

# Subject Index

## A

Accreditation programs, 217  
 Acetic acid, 34, 74  
 Acetone, 43  
 Acetylacetone, 42, 116–117  
 Acetylene, 30, 32  
 AC glow discharge source, 180, 184  
 Acid/base indicators, 148–149  
 Acid/base titrations, 152–153  
 Acid dissolution, 70–81  
     pure metals, 78  
 Acid-resistant metals, hygroscopic salts, 44  
 Acids, 31–35  
     boiling points, 75  
     non-oxidizing, 71–75  
     nonoxidizing mixtures, 74–75  
     oxidizing, 75–78  
     oxidizing mixtures, 76–77  
         additives, 77–78  
         standardization by titrants, 148  
 Acid treatment, for inclusions, 89  
 ACS grade, 29  
 Action level, 209  
 Activation analysis, 184  
 Additives, in oxidizing acid mixtures, 77–78  
 Air, 30, 32  
     ignition of organics, 82–83  
 Air baths, 25  
 Air conditioning, 5  
 Air exchange in laboratories, 5  
 Alberene stone, 12–13  
 Alchemy, ix, 3, 15  
 Alcohols, 43  
 Aldehydes, 44  
 Aliquant, 61  
 Aliquot, 61  
 Aliquoting, 204–206  
 Alkali metals, chemical behavior, 242  
 Alkali-resistant/boron-free glass, 16  
 Alloys, frequency of analyte concentrations, xiii  
 Alpha-benzoinoxime (*See* Benzoin monoxime)  
 Aluminoborosilicate glass, 16  
 Aluminosilicate glass, 15  
 Aluminum  
     after mercury cathode electrolysis, 102  
     ammonium hydroxide precipitation, 98–99  
     gravimetric measurement, 146  
     separations, 125  
         from beryllium, 104  
     spectrophotometric method, 117  
     volumetric measurement, 153  
     wet chemical methods, 111–112

Alundum (fused alumina), 19  
 Ammonia, 30, 32  
 Ammonium carbonate, 102  
 Ammonium fluoride, 41  
 Ammonium hydroxide, 35  
     precipitation, 97–100  
 Ammonium iron sulfate, 38  
 Ammonium molybdate, 43  
 Ammonium oxalate, 41  
 Ammonium perchlorate, 34  
 Ammonium peroxydisulfate, 38  
 Ammonium persulfate, 389  
 Ammonium phosphate, dibasic, 41  
 Analysis of variance (ANOVA), 67, 197–199  
 Analyte, 70  
     chemical behavior, 234–241  
     distillation, 121  
 Analytical chemistry, ix, xi  
 Analytical sampling constant, 68  
 Anion exchange systems, 119–120  
 Annealing, 63  
 ANOVA (*See* Analysis of variance)  
 Antimony  
     distillation, 121  
     separations, 125  
 Applied potential reduction, 108  
 Aqua regia, 76  
 Aqueous caustic solutions, 91  
 Arc/spark optical emission laboratory, 11  
 Argon, 31  
 Aromatic compounds, 44  
 Arsenic  
     arsine generation/spectrophotometric procedure, 105  
     distillation, 121–122  
     separations, 125–126  
 Arsenious oxide, 38  
 Asbestos, 19  
 Ascorbic acid, 39  
 ASTM D 1193, 30  
 ASTM D 3856, 219  
 ASTM E 32, 58, 65  
 ASTM E 59, 55  
 ASTM E 60, 160  
 ASTM E 172, 216  
 ASTM E 173, 217  
 ASTM E 287, 23  
 ASTM E 350, 117  
 ASTM E 356, 216  
 ASTM E 452, 44  
 ASTM E 478, 116  
 ASTM E 691, 217  
 ASTM E 877, 58  
 ASTM E 1172, 216  
 ASTM E 1479, 216  
 ASTM E 1507, 216  
 ASTM E 1601, 217

Atomic absorption, 162–167  
     calibration, 163–164  
     laboratory, 11  
 Audits  
     external, 217  
     internal, 216–217  
 Automatic titrators, 27, 156–157  
  
**B**  
 Bakelite, 16  
 Balance room, 9–10  
 Balances, analytical, 9  
 Barium  
     gravimetric measurement, 145  
     separations, 126  
 Barium chloride, 41  
 Bases, 35–36  
 Batch, 52  
 Beakers, 20  
     borosilicate, 20  
     fused silica, 20  
     plastic, 20  
     polytetrafluoroethylene, 20  
     removing residues, 203  
 Vycor, 20  
 Beer's law, 158  
 Bell jar, 25  
 Benzene, 44  
 Benzoin anti-oxime, 40  
 Benzoin monoxime, 40  
     extraction, 116  
     precipitation, 105  
 (N)-Benzoyl-N-phenylhydroxamine, 117  
 Beryllium, separations, 126  
 Bias, 191–192  
 Billets, 55  
 Bismuth, separations, 126  
 Blanking approach, 161  
 Blooms, 55  
 Boiling water baths, 26  
 Boil rod, 207  
 Bone ash, 19  
 Book molds, 53  
 Borates, 36  
 Borax, 36, 85  
 Boric acid, 36, 85  
 Boron  
     distillation, 122  
     interference evolution, 121  
     interference from Fe and Co, 111  
     separations, 126  
     volumetric measurement, 152–153  
 Boron trioxide, 36, 85  
 Borosilicate glass, 15  
     reagent storage vessels, 46  
 Bottles, 20  
 Boyle, Robert, 3

Bracketting technique (AA), 163–164

Bragg's Law, 173

Brilliant green, 43

Bromide complexes, extraction, 113

Bromine, 37–38

as additive, 77–78

separations, 135

Bromine/methanol, 89–90

Bromine/methyl acetate, 89–90

para-Bromomandelic acid, 41

precipitation, 107

Building, design, 3–6

Buna-N, 17

Burets, 22–23

automatic, 22

cleaning, 44

digital, 22–23

Butadiene-acrylonitrile copolymers, 17

(1)-Butanol (N-butanol), 43

2-Butanone, 43

Button melting, 63

Butyl acetate, 43

## C

Cadmium, separations, 126–127

Calcium, separations, 127

Calibration, 191, 194, 216

curve, 163–164

Carbon

measurement, 177–178

separations, 135

Carbon dioxide, 31–32

Carbon tetrachloride, 43

Carboys, 20

Castings, large, 55

Cation and chelating ion exchange, 120

Ceiling limit, 209

Cellulose acetate, 24

Cellulose nitrate, 24

Centroid, calibration curve, 198

Ceric ammonium nitrate, 37

Ceric sulfate, 37

Cerium, separations, 127, 135

Certification programs, 217

Certified reference material, 192–193

Cesium, separations, 127

CGA outlets and connections, 31–32

Charge-coupled device, 172

Charge injection device, 172

Chelate complexes, extraction, 115–118

Chelating agents, 115

Chemical effects, interferences, 164

Chemical inventory control, 210

Chemical waste disposal, 211

Chemistry

atomic absorption, 167

emission spectroscopy, 175–176

spectrophotometry, 160–161

Chloride

as medium for mixing elemental standards, 206

precipitation, 108

Chloride complexes, extraction, 113

Chlorinated hydrocarbons, 43

Chlorination, 90

Chlorine, 30, 32

separations, 135

Chloroform, 43

Chromate, precipitation, 109

Chromatography, 181–183

Chromium

interference evolution, 121

separations, 127

sodium hydroxide precipitation, 101

volumetric measurement, 154–155

Cinchonine, 41, 107

Cleaning labware, 203–204

Coanda effect, 204

Cobalt

separations, 127

volumetric measurement, 155

Coefficient of variation, 196

Cold vapor atomic absorption, 167

Commercial laboratories, 216

Comminution, 52, 61, 64–65

Comparative methods, x–xi, 189–190

Complexation titrations, 155–156

Complexing agents, 41–42

Composite sample, 52

Compton scattering, 175

Coning and quartering, 64

Consignment, 52

Constitution heterogeneity, 52

Continuum-source background correction

(AA), 165

Control charts, 195

records, 216

Control laboratory, 6

Coordination compounds, 115

Copper

electrogravimetric measurement, 143–144

electrogravimetric method, 105

interference on P2149 Å, 100

separations, 128

spectrophotometric measurement, 162

Corex (aluminosilicate) glass, 15

Corporate environment, 213

Correlation coefficient, 198

Crucibles

selection guide, 19

sizes, 20

(See also specific materials)

Crushing, samples, 63

Crystals, dispersion characteristics, 173

Cupferron, 40

extraction, 115

precipitation, 102–103

Curcumin, 43

Czerny-Turner configuration, 169

## D

Darkroom, 11

Davy, Sir Humphrey, 3

DC arc source, 170

DC plasma source, 172

Definitive methods, x–xi, 189

Degrees of freedom, 197

Desiccators, 25–26

cabinets, 26

Dessicants, properties and application, 26

Detection limit, 200–201

Detectors and readout (OC), 172

Deviation, 197

Diammonium phosphate, 107–108

Dianthrimide, 43

Diantripyrilmethane, 43

Dichloromethane, 43

Diethyl ether, 44

Differential spectrophotometry, 160

Diffraction gratings, 170

Diluting, 204–206

Dimethylglyoxime, 40, 43

extraction, 116

precipitation, 105–107

2,9-Dimethyl-1,10-phenanthroline, 43

Diphenylcarbazide, 43

1,5-Diphenylcarbohydrazide, 43

1,5-Diphenylthiocarbazone, 42

Dip mold, 54

Direct reading polychromators, 170

Dissolution, in acids, 70–81

pure metals, 78

Distillation/evolution, 120–122

Distillation of analytes, 121–122

Distribution heterogeneity, 52, 57

Dithiol, 43, 116

Dithizone, 42

extraction, 116

Division, 52

Dixon test, 198

DOT hazard system, 209

Dried compounds, weighing as, 145–146

Dropping bottles, 20

Drying ovens, 25

Dysprosium, 135

## E

Echelle spectrometer, 170

EDTA, 41–42

Elastic collisions, 175

Elastomers, 17

Electrical equipment, 27

Electrical fixtures, laboratory, 4–5

Electrochemical plating apparatus, 27

Electrolysis, 90–91

for inclusions, 90

mercury cathode, 110–112

Electrolytic conductivity detector, 182

Electron capture detector, 182

Electrothermal atomic absorption, 166–167

Element, weighing as, 143–144

Elemental forms, reduction to, 108

Elemental standard solutions, 44–46

Element sensitivities in atomic absorption, 163–165

Eluent suppression, 182

Emergency planning, 210–211

Emergency showers, 14

Emission spectrometry laboratory, 11

Emission sources, 170–172

Energy dispersive X-ray spectrometer, 174

Equipment

metrology, 216

miscellaneous, 27–28

Erbium, 135

Erlenmeyer flasks, 20–21

Errors, handling, 216

Ethanol, 434

Ethers, 44

extraction, 114

Europium, 135

separations, 128

Evaluation of personnel, 222

Evaporating dishes, 20

Evolution of interferences, 121

Explosion, reagents, 46

Eyewash stations, 14, 27

## F

Face shields, 27

Farrady, Michael, 3

FEP, 17

Ferrous ammonium sulfate, 38

Ferrous sulfate, 38

Filter cones, 23

- Filter paper, 23–24  
 Filter photometers, 158  
 Filter pulp, 23  
 Filtration equipment, 23–25  
 Finished product certification, 8  
 Fire assay, ix, 6, 122–123  
 Fire extinguishers, 14, 27  
 Fisher filtrator, 25  
 Flame atomic absorption, 166  
     preconcentration techniques, 112  
 Flame emission, 172  
 Flame ionization detector, 182  
 Flame photometric detector, 182  
 Flashpoint temperature, 209–210  
 Flasks  
     Erlenmeyer, 20–21  
     removing residues, 203  
     vacuum filtration, 23  
 Floors, laboratory, 4, 6  
 Flow proportional detector, 174  
 Fluoboric acid, 35, 74  
     as additive, 77  
 Fluoride  
     complexes, extraction, 113  
     precipitation, 108  
 Fluorimetry, 183  
 Fluorinated ethylene propylene (FEP), 17  
 Fluorine  
     distillation, 122  
     separations, 135  
 Fluoropolymers, 17  
 Forceps, 20  
 Formaldehyde, 44  
 Formic acid, 35  
 Fourier transform design, 185  
 "Freiburger decomposition," 85  
 F test, 197  
 Funnels  
     filter, 23–24  
     holders, 23  
     separatory, 21  
     transfer, 23  
 Furildioxime, 116  
 Fused quartz, 16, 20  
 Fused silica, 16, 20
- G**
- Gadolinium, 135  
 Gallium  
     ammonium hydroxide precipitation, 98–99  
     separations, 128  
 Gas chromatography, 181–182  
 Gases, 30–32  
     mixtures, 31  
     sampling, 59  
 Gas service, 6  
 Geiger tube, 173  
 Germanium  
     distillation, 122  
     separations, 128  
 Glass, 15–16  
     labware composition and thermal properties, 16  
 Glassblowing, 14, 20–21  
 Glasses, safety, 27, 62  
 Glass fiber filters, 23–24  
 Glass frit crucibles, 25  
     cleaning, 203  
 Glass tubing, 21  
 Gloves, safety, 27, 33  
 Glow discharge  
     emission source, 171  
     mass spectrometer, 179–181  
 Glow discharge, atomic absorption, 164, 167  
 Goggles, 62  
 Gold, 18  
     sampling in quartz, 57–58  
     separations, 128  
 Graduated cylinders, 23  
 Graduates, pharmaceutical, 23  
 Graphite, 19  
     crucibles, 19–20  
     glassy, 19  
     porous, 19  
     pyrolytic, 19  
 Gravimetry  
     other determinations, 146  
     technique, 141–143  
     weighing  
         as dried compounds, 145–146  
         as element, 143–144  
         as other ignited compounds, 145  
         as oxide, 144–145  
 Gross sample, 52  
 Gy, Pierre, 51–52, 56, 58, 61, 64, 68
- H**
- Hafnium  
     carrier for minor constituents, 103  
     separations, 135  
 Halar ECTFE, 17  
 Half-cell potentials, redox species, 39  
 Halogens, separations, 135  
 Hardware, X-ray fluorescence, 173  
 Hazards  
     associated with reagent use, 47  
     controlling, 208–210  
     limiting exposure, 209  
     management responsibilities, 208–209  
     principles, 208  
     types, 209–210  
 Heating and drying equipment, 25–26  
 Heating systems, 5  
 Helium, 31–32  
 Heterogeneity, 52  
     total, 57  
 Heteropolyacid complexes, 114  
 Hexanes, 44  
 High temperature alloy, ion exchange separation, 120  
 Hollow forms, sampling, 55  
 Holmium, 135  
 Homemade supplies, 207–208  
 Homogeneity testing  
     problems, 67  
     of standards, 193  
 Hoods  
     canopy, 13  
     ducts, 3  
     fume, 12–13  
     perchloric acid, 12  
 Hot extraction, 178  
 Hotplates, 12, 207–208  
 Hydrazine sulfate, 39  
 Hydrobromic acid, 35, 74  
 Hydrocarbons  
     chlorinated, 43  
     unsubstituted, 44  
 Hydrochloric acid, 32–33, 41, 71–72  
     anion exchange system, 119  
     mixture with  
         nitric acid, 76  
         nitric and hydrofluoric acids, 76–77  
 Hydrofluoric acid, 33–34, 41, 72–73  
 anion exchange system, 119  
 Hydrogen, 30, 32  
     measurement, 178  
     sampling for, 55–56, 63  
     separations, 135  
 Hydrogen chloride, 31–32  
 Hydrogen peroxide, 38, 43, 77–78  
 Hydrogen sulfide, 31–32  
 Hydroiodic acid, 74  
 Hydrolysis, precipitation by, 107  
 Hydropyrolysis, 90  
 Hydroquinone, 43  
 Hydrous oxides, hydrolysis, 107  
 Hydroxylamine hydrochloride, 39  
 (8)-Hydroxyquinaldine, 109, 117  
 (8)-Hydroxyquinoline, 40, 43  
     extraction, 115  
     precipitation, 103–104  
 Hypophosphorous acid, 39
- I**
- Ice machine, 27  
 Ignition of organics, 82–83  
 1,1'-Iminodianthraquinone, 43  
 Immersion samplers, 54  
 Inclusion isolation, 89–90  
 Increment, 52  
 Indicators, 148–150  
 Indium, separations, 128  
 Inductively coupled plasma mass spectrometer, 180–181  
 Inductively coupled plasma optical emission laboratory, 11  
 Inductively coupled plasma source, 171, 180  
 Inductively coupled plasma torch, cleaning, 204  
 Inelastic collisions, 175  
 Inert gases, 31–32  
 Infrared absorption spectrophotometry, 184  
 Infrared lamps, 26  
 Ingamells and Switzer, 67–69  
 In-house reference material, 192  
 Insolubles, 79  
 Instruments, specifying, 216  
 Interferences  
     in atomic absorption, 164–166  
     evolution, 121  
 Interlaboratory studies, 217  
 Iodide complexed, extraction, 113  
 Iodine, 37, 135  
 Ion association complexes, extraction, 113–114  
 Ion chromatography, 182–183  
 Ion exchange, 118–120  
 Ionic displacement, 89  
 Ionization effects, interferences, 164–165  
 Ion optics for mass spectrometry, 178–179  
 Ion sources for mass spectrometry, 179–181  
 Ion trap mass analyzer, 184  
 Iridium, separations, 128–129  
 Iron, 18  
     crucibles, 18, 20  
     in high-carbon ferrochromium, 101  
     separations, 129  
     volumetric determination, 99, 154  
     vanadium-bearing samples, 102  
 ISO 5725, 217  
 Isobutyl alcohol, 43  
 ISO Guide 31, 193

Isopropyl ether, 44  
 Isotope dilution mass spectrometry, 180–181  
 IUPAC, 51, 61

**J**

Jars, 20  
 Jaw crusher, 64  
 Johnson, C. M., 3  
 Jones reductor, 39

**K**

Kel-F, 18  
 Ketones, 43  
 Kimax glass (*See* Borosilicate glass)  
 Kynar, 24

**L**

Laboratory  
 benches, 13  
 design, 3–14  
 balance room, 9–10  
 building, 3–6  
 finished product certification, 8  
 hot metal control, 6–7  
 office areas, 9  
 purchased material verification, 7–8  
 R & D support, 8–9  
 sample preparation room, 14  
 small instrument room, 10  
 spectrometry laboratories, 10–11  
 wet lab, 11–14  
 early metals analysis, 3, 5  
 organization, 219–221  
 sinks, 13  
 style, 215  
 Laboratory information management system, 213–214  
 Laboratory sample (*See* Sample)  
 Labware  
 cleaning, 203–204  
 shapes and sizes, 20–21  
 volumetric, 21–23  
 washer, 27  
 Lactic acid, 78  
 Lanthanides, 135  
 Lanthanum, separations, 129, 135  
 Laser source, for atomic absorption, 185  
 Latex rubber, 17  
 Latex tubing, natural, 21  
 Lavoisier, Antoine, ix  
 Leaching agent, HCl as, 72  
 Lead, separations, 129  
 Leonardo da Vinci, ix  
 Liberation size, 57, 61, 66  
 Liebig, Justus, Baron von, 3  
 Lighting, laboratory, 4  
 Limiting exposure to chemical hazards, 209  
 LIMS, 213–214  
 Linear regression, 197–198  
 “Liquid fire” method, 83  
 Liquids, sampling, 59  
 Lithium, separations, 129  
 Lithium carbonate, 36, 85  
 Lithium hydroxide, 88, 360  
 Lithium metaborate, 36, 86  
 Lithium tetraborate, 36, 86  
 Lot, 52  
 Low actinic glass, 16

Lucite (*See* Polymethyl methacrylate)  
 Lutetium, 135

**M**

Magnesium  
 in aluminum alloys, 101  
 separations, 129  
 from other alkaline earths, 104  
 Magnetic stirrers, 27  
 Management, safety responsibilities, 208–209  
 Manganese  
 separations, 129  
 spectrophotometric measurement, 161–162  
 Manufacture of standards, 193–194  
 Masking agents, 112–113  
 Masking techniques, spectrophotometry, 160–161

Mass analyzer detector, 182  
 Mass spectrometer laboratory, 11  
 Mass spectrometry, 178–181  
 Material and data flow, 213–215  
 Material safety data sheets, 210  
 Matrix modifiers in atomic absorption, 164, 167  
 Membrane filters, 24  
 Membrane filter vacuum filtration, 23  
 Mercury, 19  
 separations, 129–130  
 Mercury cathode, 27  
 electrolysis, 110–112  
 Metal labware, 18  
 Metallic particles, 66  
 Metallochromic indicators, 150  
 Metals  
 analysis measurement, future, 184–185  
 compounds, solubility, 41  
 molten, sampling, 52–55  
 oxidation with *aqua regia*, 76  
 residues, removing, 203  
 Methanol, 43  
 Method of additions (AA), 163–164  
 Methyl acetate, 43  
 Methylene chloride, 43  
 Methylethylketone, 43  
 Methylisobutylketone (MIBK), 43  
 2-Methyl-1-propanol, 43  
 Microwave dissolutions, 80  
 Microwave ovens, 26, 80  
 Microwave plasma source, 172  
 Minimum sampling weight, 61  
 Mohr pipets, 22  
 Mohr’s salt, 38  
 Molecular fluorescence, 183  
 Molten metal, sampling, 52–55  
 Molten salt fluxes, 36, 83–88  
 Molybdate, precipitation, 109–334  
 Molybdenum  
 gravimetric determination, 100, 105  
 interference on Al3961 Å, 99–100  
 separations, 130  
 Monroe crucible, 25  
 Motivation of personnel, 222  
 Muffle furnaces, 25

**N**

Nascent hydrogen, 39  
 Nebulizers for ICP-OES, 171  
 cleaning, 204  
 Neocuproine, 43  
 Neodymium, 135

Neoprene, 17  
 Nerst, 112  
 NFPA hazard system, 209–210  
 Nickel, 18  
 crucibles, 18, 20  
 separations, 130  
 volumetric measurement, 156  
 Niobium  
 gravimetric measurement, 144  
 ICP/OES determination in high-temperature alloys, 103  
 separations, 130  
 and titanium, simultaneous spectrophotometric measurement, 162  
 Nitric acid, 33, 75  
 mixture with hydrochloric acid, 76  
 hydrochloric and hydrofluoric acids, 76–77  
 Nitrogen, 31–32  
 distillation, 122  
 measurement, 178  
 sampling for, 63  
 separations, 135  
 Nitrogen/phosphorus detector, 182  
 Nitron, 41, 109  
 (1)-Nitroso-2-naphthol, 108  
 extraction, 116  
 Nitroso R acid, disodium salt, 42, 116  
 Nitrous oxide, 31–32  
 Non-oxide ignited compounds, weighing as, 145  
 Nonparametric statistics, 202  
 Nylons, 17, 25

**O**

Office areas, 9  
 Optical designs for emission spectroscopy, 169–170  
 Optical emission spectroscopy, 169–172  
 Organics  
 destruction of, 82–83  
 residues, removing, 203  
 solvents, 43–44  
 Osmium  
 distillation, 122  
 separations, 130  
 Outlier test, 198  
 Oxalate precipitation, 108  
 Oxalic acid, 38  
 Oxide  
 hydrochloric acid attack, 71  
 residues, removing, 203  
 weighing as, 144–145  
 Oxidizing agents, 36–38  
 Oxygen, 31–32  
 ignition of organics, 82–83  
 measurement, 178  
 sampling, 55, 63  
 separations, 135  
 Oxygen bomb, 82–83

**P**

Paired t test, 197  
 Palladium  
 gravimetric measurement, 146  
 separations, 130–131  
 Particulates, sampling, 57–59  
 Perchlorates, 34  
 precipitation, 109  
 Perchloric acid, 34, 70, 75–76, 77

- Perchloric acid/nitric acid wet ashing, 83  
 Perfluoroalkoxy polymer, 17  
   reagent storage vessels, 46  
 Periodic acid, 38  
 Permissible exposure limits, 47, 209  
 Personnel  
   evaluation, 222  
   motivation, 222  
   training, 221–222  
 pH electrodes, 27  
 o-Phenanthroline, 43  
 (1,10)-Phenanthroline (o-phenanthroline), 43  
 pH meters, 27  
 Phosphoric acid, 34, 74  
 Phosphorus  
   separations, 131  
   spectrophotometric methods, 100  
 Photodiode array, 158, 172  
 Photographic plates, 172  
 Photoionization detector, 182  
 Photomultiplier, 158, 172  
 Phototube, 158  
 Physical effects, interferences, 164  
 Pipeting bulbs, 22  
 Pipets, 22–23  
   automatic, 22–23  
   cleaning, 44  
 Plasma dry ashing, 83  
 Plastic, 16–18  
 Platinum, 18  
   crucibles, 20  
   separations, 131  
 Plexiglass, 17  
 Polarography, 183  
 Polyacetal, 17  
 Polyallomer, 17  
 Polycarbonate, 17  
   track-etched, 24–25  
 Polychloroprene, 17  
 Polychromators, 169–170  
 Polyethylene  
   high density, 17  
   linear, low density, 17  
   low density, 17  
   stoppers, 21  
 Polymethylene terephthalate G  
   copolymer, 17  
 Polymethyl methacrylate, 17  
 Polymethylpentene, 17  
 Polypropylene, 17, 25  
 Polystyrene, 17  
 Polysulfone, 17, 25  
 Polytetrafluoroethylene, 17, 25  
   labware, cleaning, 204  
   stopcocks, 21  
 Polyurethane, 17  
 Polyvinyl chloride, 17, 25  
   tubing, 21  
 Polyvinylidene fluoride, 24  
 Pooled standard deviation, 196  
 Pooled variance, 196  
 Porcelain, 19  
   crucibles, 19, 20, 25  
 Potassium, separations, 131  
 Potassium bisulfate, fused, 36, 86–87  
 Potassium bromate, 37, 78  
 Potassium carbonate, 36, 85  
 Potassium chlorate, 78  
 Potassium chromate, 41  
 Potassium cyanide, 42  
 Potassium dichromate, 37, 78  
 Potassium ethyl xanthate, 116  
 Potassium ferrocyanide, 38  
 Potassium fluoride, 41  
 Potassium hydroxide, 36, 88  
 Potassium iodate, 37  
 Potassium metaperiodate, 38  
 Potassium permanganate, 36–37  
 Potassium pyrosulfate, 36, 86–87  
 Praseodymium, 135  
 Precipitants, 39–41  
   inorganic, 96  
   organic, 97–98  
 Precipitation, 96–97  
   alpha-benzoinoxime, 105  
   ammonium hydroxide, 97–100  
   cupferron, 102–103  
   dimethylglyoxime, 105–107  
   by hydrofluoric acid, 73  
   8-hydroxyquinoline, 103–104  
   para-bromomallic acid, 107  
   sodium hydroxide, 100–102  
   sulfide, 104–105  
 Precipitation titrations, 155  
   indicators, 150  
 Precision, 196–199  
 Preparation error, 61  
 Pressure dissolutions, 80  
 Pressure vessel, decomposition, 72  
 Priestley, Joseph, ix, 3  
 Primary comparative methods, 189–190  
 Primary standard, 192  
 Professional integrity, 195  
 Proficiency testing, 217  
 Promethium, 135  
 (1)-Propanol (N-propanol), 43  
 (2)-Propanol (isopropanol), 43  
 Protective shields, 27  
 Purity of reagents, 29  
 PVC, 17, 25  
   tubing, 21  
 Pyrex glass (*See* Borosilicate glass)  
 Pyrocatechol violet, 43  
 Pyroceram, 19–20  
 Pyrogallol, 43  
 Pyrohydrolysis, 90  
 Pyrolysis, 90
- Q**
- Quality assessment, 215–217  
 Quality assurance, 215  
 Quality control, 215–216  
 Quantitation limit, 200–201  
 Quartz, gold sampling, 57–58  
 (8)-Quinolinol (*See* 8-Hydroxyquinoline)
- R**
- Radioactive hazard, scrap, 56  
 Radioactive source, 174  
 Range statistics, 202  
 Rare earths  
   chemical behavior, 243–244  
   separations, 135  
   trace levels, X-ray fluorescence, 111  
 Raw material, verification, 7–8  
 Rayleigh scattering, 175  
 Ray-Sorb glassware, 16  
 Reactant gases, 30–32  
 Reagents, 29–47  
   acids, 31–35  
   bases, 35–36  
   complexing agents, 41–42  
   contaminated, 46  
   inert gases, 31–32  
   molten salt fluxes, 36
- organic solvents, 43–44  
 oxidizing agents, 36–38  
 precipitants, 39–41  
 purity levels, 29  
 reactant gases, 30–32  
 reducing agents, 38–39  
 spectrophotometric, 42–43  
 storage, 13–14, 46–47  
 water, 29–30  
 Recording and storing data, 214  
 Redox-indicating electrodes, 27  
 Redox indicators, 149–150  
 Redox species, half-cell potentials, 39  
 Redox standard solutions, preparation, 40  
 Redox titrations, 153–155  
 Reducing agents, 38–39  
 Reduction, to elemental forms, 108  
 Reference material, 192–194  
 Reflux dissolution, 80  
 Refrigerator, explosion-proof, 27  
 Relative standard deviation, 68, 196  
 Repeatability, 198  
   interval, 198  
 Reporting conventions, 200–202  
 Reproducibility, 198  
   interval, 198  
 Research and development support, 8–9  
 Resins, ion exchange, 118  
 Rhenium, separations, 131  
 Rhodium, separations, 131  
 Riffling, 65  
 Robotic laboratory, 7–8  
 Ross electrode, 27  
 Round robin, 217  
 Rowland circle, 170  
 Rubber stoppers, 21  
 Rubber tubing, 21  
 Rubidium, separations, 131–132  
 Ruthenium, separations, 132
- S**
- Safety, 6, 27, 46–47, 62, 208–210  
 equipment, 27  
 Samarium, 135  
 Sample, 52, 61  
   custody, 214  
   deoxidizing, 53  
   preparation  
    examination for defects, 62  
    identification codes, 61–62  
    particulates, 64–66  
    preliminary acid cleaning, 63  
    solid metals, 61–64  
 Sample preparation room, 14  
 Sampling, 51–59  
   gases, 59  
   liquids, 59  
   molten metal, 52–55  
   particulates, 57–59  
   scrap, 56–57  
   solid metal, 55–56  
 Sampling constant, 58  
 Sampling error, 51–52  
 Sampling guns, 54  
 Sampling plan, 51–52, 56  
 Sampling spoons, 53–54  
 Sampling tubes, 54  
 Sandbaths, 26, 207–208  
 Sandstone, impregnated hard, 12  
 Saturated calomel electrode, 27  
 Scandium, 135  
 Scanning monochromators, 169  
 Schöniger flask, 82

- Scintillation detector, 173–174  
 Scrap, sampling, 56–57  
 Secondary comparative methods, 190  
 Secondary standard, 192  
 Segregation, 52–53, 57  
     ferroalloys, 58  
 Selection, 52  
 Selection error, 61  
 Selenium  
     distillation, 122  
     gravimetric measurement, 143  
     separations, 132  
 Semiconductor detector, 174  
 Separatory funnels, 21  
 Serological pipets, 22  
 Sewage, handling, 5  
 Shields (*See* Face shields; Protective shields)  
 Short-term exposure levels, 209  
 Showers, emergency, 14, 27  
 Sieves, 65  
 Silicon  
     interference evolution, 121  
     separations, 132  
 Silicone rubber, 17  
 Silver, 18, 25  
     separations, 132  
 Silver diethyldithiocarbamate, 42  
 Silver nitrate, 41  
 Silver reductor, 39  
 Sinters, 88–89  
 Small instrument room, 10  
 Smith-Hieftje background correction (AA), 165–166  
 Sodium, separations, 132  
 Sodium acetate, 41  
 Sodium bisulfate, fused, 36, 87  
 Sodium bisulfite, 39  
 Sodium carbonate, 36, 84–85, 102  
 Sodium diethyldithiocarbamate, 116  
 Sodium dithionite, 39  
 Sodium hydroxide, 35–36, 88  
     precipitation, 100–102  
 Sodium meta-arsenite, 38  
 Sodium molybdate, 43  
 Sodium nitrite, 39  
 Sodium oxalate, 38  
 Sodium peroxide, 36, 87–88  
 Sodium pyrophosphate, 42  
 Sodium succinate, 41  
 Sodium sulfite, 39  
 Sodium tetraborate, 36, 85  
 Sodium tetraphenylborate, 109  
 Sodium thiocyanate, 43  
 Sodium thiosulfate, 38  
 Software  
     performance verification, 216  
     X-ray fluorescence, 174–175  
 Solid metals  
     sample preparation, 61–64  
     sampling, 55–56  
 Solid sampling for ICP-OES, 171–172  
 Solubility, metal compounds, 41  
 Solutions  
     synthetic standards, 206–207  
     transferring, 204  
 Solvent, storage shed, 14  
 Solvent extraction, 112–118  
     ion association complex, 113–114  
     masking agents, 112–113  
 Solvent extraction-atomic absorption method, 100  
 Spare equipment storage, 14  
 Spark source mass spectrometer, 179  
 Spark sources, 170–171  
 Specifications, 199–200  
 Specific ion electrodes, 27  
 Specimen, 52  
 Spectral effects, interferences, 165  
 Spectrometry laboratory, 6, 10–11  
 Spectrophotometers, ix–x, 158–159  
 Spectrophotometric determinations, 159–160  
     chemistry, 160–161  
 Spectrophotometric methods, phosphorus, 100  
 Spectrophotometric reagents, 42–43  
 Spiking technique (AA), 163–164  
 Spill control, 210–211  
     kits, 14  
 Spray chamber for ICP-OES, 171  
 Sputtering, 171  
 Stainless steels, austenitic, hood construction, 12–13  
 Standard deviation, 196–197  
 Standardization of titrants, 147–148  
 Standard operating procedures, 215–216  
 Standard reference material, 192  
 Standards, x–xi  
     manufacture, 193–194  
     use, 194–195  
 Stannous chloride, 38–39  
 Steel mill laboratory, x  
 Stoppers, 21–22  
 Strontium, separations, 132  
 Sub-boiling still, 35  
 Subsample, 52  
 Sulfate, precipitation, 108  
 Sulfide, precipitation, 104–105  
 Sulfur  
     distillation, 122  
     measurement, 177–178  
     separations, 132–133, 135  
 Sulfur dioxide, 31–32  
 Sulfuric acid, 33, 41, 73–74, 77  
 Sulfuric acid/nitric acid wet ashing, 83  
 Sulfurous acid, 39  
 Synthetic solution standards, 206–207  
 Systemic error, 191
- T**
- Tannin, 108  
 Tantalum, 18  
     gravimetric measurement, 144  
     ICP/OES determination in high-temperature alloys, 103  
     separations, 133  
 Tartaric, citric, 78  
 Teflon FEP, 17  
 Teflon PFA, 17  
     reagent storage vessels, 46  
 Teflon PTFE (*See* Polytetrafluoroethylene)  
 Teflon TFE (*See* Polytetrafluoroethylene)  
 Tefzel ETFE, 17–18  
 TEHPO, 114–115  
 Tellurium  
     gravimetric measurement, 143  
     separations, 133  
 Terbium, 135  
 Test portion, 52, 61  
     selection, 66–69  
 Test sample, 52, 61  
 Tetraphenylarsonium chloride, 114  
 Thallium, separations, 133  
 Thermal conductivity detector, 181–182  
 Thermal evolution methods, 177–178  
 Thermal shock, glass labware, 15–16
- U**
- Ultrasonic agitation, 80–81  
 Uranium, separations, 134  
 Utility service in laboratories, 5, 13  
 UV/visible molecular absorption, 158–162  
 UV/visible spectrophotometry, 185
- V**
- Vacuum fusion, 178  
 Vacuum pumps, 23  
 Validation, 191–194, 216  
 Vanadium  
     sodium hydroxide precipitation, 101  
     separations, 134  
 Variance, 196  
 V-blender, 64  
 Verification, 194  
 Vials, 20  
 Viton, 18  
 Vitreous quartz, 16, 20  
 Vitreous silica, 16, 20

Volumetric flasks, 21–22  
 Volumetric labware, 15, 21–23  
   cleaning, 203  
 Vycor glass, 15–16, 20

**W**

Wash bottles, 20  
 Waste disposal (chemical), 211  
 Watchglasses, 20  
 Water, 29–30  
   Water, distilled, 5–6, 13  
 Wavelength dispersive X-ray fluorescence spectrometer, 173  
 Wet ashing, 83  
 Wet chemical analysis laboratory, 11–14  
 Wet chemical methods, aluminum, 111–112

Williams-Steiger Occupational Safety and Health Act of 1970, 208  
 Windows, laboratory, 4

**X**

X-ray analyzer crystal, 173  
 X-ray collimator, 173  
 X-ray detectors, 173  
 X-ray fluorescence, x, 18, 24, 67, 169, 172–175  
   emission lines, 173  
   hardware, 173–174  
   software, 174–175  
   trace levels of rare earths, 111  
 X-ray fluorescence laboratory, 11  
 X-ray tube, 173–174  
 Xylene, 44

**Y**

Youden plot, 217  
 Ytterbium, 135  
 Yttrium, 135

**Z**

Zeeman background correction (AA), 165–166  
 Zinc, separations, 134  
 Zinc oxide, 102  
 Zirconia, 19  
 Zirconium, 18  
   crucibles, 18, 20  
   sodium peroxide fusion, 87  
 gravimetric measurement, 145  
 separations, 134–135