup><br>C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> | Cu <sup>2+</sup>                                    | H <sup>+</sup>  | Free Zn  |
|  | Al <sup>3+</sup>                                    | OH <sup>-</sup>                                       | Free Cu <sup>2+</sup>  |
|  | Ag <sup>+</sup>                                     | H <sub>2</sub> O                                      | Precipitation of Al(OH) <sub>3</sub>   |
|  | Ag <sup>+</sup>                                     | SiO <sub>2</sub> (amorphous)                          | Precipitation of AgCl  |
| Cl <sup>-</sup> (concentrated)<br>Ethylenediamine                            | Ag <sup>+</sup>                                     | SiO <sub>2</sub> (amorphous)                          | Differentiation of crystalline and amorphous SiO <sub>2</sub> (with CrO <sub>4</sub> <sup>2-</sup> ) |
|  | Al <sup>3+</sup>                                    | F <sup>-</sup>  | Titration of Al  |
|  | Ba <sup>2+</sup>                                    | H <sup>+</sup>  | Precipitation of BaSO <sub>4</sub> (with SO <sub>4</sub> <sup>2-</sup> )                             |
|  | Co <sup>2+</sup>                                    | Ca <sup>2+</sup>                                      | Detection of Co (with diethyldithiocarbamate)  |
| EDTA   | Mg <sup>2+</sup>                                    | F <sup>-</sup>  | Titration of Mg, Mn  |
|  | Th(IV)  | SO <sub>4</sub> <sup>2-</sup>                         | Titration of Th  |
|  | Ti(IV)  | Mg <sup>2+</sup>                                      | Precipitation of Ti (with NH <sub>3</sub> )  |
|  | Zn <sup>2+</sup>                                    | CN <sup>-</sup>                                       | Titration of Mg, Mn, Zn  |
| F <sup>-</sup>   | Many ions<br>Al(III)                                | KMO <sub>4</sub> <sup>-</sup>                         | Free ions  |
|  |   | Be(II)  | Precipitation of Al (with 8-hydroxyquinoline)  |
|  | Fe(III)<br>Hf(IV)<br>Mo(VI)<br>Sn(IV)<br>U(VI)      | OH <sup>-</sup>                                       | Precipitation of Al(OH) <sub>3</sub>   |
|  |   | OH <sup>-</sup>                                       | Precipitation of Fe(OH) <sub>3</sub>   |
|  |   | Al(III) or Be(II)                                     | Detection of Hg (with xylenol orange)  |
|  |   | H <sub>3</sub> BO <sub>3</sub>                        | Free molybdate   |
|  |   | H <sub>3</sub> BO <sub>3</sub>                        | Precipitation of Sn (with H <sub>2</sub> S)  |
|  |   | Al(III)   | Detection of U (with dibenzoylmethane)   |
|  |   | Al(III) or Be(II)                                     | Detection of Zr (with xylenol orange)  |
|  | Zr(IV)  | Ca(II)  | Detection of Ca (with alizarin S)  |
|  |   | OH <sup>-</sup>                                       | Precipitation of Zr(OH) <sub>4</sub>   |
|  |   | Fe(III)   | Free ions  |
|  | Hf(IV), Ti(IV), or Zr                               | Br <sup>-</sup>                                       | Detection of Br <sup>-</sup>   |
|  |   | H <sup>+</sup>  | Detection of Ag  |
|  |   | I <sup>-</sup>  | Detection of I and Br  |
|  |   | SiO <sub>2</sub> (amorphous)                          | Differentiation of crystalline and amorphous SiO <sub>2</sub> (with CrO <sub>4</sub> <sup>2-</sup> ) |
| H <sub>2</sub> O <sub>2</sub>  | Co(III)<br>Fe(III)<br>UO <sub>2</sub> <sup>2-</sup> | H <sup>+</sup>  | Free Co  |
|  |   | OH <sup>-</sup>                                       | Precipitation of FePO <sub>4</sub>   |
|  |   | Al(III)   | Detection of U (with dibenzoylmethane)   |
| NO <sub>2</sub> <sup>-</sup><br>PO <sub>4</sub> <sup>3-</sup>                | Fe(III)   | OH <sup>-</sup>                                       | Precipitation of Fe(OH) <sub>3</sub>   |
|  | Ba <sup>2+</sup>                                    | H <sub>2</sub> O                                      | Precipitation of BaSO <sub>4</sub>   |
|  | Ag <sup>+</sup>                                     | H <sup>+</sup>  | Free Ag <sup>+</sup>   |
|  | Cu <sup>2+</sup>                                    | OH <sup>-</sup>                                       | Detection of Cu (with PAN)   |
| Tartrate   | Al(III)   | H <sub>2</sub> O <sub>2</sub> + Cu <sup>2+</sup>      | Precipitation of Al(OH) <sub>3</sub>   |

**TABLE 11.39** Amino Acids pI and  $pK_a$  Values

This table lists the  $pK_a$  and pI (pH at the isoelectric point) values of  $\alpha$ -amino acids commonly found in proteins along with their abbreviations. The dissociation constants refer to aqueous solutions at 25°C.

| Name          | Abbreviations |          | $pK_a$ values |                               |              | pI values |
|---------------|---------------|----------|---------------|-------------------------------|--------------|-----------|
|               | 3 Letter      | 1 Letter | —COOH         | —NH <sub>3</sub> <sup>+</sup> | Other groups |           |
| Alanine       | Ala           | A        | 2.34          | 9.69                          |              | 6.00      |
| Arginine      | Arg           | R        | 2.17          | 9.04                          | 12.48        | 10.76     |
| Asparagine    | Asn           | N        | 2.01          | 8.80                          |              | 5.41      |
| Aspartic acid | Asp           | D        | 1.89          | 9.60                          | 3.65         | 2.77      |
| Cysteine      | Cys           | C        | 1.96          | 10.28                         | 8.18         | 5.07      |
| Glutamine     | Gln           | Q        | 2.17          | 9.13                          |              | 5.65      |
| Glutamic acid | Glu           | E        | 2.19          | 9.67                          | 4.25         | 3.22      |
| Glycine       | Gly           | G        | 2.34          | 9.60                          |              | 5.97      |
| Histidine     | His           | H        | 1.82          | 9.17                          | 6.00         | 7.59      |
| Isoleucine    | Ile           | I        | 2.36          | 9.60                          |              | 6.02      |
| Leucine       | Leu           | L        | 2.36          | 9.60                          | 5.98         |           |
| Lysine        | Lys           | K        | 2.18          | 8.98                          | 10.53        | 9.74      |
| Methionine    | Met           | M        | 2.28          | 9.21                          |              | 5.74      |
| Phenylalanine | Phe           | F        | 1.83          | 9.13                          |              | 5.48      |
| Proline       | Pro           | P        | 1.99          | 10.60                         |              | 6.30      |
| Serine        | Ser           | S        | 2.21          | 9.15                          |              | 5.68      |
| Threonine     | Thr           | T        | 2.09          | 9.10                          |              | 5.60      |
| Tryptophan    | Trp           | W        | 2.83          | 9.39                          |              | 5.89      |
| Tyrosine      | Tyr           | Y        | 2.20          | 9.11                          | 10.07        | 5.66      |
| Valine        | Val           | V        | 2.32          | 9.62                          |              | 5.96      |

**Source:** E. L. Smith, et al., *Principles of Biochemistry*, 7th ed., McGraw-Hill, New York, 1983; H. J. Hinz, ed., *Thermodynamic Data for Biochemistry and Biotechnology*, Springer-Verlag, Heidelberg, 1986.

**TABLE 11.40** Tolerances of Volumetric Flasks

| Capacity, mL | Tolerances,* $\pm$ mL |         | Capacity, mL | Tolerances,* $\pm$ mL |         |
|--------------|-----------------------|---------|--------------|-----------------------|---------|
|              | Class A               | Class B |              | Class A               | Class B |
| 5            | 0.02                  | 0.04    | 200          | 0.10                  | 0.20    |
| 10           | 0.02                  | 0.04    | 250          | 0.12                  | 0.24    |
| 25           | 0.03                  | 0.06    | 500          | 0.20                  | 0.40    |
| 50           | 0.05                  | 0.10    | 1000         | 0.30                  | 0.60    |
| 100          | 0.08                  | 0.16    | 2000         | 0.50                  | 1.00    |

\*Accuracy tolerances for volumetric flasks at 20°C are given by ASTM standard E288.

**TABLE 11.41** Pipet Capacity Tolerances

| Volumetric transfer pipets |                       |         | Measuring and serological pipets |                       |
|----------------------------|-----------------------|---------|----------------------------------|-----------------------|
| Capacity, mL               | Tolerances,* $\pm$ mL |         | Capacity, mL                     | Tolerances,† $\pm$ mL |
|                            | Class A               | Class B |                                  | Class B               |
| 0.5                        | 0.006                 | 0.012   | 0.1                              | 0.005                 |
| 1                          | 0.006                 | 0.012   | 0.2                              | 0.008                 |
| 2                          | 0.006                 | 0.012   | 0.25                             | 0.008                 |
| 3                          | 0.01                  | 0.02    | 0.5                              | 0.01                  |
| 4                          | 0.01                  | 0.02    | 0.6                              | 0.01                  |
| 5                          | 0.01                  | 0.02    | 1                                | 0.02                  |
| 10                         | 0.02                  | 0.04    | 2                                | 0.02                  |
| 15                         | 0.03                  | 0.06    | 5                                | 0.04                  |
| 20                         | 0.03                  | 0.06    | 10                               | 0.06                  |
| 25                         | 0.03                  | 0.06    | 25                               | 0.10                  |
| 50                         | 0.05                  | 0.10    |                                  |                       |
| 100                        | 0.08                  | 0.16    |                                  |                       |

\* Accuracy tolerances for volumetric transfer pipets are given by ASTM standard E969 and Federal Specification NNN-P-395.

† Accuracy tolerances for measuring pipets are given by Federal Specification NNN-P-350 and for serological pipets by Federal Specification NNN-P-375.

**TABLE 11.42** Tolerances of Micropipets (Eppendorf)

| Capacity, $\mu$ L | Accuracy, % | Precision, % | Capacity, $\mu$ L | Accuracy, % | Precision, % |
|-------------------|-------------|--------------|-------------------|-------------|--------------|
| 10                | 1.2         | 0.4          | 100               | 0.5         | 0.2          |
| 40                | 0.6         | 0.2          | 250               | 0.5         | 0.15         |
| 50                | 0.5         | 0.2          | 500               | 0.5         | 0.15         |
| 60                | 0.5         | 0.2          | 600               | 0.5         | 0.15         |
| 70                | 0.5         | 0.2          | 900               | 0.5         | 0.15         |
| 80                | 0.5         | 0.2          | 1000              | 0.5         | 0.15         |

**TABLE 11.43** Buret Accuracy Tolerances

| Capacity, mL | Subdivision, mL | Accuracy, $\pm$ mL           |                            |
|--------------|-----------------|------------------------------|----------------------------|
|              |                 | Class A* and precision grade | Class B and standard grade |
| 10           | 0.05            | 0.02                         | 0.04                       |
| 25           | 0.10            | 0.03                         | 0.06                       |
| 50           | 0.10            | 0.05                         | 0.10                       |
| 100          | 0.20            | 0.10                         | 0.20                       |

\* Class A conforms to specifications in ASTM E694 for standard taper stopcocks and to ASTM E287 for Teflon or polytetrafluoroethylene stopcock plugs. The 10-mL size meets the requirements for ASTM D664.

**TABLE 11.44** Factors for Simplified Computation of Volume

The volume is determined by weighing the water, having a temperature of  $t^{\circ}\text{C}$ , contained or delivered by the apparatus at the same temperature. The weight of water,  $w$  grams, is obtained with brass weights in air having a density of 1.20 mg/mL.

For apparatus made of soft glass, the volume contained or delivered at  $20^{\circ}\text{C}$  is given by

$$\nu_{20} = wf_{20} \text{ mL}$$

where  $\nu_{20}$  is the volume at  $20^{\circ}$  and  $f_{20}$  is the factor (apparent specific volume) obtained from the table below for the temperature  $t$  at which the calibration is performed. The volume at any other temperature  $t'$  may then be obtained from

$$\nu' = \nu_{20}[1 + 0.00002(t' - 20)] \text{ mL}$$

For apparatus made of any other material, the volume contained or delivered at the temperature  $t$  is

$$\nu_t = wf_t \text{ mL}$$

where  $w$  is again the weight in air obtained with brass weights (in grams), and  $f_t$  is the factor given in the third column of the table for the temperature  $t$ . The volume at any temperature  $t'$  may then be obtained from

$$\nu'_t = \nu_t[1 + \beta(t' - t)] \text{ mL}$$

where  $\beta$  is the cubical coefficient of thermal expansion of the material from which the apparatus is made. Approximate values of  $\beta$  for some frequently encountered materials are given in Table 11.45.

| $t, ^{\circ}\text{C}$ | $f_{20}$ | $f_t$    | $t, ^{\circ}\text{C}$ | $f_{20}$ | $f_t$    |
|-----------------------|----------|----------|-----------------------|----------|----------|
| 0                     | 1.001 62 | 1.001 22 | 20                    | 1.002 86 | 1.002 86 |
| 1                     | 54       | 16       | 21                    | 1.003 05 | 1.003 07 |
| 2                     | 48       | 12       | 22                    | 26       | 30       |
| 3                     | 43       | 09       | 23                    | 47       | 53       |
| 4                     | 41       | 09       | 24                    | 69       | 77       |
| 5                     | 1.001 39 | 1.001 09 | 25                    | 1.003 93 | 1.004 03 |
| 6                     | 40       | 12       | 26                    | 1.004 17 | 29       |
| 7                     | 42       | 16       | 27                    | 42       | 56       |
| 8                     | 45       | 21       | 28                    | 68       | 84       |
| 9                     | 50       | 28       | 29                    | 95       | 1.005 13 |
| 10                    | 1.001 56 | 1.001 36 | 30                    | 1.005 23 | 1.005 43 |
| 11                    | 63       | 45       | 31                    | 1.005 52 | 1.005 74 |
| 12                    | 72       | 56       | 32                    | 1.005 82 | 1.006 06 |
| 13                    | 82       | 68       | 33                    | 1.006 13 | 1.006 39 |
| 14                    | 93       | 81       | 34                    | 1.006 44 | 1.006 72 |
| 15                    | 1.002 06 | 1.001 96 | 35                    | 1.006 77 | 1.007 07 |
| 16                    | 20       | 1.002 12 | 36                    | 1.007 10 | 1.007 42 |
| 17                    | 35       | 29       | 37                    | 1.007 44 | 1.007 78 |
| 18                    | 51       | 47       | 38                    | 1.007 79 | 1.008 15 |
| 19                    | 68       | 66       | 39                    | 1.008 15 | 1.008 53 |
|                       |          |          | 40                    | 1.008 52 | 1.008 91 |

**TABLE 11.45** Cubical Coefficients of Thermal Expansion

This table lists values of  $\beta$ , the cubical coefficient of thermal expansion, taken from "Essentials of Quantitative Analysis," by Benedetti-Pichler, and from various other sources. The value of  $\beta$  represents the relative increases in volume for a change in temperature of 1°C at temperatures in the vicinity of 25°C, and is equal to  $3\alpha$ , where  $\alpha$  is the linear coefficient of thermal expansion. Data are given for the types of glass from which volumetric apparatus is most commonly made, and also for some other materials which have been or may be used in the fabrication of apparatus employed in analytical work.

| Material                            | $\beta$               |
|-------------------------------------|-----------------------|
| <b>Glasses</b>                      |                       |
| Alkali-resistant, Corning 728       | $1.90 \times 10^{-5}$ |
| Gerateglas, Schott G20              | 1.47                  |
| Kimble KG-33 (borosilicate)         | 0.96                  |
| N-51A ("Resistant")                 | 1.47                  |
| R-6 (soft)                          | 2.79                  |
| Pyrex, Corning 744                  | 0.96                  |
| Vitreous silica                     | 0.15                  |
| Vycor, Corning 790                  | 0.24                  |
| <b>Metals</b>                       |                       |
| Brass                               | ca. 5.5               |
| Copper                              | 5.0                   |
| Gold                                | 4.3                   |
| Monel metal                         | 4.0                   |
| Platinum                            | 2.7                   |
| Silver                              | 5.7                   |
| Stainless steel                     | ca. 5.3               |
| Tantalum                            | ca. 2.0               |
| Tungsten                            | 1.3                   |
| <b>Plastics and other materials</b> |                       |
| Hard rubber                         | $24 \times 10^{-5}$   |
| Polyethylene                        | 45–90                 |
| Polystyrene                         | 18–24                 |
| Porcelain                           | ca. 1.2               |
| Teflon (polytetrafluoroethylene)    | 16.5                  |

**TABLE 11.46** General Solubility Rules for Inorganic Compounds

|  |   |
|--|---|
| Nitrates                                     | All nitrates are soluble.   |
| Acetates                                     | All acetates are soluble; silver acetate is moderately soluble.   |
| Chlorides                                    | All chlorides are soluble except AgCl, PbCl <sub>2</sub> , and Hg <sub>2</sub> Cl <sub>2</sub> . PbCl <sub>2</sub> is soluble in hot water, slightly soluble in cold water.   |
| Sulfates                                     | All sulfates are soluble except barium and lead. Silver, mercury(I), and calcium are only slightly soluble.   |
| Hydrogen sulfates                            | The hydrogen sulfates are more soluble than the sulfates.   |
| Carbonates, phosphates, chromates, silicates | All carbonates, phosphates, chromates, and silicates are insoluble, except those of sodium, potassium, and ammonium. An exception is MgCrO <sub>4</sub> which is soluble.   |
| Hydroxides                                   | All hydroxides (except lithium, sodium, potassium, cesium, rubidium, and ammonia) are insoluble; Ba(OH) <sub>2</sub> is moderately soluble; Ca(OH) <sub>2</sub> and Sr(OH) <sub>2</sub> are slightly soluble.   |
| Sulfides                                     | All sulfides (except alkali metals, ammonium, magnesium, calcium, and barium) are insoluble. Aluminum and chromium sulfides are hydrolyzed and precipitate as hydroxides.   |
| Sodium, potassium, ammonium                  | All sodium, potassium, and ammonium salts are soluble. Exceptions: Na <sub>4</sub> Sb <sub>2</sub> O <sub>7</sub> , K <sub>2</sub> NaCo(NO <sub>2</sub> ) <sub>6</sub> , K <sub>2</sub> PtCl <sub>6</sub> , (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> , and (NH <sub>4</sub> ) <sub>2</sub> NaCo(NO <sub>2</sub> ) <sub>6</sub> . |
| Silver                                       | All silver salts are insoluble. Exceptions: AgNO <sub>3</sub> and AgClO <sub>4</sub> ; AgC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> and Ag <sub>2</sub> SO <sub>4</sub> are moderately soluble.   |

## 11.7 LABORATORY SOLUTIONS

**TABLE 11.47** Concentrations of Commonly Used Acids and Bases

Freshly opened bottles of these reagents are generally of the concentrations indicated in the table. This may not be true of bottles long opened and this is especially true of ammonium hydroxide, which rapidly loses its strength. In preparing volumetric solutions, it is well to be on the safe side and take a little more than the calculated volume of the concentrated reagent, since it is much easier to dilute a concentrated solution than to strengthen one that is too weak.

A concentrated C.P. reagent usually comes to the laboratory in a bottle having a label which states its molecular weight  $w$ , its density (or its specific gravity)  $d$ , and its percentage assay  $p$ . When such a reagent is used to prepare an aqueous solution of desired molarity  $M$ , a convenient formula to employ is

$$V = \frac{100 \, wM}{pd}$$

where  $V$  is the number of milliliters of concentrated reagent required for 1 liter of the dilute solution.

*Example:* Sulfuric acid has the molecular weight 98.08. If the concentrated acid assays 95.5% and has the specific gravity 1.84, the volume required for 1 liter of a 0.1 molar solution is

$$V = \frac{100 \times 98.08 \times 0.1}{95.5 \times 1.84} = 5.58 \text{ mL}$$

| Reagent                                   | Formula Weight | Density, $\text{g} \cdot \text{mL}^{-1}$ (20°C) | Weight % (approx) | Molarity | V, mL* |
|---|----------------|---|-------------------|----------|--------|
| Acetic acid                               | 60.05          | 1.05  | 99.8              | 17.45    | 57.3   |
| Ammonium hydroxide<br>(as $\text{NH}_3$ ) | 35.05<br>17.03 | 0.90  | 56.6<br>28.0      | 14.53    | 60.0   |
| Ethylenediamine                           | 60.10          | 0.899   | 100               | 15.0     | 66.7   |
| Formic acid                               | 46.03          | 1.20  | 90.5              | 23.6     | 42.5   |
| Hydrazine                                 | 32.05          | 1.011   | 95                | 30.0     | 33.3   |
| Hydriodic acid                            | 127.91         | 1.70  | 57                | 7.6      | 132    |
| Hydrobromic acid                          | 80.92          | 1.49  | 48                | 8.84     | 113    |
| Hydrochloric acid                         | 36.46          | 1.19  | 37.2              | 12.1     | 82.5   |
| Hydrofluoric acid                         | 20.0           | 1.18  | 49.0              | 28.9     | 34.5   |
| Nitric acid                               | 63.01          | 1.42  | 70.4              | 15.9     | 63.0   |
| Perchloric acid                           | 100.47         | 1.67  | 70.5              | 11.7     | 85.5   |
| Phosphoric acid                           | 97.10          | 1.70  | 85.5              | 14.8     | 67.5   |
| Pyridine                                  | 79.10          | 0.982   | 100               | 12.4     | 80.6   |
| Potassium hydroxide (soln)                | 56.11          | 1.46  | 45                | 11.7     | 85.5   |
| Sodium hydroxide (soln)                   | 40.00          | 1.54  | 50.5              | 19.4     | 51.5   |
| Sulfuric acid                             | 98.08          | 1.84  | 96.0              | 18.0     | 55.8   |
| Triethanolamine                           | 149.19         | 1.124   | 100               | 7.53     | 132.7  |

\* V, mL = volume in milliliters needed to prepare 1 liter of 1 molar solution.

TABLE 11.48 Standard Stock Solutions\*

| Element    | Procedure   |
|------------|---|
| Aluminum   | Dissolve 1.000 g Al wire in minimum amount of 2 M HCl; dilute to volume.  |
| Antimony   | Dissolve 1.000 g Sb in (1) 10 ml HNO <sub>3</sub> plus 5 ml HCl, and dilute to volume when dissolution is complete; or (2) 18 ml HBr plus 2 ml liquid Br <sub>2</sub> ; when dissolution is complete add 10 ml HClO <sub>4</sub> , heat in a well-ventilated hood while swirling until white fumes appear and continue for several minutes to expel all HBr, then cool and dilute to volume.  |
| Arsenic    | Dissolve 1.3203 g of As <sub>2</sub> O <sub>3</sub> in 3 ml 8 M HCl and dilute to volume; or treat the oxide with 2 g NaOH and 20 ml water; after dissolution dilute to 200 ml, neutralize with HCl (pH meter), and dilute to volume.   |
| Barium     | (1) Dissolve 1.7787 g BaCl <sub>2</sub> · 2H <sub>2</sub> O (fresh crystals) in water and dilute to volume. (2) Dissolve 1.516 g BaCl <sub>2</sub> (dried at 250°C for 2 hr) in water and dilute to volume. (3) Treat 1.4367 g BaCO <sub>3</sub> with 300 ml water, slowly add 10 ml of HCl and, after the CO <sub>2</sub> is released by swirling, dilute to volume.   |
| Beryllium  | (1) Dissolve 19.655 g BeSO <sub>4</sub> · 4H <sub>2</sub> O in water, add 5 ml HCl (or HNO <sub>3</sub> ), and dilute to volume. (2) Dissolve 1.000 g Be in 25 ml 2 M HCl, then dilute to volume.   |
| Bismuth    | Dissolve 1.000 g Bi in 8 ml of 10 M HNO <sub>3</sub> , boil gently to expel brown fumes, and dilute to volume.  |
| Boron      | Dissolve 5.720 g fresh crystals of H <sub>3</sub> BO <sub>3</sub> and dilute to volume.   |
| Bromine    | Dissolve 1.489 g KBr (or 1.288 g NaBr) in water and dilute to volume.   |
| Cadmium    | (1) Dissolve 1.000 g Cd in 10 ml of 2 M HCl; dilute to volume. (2) Dissolve 2.282 g 3CdSO <sub>4</sub> · 8H <sub>2</sub> O in water; dilute to volume.  |
| Calcium    | Place 2.4973 g CaCO <sub>3</sub> in volumetric flask with 300 ml water, carefully add 10 ml HCl; after CO <sub>2</sub> is released by swirling, dilute to volume.   |
| Cerium     | (1) Dissolve 4.515 g (NH <sub>4</sub> ) <sub>4</sub> Ce(SO <sub>4</sub> ) <sub>4</sub> · 2H <sub>2</sub> O in 500 ml water to which 30 ml H <sub>2</sub> SO <sub>4</sub> had been added, cool, and dilute to volume. Advisable to standardize against As <sub>2</sub> O <sub>3</sub> . (2) Dissolve 3.913 g (NH <sub>4</sub> ) <sub>2</sub> Ce(NO <sub>3</sub> ) <sub>6</sub> in 10 ml H <sub>2</sub> SO <sub>4</sub> , stir 2 min, cautiously introduce 15 ml water and again stir 2 min. Repeat addition of water and stirring until all the salt has dissolved, then dilute to volume. |
| Cesium     | Dissolve 1.267 g CsCl and dilute to volume. Standardize: Pipette 25 ml of final solution to Pt dish, add 1 drop H <sub>2</sub> SO <sub>4</sub> , evaporate to dryness, and heat to constant weight at > 800°C. Cs (in µg/ml) = (40)(0.734)(wt of residue)   |
| Chlorine   | Dissolve 1.648 g NaCl and dilute to volume.   |
| Chromium   | (1) Dissolve 2.829 g K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> in water and dilute to volume. (2) Dissolve 1.000 g Cr in 10 ml HCl, and dilute to volume.   |
| Cobalt     | Dissolve 1.000 g Co in 10 ml of 2 M HCl, and dilute to volume.  |
| Copper     | (1) Dissolve 3.929 g fresh crystals of CuSO <sub>4</sub> · 5H <sub>2</sub> O, and dilute to volume. (2) Dissolve 1.000 g Cu in 10 ml HCl plus 5 ml water to which HNO <sub>3</sub> (or 30% H <sub>2</sub> O <sub>2</sub> ) is added dropwise until dissolution is complete. Boil to expel oxides of nitrogen and chlorine, then dilute to volume.   |
| Dysprosium | Dissolve 1.1477 g Dy <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl; dilute to volume.   |
| Erbium     | Dissolve 1.1436 g Er <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl; dilute to volume.   |
| Europium   | Dissolve 1.1579 g Eu <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl; dilute to volume.   |
| Fluorine   | Dissolve 2.210 g NaF in water and dilute to volume.   |
| Gadolinium | Dissolve 1.152 g Gd <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl; dilute to volume.  |
| Gallium    | Dissolve 1.000 g Ga in 50 ml of 2 M HCl; dilute to volume.  |
| Germanium  | Dissolve 1.4408 g GeO <sub>2</sub> with 50 g oxalic acid in 100 ml of water; dilute to volume.  |

\* 1000 µg/mL as the element in a final volume of 1 liter unless stated otherwise.

From J. A. Dean and T. C. Rains, "Standard Solutions for Flame Spectrometry," in *Flame Emission and Atomic Absorption Spectrometry*, J. A. Dean and T. C. Rains (Eds.), Vol. 2, Chap. 13, Marcel Dekker, New York, 1971.

**TABLE 11.48** Standard Stock Solutions (*Continued*)

| Element      | Procedure  |
|--------------|--|
| Gold         | Dissolve 1.000 g Au in 10 ml of hot $\text{HNO}_3$ by dropwise addition of HCl, boil to expel oxides of nitrogen and chlorine, and dilute to volume. Store in amber container away from light.   |
| Hafnium      | Transfer 1.000 g Hf to Pt dish, add 10 ml of 9 M $\text{H}_2\text{SO}_4$ , and then slowly add HF dropwise until dissolution is complete. Dilute to volume with 10% $\text{H}_2\text{SO}_4$ .  |
| Holmium      | Dissolve 1.1455 g $\text{Ho}_2\text{O}_3$ in 50 ml of 2 M HCl; dilute to volume.   |
| Indium       | Dissolve 1.000 g In in 50 ml of 2 M HCl; dilute to volume.   |
| Iodine       | Dissolve 1.308 g KI in water and dilute to volume.   |
| Iridium      | (1) Dissolve 2.465 g $\text{Na}_3\text{IrCl}_6$ in water and dilute to volume. (2) Transfer 1.000 g Ir sponge to a glass tube, add 20 ml of HCl and 1 ml of $\text{HClO}_4$ . Seal the tube and place in an oven at $300^\circ\text{C}$ for 24 hr. Cool, break open the tube, transfer the solution to a volumetric flask, and dilute to volume. Observe all safety precautions in opening the glass tube. |
| Iron         | Dissolve 1.000 g Fe wire in 20 ml of 5 M HCl; dilute to volume.  |
| Lanthanum    | Dissolve 1.1717 g $\text{La}_2\text{O}_3$ (dried at $110^\circ\text{C}$ ) in 50 ml of 5 M HCl, and dilute to volume.   |
| Lead         | (1) Dissolve 1.5985 g $\text{Pb}(\text{NO}_3)_2$ in water plus 10 ml $\text{HNO}_3$ , and dilute to volume. (2) Dissolve 1.000 g Pb in 10 ml $\text{HNO}_3$ , and dilute to volume.  |
| Lithium      | Dissolve a slurry of 5.3228 g $\text{Li}_2\text{CO}_3$ in 300 ml of water by addition of 15 ml HCl; after release of $\text{CO}_2$ by swirling, dilute to volume.  |
| Lutetium     | Dissolve 1.6079 g $\text{LuCl}_3$ in water and dilute to volume.   |
| Magnesium    | Dissolve 1.000 g Mg in 50 ml of 1 M HCl and dilute to volume.  |
| Manganese    | (1) Dissolve 1.000 g Mn in 10 ml HCl plus 1 ml $\text{HNO}_3$ , and dilute to volume. (2) Dissolve 3.0764 g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ (dried at $105^\circ\text{C}$ for 4 hr) in water and dilute to volume. (3) Dissolve 1.5824 g $\text{MnO}_2$ in 10 HCl in a good hood, evaporate to gentle dryness, dissolve residue in water and dilute to volume.                                    |
| Mercury      | Dissolve 1.000 g Hg in 10 ml of 5 M $\text{HNO}_3$ and dilute to volume.   |
| Molybdenum   | (1) Dissolve 2.0425 g $(\text{NH}_4)_2\text{MoO}_4$ in water and dilute to volume. (2) Dissolve 1.5003 g $\text{MoO}_3$ in 100 ml of 2 M ammonia, and dilute to volume.  |
| Neodymium    | Dissolve 1.7373 g $\text{NdCl}_3$ in 100 ml 1 M HCl and dilute to volume.  |
| Nickel       | Dissolve 1.000 g Ni in 10 ml hot $\text{HNO}_3$ , cool, and dilute to volume.  |
| Niobium      | Transfer 1.000 g Nb (or 1.4305 g $\text{Nb}_2\text{O}_5$ ) to Pt dish, add 20 ml HF, and heat gently to complete dissolution. Cool, add 40 ml $\text{H}_2\text{SO}_4$ , and evaporate to fumes of $\text{SO}_3$ . Cool and dilute to volume with 8 M $\text{H}_2\text{SO}_4$ .   |
| Osmium       | Dissolve 1.3360 g $\text{OsO}_4$ in water and dilute to 100 ml. Prepare only as needed as solution loses strength on standing unless Os is reduced by $\text{SO}_2$ and water is replaced by 100 ml 0.1 M HCl.   |
| Palladium    | Dissolve 1.000 g Pd in 10 ml of $\text{HNO}_3$ by dropwise addition of HCl to hot solution; dilute to volume.  |
| Phosphorus   | Dissolve 4.260 g $(\text{NH}_4)_2\text{HPO}_4$ in water and dilute to volume.  |
| Platinum     | Dissolve 1.000 g Pt in 40 ml of hot aqua regia, evaporate to incipient dryness, add 10 ml HCl and again evaporate to moist residue. Add 10 ml HCl and dilute to volume.  |
| Potassium    | Dissolve 1.9067 g KCl (or 2.8415 g $\text{KNO}_3$ ) in water and dilute to volume.   |
| Praseodymium | Dissolve 1.1703 g $\text{Pr}_2\text{O}_3$ in 50 ml of 2 M HCl; dilute to volume.   |
| Rhenium      | Dissolve 1.000 g Re in 10 ml of 8 M $\text{HNO}_3$ in an ice bath until initial reaction subsides, then dilute to volume.  |
| Rhodium      | Dissolve 1.000 g Rh by the sealed-tube method described under iridium.   |
| Rubidium     | Dissolve 1.4148 g RbCl in water. Standardize as described under cesium. Rb (in $\mu\text{g/ml}$ ) = $(40)(0.320)(\text{wt of residue})$ .  |
| Ruthenium    | Dissolve 1.317 g $\text{RuO}_2$ in 15 ml of HCl; dilute to volume.   |
| Samarium     | Dissolve 1.1596 g $\text{Sm}_2\text{O}_3$ in 50 ml of 2 M HCl; dilute to volume.   |
| Scandium     | Dissolve 1.5338 g $\text{Sc}_2\text{O}_3$ in 50 ml of 2 M HCl; dilute to volume.   |

**TABLE 11.48** Standard Stock Solutions (*Continued*)

| Element   | Procedure   |
|-----------|---|
| Selenium  | Dissolve 1.4050 g SeO <sub>2</sub> in water and dilute to volume or dissolve 1.000 g Se in 5 ml of HNO <sub>3</sub> , then dilute to volume.  |
| Silicon   | Fuse 2.1393 g SiO <sub>2</sub> with 4.60 g Na <sub>2</sub> CO <sub>3</sub> , maintaining melt for 15 min in Pt crucible. Cool, dissolve in warm water, and dilute to volume. Solution contains also 2000 µg/ml sodium.  |
| Silver    | (1) Dissolve 1.5748 g AgNO <sub>3</sub> in water and dilute to volume. (2) Dissolve 1.000 g Ag in 10 ml of HNO <sub>3</sub> ; dilute to volume. Store in amber glass container away from light.   |
| Sodium    | Dissolve 2.5421 g NaCl in water and dilute to volume.   |
| Strontium | Dissolve a slurry of 1.6849 g SrCO <sub>3</sub> in 300 ml of water by careful addition of 10 ml of HCl; after release of CO <sub>2</sub> by swirling, dilute to volume.   |
| Sulfur    | Dissolve 4.122 g (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> in water and dilute to volume.   |
| Tantalum  | Transfer 1.000 g Ta (or 1.2210 g Ta <sub>2</sub> O <sub>5</sub> ) to Pt dish, add 20 ml of HF, and heat gently to complete the dissolution. Cool, add 40 ml of H <sub>2</sub> SO <sub>4</sub> and evaporate to heavy fumes of SO <sub>3</sub> . Cool and dilute to volume with 50% H <sub>2</sub> SO <sub>4</sub> . |
| Tellurium | (1) Dissolve 1.2508 g TeO <sub>2</sub> in 10 ml of HCl; dilute to volume. (2) Dissolve 1.000 g Te in 10 ml of warm HCl with dropwise addition of HNO <sub>3</sub> , then dilute to volume.  |
| Terbium   | Dissolve 1.6692 g of TbCl <sub>3</sub> in water, add 1 ml of HCl, and dilute to volume.   |
| Thallium  | Dissolve 1.3034 g TlNO <sub>3</sub> in water and dilute to volume.  |
| Thorium   | Dissolve 2.3794 g Th(NO <sub>3</sub> ) <sub>4</sub> · 4H <sub>2</sub> O in water, add 5 ml HNO <sub>3</sub> , and dilute to volume.   |
| Thulium   | Dissolve 1.142 g Tm <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl; dilute to volume.  |
| Tin       | Dissolve 1.000 g Sn in 15 ml of warm HCl; dilute to volume.   |
| Titanium  | Dissolve 1.000 g Ti in 10 ml of H <sub>2</sub> SO <sub>4</sub> with dropwise addition of HNO <sub>3</sub> ; dilute to volume with 5% H <sub>2</sub> SO <sub>4</sub> .   |
| Tungsten  | Dissolve 1.7941 g of Na <sub>2</sub> WO <sub>4</sub> · 2H <sub>2</sub> O in water and dilute to volume.   |
| Uranium   | Dissolve 2.1095 g UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O (or 1.7734 g uranyl acetate dihydrate) in water and dilute to volume.   |
| Vanadium  | Dissolve 2.2963 g NH <sub>4</sub> VO <sub>3</sub> in 100 ml of water plus 10 ml of HNO <sub>3</sub> ; dilute to volume.   |
| Ytterbium | Dissolve 1.6147 g YbCl <sub>3</sub> in water and dilute to volume.  |
| Yttrium   | Dissolve 1.2692 g Y <sub>2</sub> O <sub>3</sub> in 50 ml of 2 M HCl and dilute to volume.   |
| Zinc      | Dissolve 1.000 g Zn in 10 ml of HCl; dilute to volume.  |
| Zirconium | Dissolve 3.533 g ZrOCl <sub>2</sub> · 8H <sub>2</sub> O in 50 ml of 2 M HCl, and dilute to volume. Solution should be standardized.   |

### 11.7.1 General Reagents, Indicators, and Special Solutions

Unless otherwise stated, the term *g per liter* signifies grams of the formula indicated dissolved in water and made up to a liter of solution.

**Acetic acid**, HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>—6*N*: 350 mL glacial acetic acid per liter.

**Alcohol, amyl**, C<sub>5</sub>H<sub>11</sub>OH: use as purchased.

**Alcohol, ethyl**, C<sub>2</sub>H<sub>5</sub>OH; 95% alcohol, as purchased.

**Alizarin**, dihydroxyanthraquinone (indicator): dissolve 0.1 g in 100 mL alcohol; pH range yellow 5.5–6.8 red.

**Alizarin yellow R**, sodium *p*-nitrobenzeneazosalicylate (indicator): dissolve 0.1 g in 100 mL water; pH range yellow 10.1–violet 12.1.

**Alizarin yellow GG**, salicyl yellow, sodium *m*-nitrobenzeneazosalicylate (indicator): dissolve 0.1 g in 100 mL 50% alcohol; pH range yellow 10.0–12.0 lilac.

**Alizarin S**, alizarin carmine, sodium alizarin sulfonate (indicator): dissolve 0.1 g in 100 mL water; pH range yellow 3.7–5.2 violet.

**Aluminon** (qualitative test for aluminum). The reagent consists of 0.1% solution of the ammonium salt of aurin tricarboxylic acid. A bright red precipitate, persisting in alkaline solution, indicates aluminum.

**Aluminum chloride**,  $\text{AlCl}_3$ —0.5*N*: 22 g per liter.

**Aluminum nitrate**,  $\text{Al}(\text{NO}_3)_3 \cdot 7.5\text{H}_2\text{O}$ —0.5*N*: 58 g per liter.

**Aluminum sulfate**,  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ —0.5*N*: 55 g per liter.

**Ammonium acetate**,  $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ —3*N*: 231 g per liter.

**Ammonium carbonate**,  $(\text{NH}_4)_2\text{CO}_3 \cdot \text{H}_2\text{O}$ —3*N*: 171 g per liter; for the anhydrous salt: 144 g per liter.

**Ammonium chloride**,  $\text{NH}_4\text{Cl}$ —3*N*: 161 g per liter.

**Ammonium hydroxide**,  $\text{NH}_4\text{OH}$ —15*N*: the concentrated solution which contains 28%  $\text{NH}_3$ ; for 6*N*: 400 mL per liter.

**Ammonium molybdate**,  $(\text{NH}_4)_2\text{MoO}_4$ —*N*: dissolve 88.3 g of solid  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$  in 100 mL 6*N*  $\text{NH}_4\text{OH}$ . Add 240 g of solid  $\text{NH}_4\text{NO}_3$  and dilute to 1 liter. Another method is to take 72 g of  $\text{MoO}_3$ , add 130 mL of water and 75 mL of 15*N*  $\text{NH}_4\text{OH}$ ; stir mechanically until nearly all has dissolved, then add it to a solution of 240 mL concentrated  $\text{HNO}_3$  and 500 mL of water; stir continuously while solutions are being mixed; allow to stand 3 days, filter, and use the clear filtrate.

**Ammonium nitrate**,  $\text{NH}_4\text{NO}_3$ —*N*: 80 g per liter.

**Ammonium oxalate**,  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ —0.5*N*: 40 g per liter.

**Ammonium polysulfide** (yellow ammonium sulfide),  $(\text{NH}_4)_2\text{S}_x$ : allow the colorless  $(\text{NH}_4)_2\text{S}$  to stand, or add sulfur.

**Ammonium sulfate**,  $(\text{NH}_4)_2\text{SO}_4$ —0.5*N*: 33 g per liter; saturated: dissolve 780 g of  $(\text{NH}_4)_2\text{SO}_4$  in water and make up to a liter.

**Ammonium sulfide** (colorless),  $(\text{NH}_4)_2\text{S}$ —saturated: pass  $\text{H}_2\text{S}$  through 200 mL of concentrated  $\text{NH}_4\text{OH}$  in the cold until no more gas is dissolved, add 200 mL  $\text{NH}_4\text{OH}$  and dilute with water to a liter; the addition of 15 g of sulfur is sufficient to make the polysulfide.

**Antimony pentachloride**,  $\text{SbCl}_5$ —0.5*N*: 39 g per liter.

**Antimony trichloride**,  $\text{SbCl}_3$ —0.5*N*: 38 g per liter.

**Aqua regia**: mix 3 parts of concentrated  $\text{HCl}$  and 1 part of concentrated  $\text{HNO}_3$  just before ready to use.

**Arsenic acid**,  $\text{H}_3\text{AsO}_4 \cdot 0.5\text{H}_2\text{O}$ —0.5*N* ( $= \frac{1}{2}\text{H}_3\text{AsO}_4 \div 5$ ): 15 g per liter.

**Arsenous oxide**,  $\text{As}_2\text{O}_3$ —0.25*N*: 8 g per liter for saturation.

**Aurichloric acid**,  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ : dissolve in ten parts of water.

**Aurin**, *see* rosolic acid.

**Azolitmin solution** (indicator); make up a 1% solution of azolitmin by boiling in water for 5 minutes; it may be necessary to add a small amount of  $\text{NaOH}$  to make the solution neutral; pH range red 4.5–8.3 blue.

**Bang's reagent** (for glucose estimation): dissolve 100 g of  $\text{K}_2\text{CO}_3$ , 66 g of KCl, and 160 of  $\text{KHCO}_3$  in the order given in about 700 mL of water at  $30^\circ\text{C}$ . Add 4.4 g of copper sulfate and dilute to 1 liter after the  $\text{CO}_2$  is evolved. This solution should be shaken only in such a manner as not to allow the entry of air. After 24 hours 300 mL diluted to a liter with saturated KCl solution, shaken gently and used after 24 hours; 50 mL  $\equiv$  10 mg glucose.

**Barfoed's reagent** (test for glucose): dissolve 66 g of cupric acetate and 10 mL of glacial acetic acid in water and dilute to 1 liter.

**Barium chloride**,  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ —0.5*N*: 61 g per liter.

**Barium hydroxide**,  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ —0.2*N*: 32 g per liter for saturation.

**Barium nitrate**,  $\text{Ba}(\text{NO}_3)_2$ —0.5*N*: 65 g per liter.

**Baudisch's reagent**: *see* cupferron.

**Benedict's qualitative reagent** (for glucose): dissolve 173 g of sodium citrate and 100 g of anhydrous sodium carbonate in about 600 mL of water, and dilute to 850 mL; dissolve 17.3 g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in 100 mL of water and dilute to 150 mL; this solution is added to the citrate-carbonate solution with constant stirring. *See also* the quantitative reagent below.

**Benedict's quantitative reagent** (sugar in urine): This solution contains 18 g copper sulfate, 100 g of anhydrous sodium carbonate, 200 g of potassium citrate, 125 g of potassium thiocyanate, and 0.25 g of potassium ferrocyanide per liter; 1 mL of this solution  $\equiv$  0.002 g sugar.

**Benzidine hydrochloride solution** (for sulfate determination): mix 6.7 g of benzidine [ $\text{C}_{12}\text{H}_8(\text{NH}_2)_2$ ] or 8.0 g of the hydrochloride [ $\text{C}_{12}\text{H}_8(\text{NH}_2)_2 \cdot 2\text{HCl}$ ] into a paste with 20 mL of water; add 20 mL of HCl (sp. gr. 1.12) and dilute the mixture to 1 liter with water; each mL of this solution is equivalent to 0.00357 g  $\text{H}_2\text{SO}_4$ .

**Benzopurpurine 4B** (indicator): dissolve 0.1 g in 100 mL water; pH range blue-violet 1.3–4.0 red.

**Benzoyl auramine** (indicator): dissolve 0.25 g in 100 mL methyl alcohol; pH range violet 5.0–5.6 pale yellow. Since this compound is not stable in aqueous solution, hydrolyzing slowly in neutral medium, more rapidly in alkaline, and still more rapidly in acid solution, the indicator should not be added until one is ready to titrate. The acid quinoid form of the compound is dichroic, showing a red-violet in thick layers and blue in thin. At a pH of 5.4 the indicator appears a neutral gray color by daylight or a pale red under tungsten light. The change to yellow is easily recognized in either case. Cf. Scanlan and Reid, *Ind. Eng. Chem., Anal. Ed.* **7**:125 (1935).

**Bertrand's reagents** (glucose estimation): (a) 40 g of copper sulfate diluted to 1 liter; (b) rochelle salt 200 g, NaOH 150 g, and sufficient water to make 1 liter; (c) ferric sulfate 50 g,  $\text{H}_2\text{SO}_4$  200 g, and sufficient water to make 1 liter; (d)  $\text{KMnO}_4$  5 g and sufficient water to make 1 liter.

**Bial's reagent** (for pentoses): dissolve 1 g of orcinol in 500 mL of 30% HCl to which 30 drops of a 10% ferric chloride solution have been added.

**Bismuth chloride**,  $\text{BiCl}_3$ —0.5*N*: 52 g per liter, using 1 : 5 HCl in place of water.

**Bismuth nitrate**,  $\text{Bi}(\text{N}_2\text{O}_3)_3 \cdot 5\text{H}_2\text{O}$ —0.25*N*: 40 g per liter, using 1 : 5  $\text{HNO}_3$  in place of water.

**Bismuth standard solution** (quantitative color test for Bi): dissolve 1 g of bismuth in a mixture of 3 mL of concentrated  $\text{HNO}_3$  and 2.8 mL of  $\text{H}_2\text{O}$  and make up to 100 mL with glycerol. Also dissolve 5 g of KI in 5 mL of water and make up to 100 mL with glycerol. The two solutions are used together in the colorimetric estimation of Bi.

**Boutron-Boudet solution**: *see* soap solution.

**Bromchlorophenol blue**, dibromodichlorophenol-sulfonphthalein (indicator): dissolve 0.1 g in 8.6 mL 0.02 *N* NaOH and dilute with water to 250 mL; pH range yellow 3.2–4.8 blue.

**Bromcresol green**, tetrabromo-*m*-cresol-sulfonphthalein (indicator): dissolve 0.1 g in 7.15 mL 0.02 *N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 4.0–5.6 blue.

**Bromcresol purple**, dibromo-*o*-cresol-sulfonphthalein (indicator): dissolve 0.1 g in 9.5 mL 0.02 *N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 5.2–6.8 purple.

**Bromine water**, saturated solution: to 400 mL water add 20 mL of bromine; use a glass stopper coated with petrolatum.

**Bromphenol blue**, tetrabromophenol-sulfonphthalein (indicator): dissolve 0.1 g in 7.45 mL 0.02 *N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 3.6–4.6 violet-blue.

**Bromphenol red**, dibromophenol-sulfonphthalein (indicator): dissolve 0.1 g in 9.75 mL 0.02 *N* NaOH and dilute with water to 250 mL; pH range yellow 5.2–7.0 red.

**Bromthymol blue**, dibromothymol-sulfonphthalein (indicator): dissolve 0.1 g in 8.0 mL 0.02 *N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL of 20% alcohol; pH range yellow 6.0–7.6 blue.

**Brucke's reagent** (protein precipitant): dissolve 50 g of KI in 500 mL of water, saturate with  $\text{HgI}_2$  (about 120 g), and dilute to 1 liter.

**Cadmium chloride**,  $\text{CdCl}_2$ —0.5*N*: 46 g per liter.

**Cadmium nitrate**,  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ —0.5*N*: 77 g per liter.

**Cadmium sulfate**,  $\text{CdSO}_4 \cdot 4\text{H}_2\text{O}$ —0.5*N*: 70 g per liter.

**Calcium chloride**,  $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 55 g per liter.

**Calcium hydroxide**,  $\text{Ca}(\text{OH})_2$ —0.04*N*: 10 g per liter for saturation.

**Calcium nitrate**,  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ —0.5*N*: 59 g per liter.

**Calcium sulfate**,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ —0.03*N*: mechanically stir 10 g in a liter of water for 3 hours; decant and use the clear liquid.

**Carbon disulfide**,  $\text{CS}_2$ : commercial grade which is colorless.

**Chloride reagent**: dissolve 1.7 g of  $\text{AgNO}_3$  and 25 g  $\text{KNO}_3$  in water, add 17 mL of concentrated  $\text{NH}_4\text{OH}$  and make up to 1 liter with water.

**Chlorine water**, saturated solution: pass chlorine gas into small amounts of water as needed; solutions deteriorate on standing.

**Chloroform**,  $\text{CHCl}_3$ : commercial grade.

**Chloroplatinic acid**,  $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ —10% solution: dissolve 1 g in 9 mL of water; keep in a dropping bottle.

**Chlorphenol red**, dichlorophenol-sulfonphthalein (indicator): dissolve 0.1 g in 11.8 mL 0.02 *N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 5.2–6.6 red.

**Chromic chloride**,  $\text{CrCl}_3$ —0.5*N*: 26 g per liter.

**Chromic nitrate**,  $\text{Cr}(\text{NO}_3)_3$ —0.5*N*: 40 g per liter.

**Chromic sulfate**,  $\text{Cr}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ —0.5*N*: 60 g per liter.

**Cobaltous nitrate**,  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 73 g per liter.

**Cobaltous sulfate**,  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 70 g per liter.

**Cochineal** (indicator): triturate 1 g with 75 mL alcohol and 75 mL water, let stand for two days and filter; pH range red 4.8–6.2 violet.

**Congo red**, sodium tetrazodiphenyl-naphthionate (indicator): dissolve 0.1 g in 100 mL water; pH range blue 3.0–5.2 red.

**Corallin** (indicator): *see* rosolic acid.

**Cresol red**, *o*-cresol-sulfonphthalein (indicator): dissolve 0.1 g in 13.1 mL 0.02*N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 7.2–8.8 red.

***o*-Cresolphthalein** (indicator): dissolve 0.1 g in 250 mL alcohol; pH range colorless 8.2–10.4 red.

**Cupferron** (iron analysis): dissolve 6 g of ammonium nitrosophenyl-hydroxylamine (cupferron) in water and dilute to 100 mL. This solution is stable for about one week if protected from light.

**Cupric chloride**,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ —0.5*N*: 43 g per liter.

**Cupric nitrate**,  $\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 74 g per liter.

**Cupric sulfate**,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ —0.5*N*: 62 g per liter.

**Cuprous chloride**,  $\text{CuCl}$ —0.5*N*: 50 g per liter, using 1 : 5 HCl in place of water.

**Cuprous chloride**, acid (for gas analysis, absorption of CO): cover the bottom of a 2-liter bottle with a layer of copper oxide  $\frac{3}{8}$  inch deep, and place a bundle of copper wire an inch thick in the bottle so that it extends from the top to the bottom. Fill the bottle with HCl (sp. gr. 1.10). The bottle is shaken occasionally, and when the solution is colorless or nearly so, it is poured into half-liter bottles containing copper wire. The large bottle may be filled with hydrochloric acid, and by adding the oxide or wire when either is exhausted, a constant supply of the reagent is available.

**Cuprous chloride, ammoniacal**: this solution is used for the same purpose and is made in the same manner as the acid cuprous chloride above, except that the acid solution is treated with ammonia until a faint odor of ammonia is perceptible. Copper wire should be kept with the solution as in the acid reagent.

**Curcumin** (indicator): prepare a saturated aqueous solution; pH range yellow 6.0–8.0 brownish red.

**Dibromophenol-tetrabromophenol-sulfonphthalein** (indicator): dissolve 0.1 g in 1.21 mL 0.1*N* NaOH and dilute with water to 250 mL; pH range yellow 5.6–7.2 purple.

**Dimethyl glyoxime**,  $(\text{CH}_3\text{CNOH})_2$ —0.01*N*: 6 g in 500 mL of 95% alcohol.

**2,4-Dinitrophenol** (indicator): dissolve 0.1 g in a few mL alcohol, then dilute with water to 100 mL; pH range colorless 2.6–4.0 yellow.

**2,5-Dinitrophenol** (indicator): dissolve 0.1 g in 20 mL alcohol, then dilute with water to 100 mL; pH range colorless 4–5.8 yellow.

**2,6-Dinitrophenol** (indicator): dissolve 0.1 g in a few mL alcohol, then dilute with water to 100 mL; pH range colorless 2.4–4.0 yellow.

**Esbach's reagent** (estimation of proteins): dissolve 10 g of picric acid and 20 g of citric acid in water and dilute to 1 liter.

**Eschka's mixture** (sulfur in coal): mix 2 parts of porous calcined MgO with 1 part of anhydrous  $\text{Na}_2\text{CO}_3$ ; not a solution but a dry mixture.

**Ether**,  $(\text{C}_2\text{H}_5)_2\text{O}$ —use commercial grade.

***p*-Ethoxychrysoidine**, *p*-ethoxybenzeneazo-*m*-phenylenediamine (indicator): dissolve 0.1 g of the base in 100 mL 90% alcohol; or, 0.1 g of the hydrochloride salt in 100 mL water; pH range red 3.5–5.5 yellow.

**Ethyl bis-(2,4-dinitrophenyl) acetate** (indicator): the stock solution is prepared by saturating a solution containing equal volumes of alcohol and acetone with the indicator; pH range colorless 7.4–9.1 deep blue. This compound is available commercially. The preparation of this compound is described by Fehnel and Amstutz, *Ind. Eng. Chem., Anal. Ed.* **16**:53 (1944), and by von Richter, *Ber.* **21**:2470 (1888), who recommended it for the titration of orange- and red-colored solutions or dark oils in which the endpoint of phenol-phthalein is not easily visible. The indicator is an orange solid which after crystallization from benzene gives pale yellow crystals melting at 150–153.5°C, uncorrected.

**Fehling's solution** (sugar detection and estimation): (a) Copper sulfate solution: dissolve 34.639 g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in water and dilute to 500 mL. (b) Alkaline tartrate solution: dissolve 173 g of rochelle salts ( $\text{KNaC}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) and 125 g of KOH in water and dilute to 500 mL. Equal volumes of the two solutions are mixed just prior to use. The Methods of the Assoc. of Official Agricultural Chemists give 50 g of NaOH in place of the 125 g KOH.

**Ferric chloride**,  $\text{FeCl}_3$ —0.5*N*: 27 g per liter.

**Ferric nitrate**,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ —0.5*N*: 67 g per liter.

**Ferrous ammonium sulfate**, Mohr's salt,  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 196 g per liter.

**Ferrous sulfate**,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 80 g per liter; add a few drops of  $\text{H}_2\text{SO}_4$ .

**Folin's mixture** (for uric acid): dissolve 500 g of ammonium sulfate, 5 g of uranium acetate, and 6 mL of glacial acetic acid, in 650 mL of water. The volume is about a liter.

**Formal or Formalin**: use the commercial 40% solution of formaldehyde.

**Froehde's reagent** (gives characteristic colorations with certain alkaloids and glycosides): dissolve 0.01 g of sodium molybdate in 1 mL of concentrated  $\text{H}_2\text{SO}_4$ ; use only a freshly prepared solution.

**Gallein** (indicator): dissolve 0.1 g in 100 mL alcohol; pH range light brown-yellow 3.8–6.6 rose.

**Glyoxylic acid solution** (protein detection): cover 10 g of magnesium powder with water and slowly add 250 mL of a saturated oxalic solution, keeping the mixture cool; filter off the magnesium oxalate, acidify the filtrate with acetic acid and make up to a liter with water.

**Guaiaicum tincture**: dissolve 1 g of guaiacum in 100 mL of alcohol.

**Gunzberg's reagent** (detection of HCl in gastric juice): dissolve 4 g of phloroglucinol and 2 g of vanillin in 100 mL of absolute alcohol; use only a freshly prepared solution.

**Hager's reagent** (for alkaloids): this reagent is a saturated solution of picric acid in water.

**Hanus solution** (for determination of iodine number): dissolve 13.2 g of iodine in a liter of glacial acetic acid that will not reduce chromic acid; add sufficient bromine to double the halogen content determined by titration (3 mL is about the right amount). The iodine may be dissolved with the aid of heat, but the solution must be cold when the bromine is added.

**Hematoxylin** (indicator): dissolve 0.5 g in 100 mL alcohol; pH range yellow 5.0–6.0.

**Heptamethoxy red**, 2,4,6,2',4',2'',4''-heptamethoxytriphenyl carbinol (indicator): dissolve 0.1 g in 100 mL alcohol; pH range red 5.0–7.0 colorless.

**Hydriodic acid**, HI—0.5*N*: 64 g per liter.

**Hydrobromic acid**, HBr—0.5*N*: 40 g per liter.

**Hydrochloric acid**, HCl—5*N*: 182 g per liter; sp. gr. 1.084.

**Hydrofluoric acid**,  $\text{H}_2\text{F}_2$ —48% solution: use as purchased, and keep in the special container.

**Hydrogen peroxide**,  $\text{H}_2\text{O}_2$ —3% solution: use as purchased.

**Hydrogen sulfide**,  $\text{H}_2\text{S}$ : prepare a saturated aqueous solution.

**Indicator solutions:** a number of indicator solutions are listed in this section under the names of the indicators; e.g., alizarin, aurin, azolitmin, et al., which follow alphabetically. *See also* various index entries.

**Indigo carmine**, sodium indigodisulfonate (indicator): dissolve 0.25 g in 100 mL 50% alcohol; pH range blue 11.6–14.0 yellow.

**Indo-oxine**, 5,8-quinolinequinone-8-hydroxy-5-quinoyl-5-imide (indicator): dissolve 0.05 g in 100 mL alcohol; pH range red 6.0–8.0 blue. Cf. Berg and Becker, *Z. Anal. Chem.* **119**:81 (1940).

**Iodeosin**, tetraiodofluorescein (indicator): dissolve 0.1 g in 100 mL ether saturated with water; pH range yellow 0—about 4 rose-red; *see also* under methyl orange.

**Iodic acid**,  $\text{HIO}_3$ —0.5*N* ( $\text{HIO}_3/12$ ): 15 g per liter.

**Iodine:** *see* tincture of iodine.

**Lacmoid** (indicator): dissolve 0.5 g in 100 mL alcohol; pH range red 4.4–6.2 blue.

**Lead acetate**,  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ —0.5*N*: 95 g per liter.

**Lead chloride**,  $\text{PbCl}_2$ —saturated solution is 1/7*N*.

**Lead nitrate**,  $\text{Pb}(\text{NO}_3)_2$ —0.5*N*: 83 g per liter.

**Lime water:** *see* calcium hydroxide.

**Litmus** (indicator): powder the litmus and make up a 2% solution in water by boiling for 5 minutes; pH range red 4.5–8.3 blue.

**Magnesia mixture:** 100 g of  $\text{MgSO}_4$ , 200 g of  $\text{NH}_4\text{Cl}$ , 400 mL of  $\text{NH}_4\text{Cl}$ , 800 mL of water; each mL  $\equiv$  0.01 g phosphorus (P).

**Magnesium chloride**,  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 50 g per liter.

**Magnesium nitrate**,  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 64 g per liter.

**Magnesium sulfate**, epsom salts,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 62 g per liter; saturated solution dissolve 600 g of the salt in water and dilute to 1 liter.

**Manganous chloride**,  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ —0.5*N*: 50 g per liter.

**Manganous nitrate**,  $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 72 g per liter.

**Manganous sulfate**,  $\text{MnSO}_4 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 69 g per liter.

**Marme's reagent** (gives yellowish-white precipitate with salts of alkaloids): saturate a boiling solution of 4 parts of KI in 12 parts of water with  $\text{CdI}_2$ ; then add an equal volume of cold saturated KI solution.

**Marquis reagent** (gives a purple-red coloration, then violet, then blue with morphine, codeine, dionine, and heroine): mix 3 mL of concentrated  $\text{H}_2\text{SO}_4$  with 3 drops of a 35% formaldehyde solution.

**Mayer's reagent** (gives white precipitate with most alkaloids in a slightly acid solution): dissolve 13.55 g of  $\text{HgCl}_2$  and 50 g of KI in a liter of water.

**Mercuric chloride**,  $\text{HgCl}_2$ —0.5*N*: 68 g per liter.

**Mercuric nitrate**,  $\text{Hg}(\text{NO}_3)_2$ —0.5*N*: 81 g per liter.

**Mercuric sulfate**,  $\text{HgSO}_4$ —0.5*N*: 74 g per liter.

**Mercurous nitrate**,  $\text{HgNO}_3$ : mix 1 part of  $\text{HgNO}_3$ , 20 parts of  $\text{H}_2\text{O}$ , and 1 part of  $\text{HNO}_3$ .

**Metacresol purple**, *m*-cresol-sulfonphthalein (indicator): dissolve 0.1 g in 13.6 mL 0.02*N* NaOH and dilute with water to 250 mL; acid pH range red 0.5–2.5 yellow, alkaline pH range yellow 7.4–9.0 purple.

**Metanil yellow**, diphenylaminoazo-*m*-benzene sulfonic acid (indicator): dissolve 0.25 g in 100 mL alcohol; pH range red 1.2–2.3 yellow.

**Methyl green**, hexamethylparosaniline hydroxymethylate (component of mixed indicator): dissolve 0.1 g in 100 mL alcohol; when used with equal parts of hexamethoxytriphenyl carbinol gives color change from violet to green at a titration exponent (pI) of 4.0.

**Methyl orange**, orange III, tropeolin D, sodium *p*-dimethylaminoazobenzenesulfonate (indicator): dissolve 0.1 g in 100 mL water; pH range red 3.0–4.4 orange-yellow. If during a titration where methyl yellow is being used a precipitate forms which tends to remove the indicator from the aqueous phase, methyl orange will be found to be a more suitable indicator. This occurs, for example, in titrations of soaps with acids. The fatty acids, liberated by the titration, extract the methyl yellow so that the endpoint cannot be perceived. Likewise methyl orange is more suitable for titrations in the presence of immiscible organic solvents such as carbon tetrachloride or ether used in the extraction of alkaloids for analysis. Iodeosin (*q.v.*) has also been proposed as an indicator for such cases. Cf. Mylius and Foerster, *Ber.* **24**:1482 (1891); *Z. Anal. Chem.* **31**:240 (1892).

**Methyl red**, *p*-dimethylaminoazobenzene-*o*'-carboxylic acid (indicator): dissolve 0.1 g in 18.6 mL of 0.02*N* NaOH and dilute with water to 250 mL; or, 0.1 g in 60% alcohol; pH range red 4.4–6.2 yellow.

**Methyl violet** (indicator): dissolve 0.25 g in 100 mL water, pH range blue 1.5–3.2 violet.

**Methyl yellow**, *p*-dimethylaminoazobenzene, benzeneazodimethylaniline (indicator): dissolve 0.1 g in 200 mL alcohol; pH range red 2.9–4.0 yellow. The color change from yellow to orange can be perceived somewhat more sharply than the change of methyl orange from orange to rose, so that methyl yellow seems to deserve preference in many cases. *See also* under methyl orange.

**Methylene blue**, *N,N,N',N'*-tetramethylthionine (component of mixed indicator): dissolve 0.1 g in 100 mL alcohol; when used with equal part of methyl yellow gives color change from blue-violet to green at a titration exponent (pI) of 3.25; when used with equal part of 0.2% methyl red in alcohol gives color change from red-violet to green at a titration exponent (pI) of 5.4; when used with an equal part of neutral red gives color change from violet-blue to green at a titration exponent (pI) of 7.0.

**Millon's reagent** (gives a red precipitate with certain proteins and with various phenols): dissolve 1 part of mercury in 1 part of  $\text{HNO}_3$  (sp. gr. 1.40) with gentle heating, then add 2 parts of water; a few crystals of  $\text{KNO}_3$  help to maintain the strength of the reagent.

**Mohr's salt**: *see* ferrous ammonium sulfate.

**$\alpha$ -Naphthol solution**: dissolve 144 g of  $\alpha$ -naphthol in enough alcohol to make a liter of solution.

**$\alpha$ -Naphtholbenzein** (indicator): dissolve 0.1 g in 100 mL 70% alcohol; pH range colorless 9.0–11.0 blue.

**$\alpha$ -Naphtholphthalein** (indicator): dissolve 0.1 g in 50 mL alcohol and dilute with water to 100 mL; pH range pale yellow-red 7.3–8.7 green.

**Nessler's reagent** (for free ammonia): dissolve 50 g of KI in the least possible amount of cold water; add a saturated solution of  $\text{HgCl}_2$  until a very slight excess is indicated; add 400 mL of a 50% solution of KOH; allow to settle, make up to a liter with water, and decant.

**Neutral red**, toluylene red, dimethyldiaminophenazine chloride, aminodimethylaminotoluphenazine hydrochloride (indicator): dissolve 0.1 g in 60 mL alcohol and dilute with water to 100 mL; pH range red 6.8–8.0 yellow-orange.

**Nickel chloride**,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 59 g per liter.

**Nickel nitrate**,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 73 g per liter.

**Nickel sulfate**,  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 66 g per liter.

**Nitramine**, picrylmethylnitramine, 2,4,6-trinitrophenylmethyl nitramine (indicator): dissolve 0.1 g in 60 mL alcohol and dilute with water to 100 mL; pH range colorless 10.8–13.0 red-brown; the solution should be kept in the dark as nitramine is unstable; on boiling with alkali it decomposes quickly. Fresh solutions should be prepared every few months.

**Nitric acid**,  $\text{HNO}_3$ —5*N*: 315 g per liter; sp. gr. 1.165.

**Nitrohydrochloric acid**: *see* aqua regia.

***p*-Nitrophenol** (indicator): dissolve 0.2 g in 100 mL water; pH range colorless at about 5–7 yellow.

**Nitroso- $\beta$ -naphthol**,  $\text{HOC}_{10}\text{H}_6\text{NO}$ —saturated solution: saturate 100 mL of 50% acetic acid with the solid.

**Nylander's solution** (detection of glucose): dissolve 40 g of rochelle salt and 20 g of bismuth subnitrate in 1000 mL of an 8% NaOH solution.

**Obermayer's reagent** (detection of indoxyl in urine): dissolve 4 g of  $\text{FeCl}_3$  in a liter of concentrated HCl.

**Orange III** (indicator): *see* under methyl orange.

**Oxalic acid**,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ : dissolve in ten parts of water.

**Pavy's solution** (estimation of glucose): mix 120 mL of Fehling's solution and 300 mL of ammonium hydroxide (sp. gr. 0.88), and dilute to a liter with water.

**Perchloric acid**,  $\text{HClO}_4$ —60%: use as purchased.

**Phenol red**, phenol-sulfonphthalein (indicator): dissolve 0.1 g in 14.20 mL 0.02*N* NaOH and dilute with water to 250 mL; or, 0.1 g in 100 mL 20% alcohol; pH range yellow 6.8–8.0 red.

**Phenol solution**: dissolve 20 g of phenol (carbolic acid) in a liter of water.

**Phenol sulfonic acid** (determination of nitrogen as nitrate; water analysis for nitrate): dissolve 25 g pure, white phenol in 150 mL of pure concentrated  $\text{H}_2\text{SO}_4$ , add 75 mL of fuming  $\text{H}_2\text{SO}_4$  (15%  $\text{SO}_3$ ), stir well and heat for two hours at 100°C.

**Phenolphthalein** (indicator): dissolve 1 g in 60 mL of alcohol and dilute with water to 100 mL; pH range colorless 8.2–10.0 red.

**Phosphoric acid**, *ortho*,  $\text{H}_3\text{PO}_4$ —0.5*N*: 16 g per liter.

**Poirrer blue C4B** (indicator): dissolve 0.2 g in 100 mL water; pH range blue 11.0–13.0 red.

**Potassium acid antimonate**,  $\text{KH}_2\text{SbO}_4$ —0.1*N*: boil 23 g of the salt with 950 mL of water for 5 minutes, cool rapidly and add 35 mL of 6*N* KOH; allow to stand for one day, filter dilute filtrate to a liter.

**Potassium arsenate**,  $\text{K}_3\text{AsO}_4$ —0.5*N* ( $\text{K}_3\text{AsO}_4/10$ ): 26 g per liter.

**Potassium arsenite**,  $\text{KAsO}_2$ —0.5*N* ( $\text{KAsO}_2/6$ ): 24 g per liter.

**Potassium bromate**,  $\text{KBrO}_3$ —0.5*N* ( $\text{KBrO}_3/12$ ): 14 g per liter.

**Potassium bromide**,  $\text{KBr}$ —0.5*N*: 60 g per liter.

**Potassium carbonate**,  $K_2CO_3$ —3*N*: 207 g per liter.

**Potassium chloride**,  $KCl$ —0.5*N*: 37 g per liter.

**Potassium chromate**,  $K_2CrO_4$ —0.5*N*: 49 g per liter.

**Potassium cyanide**,  $KCN$ —0.5*N*: 33 g per liter.

**Potassium dichromate**,  $K_2Cr_2O_7$ —0.5*N* ( $K_2Cr_2O_7/8$ ): 38 g per liter.

**Potassium ferricyanide**,  $K_3Fe(CN)_6$ —0.5*N*: 55 g per liter.

**Potassium ferrocyanide**,  $K_4Fe(CN)_6 \cdot 3H_2O$ —0.5*N*: 53 g per liter.

**Potassium hydroxide**,  $KOH$ —5*N*: 312 g per liter.

**Potassium iodate**,  $KIO_3$ —0.5*N* ( $KIO_3/12$ ): 18 g per liter.

**Potassium iodide**,  $KI$ —0.5*N*: 83 g per liter.

**Potassium nitrate**,  $KNO_3$ —0.5*N*: 50 g per liter.

**Potassium nitrate**,  $KNO_2$ —6*N*: 510 g per liter.

**Potassium permanganate**,  $KMnO_4$ —0.5*N* ( $KMnO_4/10$ ): 16 g per liter.

**Potassium pyrogallate** (oxygen in gas analysis): weigh out 5 g of pyrogallol (pyrogallic acid), and pour upon it 100 mL of a  $KOH$  solution. If the gas contains less than 28% of oxygen, the  $KOH$  solution should be 500 g  $KOH$  in a liter of water; if there is more than 28% of oxygen in the gas, the  $KOH$  solution should be 120 g of  $KOH$  in 100 mL of water.

**Potassium sulfate**,  $K_2SO_4$ —0.5*N*: 44 g per liter.

**Potassium thiocyanate**,  $KCNS$ —0.5*N*: 49 g per liter.

**Precipitating reagent** (for group II, anions): dissolve 61 g of  $BaCl_2 \cdot 2H_2O$  and 52 g of  $CaCl_2 \cdot 6H_2O$  in water and dilute to 1 liter. If the solution becomes turbid, filter and use filtrate.

**Quinaldine red** (indicator): dissolve 0.1 g in 100 mL alcohol; pH range colorless 1.4–3.2 red.

**Quinoline blue**, cyanin (indicator): dissolve 1 g in 100 mL alcohol; pH range colorless 6.6–8.6 blue.

**Rosolic acid**, aurin, corallin, corallinphthalein, 4,4'-dihydroxy-fuchsone, 4,4'-dihydroxy-3-methyl-fuchsone (indicator): dissolve 0.5 g in 50 mL alcohol and dilute with water to 100 mL.

**Salicyl yellow** (indicator): *see* alizarin yellow GG.

**Scheibler's reagent** (precipitates alkaloids, albumoses and peptones): dissolve sodium tungstate in boiling water containing half its weight of phosphoric acid (sp. gr. 1.13); on evaporation of this solution, crystals of phosphotungstic acid are obtained. A 10% solution of phosphotungstic acid in water constitutes the reagent.

**Schweitzer's reagent** (dissolves cotton, linen, and silk, but not wool); add  $NH_4Cl$  and  $NaOH$  to a solution of copper sulfate. The blue precipitate is filtered off, washed, pressed, and dissolved in ammonia (sp. gr. 0.92).

**Silver nitrate**,  $AgNO_3$ —0.25*N*: 43 g per liter.

**Silver sulfate**,  $Ag_2SO_4$ —*N*/13 (saturated solution): stir mechanically 10 g of the salt in a liter of water for 3 hours; decant and use the clear liquid.

**Soap solution** (for hardness in water): (*a*) *Clark's or A.P.H.A. Stand. Methods*—prepare stock solution of 100 g of pure powdered castile soap in a liter of 80% ethyl alcohol; allow to stand over night and decant. Titrate against  $CaCl_2$  solution (0.5 g  $CaCO_3$  dissolved in a concentrated  $HCl$ , neutralized with  $NH_4OH$  to slight alkalinity using litmus as the indicator, make up to 500 mL; 1 mL of this solution is equivalent to 1 mg  $CaCO_3$ ) and dilute with 80% alcohol until 1 mL of the resulting solution is equivalent to 1 mL of the standard  $CaCl_2$  making due allowance

for the lather factor (the lather factor is that amount of standard soap solution required to produce a permanent lather in a 50-mL portion of distilled water). One milliliter of this solution after subtracting the lather factor is equivalent to 1 mg of  $\text{CaCO}_3$ . (b) *Boutron-Boudet*—dissolve 100 g of pure castile soap in about 2500 mL of 56% ethyl alcohol and adjust so that 2.4 mL will give a permanent lather with 40 mL of a solution containing 0.59 g  $\text{Ba}(\text{NO}_3)_2$  per liter of water; 2.4 mL of this solution is equivalent to 22 French degrees or 220 parts per million of hardness (as  $\text{CaCO}_3$ ) on a 40-mL sample of water.

**Sodium acetate**,  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ : dissolve 1 part of the salt in 10 parts of water.

**Sodium acetate, acid**: dissolve 100 g of sodium acetate and 30 mL of glacial acetic acid in water and dilute to 1 liter.

**Sodium bismuthate** (oxidation of manganese): heat 20 parts of NaOH nearly to redness in an iron or nickel crucible, and add slowly 10 parts of basic bismuth nitrate which has been previously dried. Add 2 parts of sodium peroxide, and pour the brownish-yellow fused mass on an iron plate to cool. When cold break up in a mortar, extract with water, and collect on an asbestos filter.

**Sodium carbonate**,  $\text{Na}_2\text{CO}_3$ —3*N*: 159 g per liter; one part  $\text{Na}_2\text{CO}_3$ , or 2.7 parts of the crystalline  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  in 5 parts of water.

**Sodium chloride**,  $\text{NaCl}$ —0.5*N*: 29 g per liter.

**Sodium chloroplatinite**,  $\text{Na}_2\text{PtCl}_4$ : dissolve 1 part of the salt in 12 parts of water.

**Sodium cobaltinitrite**,  $\text{Na}_2\text{Co}(\text{NO}_2)_6$ —0.3*N*: dissolve 230 g of  $\text{NaNO}_2$  in 500 mL of water, add 160 mL of 6*N* acetic acid and 35 g of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . Allow to stand one day, filter, and dilute the filtrate to a liter.

**Sodium hydrogen phosphate**,  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ —0.5*N*: 60 g liter.

**Sodium hydroxide**,  $\text{NaOH}$ —5*N*: 220 g per liter.

**Sodium hydroxide, alcoholic**: dissolve 20 g of NaOH in alcohol and dilute to 1 liter with alcohol.

**Sodium hypobromite**: dissolve 100 g of NaOH in 250 mL of water and add 25 mL of bromine.

**Sodium nitrate**,  $\text{NaNO}_3$ —0.5*N*: 43 g per liter.

**Sodium nitroprusside** (for sulfur detection): dissolve about 1 g of sodium nitroprusside in 10 mL of water; as the solution deteriorates on standing, only freshly prepared solutions should be used. This compound is also called sodium nitroferrocyanide and has the formula  $\text{Na}_2\text{Fe}(\text{NO})(\text{CN})_5 \cdot 2\text{H}_2\text{O}$ .

**Sodium polysulfide**,  $\text{Na}_2\text{S}_x$ : dissolve 480 g of  $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$  in 500 mL of water, add 40 g of NaOH and 18 g of sulfur, stir mechanically and dilute to 1 liter with water.

**Sodium sulfate**,  $\text{Na}_2\text{SO}_4$ —0.5*N*: 35 g per liter.

**Sodium sulfide**,  $\text{Na}_2\text{S}$ : saturate NaOH solution with  $\text{H}_2\text{S}$ , then add as much NaOH as was used in the original solution.

**Sodium sulfite**,  $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 63 g per liter.

**Sodium sulfite, acid** (saturated): dissolve 600 g of  $\text{NaHSO}_3$  in water and dilute to 1 liter; for the preparation of addition compounds with aldehydes and ketones: prepare a saturated solution of sodium carbonate in water and saturate with sulfur dioxide.

**Sodium tartrate, acid**,  $\text{NaHC}_4\text{H}_4\text{O}_6$ : dissolve 1 part of the salt in 10 parts of water.

**Sodium thiosulfate**,  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ : one part of the salt in 40 parts of water.

**Sonnenschein's reagent** (alkaloid detection): a nitric acid solution of ammonium molybdate is treated with phosphoric acid. The precipitate so produced is washed and boiled with aqua regia

until the ammonium salt is decomposed. The solution is evaporated to dryness and the residue is dissolved in 10%  $\text{HNO}_3$ .

**Stannic chloride**,  $\text{SnCl}_4$ —0.5*N*: 33 g per liter.

**Stannous chloride**,  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ —0.5*N*: 56 g per liter. The water should be acid with  $\text{HCl}$  and some metallic tin should be kept in the bottle.

**Starch solution** (iodine indicator): dissolve 5 g of soluble starch in cold water, pour the solution into 2 liters of water and boil for a few minutes. Keep in a glass-stoppered bottle.

**Starch solution** (other than soluble): make a thin paste of the starch with cold water, then stir in 200 times its weight of boiling water and boil for a few minutes. A few drops of chloroform added to the solution acts as a preservative.

**Stoke's reagent**: dissolve 30 g of ferrous sulfate and 20 g of tartaric acid in water and dilute to 1 liter. When required for use, add strong ammonia until the precipitate first formed is dissolved.

**Strontium chloride**,  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 67 g per liter.

**Strontium nitrate**,  $\text{Sr}(\text{NO}_3)_2$ —0.5*N*: 53 g per liter.

**Strontium sulfate**,  $\text{SrSO}_4$ : prepare a saturated solution.

**Sulfanilic acid** (for detection of nitrites): dissolve 8 g of sulfanilic acid in 1 liter of acetic acid (sp. gr. 1.04).

**Sulfuric acid**,  $\text{H}_2\text{SO}_4$ —5*N*: 245 g per liter, sp. gr. 1.153.

**Sulfurous acid**,  $\text{H}_2\text{SO}_3$ : saturate water with sulfur dioxide.

**Tannic acid**: dissolve 1 g tannic acid in 1 mL alcohol and make up to 10 mL with water.

**Tartaric acid**,  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ : dissolve one part of the acid in 3 parts of water; for a saturated solution dissolve 750 g of tartaric acid in water and dilute to 1 liter.

**Tetrabromophenol blue**, tetrabromophenol-tetrabromosulfonphthalein (indicator): dissolve 0.1 g in 5 mL 0.02*N*  $\text{NaOH}$  and dilute with water to 250 mL; pH range yellow 3.0–4.6 blue.

**Thymol blue**, thymol-sulfonphthalein (indicator): dissolve 0.1 g in 10.75 mL 0.02*N*  $\text{NaOH}$  and dilute with water to 250 mL; or dissolve 0.1 g in 20 mL warm alcohol and dilute with water to 100 mL; pH range (acid) red 1.2–2.8 yellow, and (alkaline) yellow 8.0–9.6 blue.

**Thymolphthalein** (indicator): dissolve 0.1 g in 100 mL alcohol; pH range colorless 9.3–10.5 blue.

**Tincture of iodine** (antiseptic): add 70 g of iodine and 50 g of  $\text{KI}$  to 50 mL of water; make up to 1 liter with alcohol.

***o*-Tolidine solution** (for residual chlorine in water analysis): dissolve 1 g of pulverized *o*-tolidine, m.p. 129°C., in 1 liter of dilute hydrochloric acid (100 mL conc.  $\text{HCl}$  diluted to 1 liter).

**Toluylene red** (indicator): *see* neutral red.

**Trichloroacetic acid**: dissolve 100 g of the acid in water and dilute to 1 liter.

**Trinitrobenzene**, 1,3,5-trinitrobenzene (indicator): dissolve 0.1 g in 100 mL alcohol; pH range colorless 11.5–14.0 orange.

**Trinitrobenzoic acid**, 2,4,6-trinitrobenzoic acid (indicator): dissolve 0.1 g in 100 mL water; pH range colorless 12.0–13.4 orange-red.

**Tropeolin D** (indicator): *see* methyl orange.

**Tropeolin O**, sodium 2,4-dihydroxyazobenzene-4-sulfonate (indicator): dissolve 0.1 g in 100 mL water; pH range yellow 11.0–13.0 orange-brown.

**Tropeolin OO**, orange IV, sodium *p*-diphenylamino-azobenzene sulfonate, sodium 4'-anilino-azobenzene-4-sulfonate (indicator): dissolve 0.1 g in 100 mL water; pH range red 1.3–3.2 yellow.

**Tropeolin OOO**, sodium  $\alpha$ -naphtholazobenzene sulfonate (indicator): dissolve 0.1 g in 100 mL water; pH range yellow 7.6–8.9 red.

**Turmeric paper** (gives a rose-brown coloration with boric acid): wash the ground root of turmeric with water and discard the washings. Digest with alcohol and filter, using the clear filtrate to impregnate white, unsized paper, which is then dried.

**Uffelmann's reagent** (gives a yellow coloration in the presence of lactic acid): add a ferric chloride solution to a 2% phenol solution until the solution becomes violet in color.

**Wagner's solution** (phosphate rock analysis): dissolve 25 g citric acid and 1 g salicylic acid in water, and make up to 1 liter. Twenty-five to fifty milliliters of this reagent prevents precipitation of iron and aluminum.

**Wijs solution** (for iodine number): dissolve 13 g resublimed iodine in 1 liter of glacial acetic acid (99.5%), and pass in washed and dried (over or through  $\text{H}_2\text{SO}_4$ ) chlorine gas until the original thio titration of the solution is not quite doubled. There should be only a slight excess of iodine and no excess of chlorine. Preserve the solution in amber colored bottles sealed with paraffin. Do not use the solution after it has been prepared for more than 30 days.

**Xylene cyanole-methyl orange indicator**, Schoepfle modification (for partially color blind operators): dissolve 0.75 g xylene cyanole FF (Eastman No. T 1579) and 1.50 g methyl orange in 1 liter of water.

***p*-Xylenol blue**, 1,4-dimethyl-5-hydroxybenzene-sulfonphthalein (indicator): dissolve 0.1 g in 250 mL alcohol; pH range (acid) red 1.2–2.8 yellow, and (alkaline) yellow 8.0–9.6 blue.

**Zinc chloride**,  $\text{ZnCl}_2$ —0.5*N*: 34 g per liter.

**Zinc nitrate**,  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ —0.5*N*: 74 g per liter.

**Zinc sulfate**,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ —0.5*N*: 72 g per liter.

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors

Exposure limits (threshold limit value or TLV) are those set by the Occupational Safety and Health Administration and represent conditions to which most workers can be exposed without adverse effects. The TLV value is expressed as a time weighted average airborne concentration over a normal 8-hour workday and 40-hour work-week.

| Substance        | Maximum allowable exposure |                                 | Toxicity          |
|------------------|----------------------------|---------------------------------|-------------------|
|                  | ppm                        | $\text{mg} \cdot \text{m}^{-3}$ |                   |
| Acetaldehyde     | 25                         | 45                              | carcinogen        |
| Acetic acid      | 10                         | 25                              |                   |
| Acetic anhydride | 5                          | 21                              |                   |
| Acetone          | 750                        | 1780                            |                   |
| Acetonitrile     | 40                         | 67                              |                   |
| Acetophenone     | 10                         | 49                              | slightly narcotic |
| Acetylene        |                            |                                 |                   |
| Acrolein         | 0.1                        | 0.23                            |                   |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance  | Maximum allowable exposure |                      | Toxicity                           |
|--|----------------------------|----------------------|------------------------------------|
|  | ppm                        | mg · m <sup>-3</sup> |                                    |
| Acrylic acid                                       | 2                          | 5.9                  |                                    |
| Acrylonitrile                                      | 2                          | 4.3                  |                                    |
| Acrylonitrile                                      | 20                         | 45                   |                                    |
| Allyl alcohol                                      | 2                          | 4.8                  |                                    |
| Allyl chloride                                     | 1                          | 3                    |                                    |
| Allyl glycidyl ether                               | 5                          | 22                   |                                    |
| Ammonia  | 25                         | 18                   | toxic                              |
| Aniline  | 2                          | 7.6                  | carcinogen                         |
| Arsine   | 0.05                       | 0.2                  | highly toxic                       |
| Benzene  | 10                         | 32                   | carcinogen                         |
| Benzenethiol                                       | 0.5                        | 2.3                  |                                    |
| <i>p</i> -Benzoquinone                             | 0.1                        |                      |                                    |
| Benzoyl chloride                                   | 0.5                        |                      |                                    |
| Benzoyl peroxide                                   |                            | 5                    |                                    |
| Benzyl acetate                                     | 10                         |                      |                                    |
| Benzyl chloride                                    | 1                          |                      | carcinogen                         |
| Biphenyl   | 0.2                        |                      |                                    |
| Bis(2-aminoethyl)amine                             | 1                          |                      |                                    |
| Bis(2-chloroethyl) ether                           | 5                          | 29                   |                                    |
| Bis(2-chloromethyl) ether                          | 0.001                      |                      | carcinogen                         |
| Bis(2-ethylhexyl) phthalate                        |                            | 5                    |                                    |
| Boron tribromide                                   | 1                          |                      |                                    |
| Boron trichloride                                  |                            |                      | toxic                              |
| Boron trifluoride                                  | 1                          | 3                    | highly toxic                       |
| Bromine  | 0.1                        | 0.7                  |                                    |
| Bromine pentafluoride                              | 0.1                        |                      | highly toxic                       |
| Bromine trifluoride                                |                            |                      | highly toxic                       |
| Bromochloromethane (Halon 1011)                    | 200                        | 1060                 |                                    |
| Bromoethane  | 5                          | 22                   | carcinogen                         |
| Bromoethylene                                      | 5                          | 22                   | slightly toxic                     |
| Bromoform  | 0.5                        | 5                    |                                    |
| Bromomethane                                       | 5                          | 19                   | highly toxic,<br>carcinogen        |
| 1,3-Butadiene                                      | 2                          |                      | slightly anesthetic,<br>carcinogen |
| Butane   | 800                        | 1900                 | slightly anesthetic                |
| 1-Butanethiol                                      | 0.5                        | 1.8                  |                                    |
| 1-Butanol  | 50                         | 152                  |                                    |
| 2-Butanol  | 100                        | 303                  |                                    |
| 2-Butanone   | 200                        | 590                  |                                    |
| 2-Butoxyethanol                                    | 25                         | 121                  |                                    |
| Butyl acetate                                      | 150                        | 710                  |                                    |
| <i>sec</i> -Butyl acetate                          | 200                        | 950                  |                                    |
| <i>tert</i> -Butyl acetate                         | 200                        | 950                  |                                    |
| Butyl acrylate                                     | 10                         |                      |                                    |
| <i>tert</i> -Butyl alcohol                         | 100                        | 300                  |                                    |
| Butylamine   | 5                          | 15                   |                                    |
| <i>tert</i> -Butyl chromate (as CrO <sub>3</sub> ) |                            | 0.1                  |                                    |
| Butyl glycidyl ether                               | 50                         | 270                  |                                    |

TABLE 11.49 TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance  | Maximum allowable exposure |                      | Toxicity          |
|--|----------------------------|----------------------|-------------------|
|  | ppm                        | mg · m <sup>-3</sup> |                   |
| Butyl mercaptan                                      | 0.5                        | 1.5                  |                   |
| <i>p</i> -tert-Butyltoluene                          | 10                         |                      |                   |
| (+)-Camphor  | 2                          | 12                   |                   |
| Caprolactam  | 5                          |                      |                   |
| Carbon dioxide                                       | 5000                       | 9000                 |                   |
| Carbon disulfide                                     | 10                         | 31                   |                   |
| Carbon monoxide                                      | 25                         | 28                   | toxic             |
| Carbon tetrachloride                                 | 10                         | 65                   |                   |
| Carbonyl chloride                                    | 0.1                        |                      |                   |
| Carbonyl fluoride                                    | 2                          |                      | toxic             |
| Chlordane  |                            | 0.5                  |                   |
| Chlorine   | 0.5                        | 1.5                  | highly toxic      |
| Chlorine dioxide                                     | 0.1                        | 0.3                  |                   |
| Chlorine trifluoride                                 | 0.1                        | 0.4                  | highly toxic      |
| Chloroacetaldehyde                                   | 1                          | 3                    |                   |
| α-Chloroacetophenone                                 | 0.05                       | 0.3                  |                   |
| Chloroacetyl chloride                                | 0.05                       |                      |                   |
| Chlorobenzene  | 10                         | 46                   |                   |
| 2-Chloro-1,3-butadiene                               | 10                         |                      | carcinogen        |
| Chlorodifluoromethane (CFC 22)                       | 1000                       | 3540                 |                   |
| Chloroethane   | 100                        | 264                  | low toxicity      |
| 2-Chloroethanol                                      | 1                          | 3.3                  |                   |
| Chloroethylene (vinyl chloride)                      | 5                          | 13                   | toxic, carcinogen |
| Chloroform (trichloromethane)                        | 10                         | 49                   |                   |
| Chloromethane  | 50                         | 103                  | toxic, carcinogen |
| 1-Chloro-1-nitropropane                              | 20                         | 100                  |                   |
| Chloropentafluoroethane (CFC 115)                    | 1000                       | 6320                 |                   |
| 3-Chloro-1-propene (allyl chloride)                  | 1                          | 3                    | carcinogen        |
| <i>o</i> -Chlorotoluene                              | 50                         | 259                  |                   |
| Chlorotrifluoroethylene                              |                            |                      | toxic             |
| Chromyl chloride (CrO <sub>2</sub> Cl <sub>2</sub> ) | 0.025                      |                      | carcinogen        |
| <i>o</i> -Cresol (also <i>m</i> -, <i>p</i> -)       | 5                          | 22                   |                   |
| <i>trans</i> -Crotonaldehyde                         | 2                          | 5.7                  |                   |
| Cyanogen   | 10                         | 20                   | highly toxic      |
| Cyanogen chloride                                    | 0.3                        |                      |                   |
| Cyclohexane  | 300                        | 1030                 |                   |
| Cyclohexanol   | 50                         | 206                  |                   |
| Cyclohexanone  | 25                         | 100                  |                   |
| Cyclohexene  | 300                        | 1015                 |                   |
| Cyclohexylamine                                      | 10                         | 41                   |                   |
| 1,3-Cyclopentadiene                                  | 75                         |                      |                   |
| Cyclopentane   | 600                        | 1720                 |                   |
| Cyclopropane   |                            |                      | anesthetic        |
| 2,4-D  |                            | 10                   |                   |
| DDT  |                            | 1                    |                   |
| Decaborane   | 0.05                       | 0.3                  |                   |
| Diacetone alcohol                                    | 50                         | 238                  |                   |
| 2,2'-Diaminodiethylamine                             | 1                          | 4.2                  |                   |
| Diazomethane   | 0.2                        |                      | carcinogen        |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance                                       | Maximum allowable exposure |                      | Toxicity                      |
|---|----------------------------|----------------------|-------------------------------|
|   | ppm                        | mg · m <sup>-3</sup> |                               |
| Diborane  | 0.1                        | 0.1                  |                               |
| Dibromodifluoromethane                          | 100                        | 860                  |                               |
| 1,2-Dibromoethane                               |                            |                      | carcinogen                    |
| Dibutyl phthalate                               |                            | 5                    |                               |
| Dichloroacetylene                               | 0.1                        |                      |                               |
| <i>o</i> -Dichlorobenzene                       | 25                         | 150                  |                               |
| <i>p</i> -Dichlorobenzene                       | 10                         | 60                   | carcinogen                    |
| Dichlorodifluoromethane (Freon 12)              | 1000                       | 4950                 |                               |
| 1,1-Dichloroethane                              | 100                        | 405                  |                               |
| 1,2-Dichloroethane                              | 10                         | 40                   | carcinogen                    |
| 1,1-Dichloroethylene                            | 5                          | 20                   | carcinogen                    |
| <i>cis</i> -1,2-Dichloroethylene                | 200                        | 793                  |                               |
| <i>trans</i> -1,2-Dichloroethylene              | 200                        | 793                  |                               |
| Dichlorofluoromethane (Freon 21)                | 10                         | 42                   |                               |
| Dichloromethane                                 | 50                         | 174                  | carcinogen                    |
| 1,1-Dichloro-1-nitroethane                      | 10                         | 60                   |                               |
| 1,2-Dichloropropane                             | 75                         | 347                  | carcinogen                    |
| 1,3-Dichloropropene                             | 1                          |                      | carcinogen                    |
| Dichlorosilane                                  |                            |                      | highly toxic                  |
| 1,2-Dichlorotetrafluoroethane (Freon 114)       | 1000                       | 7000                 |                               |
| Dieldrin  |                            | 0.25                 |                               |
| Diethanolamine                                  | 0.46                       |                      |                               |
| Diethylamine                                    | 5                          | 15                   |                               |
| Diethyl ether                                   | 400                        | 1210                 |                               |
| Diglycidyl ether                                | 0.5                        | 2.8                  |                               |
| Diisobutyl ketone                               | 25                         | 150                  |                               |
| Diisopropylamine                                | 5                          | 20                   |                               |
| Diisopropyl ether                               | 250                        | 1040                 |                               |
| Dimethoxymethane                                | 1000                       | 3110                 |                               |
| <i>N,N</i> -Dimethylacetamide                   | 10                         | 35                   |                               |
| Dimethylamine                                   | 5                          | 9.2                  | highly toxic                  |
| <i>N,N</i> -Dimethylaniline                     | 5                          | 25                   |                               |
| Dimethyl 1,2-dibromo-2,2-dichloroethylphosphate |                            | 3                    |                               |
| Dimethyl ether                                  |                            |                      | slightly toxic,<br>anesthetic |
| 1-(1,1-Dimethylethyl)-4-methylbenzene           | 1                          | 6.1                  |                               |
| <i>N,N</i> -Dimethylformamide                   | 10                         | 30                   |                               |
| 2,6-Dimethyl-4-heptanone                        | 25                         |                      |                               |
| 1,1-Dimethylhydrazine                           | 0.5                        | 1                    | carcinogen                    |
| Dimethyl phthalate                              |                            | 5                    |                               |
| 2,2-Dimethylpropane                             |                            |                      | probably anesthetic           |
| Dimethyl sulfate                                | 0.1                        | 0.5                  | carcinogen                    |
| Dinitrobenzene                                  | 0.15                       | 1                    |                               |
| Dinitro- <i>o</i> -cresol                       |                            | 0.2                  |                               |
| Dinitrotoluene                                  |                            | 1.5                  |                               |
| 1,4-Dioxane                                     | 25                         | 90                   | carcinogen                    |
| Diphenyl  | 0.2                        | 1                    |                               |
| Diphenyl ether                                  | 1                          | 7                    |                               |
| Dipropylene glycol methyl ether—skin            | 100                        | 600                  |                               |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance                         | Maximum allowable exposure |                      | Toxicity          |
|-----------------------------------|----------------------------|----------------------|-------------------|
|                                   | ppm                        | mg · m <sup>-3</sup> |                   |
| Endrin—skin                       |                            | 0.1                  |                   |
| Epichlorohydrin                   | 2                          | 7.6                  | carcinogen        |
| 2,3-Epoxy-1-propanol (glycidol)   | 50                         | 150                  |                   |
| 1,2-Ethanediamine                 | 10                         | 25                   |                   |
| Ethanethiol                       | 0.5                        |                      |                   |
| Ethanol                           | 1000                       | 1880                 |                   |
| Ethanolamine                      | 3                          | 7.5                  |                   |
| 2-Ethoxyethanol (Cellosolve)      | 5                          | 18                   |                   |
| 2-Ethoxyethyl acetate             | 5                          | 27                   |                   |
| Ethyl acetate                     | 400                        | 1400                 |                   |
| Ethyl acrylate                    | 5                          | 20                   |                   |
| Ethylamine                        | 5                          | 9.2                  | highly toxic      |
| Ethylbenzene                      | 100                        | 435                  |                   |
| Ethylene                          |                            |                      | anesthetic        |
| Ethylene glycol                   | 39                         |                      |                   |
| Ethylene glycol dinitrate         | 0.2                        |                      |                   |
| Ethyleneimine                     | 0.05                       |                      | carcinogen        |
| Ethylene oxide                    | 1                          |                      | toxic, carcinogen |
| Ethyl formate                     | 100                        | 300                  |                   |
| Ethyl mercaptan                   | 0.1                        | 1                    |                   |
| Ethyl silicate                    | 100                        | 850                  |                   |
| Fluorine                          | 1                          | 2                    | highly toxic      |
| Fluorotrichloromethane (Freon 11) | 1000                       | 5600                 |                   |
| Formaldehyde                      | 0.3                        |                      | carcinogen        |
| Formamide                         | 10                         | 18                   |                   |
| Formic acid                       | 5                          | 9.4                  |                   |
| 2-Furancarboxaldehyde (furfural)  | 2                          | 7.9                  |                   |
| 2-Furanmethanol                   | 10                         | 40                   |                   |
| Glycerol                          |                            | 10                   |                   |
| Heptachlor                        |                            | 0.5                  |                   |
| Heptane                           | 400                        | 1640                 |                   |
| 2-Heptanone                       | 50                         | 233                  |                   |
| 3-Heptanone                       | 50                         | 234                  |                   |
| Hexachloro-1,3-butadiene          | 0.02                       |                      | carcinogen        |
| Hexachlorocyclohexane (lindane)   |                            | 0.5                  |                   |
| Hexachloroethane                  | 1                          |                      | carcinogen        |
| Hexachloronaphthalene             |                            | 0.2                  |                   |
| Hexamethylphosphoric triamide     |                            |                      | carcinogen        |
| Hexane                            | 50                         | 176                  |                   |
| 2-Hexanone                        | 5                          | 20                   |                   |
| sec-Hexyl acetate                 | 50                         | 300                  |                   |
| Hexylene glycol                   | 25                         |                      |                   |
| Hydrazine                         | 0.01                       | 0.1                  | carcinogen        |
| Hydrogen bromide                  | 3                          | 10                   | highly toxic      |
| Hydrogen chloride                 | 5                          | 7                    | highly toxic      |
| Hydrogen cyanide                  | 4.7                        |                      | highly toxic      |
| Hydrogen fluoride                 | 3                          | 2                    | highly toxic      |
| Hydrogen iodide                   |                            |                      | highly toxic      |
| Hydrogen peroxide (90%)           | 1                          | 1.4                  |                   |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance                                 | Maximum allowable exposure |                      | Toxicity     |
|---|----------------------------|----------------------|--------------|
|   | ppm                        | mg · m <sup>-3</sup> |              |
| Hydrogen selenide                         | 0.05                       | 0.2                  | highly toxic |
| Hydrogen sulfide                          | 10                         | 15                   | highly toxic |
| 4-Hydroxy-4-methyl-2-pentanone            | 50                         | 238                  |              |
| Indene                                    | 10                         |                      |              |
| Iodine                                    | 0.1                        | 1                    |              |
| Iodine pentafluoride                      |                            |                      | highly toxic |
| Iodomethane                               | 2                          | 12                   |              |
| Isobutyl acetate                          | 150                        | 700                  |              |
| Isobutyl alcohol                          | 50                         | 150                  |              |
| Isopentyl acetate                         | 100                        | 525                  |              |
| Isopentyl alcohol                         | 100                        | 360                  |              |
| Isophorone                                | 5                          | 28                   |              |
| Isopropyl acetate                         | 250                        | 1040                 |              |
| Isopropylamine                            | 5                          | 12                   |              |
| Isopropylbenzene (cumene)                 | 50                         | 246                  |              |
| Isopropyl glycidyl ether                  | 50                         | 240                  |              |
| Ketene                                    | 0.5                        | 0.9                  |              |
| Lindane                                   |                            | 0.5                  |              |
| Liquified petroleum gas                   | 1000                       | 1800                 |              |
| Malathion                                 |                            | 10                   |              |
| Maleic anhydride                          | 0.25                       | 1                    |              |
| Malononitrile                             | 0.05                       | 0.4                  |              |
| Mesityl oxide                             | 15                         | 60                   |              |
| Methacrylic acid                          | 20                         | 70                   |              |
| Methanethiol                              | 0.5                        |                      |              |
| Methanol                                  | 200                        | 262                  |              |
| 2-Methoxyaniline (also 4-)                | 0.1                        |                      | carcinogen   |
| 2-Methoxyethanol                          | 5                          | 16                   |              |
| 2-Methoxyethyl acetate                    | 5                          | 24                   |              |
| Methyl acetate                            | 200                        | 610                  |              |
| Methyl acetylene-propadiene (MAPP)        | 1000                       | 1800                 |              |
| Methyl acrylate                           | 10                         | 35                   |              |
| Methylacrylonitrile                       | 1                          |                      |              |
| Methylamine                               | 5                          | 6.4                  | highly toxic |
| <i>o</i> -Methylaniline (also <i>p</i> -) | 2                          |                      | carcinogen   |
| <i>m</i> -Methylaniline                   | 2                          |                      |              |
| <i>N</i> -Methylaniline                   | 0.5                        | 2.2                  |              |
| 3-Methyl-1-butanol                        | 100                        | 361                  |              |
| Methyl <i>tert</i> -butyl ether           | 40                         |                      |              |
| Methylcyclohexane                         | 400                        | 1600                 |              |
| 1-Methylcyclohexanol                      | 50                         | 234                  |              |
| <i>cis</i> -2-Methylcyclohexanol          | 50                         | 234                  |              |
| <i>trans</i> -2-Methylcyclohexanol        | 50                         | 234                  |              |
| <i>cis</i> -3-Methylcyclohexanol          | 50                         | 234                  |              |
| <i>trans</i> -3-Methylcyclohexanol        | 50                         | 234                  |              |
| <i>cis</i> -4-Methylcyclohexanol          | 50                         | 234                  |              |
| <i>trans</i> -4-Methylcyclohexanol        | 50                         | 234                  |              |
| Methyl formate                            | 100                        | 250                  |              |
| 5-Methyl-2-hexanone                       | 50                         | 234                  |              |

TABLE 11.49 TLV Concentration Limits for Gases and Vapors (Continued)

| Substance   | Maximum allowable exposure |                      | Toxicity     |
|---|----------------------------|----------------------|--------------|
|   | ppm                        | mg · m <sup>-3</sup> |              |
| Methyl hydrazine                                      | 0.01                       |                      |              |
| Methyl isocyanate                                     | 0.02                       | 0.05                 |              |
| Methyl mercaptan                                      | 0.5                        | 1                    | highly toxic |
| Methyl methacrylate                                   | 100                        | 410                  |              |
| Methyl oxirane  | 20                         |                      | carcinogen   |
| 4-Methyl-2-pentanol                                   | 25                         | 104                  |              |
| 4-Methyl-2-pentanone                                  | 50                         | 205                  |              |
| 2-Methyl-2,4-pentanediol                              | 25                         | 121                  |              |
| 2-Methyl-1-propanol                                   | 50                         | 152                  |              |
| 2-Methyl-2-propanol                                   | 100                        | 303                  |              |
| 2-Methyl-2-propenenitrile                             | 1                          | 2.7                  |              |
| <i>o</i> -Methylstyrene (also <i>m</i> -, <i>p</i> -) | 50                         |                      |              |
| Morpholine  | 20                         | 70                   |              |
| Naphthalene   | 10                         | 50                   |              |
| Nickel carbonyl [Ni(CO) <sub>4</sub> ]                | 0.05                       | 0.35                 | carcinogen   |
| Nicotine  |                            | 0.5                  |              |
| Nitric acid   | 2                          | 5                    |              |
| Nitric oxide  | 25                         | 30                   | highly toxic |
| Nitrobenzene  | 1                          | 5                    |              |
| <i>p</i> -Nitrochlorobenzene                          |                            | 1                    |              |
| Nitroethane   | 100                        | 310                  |              |
| Nitrogen dioxide                                      | 3                          |                      | highly toxic |
| Nitrogen trifluoride                                  | 10                         |                      |              |
| Nitrogen trioxide                                     | 10                         | 29                   | highly toxic |
| Nitroglycerine  | 0.2                        | 2                    |              |
| Nitromethane  | 100                        | 250                  |              |
| 1-Nitropropane  | 25                         | 90                   |              |
| 2-Nitropropane  | 10                         | 36                   |              |
| Nitrosyl chloride                                     |                            |                      | highly toxic |
| <i>o</i> -Nitrotoluene (also <i>m</i> -, <i>p</i> -)  | 2                          |                      |              |
| Nonane  | 200                        | 1050                 |              |
| Octachloronaphthalene                                 |                            | 0.1                  |              |
| Octane  | 300                        | 1450                 |              |
| Oxalic acid   |                            | 1                    |              |
| 2-Oxetanone   | 0.05                       |                      | carcinogen   |
| Oxygen difluoride                                     | 0.05                       | 0.1                  |              |
| Ozone   | 0.1                        | 0.2                  |              |
| Parathion   |                            | 0.1                  |              |
| Pentaborane   | 0.005                      | 0.01                 |              |
| Pentachloronaphthalene                                |                            | 0.5                  |              |
| Pentachlorophenol                                     |                            | 0.5                  |              |
| Pentanal  | 50                         |                      |              |
| Pentane   | 600                        | 1770                 |              |
| 2-Pentanone   | 200                        | 700                  |              |
| 3-Pentanone   | 200                        | 700                  |              |
| Pentyl acetate  | 100                        | 530                  |              |
| Perchloroethylene                                     | 100                        | 670                  |              |
| Perchloromethyl mercaptan                             | 0.1                        | 0.8                  |              |
| Perchloryl fluoride                                   | 3                          | 14                   |              |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance                  | Maximum allowable exposure |                      | Toxicity     |
|----------------------------|----------------------------|----------------------|--------------|
|                            | ppm                        | mg · m <sup>-3</sup> |              |
| Perfluoroacetone           | 0.1                        |                      |              |
| Phenol                     | 5                          | 19                   |              |
| <i>p</i> -Phenylenediamine |                            | 0.1                  |              |
| Phenylhydrazine            | 0.1                        | 0.4                  | carcinogen   |
| Phosgene                   | 0.1                        | 0.4                  | highly toxic |
| Phosphine                  | 0.3                        | 0.4                  | highly toxic |
| Phosphoric acid            |                            | 1                    |              |
| Phosphorus pentachloride   |                            | 1                    |              |
| Phosphorus pentafluoride   |                            |                      | highly toxic |
| Phosphorus pentasulfide    |                            | 1                    |              |
| Phosphorus trichloride     | 0.5                        | 3                    |              |
| Phosphoryl chloride        | 0.1                        |                      |              |
| Phthalic anhydride         | 1                          | 6                    |              |
| Picric acid—skin           |                            | 0.1                  |              |
| Propane                    | 1000                       | 1800                 | low toxicity |
| Propanoic acid             | 10                         | 30                   |              |
| 1-Propanol                 | 200                        | 500                  |              |
| 2-Propanol                 | 400                        | 980                  |              |
| Propenal                   | 0.1                        |                      |              |
| Propenenitrile             | 2                          |                      | carcinogen   |
| Propenoic acid             | 2                          |                      |              |
| Propyl acetate             | 200                        | 835                  |              |
| Propyleneimine             | 2                          | 5                    | carcinogen   |
| Propylene oxide            | 100                        | 240                  | toxic        |
| Propyl nitrate             | 25                         | 110                  |              |
| Propyne                    | 1000                       | 1650                 |              |
| 2-Propyn-1-ol              | 1                          | 2.3                  |              |
| Pyridine                   | 5                          | 15                   |              |
| Quinone                    | 0.1                        | 0.4                  |              |
| Selenium compounds (as Se) |                            | 0.2                  |              |
| Selenium hexafluoride      | 0.05                       | 0.4                  |              |
| Silane                     | 5                          | 7                    | highly toxic |
| Silicon tetrafluoride      |                            |                      | highly toxic |
| Stibine                    | 0.1                        |                      |              |
| Stoddard solvent           | 100                        | 575                  |              |
| Strychnine                 |                            | 0.15                 |              |
| Styrene                    | 50                         | 213                  | carcinogen   |
| Sulfur dioxide             | 2                          |                      | highly toxic |
| Sulfur hexafluoride        | 1000                       | 6000                 | low toxicity |
| Sulfuric acid              |                            | 1                    |              |
| Sulfur monochloride        | 1                          | 6                    |              |
| Sulfur pentafluoride       | 0.01                       |                      |              |
| Sulfur tetrafluoride       | 0.1                        | 0.4                  |              |
| Sulfuryl fluoride          | 5                          | 20                   | highly toxic |
| Tellurium hexafluoride     | 0.02                       | 0.2                  |              |
| Terphenyls                 | 1                          | 9                    |              |
| 1,1,2,2-Tetrabromoethane   | 1                          | 14                   |              |

**TABLE 11.49** TLV Concentration Limits for Gases and Vapors (*Continued*)

| Substance   | Maximum allowable exposure |                      | Toxicity            |
|---|----------------------------|----------------------|---------------------|
|   | ppm                        | mg · m <sup>-3</sup> |                     |
| Tetrabromomethane   | 0.1                        |                      |                     |
| 1,1,1,2-Tetrachloro-2,2-difluoroethane                        | 500                        | 4170                 |                     |
| 1,1,2,2-Tetrachloro-1,2-difluoroethane                        | 500                        | 4170                 |                     |
| 1,1,2,2-Tetrachloroethane                                     | 1                          | 6.9                  | carcinogen          |
| Tetrachloroethylene   | 25                         | 170                  | carcinogen          |
| Tetrachloromethane  | 5                          | 31                   | carcinogen          |
| 1,2,3,4-Tetrachloronaphthalene                                |                            | 2                    |                     |
| Tetraethyllead (as Pb)  |                            | 0.100                |                     |
| Tetrafluoromethane  |                            |                      | low toxicity        |
| Tetrahydrofuran   | 200                        | 590                  |                     |
| Tetramethyllead (as Pb)                                       |                            | 0.150                |                     |
| Tetramethylsuccinonitrile                                     | 0.5                        | 3                    |                     |
| Tetranitromethane   | 1                          | 8                    |                     |
| Thionyl chloride  | 1                          |                      |                     |
| Thiram  |                            | 5                    |                     |
| Toluene   | 50                         | 188                  |                     |
| Toluene-2,4-diisocyanate                                      | 0.02                       | 0.14                 |                     |
| <i>o</i> -Toluidine (also <i>m</i> -, <i>p</i> -)             | 2                          | 8.8                  |                     |
| Tribromomethane   | 0.5                        | 5.2                  |                     |
| Tributyl phosphate  | 0.2                        | 2.2                  |                     |
| 1,2,4-Trichlorobenzene  | 5                          |                      |                     |
| 1,1,1-Trichloroethane   | 350                        | 1910                 |                     |
| 1,1,2-Trichloroethane   | 10                         | 55                   | carcinogen          |
| Trichloroethylene   | 50                         | 270                  | carcinogen          |
| Trichlorofluoromethane  | 1000                       | 5600                 |                     |
| Trichloromethane  | 10                         | 49                   | carcinogen          |
| 1,2,3-Trichloropropane  | 10                         | 60                   |                     |
| 1,1,2-Trichlorotrifluoroethane                                | 1000                       |                      |                     |
| Tri- <i>o</i> -cresol phosphate (also <i>m</i> -, <i>p</i> -) |                            | 0.1                  |                     |
| Triethanolamine   | 0.5                        |                      |                     |
| Triethylamine   | 1                          |                      |                     |
| Trifluorobromomethane (Freon 13B1)                            | 1000                       | 6100                 |                     |
| 1,1,2-Trifluorotrichloroethane                                | 1000                       | 7600                 |                     |
| Triiodomethane  | 0.6                        |                      |                     |
| Trimethylamine  | 5                          | 12                   | highly toxic        |
| 1,2,3-Trimethylbenzene  | 25                         | 123                  |                     |
| 1,2,4-Trimethylbenzene (pseudocumene)                         | 25                         | 123                  |                     |
| 1,3,5-Trimethylbenzene (mesitylene)                           | 25                         | 123                  |                     |
| Trinitrotoluene (TNT)   |                            | 1.5                  |                     |
| Triphenyl phosphate   |                            | 3                    |                     |
| Turpentine  | 100                        | 560                  |                     |
| Vinyl acetate   | 10                         | 35                   | carcinogen          |
| Vinyl methyl ether  |                            |                      | probably anesthetic |
| Warfarin  |                            | 0.1                  |                     |
| <i>o</i> -Xylene (also <i>m</i> -, <i>p</i> -)                | 100                        | 434                  |                     |
| 2,3-Xylidine (also 2,4-, 2,5-, 2,6-, 3,4-, 3,5-)              | 0.5                        | 2.5                  |                     |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals

| Chemical             | Keep out of contact with   |
|----------------------|--|
| Acetic acid          | Chromium(VI) oxide, chlorosulfonic acid, ethylene glycol, ethyleneimine, hydroxyl compounds, nitric acid, oleum, perchloric acid, peroxides, permanganates, potassium <i>tert</i> -butoxide, $\text{PCl}_3$  |
| Acetylene            | Bromine, chlorine, brass, copper and copper salts, fluorine, mercury and mercury salts, nitric acid, silver and silver salts, alkali hydrides, potassium metal   |
| Alkali metals        | Moisture, acetylene, metal halides, ammonium salts, oxygen and oxidizing agents, halogens, carbon tetrachloride, carbon, carbon dioxide, carbon disulfide, chloroform, chlorinated hydrocarbons, ethylene oxide, boric acid, sulfur, tellurium   |
| Aluminum             | Chlorinated hydrocarbons, halogens, steam  |
| Ammonia, anhydrous   | Mercury, halogens, hypochlorites, chlorites, chlorine(I) oxide, hydrofluoric acid (anhydrous), hydrogen peroxide, chromium(VI) oxide, nitrogen dioxide, chromyl(VI) chloride, sulfinyl chloride, magnesium perchlorate, peroxodisulfates, phosphorus pentoxide, acetaldehyde, ethylene oxide, acrolein, gold(III) chloride   |
| Ammonium nitrate     | Acids, metal powders, flammable liquids, chlorates, nitrites, sulfur, finely divided organic or combustible materials, perchlorates, urea  |
| Ammonium perchlorate | Hot copper tubing, sugar, finely divided organic or combustible materials, potassium periodate and permanganate, powdered metals, carbon, sulfur   |
| Aniline              | Nitric acid, peroxides, oxidizing materials, acetic anhydride, chlorosulfonic acid, oleum, ozone   |
| Benzoyl peroxide     | Direct sunlight, sparks and open flames, shock and friction, acids, alcohols, amines, ethers, reducing agents, polymerization catalysts, metallic naphthenates   |
| Bromine              | Ammonia, carbides, dimethylformamide, fluorine, ozone, olefins, reducing materials including many metals, phosphine, silver azide  |
| Calcium carbide      | Moisture, selenium, silver nitrate, sodium peroxide, tin(II) chloride, potassium hydroxide plus chlorine, HCl gas, magnesium   |
| Carbon, activated    | Calcium hypochlorite, all oxidizing agents, unsaturated oils   |
| Chlorates            | Ammonium salts, acids, metal powders, sulfur, finely divided organic or combustible materials, cyanides, metal sulfides, manganese dioxide, sulfur dioxide, organic acids  |
| Chlorine             | Ammonia, acetylene, alcohols, alkanes, benzene, butadiene, carbon disulfide, dibutyl phthalate, ethers, fluorine, glycerol, hydrocarbons, hydrogen, sodium carbide, finely divided metals, metal acetylides and carbides, nitrogen compounds, nonmetals, nonmetal hydrides, phosphorus compounds, polychlorobiphenyl, silicones, steel, sulfides, synthetic rubber, turpentine |
| Chlorine dioxide     | Ammonia, carbon monoxide, hydrogen, hydrogen sulfide, methane, mercury, nonmetals, phosphine, phosphorus pentachloride   |
| Chlorites            | Ammonia, organic matter, metals  |
| Chloroform           | Aluminum, magnesium, potassium, sodium, aluminum chloride, ethylene, powerful oxidants   |
| Chlorosulfonic acid  | Saturated and unsaturated acids, acid anhydrides, nitriles, acrolein, alcohols, ammonia, esters, HCl, HF, ketones, hydrogen peroxide, metal powders, nitric acid, organic materials, water   |
| Chromic(VI) acid     | Acetic acid, acetic anhydride, acetone, alcohols, alkali metals, ammonia, dimethylformamide, camphor, glycerol, hydrogen sulfide, phosphorus, pyridine, selenium, sulfur, turpentine, flammable liquids in general   |
| Cobalt               | Acetylene, hydrazinium nitrate, oxidants   |
| Copper               | Acetylene and alkynes, ammonium nitrate, azides, bromates, chlorates, iodates, chlorine, ethylene oxide, fluorine, peroxides, hydrogen sulfide, hydrazinium nitrate  |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals (*Continued*)

| Chemical              | Keep out of contact with   |
|-----------------------|--|
| Copper(II) sulfate    | Hydroxylamine, magnesium   |
| Cumene hydroperoxide  | Acids (inorganic or organic)   |
| Cyanides              | Acids, water or steam, fluorine, magnesium, nitric acid and nitrates, nitrites   |
| Cyclohexanol          | Oxidants   |
| Cyclohexanone         | Hydrogen peroxide, nitric acid   |
| Decaborane-14         | Dimethyl sulfoxide, ethers, halocarbons  |
| Diazomethane          | Alkali metals, calcium sulfate   |
| 1,1-Dichloroethylene  | Air, chlorotrifluoroethylene, ozone, perchloryl fluoride   |
| Dimethylformamide     | Halocarbons, inorganic and organic nitrates, bromine, chromium(VI) oxide, aluminum trimethyl, phosphorus trioxide  |
| 1,1-Dimethylhydrazine | Air, hydrogen peroxide, nitric acid, nitrous oxide   |
| Dimethylsulfoxide     | Acyl and aryl halides, boron compounds, bromomethane, nitrogen dioxide, magnesium perchlorate, periodic acid, silver difluoride, sodium hydride, sulfur trioxide   |
| Dinitrobenzenes       | Nitric acid  |
| Dinitrotoluenes       | Nitric acid  |
| 1,4-Dioxane           | Silver perchlorate   |
| Esters                | Nitrates   |
| Ethylamine            | Cellulose, oxidizers   |
| Ethers                | Oxidizing materials, boron triiodide   |
| Ethylene              | Aluminum trichloride, carbon tetrachloride, chlorine, nitrogen oxides, tetrafluoroethylene   |
| Ethylene oxide        | Acids and bases, alcohols, air, 1,3-nitroaniline, aluminum chloride, aluminum oxide, ammonia, copper, iron chlorides and oxides, magnesium perchlorate, mercaptans, potassium, tin chlorides, alkane thiols        |
| Ethyl ether           | Liquid air, chlorine, chromium(VI) oxide, lithium aluminum hydride, ozone, perchloric acid, peroxides  |
| Ethyl sulfate         | Oxidizing materials, water   |
| Flammable liquids     | Ammonium nitrate, chromic acid, the halogens, hydrogen peroxide, nitric acid   |
| Fluorine              | Isolate from everything; only lead and nickel resist prolonged attack  |
| Formamide             | Iodine, pyridine, sulfur trioxide  |
| Freon 113             | Aluminum, barium, lithium, samarium, NaK alloy, titanium   |
| Glycerol              | Acetic anhydride, hypochlorites, chromium(VI) oxide, perchlorates, alkali peroxides, sodium hydride  |
| Hydrazine             | Alkali metals, ammonia, chlorine, chromates and dichromates, copper salts, fluorine, hydrogen peroxide, metallic oxides, nickel, nitric acid, liquid oxygen, zinc diethyl  |
| Hydrides              | Powerful oxidizing agents, moisture  |
| Hydrocarbons          | Halogens, chromium(VI) oxide, peroxides  |
| Hydrogen              | Halogens, lithium, oxidants, lead trifluoride  |
| Hydrogen bromide      | Fluorine, iron(III) oxide, ammonia, ozone  |
| Hydrogen chloride     | Acetic anhydride, aluminum, 2-aminoethanol, ammonia, chlorosulfonic acid, ethylenediamine, fluorine, metal acetylides and carbides, oleum, perchloric acid, potassium permanganate, sodium, sulfuric acid          |
| Hydrogen fluoride     | Acetic anhydride, 2-aminoethanol, ammonia, arsenic trioxide, chlorosulfonic acid, ethylenediamine, ethyleneimine, fluorine, HgO, oleum, phosphorus trioxide, propylene oxide, sodium, sulfuric acid, vinyl acetate |
| Hydrogen iodide       | Fluorine, nitric acid, ozone, metals   |
| Hydrogen peroxide     | Copper, chromium, iron, most metals or their salts, alcohols, acetone, organic materials, flammable liquids, combustible materials   |
| Hydrogen selenide     | Hydrogen peroxide, nitric acid   |
| Hydrogen sulfide      | Fuming nitric acid, oxidizing gases, peroxides   |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals (*Continued*)

| Chemical                  | Keep out of contact with  |
|---------------------------|---|
| Hydroquinone              | Sodium hydroxide  |
| Hydroxylamine             | Barium oxide and peroxide, carbonyls, chlorine, copper(II) sulfate, dichromates, lead dioxide, phosphorus trichloride and pentachloride, permanganates, pyridine, sodium, zinc  |
| Hypochlorites, salts of   | Urea, amines, anthracene, carbon, carbon tetrachloride, ethanol, glycerol, mercaptans, organic sulfides, sulfur, thiols   |
| Indium                    | Acetonitrile, nitrogen dioxide, mercury(II) bromide, sulfur   |
| Iodine                    | Acetaldehyde, acetylene, aluminum, ammonia (aqueous or anhydrous), antimony, bromine pentafluoride, carbides, cesium oxide, chlorine, ethanol, fluorine, formamide, lithium, magnesium, phosphorus, pyridine, silver azide, sulfur trioxide       |
| Iodine monochloride       | Aluminum foil, organic matter, metal sulfides, phosphorus, potassium, rubber, sodium  |
| Iodoform                  | Acetone, lithium, mercury(II) oxide, mercury(I) chloride, silver nitrate  |
| Iodomethane               | Silver chlorite, sodium   |
| Iron disulfide            | Water, powdered pyrites   |
| Isothiourea               | Acrylaldehyde, hydrogen peroxide, nitric acid   |
| Ketones                   | Aldehydes, nitric acid, perchloric acid   |
| Lactonitrile              | Oxidizing materials   |
| Lead                      | Ammonium nitrate, chlorine trifluoride, hydrogen peroxide, sodium azide and carbide, zirconium, oxidants  |
| Lead(II) azide            | Calcium stearate, copper, zinc, brass, carbon disulfide   |
| Lead chromate             | Iron hexacyanoferrate(4-)   |
| Lead dioxide              | Aluminum carbide, hydrogen peroxide, hydrogen sulfide, hydroxylamine, nitroalkanes, nitrogen compounds, nonmetal halides, peroxyformic acid, phosphorus, phosphorus trichloride, potassium, sulfur, sulfur dioxide, sulfides, tungsten, zirconium |
| Lead(II) oxide            | Chlorinated rubber, chlorine, ethylene, fluorine, glycerol, metal acetylides, perchloric acid   |
| Lead(II,IV) oxide         | Same as for lead dioxide  |
| Lithium hydride           | Nitrous oxide, oxygen   |
| Magnesium                 | Air, beryllium fluoride, ethylene oxide, halogens, halocarbons, HI, metal cyanides, metal oxides, metal oxosalts, methanol, oxidants, peroxides, sulfur, tellurium  |
| Maleic anhydride          | Alkali metals, amines, KOH, NaOH, pyridine  |
| Manganese dioxide         | Aluminum, hydrogen sulfide, oxidants, potassium azide, hydrogen peroxide, peroxosulfuric acid, sodium peroxide  |
| Mercaptans                | Powerful oxidizers  |
| Mercury                   | Acetylenic compounds, chlorine, fulminic acid, ammonia, ethylene oxide, metals, methyl azide, oxidants, tetracarbonylnickel   |
| Mercury(II) cyanide       | Fluorine, hydrogen cyanide, magnesium, sodium nitrite   |
| Mercury(I) nitrate        | Phosphorus  |
| Mercury(II) nitrate       | Acetylene, aromatics, ethanol, hypophosphoric acid, phosphine, unsaturated organic compounds  |
| Mercury(II) oxide         | Chlorine, hydrazine hydrate, hydrogen peroxide, hypophosphorous acid, magnesium, phosphorus, sulfur, butadiene, hydrocarbons, methanethiol  |
| Mesityl oxide             | 2-Aminoethanol, chlorosulfonic acid, nitric acid, ethylenediamine, sulfuric acid  |
| Methanol                  | Beryllium dihydride, chloroform, oxidants, potassium <i>tert</i> -butoxide  |
| Methylamine               | Nitromethane  |
| <i>N</i> -Methylformamide | Benzenesulfonyl chloride  |
| Methyl isobutyl ketone    | Potassium <i>tert</i> -butoxide   |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals (*Continued*)

| Chemical                 | Keep out of contact with  |
|--------------------------|---|
| Methyl methacrylate      | Air, benzoyl peroxide   |
| 4-Methylnitrobenzene     | Sulfuric acid, tetranitromethane  |
| 2-Methylpyridine         | Hydrogen peroxide, iron(II) sulfate, sulfuric acid  |
| Methylsodium             | 4-Chloronitrobenzene  |
| Molybdenum trioxide      | Chlorine trifluoride, interhalogens, metals   |
| Naphthalene              | Chromium trioxide, dinitrogen pentaoxide  |
| 2-Naphthol               | Antipyrine, camphor, phenol, iron(III) salts, menthol, oxidizing materials, permanganates, urethane   |
| Neodymium                | Phosphorus  |
| Nickel                   | Aluminum, aluminum(III) chloride, ethylene, 1,4-dioxan, hydrogen, methanol, nonmetals, oxidants, sulfur compounds   |
| Nickel carbonyl          | Air, bromine, oxidizing materials   |
| Niobium                  | Bromine trifluoride, chlorine, fluorine   |
| Nitrates                 | Aluminum, BP, cyanides, esters, phosphorus, tin(II) chloride, sodium hypophosphite, thiocyanates  |
| Nitric acid, fuming      | Organic matter, nonmetals, most metals, ammonia, chlorosulfonic acid, chromium trioxide, cyanides, dichromates, hydrazines, hydrides, HCN, HI, hydrogen sulfide, sulfur dioxide, sulfur halides, sulfuric acid, flammable liquids and gases |
| Nitric oxide             | Aluminum, BaO, boron, carbon disulfide, chromium, many chlorinated hydrocarbons, fluorine, hydrocarbons, ozone, phosphine, phosphorus, hydrazine, acetic anhydride, ammonia, chloroform, Fe, K, Mg, Mn, Na, sulfur                          |
| Nitrites                 | Organic nitrites in contact with ammonium salts, cyanides   |
| Nitrobenzene             | Nitric acid, nitrous oxide, silver perchlorate  |
| Nitroethane              | Hydroxides, hydrocarbons, metal oxides  |
| Nitrogen trichloride     | Ammonia, As, hydrogen sulfide, nitrogen dioxide, organic matter, ozone, phosphine, phosphorus, KCN, KOH, Se, dibutyl ether  |
| Nitrogen dioxide         | Cyclohexane, fluorine, formaldehyde, alcohols, nitrobenzene, petroleum, toluene   |
| Nitrogen triiodide       | Acids, bromine, chlorine, hydrogen sulfide, ozone   |
| $\alpha$ -Nitroguanidine | Complex salts of mercury and silver   |
| Nitromethane             | Acids, alkylmetal halides, hydroxides, hydrocarbons, organic amines, formaldehyde, nitric acid, perchlorates  |
| 1-Nitropropane           | See under Nitromethane; chlorosulfonic acid, oleum  |
| Nitrosyl fluoride        | Haloalkenes, metals, nonmetals  |
| Nitrosyl perchlorate     | Acetones, amines, diethyl ether, metal salts, organic materials   |
| Nitrourea                | Mercury(II) and silver salts  |
| Nitrous acid             | Phosphine, phosphorus trichloride, silver nitrate, semicarbazone  |
| Nitryl chloride          | Ammonia, sulfur trioxide, tin(IV) bromide and iodide  |
| Oxalic acid              | Furfuryl alcohol, silver, mercury, sodium chlorate, sodium chlorite, sodium hypochlorite  |
| Oxygen                   | Acetaldehyde, acetone, alcohols, alkali metals, alkaline earth metals, Al-Ti alloys, ether, carbon disulfide, halocarbons, hydrocarbons, metal hydrides, 1,3,5-trioxane   |
| Ozone                    | Alkenes, aromatic compounds, bromine, diethyl ether, ethylene, HBr, HI, nitric oxide, nitrogen dioxide, rubber, stibine   |
| Palladium                | Arsenic, carbon, ozonides, sulfur, sodium tetrahydridoborate  |
| Paraformaldehyde         | Liquid oxygen   |
| Paraldehyde              | Alkalies, HCN, iodides, nitric acid, oxidizers  |
| Pentaborane-9            | Dimethylsulfoxide   |
| Pentacarbonyliron        | Acetic acid, nitric oxide, transition metal halides, water, zinc  |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals (*Continued*)

| Chemical                        | Keep out of contact with   |
|---------------------------------|--|
| 2-Pentanone                     | Bromine trifluoride  |
| 3-Pentanone                     | Hydrogen peroxide, nitric acid   |
| Perchlorates                    | Carbonaceous materials, finely divided metals particularly magnesium and aluminum, sulfur, benzene, olefins, ethanol, sulfur, sulfuric acid  |
| Perchloric acid                 | Acetic acid, acetic anhydride, alcohols, antimony compounds, azo pigments, bismuth and its alloys, methanol, carbonaceous materials, carbon tetrachloride, cellulose, dehydrating agents, diethyl ether, glycols and glycolethers, HCl, HI, hypophosphites, ketones, nitric acid, pyridine, steel, sulfoxides, sulfuric acid |
| Permanganates                   | All reducing agents, organic materials   |
| Peroxides                       | Reducing agents, organic materials, thiocyanates   |
| Peroxoacetic acid               | Acetic anhydride, olefins, organic matter  |
| Peroxo benzoic acid             | Olefins, reducing materials  |
| Peroxoformic acid               | Metals and nonmetals, organic materials  |
| Peroxo sulfuric acid            | Acetone, alcohols, aromatic compounds, catalysts   |
| Phenol                          | Butadiene, peroxodisulfuric acid, peroxosulfuric acid, aluminum chloride plus nitrobenzene   |
| Phenylhydrazine                 | Lead dioxide, oxidizers  |
| Phosgene                        | Aluminum, alkali metals, 2-propanol  |
| Phosphine                       | Air, boron trichloride, bromine, chlorine, nitric acid, nitrogen oxides, nitrous acid, oxygen, silver nitrate  |
| Phosphorus pentachloride        | Aluminum, chlorine, chlorine dioxide, chlorine trioxide, fluorine, magnesium oxide, nitrobenzene, diphosphorus trioxide, potassium, sodium, urea, water  |
| Phosphorus pentafluoride        | Water or steam   |
| Phosphorus pentasulfide         | Air, alcohols, water   |
| Phosphorus pentoxide            | Formic acid, HF, inorganic bases, metals, oxidants, water  |
| Phosphorus, red                 | Organic materials  |
| Phosphorus tribromide           | Potassium, ruthenium tetroxide, sodium, water  |
| Phosphorus trichloride          | Acetic acid, aluminum, chromyl dichloride, dimethylsulfoxide, hydroxylamine, lead dioxide, nitric acid, nitrous acid, organic matter, potassium, sodium water  |
| Phosphorus, white               | Air, oxidants of all types, halogens, metals   |
| Phosphoryl chloride             | Carbon disulfide, <i>N,N</i> -dimethylformamide, 2,5-dimethylpyrrole, 2,6-dimethylpyridine 1-oxide, dimethylsulfoxide, water, zinc   |
| Phthalic acid                   | Nitric acid, sodium nitrite  |
| Piperazine                      | Oxidizers  |
| Platinum                        | Acetone, arsenic, hydrazine, lithium, peroxosulfuric acid, phosphorus, selenium, tellurium   |
| Potassium                       | <i>See under</i> Alkali metals   |
| Potassium <i>tert</i> -butoxide | Organic compounds, sulfuric acid   |
| Potassium hydride               | Air, chlorine, acetic acid, acrolein, acrylonitrile, maleic anhydride, nitroparaffins, <i>N</i> -nitrosomethylurea, tetrahydrofuran, water   |
| Potassium perchlorate           | Aluminum plus magnesium, carbon, nickel plus titanium, reducing agents, sulfur, sulfuric acid  |
| Potassium permanganate          | Organic or readily oxidizable materials  |
| Potassium sodium alloy          | Air, carbon dioxide, carbon disulfide, halocarbons, metal oxides   |
| 2-Propyn-1-ol                   | Alkali metals, mercury(II) sulfate, oxidizing materials, phosphorus pentoxide, sulfuric acid   |
| Pyridine                        | Chlorosulfonic acid, chromium trioxide, formamide, maleic anhydride, nitric acid, oleum, perchromates, silver perchlorate, sulfuric acid   |
| Pyrrolidine                     | Oxidizing materials  |

**TABLE 11.50** Some Common Reactive and Incompatible Chemicals (*Continued*)

| Chemical              | Keep out of contact with  |
|-----------------------|---|
| Quinoline             | Dinitrogen tetroxide, linseed oil, maleic anhydride, thionyl chloride   |
| Salicylic acid        | Iodine, iron salts, lead acetate  |
| Silicon               | Alkali carbonates, calcium, chlorine, cobalt(II) fluoride, manganese trifluoride, oxidants, silver fluoride, sodium-potassium alloy   |
| Silver                | Acetylene, ammonium compounds, ethyleneimine, hydrogen peroxide, oxalic acid, sulfuric acid, tartaric acid  |
| Sodium                | <i>See under Alkali metals</i>  |
| Sodium peroxide       | Glacial acetic acid, acetic anhydride, aniline, benzene, benzaldehyde, carbon disulfide, diethyl ether, ethanol or methanol, ethylene glycol, ethyl acetate, furfural, glycerol, metals, methyl acetate, organic matter |
| Sulfides              | Acids, powerful oxidizers, moisture   |
| Sulfur                | Oxidizing materials, halogens   |
| Sulfur dioxide        | Halogens, metal oxides, polymeric tubing, potassium chlorate, sodium hydride  |
| Sulfuric acid         | Chlorates, metals, HCl, organic materials, perchlorates, permanganates, water   |
| Sulfuryl dichloride   | Alkalies, diethyl ether, dimethylsulfoxide, dinitrogen tetroxide, lead dioxide, phosphorus  |
| Tellurium             | Halogens, metals  |
| Tetrahydrofuran       | Tetrahydroaluminates, KOH, NaOH   |
| Tetranitroaniline     | Reducing materials  |
| Tetranitromethane     | Aluminum, cotton, aromatic nitro compounds, hydrocarbons, cotton, toluene   |
| Thiocyanates          | Chlorates, nitric acid, peroxides   |
| Thionyl chloride      | Ammonia, dimethylsulfoxide, linseed oil, quinoline, sodium  |
| Thiophene             | Nitric acid   |
| Thymol                | Acetanilide, antipyrine, camphor, chlorohydrate, menthol, quinine sulfate, urethane   |
| Tin(II) chloride      | Boron trifluoride, ethylene oxide, hydrazine hydrate, nitrates, Na, K, hydrogen peroxide  |
| Tin(IV) chloride      | Alkyl nitrates, ethylene oxide, K, Na turpentine  |
| Titanium              | Aluminum, boron trifluoride, carbon dioxide, CuO, halocarbons, halogens, PbO, nitric acid, potassium chlorate, potassium nitrate, potassium permanganate, steam at high temperatures, water                             |
| Toluene               | Sulfuric plus nitric acids, nitrogen dioxide, silver perchlorate, uranium hexafluoride  |
| Toluidines            | Nitric acid   |
| 2,4,6-Trinitrotoluene | Sodium dichromate, sulfuric acid  |
| 1,3,5-Trioxane        | Oxidizing materials, acids  |
| Urea                  | Sodium nitrite, phosphorus pentachloride  |
| Vinylidene chloride   | Chlorosulfonic acid, nitric acid, oleum   |

**TABLE 11.51** Chemicals Recommended for Refrigerated Storage

| A. Due to chemical decomposition or polymerization |  |
|--|--|
| Acetaldehyde                                       | Isoprene                               |
| Acrolein   | Lecithin                               |
| Adenosinetriphosphoric acid                        | Mercaptoacetic acid                    |
| Bromacetaldehyde, diethyl acetal                   | Methyl acrylate                        |
| Bromosuccinimide                                   | 2-Methyl-1-butene                      |
| 3-Buten-2-one                                      | Methylenedi-1,4-phenylene diisocyanate |
| <i>tert</i> -Butyl hydroperoxide                   | 4-Methyl-1-pentene                     |
| 2-Chlorocyclohexanone                              | $\alpha$ -Methylstyrene                |
| Cupferron  | 1-Naphthyl isocyanate                  |
| 1,3-Cyclohexadiene                                 | 1-Pentene                              |
| 1,3-Dihydroxy-2-propanone                          | Isopentyl acetate                      |
| Divinylbenzene                                     | Pyruvic acid                           |
| Ethyl methacrylate, monomer                        | Styrene, stabilized                    |
| Glutathione  | Tetramethylsilane                      |
| Glycidol   | Thioacetamide                          |
| Histamine, base                                    | Veratraldehyde                         |
| Hydrocinnamaldehyde                                | Vitamin E (and the acetate)            |
| B. Due to flammability and high volatility         |  |
| Acetaldehyde                                       | Iodomethane                            |
| Bromoethane  | Isoprene                               |
| <i>tert</i> -Butylamine                            | Isopropylamine                         |
| Carbon disulfide                                   | Methylal                               |
| 1-Chloropropane                                    | 2-Methylbutane                         |
| 3-Chloropropane                                    | 2-Methyl-2-butene                      |
| Cyclopentane                                       | Methyl formate                         |
| Diethyl ether                                      | Pentane                                |
| 2,2-Dimethylbutane                                 | Propylamine                            |
| Dimethyl sulfide                                   | Propylene oxide                        |
| Furan  | Trichlorosilane                        |

**TABLE 11.52** Chemicals Which Polymerize or Decompose on Extended Refrigeration

|  |                   |
|--|-------------------|
| Formaldehyde                           | Sodium methoxide  |
| Hydrogen peroxide                      | Sodium nitrate    |
| Sodium chlorite [sodium chlorate (IV)] | Sodium peroxide   |
| Sodium chromate(VI)                    | Strontium nitrate |
| Sodium dithionite                      | Urea              |
| Sodium ethoxide                        |                   |

## 11.8 SIEVES AND SCREENS

**TABLE 11.53** U.S. Standard Sieve Series

| Sieve no. | Sieve opening |        | Sieve no. | Sieve opening |        |
|-----------|---------------|--------|-----------|---------------|--------|
|           | mm            | inch   |           | mm            | inch   |
|           | 125           | 5.00   | 10        | 2.00          | 0.0787 |
|           | 106           | 4.24   | 12        | 1.70          | 0.0661 |
|           | 90            | 3.50   | 14        | 1.40          | 0.0555 |
|           | 75            | 3.00   | 16        | 1.18          | 0.0469 |
|           | 63            | 2.50   | 18        | 1.00          | 0.0394 |
|           | 53            | 2.12   | 20        | 0.850         | 0.0331 |
|           | 45            | 1.75   | 25        | 0.710         | 0.0278 |
|           | 37.5          | 1.50   | 30        | 0.600         | 0.0234 |
|           | 31.5          | 1.25   | 35        | 0.500         | 0.0197 |
|           | 26.5          | 1.06   | 40        | 0.425         | 0.0165 |
|           | 22.4          | 0.875  | 45        | 0.355         | 0.0139 |
|           | 19.0          | 0.75   | 50        | 0.300         | 0.0117 |
|           | 16.0          | 0.625  | 60        | 0.250         | 0.0098 |
|           | 13.2          | 0.530  | 70        | 0.212         | 0.0083 |
|           | 11.2          | 0.438  | 80        | 0.180         | 0.0070 |
|           | 9.5           | 0.375  | 100       | 0.150         | 0.0059 |
|           | 8.0           | 0.312  | 120       | 0.125         | 0.0049 |
|           | 6.7           | 0.265  | 140       | 0.106         | 0.0041 |
| 3.5       | 5.60          | 0.223  | 170       | 0.090         | 0.0035 |
| 4         | 4.75          | 0.187  | 200       | 0.075         | 0.0029 |
| 5         | 4.00          | 0.157  | 230       | 0.063         | 0.0025 |
| 6         | 3.35          | 0.132  | 270       | 0.053         | 0.0021 |
| 7         | 2.80          | 0.111  | 325       | 0.045         | 0.0017 |
| 8         | 2.36          | 0.0937 | 400       | 0.038         | 0.0015 |

Specifications are from ASTM E.11-81/ISO 565. The sieve numbers are the approximate number of openings per linear inch.

## 11.9 THERMOMETRY

### 11.9.1 Temperature and Its Measurement

The new international temperature scale, known as ITS-90, was adopted in September 1989. However, neither the definition of thermodynamic temperature nor the definition of the kelvin or the Celsius temperature scales has changed; it is the way in which we are to realize these definitions that has changed. The changes concern the recommended thermometers to be used in different regions of the temperature scale and the list of secondary standard fixed points. The changes in temperature determined using ITS-90 from the previous IPTS-68 are always less than 0.4 K, and almost always less than 0.2 K, over the range 0–1300 K.

The ultimate definition of thermodynamic temperature is in terms of  $pV$  (pressure  $\times$  volume) in a gas thermometer extrapolated to low pressure. The kelvin (K), the unit of thermodynamic temperature, is defined by specifying the temperature of one fixed point on the scale—the triple point

of water which is defined to be 273.16 K. The Celsius temperature scale ( $^{\circ}\text{C}$ ) is defined by the equation

$$^{\circ}\text{C} = \text{K} - 273.15$$

where the freezing point of water at 1 atm is 273.15 K.

The fixed points in the ITS-90 are given in Table 11.39. Platinum resistance thermometers are recommended for use between 14 K and 1235 K (the freezing point of silver), calibrated against the fixed points. Below 14 K either the vapor pressure of helium or a constant-volume gas thermometer is to be used. Above 1235 K radiometry is to be used in conjunction with the Planck radiation law,

$$L_{\lambda} = c_1 \lambda^{-5} (e^{c_2/\lambda T} - 1)^{-1}$$

where  $L_{\lambda}$  is the spectral radiance at wavelength  $\lambda$ . The first radiation constant,  $c_1$ , is  $3.741\,83 \times 10^{-16} \text{ W} \cdot \text{m}^2$  and the second radiation constant,  $c_2$ , has a value of  $0.014\,388 \text{ m} \cdot \text{K}$ .

**TABLE 11.54** Fixed Points in the ITS-90

| Fixed points  | $T$ , K  | $t$ , $^{\circ}\text{C}$ |
|---|----------|--------------------------|
| Triple point of hydrogen                                  | 13.8033  | −259.3467                |
| Boiling point of hydrogen at 33 321.3 Pa                  | 17.035   | −256.115                 |
| Boiling point of hydrogen at 101 292 Pa                   | 20.27    | −252.88                  |
| Triple point of neon                                      | 24.5561  | −248.5939                |
| Triple point of oxygen                                    | 54.3584  | −218.7916                |
| Triple point of argon                                     | 83.8058  | −189.3442                |
| Triple point of mercury                                   | 234.3156 | −38.8344                 |
| Triple point of water                                     | 273.16   | 0.01                     |
| Melting point of gallium                                  | 302.9146 | 29.7646                  |
| Freezing point of indium                                  | 429.7458 | 156.5985                 |
| Freezing point of tin                                     | 505.078  | 231.928                  |
| Freezing point of zinc                                    | 692.677  | 419.527                  |
| Freezing point of aluminum                                | 933.473  | 660.323                  |
| Freezing point of silver                                  | 1234.93  | 961.78                   |
| Freezing point of gold                                    | 1337.33  | 1064.18                  |
| Freezing point of copper                                  | 1357.77  | 1084.62                  |
| Secondary reference points to extend the scale (IPTS-68): |          |                          |
| Freezing point of platinum                                | 2042     | 1769                     |
| Freezing point of rhodium                                 | 2236     | 1963                     |
| Freezing point of iridium                                 | 2720     | 2447                     |
| Melting point of tungsten                                 | 3660     | 3387                     |

## 11.10 THERMOCOUPLES

The thermocouple reference data in Tables 11.55 to 11.63 give the thermoelectric voltage in millivolts with the reference junction at  $0^{\circ}\text{C}$ . Note that the temperature for a given entry is obtained by adding the corresponding temperature in the top row to that in the left-hand column, regardless of whether the latter is positive or negative.

The noble metal thermocouples, Types B, R, and S, are all platinum or platinum-rhodium thermocouples and hence share many of the same characteristics. Metallic vapor diffusion at high temperatures can readily change the platinum wire calibration, hence platinum wires should only be used inside a nonmetallic sheath such as high-purity alumina.

Type B thermocouples (Table 11.56) offer distinct advantages of improved stability, increased mechanical strength, and higher possible operating temperatures. They have the unique advantage that the reference junction potential is almost immaterial, as long as it is between 0°C and 40°C. Type B is virtually useless below 50°C because it exhibits a double-value ambiguity from 0°C to 42°C.

Type E thermoelements (Table 11.57) are very useful down to about liquid hydrogen temperatures and may even be used down to liquid helium temperatures. They are the most useful of the commercially standardized thermocouple combinations for subzero temperature measurements because of their high Seebeck coefficient (58  $\mu\text{V}/^\circ\text{C}$ ), low thermal conductivity, and corrosion resistance. They also have the largest Seebeck coefficient (voltage response per degree Celsius) above 0°C of any of the standardized thermocouples which makes them useful for detecting small temperature changes. They are recommended for use in the temperature range from  $-250$  to  $871^\circ\text{C}$  in oxidizing or inert atmospheres. They should not be used in sulfurous, reducing, or alternately reducing and oxidizing atmospheres unless suitably protected with tubes. They should not be used in vacuum at high temperatures for extended periods of time.

Type J thermocouples (Table 11.58) are one of the most common types of industrial thermocouples because of the relatively high Seebeck coefficient and low cost. They are recommended for use in the temperature range from 0 to  $760^\circ\text{C}$  (but never above  $760^\circ\text{C}$  due to an abrupt magnetic transformation that can cause decalibration even when returned to lower temperatures). Use is permitted in vacuum and in oxidizing, reducing, or inert atmospheres, with the exception of sulfurous atmospheres above  $500^\circ\text{C}$ . For extended use above  $500^\circ\text{C}$ , heavy-gauge wires are recommended. They are not recommended for subzero temperatures. These thermocouples are subject to poor conformance characteristics because of impurities in the iron.

The Type K thermocouple (Table 11.59) is more resistant to oxidation at elevated temperatures than the Type E, J, or T thermocouple, and consequently finds wide application at temperatures above  $500^\circ\text{C}$ . It is recommended for continuous use at temperatures within the range  $-250$  to  $1260^\circ\text{C}$  in inert or oxidizing atmospheres. It should not be used in sulfurous or reducing atmospheres, or in vacuum at high temperatures for extended times.

The Type N thermocouple (Table 11.60) is similar to Type K but it has been designed to minimize some of the instabilities in the conventional Chromel-Alumel combination. Changes in the alloy content have improved the order/disorder transformations occurring at  $500^\circ\text{C}$  and a higher silicon content of the positive element improves the oxidation resistance at elevated temperatures.

The Type R thermocouple (Table 11.61) was developed primarily to match a previous platinum–10% rhodium British wire which was later found to have 0.34% iron impurity in the rhodium. Comments on Type S also apply to Type R.

The Type S thermocouple (Table 11.62) is so stable that it remains the standard for determining temperatures between the antimony point ( $630.74^\circ\text{C}$ ) and the gold point ( $1064.43^\circ\text{C}$ ). The other fixed point used is that of silver. The Type S thermocouple can be used from  $-50^\circ\text{C}$  continuously up to about  $1400^\circ\text{C}$ , and intermittently at temperatures up to the freezing point of platinum ( $1769^\circ\text{C}$ ). The thermocouple is most reliable when used in a clean oxidizing atmosphere, but may also be used in inert gaseous atmospheres or in a vacuum for short periods of time. It should not be used in reducing atmospheres, nor in those containing metallic vapor (such as lead or zinc), nonmetallic vapors (such as arsenic, phosphorus, or sulfur), or easily reduced oxides, unless suitably protected with nonmetallic protecting tubes.

The Type T thermocouple (Table 11.63) is popular for the temperature region below  $0^\circ\text{C}$  (but see under Type E). It can be used in vacuum, or in oxidizing, reducing, or inert atmospheres.

**TABLE 11.55** Thermoelectric Values in Millivolts at Fixed Points for Various Thermocouples

| <i>Abbreviations Used in the Table</i> |            |                           |          |                   |          |         |          |          |          |
|--|------------|---------------------------|----------|-------------------|----------|---------|----------|----------|----------|
|  |            | FP, freezing point        |          | BP, boiling point |          |         |          |          |          |
|  |            | NBP, normal boiling point |          | TP, triple point  |          |         |          |          |          |
| Fixed point                            | °C         | Type B                    | Type E   | Type J            | Type K   | Type N  | Type R   | Type S   | Type T   |
| Helium NPB                             | − 268.934  |                           | − 9.8331 |                   | − 6.4569 | − 4.345 |          |          | − 6.2563 |
| Hydrogen TP                            | − 259.347* |                           | − 9.7927 |                   | − 6.4393 | − 4.334 |          |          | − 6.2292 |
| Hydrogen NBP                           | − 252.88*  |                           | − 9.7447 |                   | − 6.4167 | − 4.321 |          |          | − 6.1977 |
| Neon TP                                | − 248.594* |                           | − 9.7046 |                   | − 6.3966 | − 4.271 |          |          | − 6.1714 |
| Neon NBP                               | − 246.048  |                           | − 9.6776 |                   | − 6.3827 | − 4.300 |          |          | − 6.1536 |
| Oxygen TP                              | − 218.792* |                           | − 9.2499 |                   | − 6.1446 | − 4.153 |          |          | − 5.8730 |
| Nitrogen TP                            | − 210.001  |                           | − 9.0629 | − 8.0957          | − 6.0346 | − 4.083 |          |          | − 5.7533 |
| Nitrogen NBP                           | − 195.802  |                           | − 8.7168 | − 7.7963          | − 5.8257 | − 3.947 |          |          | − 5.5356 |
| Oxygen NBP                             | − 182.962  |                           | − 8.3608 | − 7.4807          | − 5.6051 | − 3.802 |          |          | − 5.3147 |
| Carbon dioxide SP                      | − 78.474   |                           | − 4.2275 | − 3.7187          | − 2.8696 | − 1.939 |          |          | − 2.7407 |
| Mercury TP                             | − 38.834*  |                           | − 2.1930 | − 1.4849          |          | − 0.985 | − 0.1830 | − 0.1895 | − 1.4349 |
| Ice point                              | 0.000      | − 0.000                   | 0.000    | 0.000             | 0.000    | 0.000   | 0.000    | 0.000    | 0.000    |
| Diphenyl ether TP                      | 26.87      | − 0.0024                  | 1.6091   | 1.3739            | 1.076    | 0.698   | 0.1517   | 0.1537   | 1.0679   |
| Water BP                               | 100.00     | 0.0332                    | 6.3171   | 5.2677            | 4.0953   | 2.774   | 0.6472   | 0.6453   | 4.2773   |

|                   |          |        |        |        |        |        |        |        |        |
|-------------------|----------|--------|--------|--------|--------|--------|--------|--------|--------|
| Benzoic acid TP   | 122.37   | 0.0561 | 7.8468 | 6.4886 | 5.0160 | 3.446  | 0.8186 | 0.8129 | 5.3414 |
| Indium FP         | 156.598* | 0.1019 | 10.260 | 8.3743 | 6.0404 | 4.508  | 1.0956 | 1.0818 | 7.0364 |
| Tin FP            | 231.928* | 0.2474 | 15.809 | 12.552 | 9.4201 | 6.980  | 1.7561 | 1.7146 | 11.013 |
| Bismuth FP        | 271.442  | 0.3477 | 18.821 | 14.743 | 11.029 | 8.336  | 2.1250 | 2.0640 | 13.219 |
| Cadmium FP        | 321.108  | 0.4971 | 22.684 | 17.493 | 13.085 | 10.092 | 2.6072 | 2.5167 | 16.095 |
| Lead FP           | 327.502  | 0.5182 | 23.186 | 17.846 | 13.351 | 10.322 | 2.6706 | 2.5759 | 16.473 |
| Mercury BP        | 356.66   | 0.6197 | 25.489 | 19.456 | 14.571 |        | 2.9630 | 2.8483 | 18.218 |
| Zinc FP           | 419.527* | 0.8678 | 30.513 | 22.926 | 17.223 |        | 3.6113 | 3.4479 |        |
| Cu-Al eutectic FP | 548.23   | 1.4951 | 40.901 | 30.109 | 22.696 |        | 5.0009 | 4.7140 |        |
| Antimony FP       | 630.74   | 1.9784 | 47.561 | 34.911 | 26.207 |        | 5.9331 | 5.5521 |        |
| Aluminum FP       | 660.37   | 2.1668 | 49.941 | 36.693 | 27.461 |        | 6.2759 | 5.8591 |        |
| Silver FP         | 961.93*  | 4.4908 | 73.495 | 55.669 | 39.779 |        | 10.003 | 9.1482 |        |
| Gold FP           | 1064.43* | 5.4336 |        | 61.716 | 43.755 |        | 11.364 | 10.334 |        |
| Copper FP         | 1084.5   | 5.6263 |        | 62.880 | 44.520 |        | 11.635 | 10.570 |        |
| Nickel FP         | 1455     | 9.5766 |        |        |        |        | 16.811 | 15.034 |        |
| Cobalt FP         | 1494     | 10.025 |        |        |        |        | 17.360 | 15.504 |        |
| Palladium FP      | 1554     | 10.721 |        |        |        |        | 18.212 | 16.224 |        |
| Platinum FP       | 1772     | 13.262 |        |        |        |        | 21.103 | 18.694 |        |

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\* Defining fixed points of the International Temperature Scale of 1990 (ITS-90). Except for the triple points, the assigned values of temperature are for equilibrium states at a pressure of one standard atmosphere (101 325 Pa).

**TABLE 11.56** Type B Thermocouples: Platinum–30% Rhodium Alloy vs. Platinum–6% Rhodium Alloy  
*Thermoelectric voltage in millivolts; reference junction at 0°C.*

| °C   | 0       | 10      | 20      | 30      | 40      | 50      | 60      | 70      | 80      | 90      |
|------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 0    | 0.00    | −0.0019 | −0.0026 | −0.0021 | −0.0005 | 0.0023  | 0.0062  | 0.0112  | 0.0174  | 0.0248  |
| 100  | 0.0332  | 0.0427  | 0.0534  | 0.0652  | 0.0780  | 0.0920  | 0.1071  | 0.1232  | 0.1405  | 0.1588  |
| 200  | 0.1782  | 0.1987  | 0.2202  | 0.2428  | 0.2665  | 0.2912  | 0.3170  | 0.3438  | 0.3717  | 0.4006  |
| 300  | 0.4305  | 0.4615  | 0.4935  | 0.5266  | 0.5607  | 0.5958  | 0.6319  | 0.6690  | 0.7071  | 0.7462  |
| 400  | 0.7864  | 0.8275  | 0.8696  | 0.9127  | 0.9567  | 1.0018  | 1.0478  | 1.0948  | 1.1427  | 1.1916  |
| 500  | 1.2415  | 1.2923  | 1.3440  | 1.3967  | 1.4503  | 1.5048  | 1.5603  | 1.6166  | 1.6739  | 1.7321  |
| 600  | 1.7912  | 1.8512  | 1.9120  | 1.9738  | 2.0365  | 2.1000  | 2.1644  | 2.2296  | 2.2957  | 2.3627  |
| 700  | 2.4305  | 2.4991  | 2.5686  | 2.6390  | 2.7101  | 2.7821  | 2.8548  | 2.9284  | 3.0028  | 3.0780  |
| 800  | 3.1540  | 3.2308  | 3.3084  | 3.3867  | 3.4658  | 3.5457  | 3.6264  | 3.7078  | 3.7899  | 3.8729  |
| 900  | 3.9565  | 4.0409  | 4.1260  | 4.2119  | 4.2984  | 4.3857  | 4.4737  | 4.5624  | 4.6518  | 4.7419  |
| 1000 | 4.8326  | 4.9241  | 5.0162  | 5.1090  | 5.2025  | 5.2966  | 5.3914  | 5.4868  | 5.5829  | 5.6796  |
| 1100 | 5.7769  | 5.8749  | 5.9734  | 6.0726  | 6.1724  | 6.2728  | 6.3737  | 6.4753  | 6.5774  | 6.6801  |
| 1200 | 6.7833  | 6.8871  | 6.9914  | 7.0963  | 7.2017  | 7.3076  | 7.4140  | 7.5210  | 7.6284  | 7.7363  |
| 1300 | 7.8446  | 7.9534  | 8.0627  | 8.1724  | 8.2826  | 8.3932  | 8.5041  | 8.6155  | 8.7273  | 8.8394  |
| 1400 | 8.9519  | 9.0648  | 9.1780  | 9.2915  | 9.4053  | 9.5194  | 9.6338  | 9.7485  | 9.8634  | 9.9786  |
| 1500 | 10.0940 | 10.2097 | 10.3255 | 10.4415 | 10.5577 | 10.6740 | 10.7905 | 10.9071 | 11.0237 | 11.1405 |
| 1600 | 11.2574 | 11.3743 | 11.4913 | 11.6082 | 11.7252 | 11.8422 | 11.9591 | 12.0761 | 12.1929 | 12.3100 |
| 1700 | 12.4263 | 12.5429 | 12.6594 | 12.7757 | 12.8918 | 13.0078 | 13.1236 | 13.2391 | 13.3545 | 13.4696 |
| 1800 | 13.5845 | 13.6991 | 13.8135 |         |         |         |         |         |         |         |



**TABLE 11.58** Type J Thermocouples: Iron vs. Copper-Nickel Alloy*Thermoelectric voltage in millivolts; reference junction at 0°C.*

| °C   | 0      | 10     | 20     | 30     | 40     | 50     | 60     | 70     | 80     | 90     |
|------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| −200 | −7.890 | −8.096 |        |        |        |        |        |        |        |        |
| −100 | −4.632 | −5.036 | −5.426 | −5.801 | −6.159 | −6.499 | −6.821 | −7.122 | −7.402 | −7.659 |
| −0   | 0.000  | −0.501 | −0.995 | −1.481 | −1.960 | −2.431 | −2.892 | −3.344 | −3.785 | −4.215 |
| 0    | 0.000  | 0.507  | 1.019  | 1.536  | 2.058  | 2.585  | 3.115  | 3.649  | 4.186  | 4.725  |
| 100  | 5.268  | 5.812  | 6.359  | 6.907  | 7.457  | 8.008  | 8.560  | 9.113  | 9.667  | 10.222 |
| 200  | 10.777 | 11.332 | 11.887 | 12.442 | 12.998 | 13.553 | 14.108 | 14.663 | 15.217 | 15.771 |
| 300  | 16.325 | 16.879 | 17.432 | 17.984 | 18.537 | 19.089 | 19.640 | 20.192 | 20.743 | 21.295 |
| 400  | 21.846 | 22.397 | 22.949 | 23.501 | 24.054 | 24.607 | 25.161 | 25.716 | 26.272 | 26.829 |
| 500  | 27.388 | 27.949 | 28.511 | 29.075 | 29.642 | 30.210 | 30.782 | 31.356 | 31.933 | 32.513 |
| 600  | 33.096 | 33.683 | 34.273 | 34.867 | 35.464 | 36.066 | 36.671 | 37.280 | 37.893 | 38.510 |
| 700  | 39.130 | 39.754 | 40.482 | 41.013 | 41.647 | 42.283 | 42.922 |        |        |        |

**TABLE 11.59** Type K Thermocouples: Nickel-Chromium Alloy vs. Nickel-Aluminum Alloy*Thermoelectric voltage in millivolts; reference junction at 0°C.*

| °C   | 0      | 10     | 20     | 30     | 40     | 50     | 60     | 70     | 80     | 90     |
|------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| −200 | −5.891 | −6.035 | −6.158 | −6.262 | −6.344 | −6.404 | −6.441 | −6.458 |        |        |
| −100 | −3.553 | −3.852 | −4.138 | −4.410 | −4.669 | −4.912 | −5.141 | −5.354 | −5.550 | −5.730 |
| −0   | 0.000  | −0.392 | −0.777 | −1.156 | −1.517 | −1.889 | −2.243 | −2.586 | −2.920 | −3.242 |
| 0    | 0.000  | 0.397  | 0.798  | 1.203  | 1.611  | 2.022  | 2.436  | 2.850  | 3.266  | 3.681  |
| 100  | 4.095  | 4.508  | 4.919  | 5.327  | 5.733  | 6.137  | 6.539  | 6.939  | 7.338  | 7.737  |
| 200  | 8.137  | 8.537  | 8.938  | 9.341  | 9.745  | 10.151 | 10.560 | 10.969 | 11.381 | 11.793 |
| 300  | 12.207 | 12.623 | 13.039 | 13.456 | 13.874 | 14.292 | 14.712 | 15.132 | 15.552 | 15.974 |
| 400  | 16.395 | 16.818 | 17.241 | 17.664 | 18.088 | 18.513 | 18.839 | 19.363 | 19.788 | 20.214 |
| 500  | 20.640 | 21.066 | 21.493 | 21.919 | 22.346 | 22.772 | 23.198 | 23.624 | 24.050 | 24.476 |
| 600  | 24.902 | 25.327 | 25.751 | 26.176 | 26.599 | 27.022 | 27.445 | 27.867 | 28.288 | 28.709 |
| 700  | 29.128 | 29.547 | 29.965 | 30.383 | 30.799 | 31.214 | 31.629 | 32.042 | 32.455 | 32.866 |
| 800  | 33.277 | 33.686 | 34.095 | 34.502 | 34.909 | 35.314 | 35.718 | 36.121 | 36.524 | 36.925 |
| 900  | 37.325 | 37.724 | 38.122 | 38.519 | 38.915 | 39.310 | 39.703 | 40.096 | 40.488 | 40.879 |
| 1000 | 41.269 | 41.657 | 42.045 | 42.432 | 42.817 | 43.202 | 43.585 | 43.968 | 44.349 | 44.729 |
| 1100 | 45.108 | 45.486 | 45.863 | 46.238 | 46.612 | 46.985 | 47.356 | 47.726 | 48.095 | 48.462 |
| 1200 | 48.828 | 49.129 | 49.555 | 49.916 | 50.276 | 50.633 | 50.990 | 51.344 | 51.697 | 52.049 |
| 1300 | 52.398 | 52.747 | 53.093 | 53.439 | 53.782 | 54.125 | 54.466 | 54.807 |        |        |



**TABLE 11.61** Type R Thermocouples: Platinum–13% Rhodium Alloy vs. Platinum*Thermoelectric voltage in millivolts; reference junction at 0°C.*

| °C           | 0       | 10      | 20      | 30      | 40      | 50      | 60      | 70      | 80      | 90      |
|--------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| (Below zero) |         | −0.0515 | −0.100  | −0.1455 | −0.1877 | −0.2264 |         |         |         |         |
| 0            | 0.0000  | 0.0543  | 0.1112  | 0.1706  | 0.2324  | 0.2965  | 0.3627  | 0.4310  | 0.5012  | 0.5733  |
| 100          | 0.6472  | 0.7228  | 0.8000  | 0.8788  | 0.9591  | 1.0407  | 1.1237  | 1.2080  | 1.2936  | 1.3803  |
| 200          | 1.4681  | 1.5571  | 1.6471  | 1.7381  | 1.8300  | 1.9229  | 2.0167  | 2.1113  | 2.2068  | 2.3030  |
| 300          | 2.4000  | 2.4978  | 2.5963  | 2.6954  | 2.7953  | 2.8957  | 2.9968  | 3.0985  | 3.2009  | 3.3037  |
| 400          | 3.4072  | 3.5112  | 3.6157  | 3.7208  | 3.8264  | 3.9325  | 4.0391  | 4.1463  | 4.2539  | 4.3620  |
| 500          | 4.4706  | 4.5796  | 4.6892  | 4.7992  | 4.9097  | 5.0206  | 5.1320  | 5.2439  | 5.3562  | 5.4690  |
| 600          | 5.5823  | 5.6960  | 5.8101  | 5.9246  | 6.0398  | 6.1554  | 6.2716  | 6.3883  | 6.5054  | 6.6230  |
| 700          | 6.7412  | 6.8598  | 6.9789  | 7.0984  | 7.2185  | 7.3390  | 7.4600  | 7.5815  | 7.7035  | 7.8259  |
| 800          | 7.9488  | 8.0722  | 8.1960  | 8.3203  | 8.4451  | 8.5703  | 8.6960  | 8.8222  | 8.9488  | 9.0758  |
| 900          | 9.2034  | 9.3313  | 9.4597  | 9.5886  | 9.7179  | 9.8477  | 9.9779  | 10.1086 | 10.2397 | 10.3712 |
| 1000         | 10.5032 | 10.6356 | 10.7684 | 10.9017 | 11.0354 | 11.1695 | 11.3041 | 11.4391 | 11.5745 | 11.7102 |
| 1100         | 11.8463 | 11.9827 | 12.1194 | 12.2565 | 12.3939 | 12.5315 | 12.6695 | 12.8077 | 12.9462 | 13.0849 |
| 1200         | 13.2239 | 13.3631 | 13.5025 | 13.6421 | 13.7818 | 13.9218 | 14.0619 | 14.2022 | 14.3426 | 14.4832 |
| 1300         | 14.6239 | 14.7647 | 14.9056 | 15.0465 | 15.1876 | 15.3287 | 15.4699 | 15.6110 | 15.7522 | 15.8935 |
| 1400         | 16.0347 | 16.1759 | 16.3172 | 16.4583 | 16.5995 | 16.7405 | 16.8816 | 17.0225 | 17.1634 | 17.3041 |
| 1500         | 17.4447 | 17.5852 | 17.7256 | 17.8659 | 18.0059 | 18.1458 | 18.2855 | 18.4251 | 18.5644 | 18.7035 |
| 1600         | 18.8424 | 18.9810 | 19.1194 | 19.2575 | 19.3953 | 19.5329 | 19.6702 | 19.8071 | 19.9437 | 20.0797 |
| 1700         | 20.2151 | 20.3497 | 20.4834 | 20.6161 | 20.7475 | 20.8777 | 21.0064 |         |         |         |

**TABLE 11.62** Type S Thermocouples: Platinum–10% Rhodium Alloy vs. Platinum  
*Thermoelectric voltage in millivolts; reference junction at 0°C.*

| °C           | 0       | 10      | 20      | 30      | 40      | 50      | 60      | 70      | 80      | 90      |
|--------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| (Below zero) |         | −0.0527 | −0.1028 | −0.1501 | −0.1944 | −0.2357 |         |         |         |         |
| 0            | 0.0000  | 0.0552  | 0.1128  | 0.1727  | 0.2347  | 0.2986  | 0.3646  | 0.4323  | 0.5017  | 0.5728  |
| 100          | 0.6453  | 0.7194  | 0.7948  | 0.8714  | 0.9495  | 1.0287  | 1.1089  | 1.1902  | 1.2726  | 1.3558  |
| 200          | 1.4400  | 1.5250  | 1.6109  | 1.6975  | 1.7849  | 1.8729  | 1.9617  | 2.0510  | 2.1410  | 2.2316  |
| 300          | 2.3227  | 2.4143  | 2.5065  | 2.5991  | 2.6922  | 2.7858  | 2.8798  | 2.9742  | 3.0690  | 3.1642  |
| 400          | 3.2597  | 3.3557  | 3.4519  | 3.5485  | 3.6455  | 3.7427  | 3.8403  | 3.9382  | 4.0364  | 4.1348  |
| 500          | 4.2336  | 4.3327  | 4.4320  | 4.5316  | 4.6316  | 4.7318  | 4.8323  | 4.9331  | 5.0342  | 5.1356  |
| 600          | 5.2373  | 5.3394  | 5.4417  | 5.5445  | 5.6477  | 5.7513  | 5.8553  | 5.9595  | 6.0641  | 6.1690  |
| 700          | 6.2743  | 6.3799  | 6.4858  | 6.5920  | 6.6986  | 6.8055  | 6.9127  | 7.0202  | 7.1281  | 7.2363  |
| 800          | 7.3449  | 7.4537  | 7.5629  | 7.6724  | 7.7823  | 7.8925  | 8.0030  | 8.1138  | 8.2250  | 8.3365  |
| 900          | 8.4483  | 8.5605  | 8.6730  | 8.7858  | 8.8989  | 9.0124  | 9.1262  | 9.2403  | 9.3548  | 9.4696  |
| 1000         | 9.5847  | 9.7002  | 9.8159  | 9.9320  | 10.0485 | 10.1652 | 10.2823 | 10.3997 | 10.5174 | 10.6354 |
| 1100         | 10.7536 | 10.8720 | 10.9907 | 11.1095 | 11.2286 | 11.3479 | 11.4674 | 11.5871 | 11.7069 | 11.8269 |
| 1200         | 11.9471 | 12.0674 | 12.1878 | 12.3084 | 12.4290 | 12.5498 | 12.6707 | 12.7917 | 12.9127 | 13.0338 |
| 1300         | 13.1550 | 13.2762 | 13.3975 | 13.5188 | 13.6401 | 13.7614 | 13.8828 | 14.0041 | 14.1254 | 14.2467 |
| 1400         | 14.3680 | 14.4892 | 14.6103 | 14.7314 | 14.8524 | 14.9734 | 15.0942 | 15.2150 | 15.3356 | 15.4561 |
| 1500         | 15.5765 | 15.6967 | 15.8168 | 15.9368 | 16.0566 | 16.1762 | 16.2956 | 16.4148 | 16.5338 | 16.6526 |
| 1600         | 16.7712 | 16.8895 | 17.0076 | 17.1255 | 17.2431 | 17.3604 | 17.4474 | 17.5942 | 17.7105 | 17.8264 |
| 1700         | 17.9417 | 18.0562 | 18.1698 | 18.2823 | 18.3937 | 18.5038 | 18.6124 |         |         |         |



### 11.11 CORRECTION FOR EMERGENT STEM OF THERMOMETERS

When a thermometer which has been standardized for total immersion is used with a part of the liquid column at a temperature below that of the bulb, the reading is low and a correction must be applied. The stem correction, in degrees Celsius, is given by

$$KL(t_o - t_m) = \text{degrees Celsius}$$

where  $K$  = constant, characteristic of the particular kind of glass and temperature (see Table 11.49)

$L$  = length of exposed thermometer, °C (that is, the length not in contact with vapor or liquid being measured)

$t_o$  = observed temperature on thermometer

$t_m$  = mean temperature of exposed column (obtained by placing an auxiliary thermometer alongside with its bulb midpoint)

For thermometers containing organic liquids, it is sufficient to use the approximate value,  $K = 0.001$ . In such thermometers the value of  $K$  is practically independent of the kind of glass.

**TABLE 11.64** Values of  $K$  for Stem Correction of Thermometers

| Temperature, °C | Soft glass | Heat-resistant glass |
|-----------------|------------|----------------------|
| 0–150           | 0.000 158  | 0.000 165            |
| 200             | 0.000 159  | 0.000 167            |
| 250             | 0.000 161  | 0.000 170            |
| 300             | 0.000 164  | 0.000 174            |
| 350             |            | 0.000 178            |
| 400             |            | 0.000 183            |
| 450             |            | 0.000 188            |