<table>
<thead>
<tr>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abbé refractometer, 223–226</td>
</tr>
<tr>
<td>compensating prism adjust drum, 223–224</td>
</tr>
<tr>
<td>eyepiece, 223</td>
</tr>
<tr>
<td>hinged sample prisms, 224</td>
</tr>
<tr>
<td>light source, 224</td>
</tr>
<tr>
<td>light source swivel-arm pivot and lock, 224</td>
</tr>
<tr>
<td>line cord on–off switch, 224</td>
</tr>
<tr>
<td>recirculating water inlet and outlet, 224</td>
</tr>
<tr>
<td>scale adjust knob, 224</td>
</tr>
<tr>
<td>scale/sample field switch, 224</td>
</tr>
<tr>
<td>using, 224–226</td>
</tr>
<tr>
<td>Accidents, 5</td>
</tr>
<tr>
<td>Acetaminophen (Tylenol) preparation, 7</td>
</tr>
<tr>
<td>Activated charcoal, 94, 101–102</td>
</tr>
<tr>
<td>Adapters, 35–38</td>
</tr>
<tr>
<td>with different names, 35–36</td>
</tr>
<tr>
<td>errors in using, 35–36</td>
</tr>
<tr>
<td>thermometer adapter, 36</td>
</tr>
<tr>
<td>Addition, 186–189</td>
</tr>
<tr>
<td>addition funnel, 186</td>
</tr>
<tr>
<td>by Claisen tube, 186</td>
</tr>
<tr>
<td>separatory funnels, 186</td>
</tr>
<tr>
<td>stem, 186</td>
</tr>
<tr>
<td>by three neck flask, 189</td>
</tr>
<tr>
<td>Adsorbents, 199–200</td>
</tr>
<tr>
<td>Adsorption column, in gas chromatography, 230</td>
</tr>
<tr>
<td>Air condensers, 176</td>
</tr>
<tr>
<td>Air peak, in gas chromatography, 229</td>
</tr>
<tr>
<td>Amelioration of Pasteur pipet, 58</td>
</tr>
<tr>
<td>Analytical balance, 46–47</td>
</tr>
<tr>
<td>Anharmonic oscillator, 248</td>
</tr>
<tr>
<td>Anharmonic potential, 248</td>
</tr>
<tr>
<td>Anhydrous calcium chloride, 68</td>
</tr>
<tr>
<td>Anhydrous magnesium sulfate, 69</td>
</tr>
<tr>
<td>Anhydrous potassium carbonate, 69</td>
</tr>
<tr>
<td>Anhydrous sodium carbonate, 69</td>
</tr>
<tr>
<td>Anhydrous sodium sulfate, 69</td>
</tr>
<tr>
<td>Anisotropic field, 283</td>
</tr>
<tr>
<td>Attenuated Total Reflectance (ATR), 268</td>
</tr>
<tr>
<td>Automatic injector, in HPLC, 236</td>
</tr>
<tr>
<td>Autosampler, in HPLC, 241</td>
</tr>
<tr>
<td>Azeotropes, 168</td>
</tr>
<tr>
<td>maximum-boiling azeotrope, 168</td>
</tr>
<tr>
<td>minimum-boiling azeotrope, 168</td>
</tr>
<tr>
<td>Baby Bear, 111</td>
</tr>
<tr>
<td>Back-extraction, 120, 122, 172</td>
</tr>
<tr>
<td>Back-pressure, 244</td>
</tr>
<tr>
<td>Balance analytical balance, 46</td>
</tr>
<tr>
<td>electronic analytical balance, 46–47</td>
</tr>
<tr>
<td>top-loading balance, 46</td>
</tr>
<tr>
<td>Beakers, 53–54</td>
</tr>
<tr>
<td>Benzyl alcohol, NMR of, 288, 290</td>
</tr>
<tr>
<td>Blue Drierite, 69</td>
</tr>
<tr>
<td>Boiling range, 155</td>
</tr>
<tr>
<td>Boiling stones, 131–132, 151</td>
</tr>
<tr>
<td>Boltzmann equation, 274</td>
</tr>
<tr>
<td>Bonded reversed-phase column, 242</td>
</tr>
<tr>
<td>Brand-new boiling stone, 132</td>
</tr>
<tr>
<td>1-Bromobutane preparation, 7</td>
</tr>
<tr>
<td>Bubble trap, 238</td>
</tr>
<tr>
<td>Büchi rotovap, 196</td>
</tr>
<tr>
<td>condenser, 179–180</td>
</tr>
<tr>
<td>heating bath, 181</td>
</tr>
<tr>
<td>lab jack, 181</td>
</tr>
<tr>
<td>on–offspeed control, 181</td>
</tr>
<tr>
<td>rotovap splash trap, 181</td>
</tr>
<tr>
<td>socket joint clip with screw lock, 180</td>
</tr>
<tr>
<td>solvent-sucking inlet, 179</td>
</tr>
<tr>
<td>splash guard, 108, 181</td>
</tr>
<tr>
<td>splash trap, 179</td>
</tr>
<tr>
<td>stopcock at the top, 179</td>
</tr>
<tr>
<td>vacuum connection, 180</td>
</tr>
<tr>
<td>Buchner funnel, 53, 61, 95, 97–100, 103</td>
</tr>
<tr>
<td>Bumping, 153</td>
</tr>
<tr>
<td>Bunsen burner, 133–135</td>
</tr>
<tr>
<td>movable collar, 134</td>
</tr>
<tr>
<td>needle valve, 134</td>
</tr>
<tr>
<td>C-13 NMR, 272</td>
</tr>
<tr>
<td>Caffeine, isolation and purification of, 7</td>
</tr>
</tbody>
</table>
Calcium chloride dust, 68
Calibration of Pasteur pipet, 56–57
Cap branch out, 38
Capillary gas delivery tube, 48–49, 51
Capillary tubes, 75
Carbon tetrachloride (CCl₄), in NMR, 277
Cardboard separator, 11
Carrier gas, 227, 231
CAS Registry Number, 26
Centrifuging the Craig tube, 113
Chaser solvent, 167
Chemical shift, 274–275, 280
Chromatography, 199–201, See also
Gas chromatography (GC); Thin-layer chromatography (TLC); Wet-column chromatography
adsorbents, 199–200
eluotropic series, 200–201
euants, 200
separation or development, 200
Claisen adapter, 163, 188
Clamps/Clamping, 140–149, 151
clamp fastener, 140
clamp holder
thumbscrew, 140
clamping a distillation setup, 142–147
distillation setup, clipping, 147–149
extension clamp, 140, 141
ring-stand thumbscrew, 140
simple buret clamp, 140
three-fingered extension clamp, 142
Classical harmonic oscillator, 245
Clausius–Clapeyron equation, 161
Clay triangle, 96
Cleaning, 66–67
of Buchner funnel, 66
detergent, 66
ebrow grease, 66
solvent, 66
washing, 66
Clear, meaning, 27
Closed-end manometer, 157
Closing off melting-point tubes, 76–77
Cloudiness, 27, 103
Coarse attenuator, 232–233
Cold-finger condenser, 192
Cold recrystallization solvent, 98–99
Colorless, 27
Column holdup, 167
Column of adsorbent, 214
Column oven, 234
Column packing, 164
Column(s), 34, 152
Condensation, 150
Condensers, 35, 154
distilling column versus, 35
Conical vial, 45–47
heating, 47
Conne’s advantage (frequency precision), of FTIR, 265
Constant boiling point, 168
Continuous wave (CW) instrument, 272
Corking a vessel, 40
pressing or rolling, 40
Correlation chart, in NMR, 288–290
garden-variety NMR correlation chart, 289
Correlation tables, 263
Cospotting, in TLC, 210
Coupling constant, 287
Craig tube crystallization, 109–112
Baby Bear, 111
centrifuging Craig tube, 113
Mama Bear, 112
Papa Bear, 112
wire loop for preparing, 110
Crease, 105
Crystallizing dish, 47
Cutting, pipet, 58–60
D
Dampened pulses, 239
Data emphasis, 26
Degenerate protons, 273
Deshielded protons, 280
Destructive visualization, in TLC, 206
Detector oven, 235
Deuterated solvents, 277
Deuterium lock signal, 278
Dipole moment, 248
Dissonant oscillator, 247–248
Distillation, 137, 150–173,
See also Fractional distillation; Microscale distillation; Simple distillation; Steam distillation; Vacuum distillation
boiling range, 155
forerun of, 155
fractional distillation, 152
mistakes in, 155–156
simulated bulb-to-bulb distillation, 172–173
Distillation setup, clamping, 142–147
clamp fastener, 142
extension clamp, 142
ring stand, 142
three-way adapter, 144
vacuum adapter, 145
Distilling column versus condenser, 35
Distilling flask, 152–153
Distribution, 126
Double resonance experiment, 285–288
Downfield shift, 280
Drierite, 69
blue Drierite, 69
Dry-column chromatography, 199
Dry reflux, 185–186
drying tube, 185
indicating Drierite, 186
inlet adapter, 185
Drying, 66–67
drying agents, 68–71
drying tubes, 35, 53, 185
when in use, 67
when not in use, 67
Dual-beam spectrometer, 269
Duplicate carbonless notebooks, 11
Duty cycle, 138
E
Electronic analytical balance, 46–47
Electronic integrations, 282
Electronic interlude, in gas chromatography, 232–233
Electronic stepless controller, 138
Eluatropic series, 200–201
Eluents, 200
Elution solvent, 219
Emulsion, 124–125
Energy levels, quantization of, 247
Equilibrium steps, 166
Erlenmeyer flask, 53, 96
Estimation, 24
Ethanol—Water System, 103–104
Ethylbenzene, NMR of, 285–386
Eutectic mixture, 75
Extension clamp, 142–143
External steam distillation, 168–170
Extraction, 114, 128–130,
See also Microscale extraction and washing; Separatory funnels
back-extraction, 120–121
neutral organic, 120
organic base, 120
performing, 123–125
points to consider, 116–117
starting, 115–116
strong organic acid, 119–120
theory of extraction, 125–127
weakly acidic organic acid, 120
F
F-19 NMR, 272
Fakelrohr setups, 172
jointware, 173
microscale, 173
Williamson test tube, 173
Fan-folded fluted paper, 105–107
Fellgett’s advantage (multiplex advantage), of FTIR, 265
Filament current, 231
Filter adapter, 98
Filter cone, 95
Filter flask, 53, 97–100
Filtering, pipet, 60–62
liquids, 60–61
solids, 61–62
Fingerprint region, in IR spectroscopy, 254
Fisher-Johns apparatus, 82–83
eyepiece, 83
fuse, 82
hot stage, 82
line cord, 82
on–off switch, 82
operation of, 83
stage light, 83
thermometer, 83
thermometer end cap, 83
voltage control, 83
Flash chromatography, 219–220
columns, 220
microscale, 220
Flask, heating, 34
Flasks, 53–54
Flexible double-ended stainless steel spatula, 54–55
transferring powdered solid, 55
Fluted filter paper, 95, 101
Fold, 105
Forerun of distillation, 155
Fourier transform infrared (FTIR) spectroscopy, 264–267
advantages, 265
instrument configuration, 269–271
Michelson interferometer optical system for, 264
optical system, 264–267
reflectance attachment, 268
sample (and reference) cells, 270
solvents, 270–271
Fractional distillation, 152, 153, 164–167, 176
setup, 166
working, 164–167
Free induction decay (FID) signal, 276
Functional group analysis, in IR spectroscopy, 254–256
Fundamental absorption, 248
Funnels, 53–54
funnel stem, 35
G
Garden-variety separatory funnels, 118
Gas chromatography (GC), 227–235, 242
electronic interlude, 232–233
gas–liquid chromatography (GLC), 227, 231
mobile phase, gas, 227–228
parameters, 234–235
sample at the detector, 231–232
sample in the column, 230–231
sample introduction, 228–230
sample on the computer, 233–234
sample preparation, 228
setup, 228
vapor-phase chromatography (VPC), 227, 230
Gas collection apparatus, 48–51
capillary gas delivery tube, 48, 49
from a condenser, 50
directly from a conical vial, 50
gas collection reservoir, 48, 49
generating the gas, 49–51
isolating the product, 51
Gas collection reservoir, 48
Gas inlet tube, 177
Gas–liquid chromatography (GLC), 227, 231
Generic hazardous waste disposal, 6
Google, 26–29, See also Wiki
data emphasis, 26
Terphenyl anomaly, 29
Gradient elution systems, 237
Graduated cylinders, 53
Granular hydrate, 69
Gravity filtration, 94, 95–97
clay triangle, 96
filter cone, 95
fluted filter paper, 95
stemless funnel, 95
wire triangle, 96–97
Greasing the joints, 38–39
to grease or not to grease, 38
into the grease pit, 39
preparation of the joints, 39
Green chemistry and planning an organic synthesis, 8–9
microscale, 9
miniscale, 9
Grignard reaction, 26
Ground-glass joint, 35, 192
Guard column, 239

H
Halogenated organic compounds disposal, 6
Harmonic potential, 245
Heat lamps, 100
Heat sources, 131–139
boiling stones, 131–132
brand-new boiling stone, 132
duty cycle, 138
electronic stepless controller, 138
heating mantle, 135–137
mechanical stepless controller, 137–138
proportional heaters, 137–138
in simple distillation, 151
steam bath, 132–133
stepless controllers, 137–138
thermal lag, 138
variable-voltage transformer, 137
Heating mantle, 135–137
thermowell heating mantle, 136
traditional fiberglass heating mantle, 136
variable-voltage transformer, 135
Heating vials, 47
Hexamethyldisiloxane (HMDS), 278
Hickman still, 176–178
heating, 177–178
recovering product, 178
setup, 176–177
High-performance liquid chromatography (HPLC), 236–244
automatic injector, 236
injection port, 236
moving liquid phase, 237–239
parameters, 243–244
sample at the detector, 242–243
sample in the column, 242
sample preparation, 239–240
sample introduction, 241–242
sample on the computer, 243
setup, 240
Hirsh funnel, 53, 95, 101
Hood, working in, 3
Hot stage, 82–83
Hydrated salt, 68
Hydrogen-1 NMR (1H-NMR), 272

I
Ice bath, 154
Index of refraction, 223
Indicating Drierite, 186
Infrared (IR) spectroscopy, 245–271
asymmetric stretch, 249, 250
of 2-butane, 255
Index

bindex.indd   295
8/29/2015   8:56:32 AM

INDEX  295

correlation tables, 250
of cyclohexanol, 251
of cyclohexone, 255
dipole moment, 248
dissonant oscillator, 247–248
fingerprint region, 254
finishing touches, 263
functional group analysis, 254, 256
functional groups, 253–254
in-plane rocking, 250
in-plane scissoring, 250
interpreting IR spectra, 263
KBr disk, pressing, 261–262
liquid samples
preparation, 258–259
micrometers, 251
normal modes of vibration, 248
out-of-plane twisting, 249
out-of-plane wagging, 250
reciprocal centimeters, 254
running the spectrum, 262–263
sample preparation, 258–262
solid samples preparation, 259–262
stretches and a bends, 249
symmetric stretch, 249, 250
systematic interpretation, region wise, 256–257
of tert-butyl alcohol, 251
troughs and reciprocal centimeters, 254
wavenumbers, 254
Injection loop, in HPLC, 241
Injection port
in gas chromatography, 229
in HPLC, 236, 241
Injector oven, 235
Insoluble, 28, 115
insoluble impurities, 151
Interface, 125
Interferogram, 264
Internal steam distillation, 170–171
Internet, 92
Ionic strength, 105
Isochratic chromatogram, 237
IUPAC (International Union of Pure and Applied Chemistry) name, 26
J
J values, 287
Jacquinot’s advantage (throughput advantage), of FTIR, 265
Jointware, 30–40, 173,
 See also Greasing the joints; Microscale jointware adapters with different names, 35–38
cap branch out, 38
columns, 34–35
condensers, 34–35
corking a vessel, 40
heating a flask, 34
leaking joints, 31
O-ring, 38
round-bottom (R.B.) jointware flasks, 34
standard taper, 30
standard taper jointware, 30
sticking stoppers, 40
stoppers with only one number, 31–33
storing stuff, 40
K
Keck clips, 147–148
Kugelrohr bulb-to-bulb distillation apparatus, 172
L
Lab jack, 181
Larmor frequency, 273
Layers, 115
Leaking joints, 31
Liquid, 41
liquid product problems, 72–73
Loading the melting-point tube, 75–76
Long-stem funnel, 53
Luer-Lok, 63
Luer tip, 64
M
Magnetic spinning vane, 128
Magnetic stirrer, 162
Magnetic stirring bar, 162
Magnetic stirring vane, 108
Mama Bear, 112
Material safety data sheet (MSDS), 8, 28
Maximum-boiling azeotropes, 168
Mechanical stepless controller, 137
Meker burners, 133
Mel-Temp apparatus, 77–78
eyepiece, 78
fuse, 77
light source, 78
line cord, 77
on–off switch, 77
operation of, 79–80
thermometer, 78
voltage control, 78
Melting-point capillaries, 75, 203
Melting-point experiment, 74–91
closing off melting-point tubes, 76–77
Fisher-Johns apparatus, 82–83, See also individual entry
loading the melting-point tube, 75–76
(Continued)
Mel-Temp apparatus, 77–78, See also individual entry
melting-point hints, 77
purity determination by, 74
sample preparation, 75–77
SRS DigiMelt, 81–82
Thiele tube, 88–91, See also individual entry
Thomas-Hoover apparatus, 84–87, See also individual entry
unknowns identification by, 74–75
Melting-point tubes, 75–76
Melting range, 74
Michelson interferometer optical system for FTIR, 264
Microboiling stone, 108
Microliter syringe, 237
Micrometers, 254
Microscale, 9, 173
Microscale, wet-column chromatography, 218–219
Microscale boiling point, 195–198
apparatus, 196
ultramicroscale, 197–198
Microscale distillation, 175–178
Hickman still, 176–178
Microscale drying agents, 70
Microscale drying tube, 48
Microscale extraction and washing, 128–130
mixing, 128
removing both layers, 130
removing the bottom layer, 128–129
removing the top layer, 129–130
Microscale flash chromatography, 220
Microscale jointware, 41–51, See also O-ring cap seal
conical vial, 45–47
microscale drying tube, 47
vacuum-tight seals, 44
Microscale recrystallizations, 108–113, See also Craig tube crystallization
isolating the crystals, 109
Microscale reflux, 190–191
addition and reflux, 190–191
dry, 191
wet, 191
Millimeters of mercury (torr), 158
Mineral oil mull, 259
Minimum-boiling azeotropes, 168
Minipress, 261–262
Miniscale, 9
Miscibility, 103
Miscible liquids, 67, 93
Mixed-solvent recrystallization, 93
Mixed-solvent system advantages, 103–104
disadvantages, 104
Mixed waste disposal, 7–8
acetaminophen (Tylenol) preparation, 7
1-bromobutane preparation, 7
caffeine, isolation and purification of, 7
Mixing, 128
Movable collar, 133
Multiple spotting, in TLC, 209
N
Needle valve, 133
Needles, 63–65
Net weight of products, 73
Neutral organic, extraction and washing of, 120
Nondestructive visualization, in TLC, 206–207
Nonhazardous insoluble liquid waste disposal, 6
Nonhazardous insoluble waste disposal, 5
Nonhazardous soluble liquid waste disposal, 6
Nonhazardous soluble solid waste disposal, 6
Nonjoint, 35
Notebook, keeping, 11–24, 92
duplicate carbonless notebooks, 11
estimation, 24
research notebook, 11
technique experiment, 12–13, See also Synthesis experiment
Nuclear magnetic resonance (NMR), 272–290
basic FT-NMR, 276
C-13 NMR, 272
carbon tetrachloride (CCl4), 277
chemical shift, 274–275
continuous-wave NMR instrumentation, 272
degenerate protons, 273
F-19 NMR, 272
Fourier transform instrumentation, 272
hydrogen-1 NMR (1H-NMR), 272
magnetic catch, 273
P-31 NMR, 272
Proton NMR (PNMR), 272
relaxation processes in, 273
sample preparation, 276–279
terms and interpretations, 280–288
Nujol mull, 259–260
Nujol spectrum, 260

O
Oiling out, 104
Operation of Pasteur pipet, 57–58
Organic base, extraction and washing of, 120
O-ring, 38
O-ring cap seal, 42–44
not-so-skinny apparatus, 43
sizing up the situation, 43–44
skinny apparatus, 42–43
Overtone absorptions, 248
Oxidizing agents disposal, 6

P
P-31 NMR, 272
Papa Bear, 112
Partition coefficient, 126
Pasteur pipet, 57
Pasteur filter pipet, 59
Pasteur pipet wet-column chromatography, 219
pre-preparing, 56–58
p-dichlorobenzene, NMR of, 283
Percent transmission, 254
Percent yield calculation, 22–24
mass calculation, 23
moles calculation, 23
percent recovery, 22
significant figures, 24
stoichiometric factor to get moles, 23
volume of product conversion into the mass, 23
Permissible Exposure Limit (PEL), 8
Phenyl groups, 283
Physical properties, 12
Pipet tips, 56–62, See also Pasteur pipet
cutting, 58, 60
filtering, 60–62
Plate spotter in TLC, 203–204
Potassium bromide (KBr), 258, 260–262, See also under Infrared (IR) spectroscopy
Powder funnel, 53, 96
Powder packing, in wet-column chromatography, 216
Precolumn filter, 239–240
Preparation TLC, 212
Pressure corrections, in vacuum distillation, 159–162
Pressure-equalizing addition funnel, 186
Pressure measurement, in vacuum distillation, 157–158
Product vial, 100
Products, 72–73
liquid product problems, 72–73
net weight, 73
sample vial, 73
solid product problems, 72
tare, 73
Proportional heaters, 137–138
Proton NMR (PNMR), 272
Pulse dampeners, 237, 239
Purification, 92
Purity determination by melting-point experiment, 74
Pusher solvent, 167

Q
Quantum mechanics, 247
Quartet in NMR, 285–286

R
Receiving flask, 154
Reciprocal centimeters, 254
Reciprocating pump, 239
Reciprocating ruby rod, 239
Recovery, 120
Recrystallization, 92–107, See also Microscale recrystallizations
activated charcoal, 94, 101–102
Buchner funnel, 97–100
ethanol—water system, 103–104
fan-folded fluted paper, 105–107
filter flask, 97–100
guidelines for, 94–95
Hirsch funnel, 101
ionic strength, 105
mixed-solvent system, 103–104
oiling out, 104
salting out, 105
solvent, selecting, 93
trituration, 104
water aspirator, 102
Reducing agents disposal, 6
Reference, 231, 242
detector, in gas chromatography, 231
in gas chromatography, 231
Reflectance attachment, 268
Reflux, 137, 183, 186–189, See also Microscale reflux and addition, 186–189
dry, 185–186
standard, 183–184
Reflexing liquid, 177
Refractive index, 27, 223, 225, 242
Refractometry, 222–226, See also Abbé refractometer
Relaxation, 273
in NMR, 275
Research notebook, 11
R value, 208
Ring stand, 142
Ring-stand thumbscrew, 140
Rotary evaporator, 179–182,
  See also Büchi rotovap
Round-bottom (R.B.)
  jointware flasks, 34
  star cracks, 34
Rubber dam, 99
Rubber septum, 65
Rubber stopper, 98

S
Safety, importance, 1–10
  accidents, 5
  allergies, 5
  benchtops, 4–5
  carelessness, 2
  carrying intermediate
  products around, 4
  disobeying safety rules,
  penalties for, 1
  dress code, 2–3
  drive defensively, 2
  getting rid of chemicals, 2
  goggles, 1
  handling, 4
  label on reagent bottle,
  reading correctly, 4
  material safety data sheet
  (MSDS), 8
  medical help, 4
  never work alone, 2
  no flames, 3
  no food in the ice
  machine, 2
  safety devices, 2
  taking food, 2
  waste disposal, 5–8, See
  also individual entry
  working in the hood, 3
  ziploc bags, 10
Salting out, 105
Sample detector, in gas
  chromatography, 231
Sample preparation
  for melting-point
  experiment, 75–77
Sample vial, 73
  neat new label, 73
Scoopula, 53
Selection rule, 247
Semidestructive
  visualization, 207
Sep funnel, 114
Separation
  removing both layers, 130
  removing the bottom
  layer, 128–129
  removing the top layer,
  129–130
Separatory funnels,
  117–119, 186
  garden-variety separatory
  funnels, 118
  glass stopcock, 117–118
  plastic stopper, 118
  stopper, 117
  standard taper glass
  stopper, 117
  Teflon stopcock, 118–119
Septa, 63–65
  rubber septum, 65
Short-stem funnel, 96
Side-arm test tube, 192
Significant figures, 24
Simple buret clamp, 140
Simple distillation, 142,
  151–154, 175
  condenser, 154
  distilling flask, 152–153
  heat sources, 151
  ice bath, 154
  receiving flask, 154
  thermometer, 154
  thermometer adapter, 153
  three-way adapter, 152
  ubiquitous clamp, 153
  vacuum adapter, 154
Signal-to-Noise ratio (SIN),
  269
Skinny apparatus, O-ring
  cap seal for, 42–43
Slurry packing, in
  wet-column
  chromatography, 216
Solid, 41
Solid product problems, 71
Soluble impurities, 151
Solubility data, 28
Solvent, 27–28
Solvent reservoir, 237
Solvent, selecting, 93
Spectra, 28–29
Spin-lattice relaxation, 275
Spin-spin relaxation, 275
Spin–spin splitting, 285
Splash guard, 102
Spotter in TLC, 203–204
SRS DigiMelt, 80–82
  “data” LED, 82
  control panel, 81
  line cord, 80
  melting point tube, 81
  on–off switch, 80
  sample preparation, 81
  temperature data, 80–82
Standard reflux, 183–184
Standard taper, 30
Standard taper jointware, 30
Star cracks, in round-bottom
  (R.B.) jointware flasks,
  34
Steam bath, 64, 132–133
Steam distillation, 150,
  168–171, 175
  external steam
  distillation, 168–170
  internal steam distillation,
  170–171
  steam tap, 168
Steam inlet, 169
Stem, 186
Stemless funnel, 95
Stepless controllers, 137–138
Sticking stoppers, 40
Stoppers with only one
  number, 31
Storing stuff, 40
Strong organic acid,
  extraction and washing
  of, 119–120
Sublimation, 192–194
  inexpensive microscale
  sublimation apparatus,
  193
king-size apparatus, 193
miniature apparatus, 193

Suck-back, 102
Suction flask, See Filter flask
Swinney adapter, 239
Synthesis experiment, 16–19
notebook notes, 17–19
physical constants, 16

Syringes, 63–65
handling rules, 63

Systematic name, See IUPAC (International Union of Pure and Applied Chemistry) name

T
Tare, 73
r-butyl alcohol, NMR of, 282

Technique experiment, 12–13, 20–21
chemical reaction, 20
in-lab, 21
objective, 20
physical constants, 20
post-lab, 21
pre-lab, 20
title, 20

Teflon stopcock, 118–119
leakage in, 119

Temperature corrections, in vacuum distillation, 159–162

Tetramethysilane (TMS), 278
TMS zero, 280–282

Theory of extraction, 125–127

Thermal conductivity detector, 231
Thermal lag, 138
Thermochemistry, 28
Thermometer, 37, 78, 83, 85
Thermometer adapter, 36, 90, 153
Thermowell heating mantle, 136
Thiele tube, 84, 88–91
cleaning the tube, 89
dunking the melting-point tube, 90
getting the sample ready, 89
heating the sample, 91
thermometer adapter, 90
Thin-layer chromatography (TLC), 199, 202–212
concentration effect on a separation, 211
cospotting, 210
developing a plate, 205–206
interpretation, 207–209
multiple spotting, 209
plate spotter, 203–204
preparative TLC, 212
pre-prepared TLC plates, 203
problems in, 210–212
spot too close to the edge, effect of, 212
spots being too close together effect, 211
visualization, 206–207

Thomas-Hoover apparatus, 84–85
capillary tube stage, 85
fluorescent light switches, 84
heat, 85
light box, 84
line cords, 85
operation of, 85–87
power on–off switch, 85
sample viewing area, 85
stirrer motor control, 85
thermometer, 85
thermometer periscope, 85
vibrator on–off switch, 85
Three-fingered extension clamp, 142
Three-neck flask, 163, 188
Three-way adapter, 144, 152

Threshold Limit Value (TLV), 8

Tirrill burners, 133
Toluene, NMR of, 283–284

Top-loading balance, 46
Torr, 158
Total reflux, 166

Toxic heavy metals disposal, 6

Traditional fiberglass heating mantle, 136

Trap drain, 169

Triplet in NMR, 285

Trituration, 104

Troughs, 254

U
Ubiquitous clamp, 153

Ultramicroscale boiling point, 197–198

Ultra violet-visible (UV-Vis) instrumentation, 269

Unknowns identification by melting-point experiment, 74–75
Upfield shift, 280

V
Vacuum adapter, 145, 154, 163

Vacuum distillation, 102, 150, 156–164, 175
air leaks, 158
closed-end manometer, 157
leaks, 158–159
pressure and temperature corrections, 159–164
pressure measurement, 157–158
Vacuum filtration, 102

Vacuum pump, 163

Vacuum source, 163

Vacuum-tight seals, 44

Vaporization, 150

Vapor-phase chromatography (VPC), 227, 230
Variable-voltage transformer, 135–137
Visualization, in TLC, 206–207

W
Washing, 114–127, See also Microscale extraction and washing performing, 123–124
Waste disposal, 5–8
  generic hazardous waste, 6
  halogenated organic compounds, 6
  mixed waste, 7–8
  nonhazardous insoluble liquid waste, 6
  nonhazardous insoluble waste, 5
  nonhazardous soluble liquid waste, 6
  nonhazardous soluble solid waste, 6
  oxidizing agents, 6
  reducing agents, 6
  strong inorganic acids and bases, 6
  toxic heavy metals, 6
Water aspirator, 98, 100, 102, 163, 193
  splash guard, 102
  vacuum distillation, 102
  water trap, 102
Water of crystallization, 68
Water trap, 98–99, 102–103
Wavenumbers, 254
Weakly acidic organic acid, extraction and washing of, 120
Weighing by difference, 100
Wet-column chromatography, 199, 214–219
  compounds on the column, 216–217
  microscale, 218–219
Pasteur pipet, 219
preparing the column, 214–216
professional flash chromatography columns, 220
setup, 215
visualization and collection, 217–218
Wiki, 26–29
  appearance, 26–27
  boiling point, 27
  density, 27
  hazards, 28
  index, 26
  melting point, 27
  molar mass, 26
  molecular formula, 26
  physical properties, 26–27
  refractive index, 27
  solubility data, 28
  solvents, 27–28
  spectra, 28–29
  structures, 26
  systematic name, 26
  text, 26
  thermochemistry, 28
Williamson test tube, 173
Wire triangle, 97

Z
Ziploc bags, 10