
Appendices

1 APPENDIX 1

Tables of Unknowns and Derivatives

More extensive tables of unknowns may be found in Z. Rappoport, ed. *Handbook of Tables for Organic Compound Identification*, 3rd ed. CRC Press: Boca Raton FL, 1967.

ALDEHYDES

Compound	BP	MP	Semi-carbazone*	2,4-Dinitrophenyl-hydrazone*
Ethanal (acetaldehyde)	21	—	162	168
Propanal (propionaldehyde)	48	—	89	148
Propenal (acrolein)	52	—	171	165
2-Methylpropanal (isobutyraldehyde)	64	—	125	187
Butanal (butyraldehyde)	75	—	95	123
3-Methylbutanal (isovaleraldehyde)	92	—	107	123
Pentanal (valeraldehyde)	102	—	—	106
2-Butenal (crotonaldehyde)	104	—	199	190
2-Ethylbutanal (diethylacetaldehyde)	117	—	99	95
Hexanal (caproaldehyde)	130	—	106	104
Heptanal (heptaldehyde)	153	—	109	108
2-Furaldehyde (furfural)	162	—	202	212
2-Ethylhexanal	163	—	254	114
Octanal (caprylaldehyde)	171	—	101	106
Benzaldehyde	179	—	222	237
Nonanal (nonyl aldehyde)	185	—	100	100
Phenylethanal (phenylacetaldehyde)	195	33	153	121
2-Hydroxybenzaldehyde (salicylaldehyde)	197	—	231	248
4-Methylbenzaldehyde (<i>p</i> -tolualdehyde)	204	—	234	234
3,7-Dimethyl-6-octenal (citronellal)	207	—	82	77
Decanal (decyl aldehyde)	207	—	102	104
2-Chlorobenzaldehyde	213	11	229	213
3-Chlorobenzaldehyde	214	18	228	248
3-Methoxybenzaldehyde (<i>m</i> -anisaldehyde)	230	—	233 d.	—
3-Bromobenzaldehyde	235	—	205	—
4-Methoxybenzaldehyde (<i>p</i> -anisaldehyde)	248	2.5	210	253
<i>trans</i> -Cinnamaldehyde	250 d.	—	215	255
3,4-Methylenedioxybenzaldehyde (piperonal)	263	37	230	266 d.
2-Methoxybenzaldehyde (<i>o</i> -anisaldehyde)	245	38	215 d.	254
3,4-Dimethoxybenzaldehyde	—	44	177	261
2-Nitrobenzaldehyde	—	44	256	265
4-Chlorobenzaldehyde	—	48	230	254
4-Bromobenzaldehyde	—	57	228	257
3-Nitrobenzaldehyde	—	58	246	293

ALDEHYDES (Cont.)

Compound	BP	MP	Semi-carbazone*	2,4-Dinitro-phenyl-hydrazone*
2,4-Dimethoxybenzaldehyde	—	71	—	—
2,4-Dichlorobenzaldehyde	—	72	—	—
4-Dimethylaminobenzaldehyde	—	74	222	325
4-Hydroxy-3-methoxybenzaldehyde (vanillin)	—	82	230	271
3-Hydroxybenzaldehyde	—	104	198	259
5-Bromo-2-hydroxybenzaldehyde (5-bromosalicylaldehyde)	—	106	297 d.	—
4-Nitrobenzaldehyde	—	106	221	320 d.
4-Hydroxybenzaldehyde	—	116	224	280 d.
(±)-Glyceraldehyde	—	142	160 d.	167

Note: "d" indicates "decomposition."

*See Appendix 2, "Procedures for Preparing Derivatives."

KETONES

Compound	BP	MP	Semi-carbazone*	2,4-Dinitro-phenyl-hydrazone*
2-Propanone (acetone)	56	—	187	126
2-Butanone (methyl ethyl ketone)	80	—	146	117
3-Buten-2-one (methyl vinyl ketone)	81	—	140	—
3-Methyl-2-butanone (isopropyl methyl ketone)	94	—	112	120
2-Pentanone (methyl propyl ketone)	102	—	112	143
3-Pentanone (diethyl ketone)	102	—	138	156
3,3-Dimethyl-2-butanone (pinacolone)	106	—	157	125
4-Methyl-2-pentanone (isobutyl methyl ketone)	117	—	132	95
2,4-Dimethyl-3-pentanone (diisopropyl ketone)	124	—	160	86
3-Hexanone	125	—	113	130
2-Hexanone (methyl butyl ketone)	128	—	121	106
4-Methyl-3-penten-2-one (mesityl oxide)	130	—	164	200
Cyclopentanone	131	—	210	146
5-Hexen-2-one	131	—	102	108
2,3-Pentanedione	134	—	122 (mono) 209 (di)	209
5-Methyl-3-hexanone	136	—	—	—
2,4-Pentanedione (acetylacetone)	139	—	122 (mono)	209

KETONES (Cont.)

Compound	BP	MP	Semi-carbazone*	2,4-Dinitro-phenyl-hydrazone*
			209 (di)	
4-Heptanone (dipropyl ketone)	144	—	132	75
5-Methyl-2-hexanone	145	—	—	—
1-Hydroxy-2-propanone (hydroxyacetone, acetol)	146	—	196	129
3-Heptanone	148	—	101	—
2-Heptanone (methyl amyl ketone)	151	—	123	89
Cyclohexanone	156	—	166	162
2-Methylcyclohexanone	165	—	191	136
3-Octanone	167	—	—	—
2,6-Dimethyl-4-heptanone (diisobutyl ketone)	168	—	122	66
2-Octanone	173	—	122	92
Cycloheptanone	181	—	163	148
Ethyl acetoacetate	181	—	129 d.	93
5-Nonanone	186	—	90	—
3-Nonanone	187	—	112	—
2,5-Hexanedione (acetylacetone)	191	-9	185 (mono) 224 (di)	257 (di)
2-Nonanone	195	-8	118	—
Acetophenone (methyl phenyl ketone)	202	20	198	238
2-Hydroxyacetophenone	215	28	210	212
1-Phenyl-2-propanone (phenylacetone)	216	27	198	156
Propiophenone (1-phenyl-1-propanone)	218	21	173	191
Isobutyrophenone (2-methyl-1-phenyl-1-propanone)	222	—	181	163
1-Phenyl-2-butanone	226	—	135	—
4-Methylacetophenone	226	28	205	258
3-Chloroacetophenone	228	—	232	—
2-Chloroacetophenone	229	—	160	—
Butyrophenone (1-phenyl-1-butanone)	230	12	187	190
2-Undecanone	231	12	122	63
4-Chloroacetophenone	232	12	204	231
4-Phenyl-2-butanone (benzylacetone)	235	—	142	127
2-Methoxyacetophenone	239	—	183	—
3-Methoxyacetophenone	240	—	196	—
Valerophenone (1-phenyl-1-pentanone)	248	—	160	166
4-Chloropropiophenone	—	36	176	—
4-Phenyl-3-buten-2-one (benzalacetone)	—	37	187	227
4-Methoxyacetophenone	—	38	198	220
3-Bromopropiophenone	—	40	183	—
1-Indanone	—	41	233	258

KETONES (Cont.)

Compound	BP	MP	Semi-carbazone*	2,4-Dinitro-phenyl-hydrazone*
Benzophenone	—	48	164	238
4-Bromoacetophenone	—	51	208	230
3,4-Dimethoxyacetophenone	—	51	218	207
2-Acetonaphthone (methyl 2-naphthyl ketone)	—	53	234	262 d.
Desoxybenzoin (benzyl phenyl ketone)	—	60	148	204
1,1-Diphenylacetone	—	61	170	—
4-Chlorobenzophenone	—	76	—	185
3-Nitroacetophenone	—	80	257	228
4-Nitroacetophenone	—	80	—	—
4-Bromobenzophenone	—	82	350	230
Fluorenone	—	83	—	283
4-Hydroxyacetophenone	—	109	199	210
Benzoin	—	136	206	245
4-Hydroxypropiophenone	—	148	—	229
(±)-Camphor	—	179	237	164

Note: "d" indicates "decomposition."

*See Appendix 2, "Procedures for Preparing Derivatives."

CARBOXYLIC ACIDS

Compound	BP	MP	<i>p</i> -Toluidide*	Anilide*	Amide*
Methanoic acid (formic acid)	101	8	53	47	43
Ethanoic acid (acetic acid)	118	17	148	114	82
Propenoic acid (acrylic acid)	139	13	141	104	85
Propanoic acid (propionic acid)	141	—	124	103	81
2-Methylpropanoic acid (isobutyric acid)	154	—	104	105	128
Butanoic acid (butyric acid)	162	—	72	95	115
3-Butenoic acid (vinylacetic acid)	163	—	—	58	73
2-Methylpropenoic acid (methacrylic acid)	163	16	—	87	102
Pyruvic acid	165 d.	14	109	104	124
3-Methylbutanoic acid (isovaleric acid)	176	—	106	109	135
3,3-Dimethylbutanoic acid	185	—	134	132	132
Pentanoic acid (valeric acid)	186	—	74	63	106
2-Chloropropanoic acid	186	—	124	92	80

CARBOXYLIC ACIDS (Cont.)

Compound	BP	MP	<i>p</i> -Toluidide*	Anilide*	Amide*
Dichloroacetic acid	194	6	153	118	98
2-Methylpentanoic acid	195	—	80	95	79
Hexanoic acid (caproic acid)	205	—	75	95	101
2-Bromopropanoic acid	205 d.	24	125	99	123
Heptanoic acid	223	—	81	70	96
2-Ethylhexanoic acid	228	—	—	—	102
Cyclohexanecarboxylic acid	233	31	—	146	186
Octanoic acid (caprylic acid)	237	16	70	57	107
Nonanoic acid	254	12	84	57	99
Decanoic acid (capric acid)	—	32	78	70	108
4-Oxopentanoic acid (levulinic acid)	—	33	108	102	108 d.
Trimethylacetic acid (pivalic acid)	—	35	120	130	155
3-Chloropropanoic acid	—	40	—	—	101
Dodecanoic acid (lauric acid)	—	43	87	78	100
3-Phenylpropanoic acid (hydrocinnamic acid)	—	48	135	98	105
Bromoacetic acid	—	50	—	131	91
4-Phenylbutanoic acid	—	52	—	—	84
Tetradecanoic acid (myristic acid)	—	54	93	84	103
Trichloroacetic acid	—	57	113	97	141
3-Bromopropanoic acid	—	61	—	—	111
Hexadecanoic acid (palmitic acid)	—	62	98	90	106
Chloroacetic acid	—	63	162	137	121
Cyanoacetic acid	—	66	—	198	120
Octadecanoic acid (stearic acid)	—	69	102	95	109
<i>trans</i> -2-Butenoic acid (crotonic acid)	—	72	132	118	158
Phenylacetic acid	—	77	136	118	156
α -Methyl- <i>trans</i> -cinnamic acid	—	81	—	—	128
4-Methoxyphenylacetic acid	—	87	—	—	189
3,4-Dimethoxyphenyl acetic acid	—	97	—	—	147
Pentanedioic acid (glutaric acid)	—	98	218 (di)	224 (di)	176 (di)
Phenoxyacetic acid	—	99	—	99	102
2-Methoxybenzoic acid (<i>o</i> -anisic acid)	—	100	—	131	129
2-Methylbenzoic acid (<i>o</i> -toluic acid)	—	104	144	125	142
Nonanedioic acid (azelaic acid)	—	106	201 (di)	107 (mono) 186 (di)	93 (mono) 175 (di)
3-Methoxybenzoic acid (<i>m</i> -anisic acid)	—	107	—	—	136
3-Methylbenzoic acid (<i>m</i> -toluic acid)	—	111	118	126	94
4-Bromophenylacetic acid	—	117	—	—	194
(\pm)-Phenylhydroxyacetic acid (mandelic acid)	—	118	172	151	133
Benzoic acid	—	122	158	163	130
2,4-Dimethylbenzoic acid	—	126	—	141	180
2-Benzoylbenzoic acid	—	127	—	195	165

CARBOXYLIC ACIDS (Cont.)

Compound	BP	MP	<i>p</i> -Toluidide*	Anilide*	Amide*
Maleic acid	—	130	142 (di)	198 (mono) 187 (di)	172 (mono) 260 (di)
Decanedioic acid (sebacic acid)	—	133	201 (di)	122 (mono) 200 (di)	170 (mono) 210 (di)
3-Chlorocinnamic acid	—	133	142	135	76
2-Furoic acid	—	133	170	124	143
<i>trans</i> -Cinnamic acid	—	133	168	153	147
2-Acetylsalicylic acid (aspirin)	—	138	—	136	138
5-Chloro-2-nitrobenzoic acid	—	139	—	164	154
2-Chlorobenzoic acid	—	140	131	118	139
3-Nitrobenzoic acid	—	140	162	155	143
4-Chloro-2-nitrobenzoic acid	—	142	—	—	172
2-Nitrobenzoic acid	—	146	—	155	176
2-Aminobenzoic acid (anthranilic acid)	—	146	151	131	109
Diphenylacetic acid	—	148	172	180	167
2-Bromobenzoic acid	—	150	—	141	155
Benzilic acid	—	150	190	175	154
Hexanedioic acid (adipic acid)	—	152	239	151 (mono) 241 (di)	125 (mono) 220 (di)
Citric acid	—	153	189 (tri)	198 (tri)	210 (tri)
4-Nitrophenylacetic acid	—	153	—	198	198
2,5-Dichlorobenzoic acid	—	153	—	—	155
3-Chlorobenzoic acid	—	156	—	123	134
2,4-Dichlorobenzoic acid	—	158	—	—	194
4-Chlorophenoxyacetic acid	—	158	—	125	133
2-Hydroxybenzoic acid (salicylic acid)	—	158	156	136	142
5-Bromo-2-hydroxybenzoic acid (5-bromosalicylic acid)	—	165	—	222	232
3,4-Dimethylbenzoic acid	—	165	—	104	130
2-Chloro-5-nitrobenzoic acid	—	166	—	—	178
Methylenesuccinic acid (itaconic acid)	—	166 d.	—	152 (mono)	191 (di)
(+)-Tartaric acid	—	169	—	180 (mono) 264 (di)	171 (mono) 196 (di)
5-Chlorosalicylic acid	—	172	—	—	227
4-Methylbenzoic acid (<i>p</i> -toluic acid)	—	180	160	145	160
4-Chloro-3-nitrobenzoic acid	—	182	—	131	156
4-Methoxybenzoic acid (<i>p</i> -anisic acid)	—	184	186	169	167
Butanedioic acid (succinic acid)	—	188	180 (mono) 255 (di)	143 (mono) 230 (di)	157 (mono) 260 (di)
4-Ethoxybenzoic acid	—	198	—	170	202
Fumaric acid	—	200 s.	—	233 (mono) 314 (di)	270 (mono) 266 (di)

CARBOXYLIC ACIDS (Cont.)

Compound	BP	MP	<i>p</i> -Toluidide*	Anilide*	Amide*
3-Hydroxybenzoic acid	—	201 s.	163	157	170
3,5-Dinitrobenzoic acid	—	202	—	234	183
3,4-Dichlorobenzoic acid	—	209	—	—	133
Phthalic acid	—	210 d.	150 (mono) 201 (di)	169 (mono) 253 (di)	144 (mono) 220 (di)
4-Hydroxybenzoic acid	—	214	204	197	162
3-Nitrophthalic acid	—	215	226 (di)	234 (di)	201 (di)
Pyridine-3-carboxylic acid (nicotinic acid)	—	236	150	132	128
4-Nitrobenzoic acid	—	240	204	211	201
4-Chlorobenzoic acid	—	242	—	194	179
4-Bromobenzoic acid	—	251	—	197	190

Note: "d" indicates "decomposition"; "s" indicates "sublimation."

*See Appendix 2, "Procedures for Preparing Derivatives."

PHENOLS[†]

Compound	BP	MP	α -Naphthyl- urethane*	Bromo Derivative*			
				Mono	Di	Tri	Tetra
2-Chlorophenol	176	7	120	48	76	—	—
3-Methylphenol (<i>m</i> -cresol)	203	12	128	—	—	84	—
2-Ethylphenol	207	—	—	—	—	—	—
2,4-Dimethylphenol	212	23	135	—	—	—	—
2-Methylphenol (<i>o</i> -cresol)	191	32	142	—	56	—	—
2-Methoxyphenol (guaiacol)	204	32	118	—	—	116	—
4-Methylphenol (<i>p</i> -cresol)	202	35	146	—	49	—	198
3-Chlorophenol	214	35	158	—	—	—	—
4-Methyl-2-nitrophenol	—	35	—	—	—	—	—
2,4-Dibromophenol	238	40	—	95	—	—	—
Phenol	181	42	133	—	—	95	—
4-Chlorophenol	217	43	166	33	90	—	—
4-Ethylphenol	219	45	128	—	—	—	—
2-Nitrophenol	216	45	113	—	117	—	—
2-Isopropyl-5-methylphenol (thymol)	234	51	160	55	—	—	—
4-Methoxyphenol	243	56	—	—	—	—	—
3,4-Dimethylphenol	225	64	141	—	—	171	—
4-Bromophenol	238	64	169	—	—	—	—
4-Chloro-3-methylphenol	235	66	153	—	—	—	—
3,5-Dimethylphenol	220	68	—	—	—	166	—
2,6-Di- <i>tert</i> -butyl-4-methylphenol	—	70	—	—	—	—	—

PHENOLS[†] (Cont.)

Compound	BP	MP	α -Naphthyl- urethane*	Bromo Derivative*			
				Mono	Di	Tri	Tetra
2,4,6-Trimethylphenol	232	72	—	—	—	—	—
2,5-Dimethylphenol	212	75	173	—	—	178	—
1-Naphthol (α -naphthol)	278	94	152	—	105	—	—
2-Methyl-4-nitrophenol	186	96	—	—	—	—	—
2-Hydroxyphenol (catechol)	245	104	175	—	—	—	192
2-Chloro-4-nitrophenol	—	106	—	—	—	—	—
3-Hydroxyphenol (resorcinol)	—	109	—	—	—	112	—
4-Nitrophenol	—	112	150	—	142	—	—
2-Naphthol (β -naphthol)	—	123	157	84	—	—	—
3-Methyl-4-nitrophenol	—	129	—	—	—	—	—
1,2,3-Trihydroxybenzene (pyrogallol)	—	133	—	—	158	—	—
4-Phenylphenol	—	164	—	—	—	—	—

*See Appendix 2, "Procedures for Preparing Derivatives."

[†]Also check:

- Salicylic acid (2-hydroxybenzoic acid)
- Esters of salicylic acid (salicylates)
- Salicylaldehyde (2-hydroxybenzaldehyde)
- 4-Hydroxybenzaldehyde
- 4-Hydroxypropiophenone
- 3-Hydroxybenzoic acid
- 4-Hydroxybenzoic acid
- 4-Hydroxybenzophenone

PRIMARY AMINES[†]

Compound	BP	MP	Benzamide*	Picrate*	Acetamide*
<i>t</i> -Butylamine	46	—	134	198	101
Propylamine	48	—	84	135	—
Allylamine	56	—	—	140	—
<i>sec</i> -Butylamine	63	—	76	139	—
Isobutylamine	69	—	57	150	—
Butylamine	78	—	42	151	—
Isopentylamine (ioamylamine)	96	—	—	138	—
Pentylamine (amylamine)	104	—	—	139	—
Ethylenediamine	118	—	244 (di)	233 (di)	172 (di)
Hexylamine	132	—	40	126	—
Cyclohexylamine	135	—	149	—	101
1,3-Diaminopropane	140	—	148 (di)	250	126 (di)
Furfurylamine	145	—	—	150	—
Heptylamine	156	—	—	121	—

PRIMARY AMINES[†] (Cont.)

Compound	BP	MP	Benzamide*	Picrate*	Acetamide*
Octylamine	180	—	—	112	—
Benzylamine	184	—	105	194	65
Aniline	184	—	163	180	114
2-Methylaniline (<i>o</i> -toluidine)	200	—	144	213	110
3-Methylaniline (<i>m</i> -toluidine)	203	—	125	200	65
2-Chloroaniline	208	—	99	134	87
2,6-Dimethylaniline	216	11	168	180	177
2,5-Dimethylaniline	216	14	140	171	139
3,5-Dimethylaniline	220	—	144	225	—
4-Isopropylaniline	225	—	162	—	102
2-Methoxyaniline (<i>o</i> -anisidine)	225	6	60	200	85
3-Chloroaniline	230	—	120	177	74
2-Ethoxyaniline (<i>o</i> -phenetidine)	231	—	104	—	79
4-Chloro-2-methylaniline	241	29	142	—	140
4-Ethoxyaniline (<i>p</i> -phenetidine)	250	2	173	69	137
3-Bromoaniline	251	18	120	180	87
2-Bromoaniline	250	31	116	129	99
2,6-Dichloroaniline	—	39	—	—	—
4-Methylaniline (<i>p</i> -toluidine)	200	43	158	182	147
2-Ethylaniline	210	47	147	194	111
2,5-Dichloroaniline	251	50	120	86	132
4-Methoxyaniline (<i>p</i> -anisidine)	—	58	154	170	130
2,4-Dichloroaniline	245	62	117	106	145
4-Bromoaniline	245	64	204	180	168
4-Chloroaniline	—	72	192	178	179
2-Nitroaniline	—	72	110	73	92
2,4,6-Trichloroaniline	262	75	174	83	204
Ethyl <i>p</i> -aminobenzoate	—	89	148	—	110
<i>o</i> -Phenylenediamine	258	102	301 (di)	208	185 (di)
2-Methyl-5-nitroaniline	—	106	186	—	151
4-Aminoacetophenone	—	106	205	—	167
2-Chloro-4-nitroaniline	—	108	161	—	139
3-Nitroaniline	—	114	157	143	155
4-Methyl-2-nitroaniline	—	116	148	—	99
4-Chloro-2-nitroaniline	—	118	133	—	104
2,4,6-Tribromoaniline	—	120	200	—	232
2-Methyl-4-nitroaniline	—	130	—	—	202
2-Methoxy-4-nitroaniline	—	138	149	—	153
<i>p</i> -Phenylenediamine	—	140	128 (mono) 300 (di)	—	162 (mono) 304 (di)
4-Nitroaniline	—	148	199	100	215
4-Aminoacetanilide	—	162	—	—	304
2,4-Dinitroaniline	—	180	202	—	120

*See Appendix 2, "Procedures for Preparing Derivatives."

[†]Also check 4-aminobenzoic acid and its esters.

SECONDARY AMINES

Compound	BP	MP	Benzamide*	Picrate*	Acetamide*
Diethylamine	56	—	42	155	—
Diisopropylamine	84	—	—	140	—
Pyrrolidine	88	—	Oil	112	—
Piperidine	106	—	48	152	—
Dipropylamine	110	—	—	75	—
Morpholine	129	—	75	146	—
Diisobutylamine	139	—	—	121	86
<i>N</i> -Methylcyclohexylamine	148	—	85	170	—
Dibutylamine	159	—	—	59	—
Benzylmethylamine	184	—	—	117	—
<i>N</i> -Methylaniline	196	—	63	145	102
<i>N</i> -Ethylaniline	205	—	60	132	54
<i>N</i> -Ethyl- <i>m</i> -toluidine	221	—	72	—	—
Dicyclohexylamine	256	—	153	173	103
<i>N</i> -Benzylaniline	298	37	107	48	58
Indole	254	52	68	—	157
Diphenylamine	302	52	180	182	101
<i>N</i> -Phenyl-1-naphthylamine	335	62	152	—	115

*See Appendix 2, "Procedures for Preparing Derivatives."

TERTIARY AMINES[†]

Compound	BP	MP	Picrate*	Methiodide*
Triethylamine	89	—	173	280
Pyridine	115	—	167	117
2-Methylpyridine (α -picoline)	129	—	169	230
2,6-Dimethylpyridine (2,6-lutidine)	143	—	168	233
4-Methylpyridine (4-picoline)	143	—	167	—
3-Methylpyridine (β -picoline)	144	—	150	92
Tripropylamine	157	—	116	207
<i>N,N</i> -Dimethylbenzylamine	183	—	93	179
<i>N,N</i> -Dimethylaniline	193	—	163	228 d.
Tributylamine	216	—	105	186
<i>N,N</i> -Diethylaniline	217	—	142	102
Quinoline	237	—	203	72/133

Note: "d" indicates "decomposition."

*See Appendix 2, "Procedures for Preparing Derivatives."

[†]Also check nicotinic acid and its esters.

ALCOHOLS

Compound	BP	MP	3,5-Di-nitrobenzoate*	Phenyl-urethane*
Methanol	65	—	108	47
Ethanol	78	—	93	52
2-Propanol (isopropyl alcohol)	82	—	123	88
2-Methyl-2-propanol (<i>t</i> -butyl alcohol)	83	26	142	136
3-Buten-2-ol	96	—	54	—
2-Propen-1-ol (allyl alcohol)	97	—	49	70
1-Propanol	97	—	74	57
2-Butanol (<i>sec</i> -butyl alcohol)	99	—	76	65
2-Methyl-2-butanol (<i>t</i> -pentyl alcohol)	102	-8.5	116	42
2-Methyl-3-butyn-2-ol	104	—	112	—
2-Methyl-1-propanol (isobutyl alcohol)	108	—	87	86
3-Buten-1-ol	113	—	59	25
3-Methyl-2-butanol	114	—	76	68
2-Propyn-1-ol (propargyl alcohol)	114	—	—	—
3-Pentanol	115	—	101	48
1-Butanol	118	—	64	61
2-Pentanol	119	—	62	—
3,3-Dimethyl-2-butanol	120	—	107	77
2,3-Dimethyl-2-butanol	121	—	111	65
2-Methyl-2-pentanol	123	—	72	—
3-Methyl-3-pentanol	123	—	96	43
2-Methoxyethanol	124	—	—	(113) [†]
2-Methyl-3-pentanol	128	—	85	50
2-Chloroethanol	129	—	95	51
3-Methyl-1-butanol (isoamyl alcohol)	132	—	61	56
4-Methyl-2-pentanol	132	—	65	143
2-Ethoxyethanol	135	—	75	(67) [†]
3-Hexanol	136	—	97	—
1-Pentanol	138	—	46	46
2-Hexanol	139	—	39	(61) [†]
2,4-Dimethyl-3-pentanol	140	—	—	95
Cyclopentanol	140	—	115	132
2-Ethyl-1-butanol	146	—	51	—
2,2,2-Trichloroethanol	151	—	142	87
1-Hexanol	157	—	58	42
2-Heptanol	159	—	49	(54) [†]
Cyclohexanol	160	—	113	82
3-Chloro-1-propanol	161	—	77	38
(2-Furyl)-methanol (furfuryl alcohol)	170	—	80	45
1-Heptanol	176	—	47	60
2-Octanol	179	—	32	114
2-Ethyl-1-hexanol	185	—	—	(61) [†]
1-Octanol	195	—	61	74

ALCOHOLS (Cont.)

Compound	BP	MP	3,5-Di-nitrobenzoate*	Phenyl-urethane*
3,7-Dimethyl-1,6-octadien-3-ol (linalool)	196	—	—	66
2-Nonanol	198	—	43	(56) [†]
Benzyl alcohol	204	—	113	77
1-Phenylethanol	204	20	92	95
1-Nonanol	214	—	52	62
1,3-Propanediol	215	—	178 (di)	137 (di)
2-Phenylethanol	219	—	108	78
1-Decanol	231	7	57	59
3-Phenylpropanol	236	—	45	92
1-Dodecanol (lauryl alcohol)	—	24	60	74
3-Phenyl-2-propen-1-ol (cinnamyl alcohol)	250	34	121	90
α -Terpineol	221	36	78	112
1-Tetradecanol (myristyl alcohol)	—	39	67	74
(-)-Menthol	212	41	158	111
1-Hexadecanol (cetyl alcohol)	—	49	66	73
2,2-Dimethyl-1-propanol (neopentyl alcohol)	113	56	—	144
4-Methylbenzyl alcohol	217	59	117	79
1-Octadecanol (stearyl alcohol)	—	59	77	79
Diphenylmethanol (benzhydrol)	—	68	141	139
4-Nitrobenzyl alcohol	—	93	157	—
Benzoin	—	136	—	165
Cholesterol	—	147	—	168
Triphenylmethanol	—	161	—	—
(+)-Borneol	—	208	154	138

*See Appendix 2, "Procedures for Preparing Derivatives."

[†] α -Naphthylurethane.**ESTERS**

Compound	BP	MP	Compound	BP	MP
Methyl formate	32	—	Ethyl chloroformate	93	—
Ethyl formate	54	—	Methyl isobutyrate	93	—
Methyl acetate	57	—	(methyl 2-methylpropanoate)		
Isopropyl formate	71	—	2-Propenyl acetate (isopropenyl acetate)	94	—
Vinyl acetate	72	—	<i>tert</i> -Butyl acetate		
Ethyl acetate	77	—	(1,1-dimethylethyl acetate)	98	—
Methyl propionate (methyl propanoate)	80	—	Ethyl propionate (ethyl propanoate)	99	—
Methyl acrylate	80	—	Methyl methacrylate		
Propyl formate	81	—	(methyl 2-methylpropenoate)	100	—
Isopropyl acetate	89	—	Methyl pivalate		
			(methyl trimethyl acetate)	101	—

ESTERS (Cont.)

Compound	BP	MP	Compound	BP	MP
Ethyl acrylate (ethyl propenoate)	101	—	Ethyl heptanoate	187	—
Propyl acetate	102	—	Heptyl acetate	192	—
Methyl butyrate (methyl butanoate)	102	—	Dimethyl succinate	196	—
Ethyl isobutyrate			Phenyl acetate	197	—
(ethyl 2-methylpropanoate)	110	—	Diethyl malonate	199	—
Isopropyl propionate			Methyl benzoate	199	—
(isopropyl propanoate)	110	—	Dimethyl maleate	204	—
2-Butyl acetate (<i>sec</i> -butyl acetate)	111	—	Ethyl levulinate	206	—
Methyl isovalerate	117	—	Ethyl octanoate	208	—
(methyl 3-methylbutanoate)			Ethyl cyanoacetate	208	—
Isobutyl acetate			Ethyl benzoate	212	—
(2-methylpropyl acetate)	117	—	Benzyl acetate	217	—
Ethyl pivalate			Diethyl succinate	217	—
(ethyl 2,2-dimethylpropanoate)	118	—	Diethyl fumarate	219	—
Methyl crotonate (methyl 2-butenolate)	119	—	Methyl phenylacetate	220	—
Ethyl butyrate (ethyl butanoate)	121	—	Methyl salicylate	224	—
Propyl propionate (propyl propanoate)	123	—	Diethyl maleate	224	—
Butyl acetate	126	—	Ethyl phenylacetate	228	—
Methyl valerate (methyl pentanoate)	128	—	Propyl benzoate	231	—
Methyl methoxyacetate	130	—	Ethyl salicylate	234	—
Methyl chloroacetate	130	—	Dimethyl suberate	268	—
Ethyl isovalerate			Ethyl cinnamate	271	—
(ethyl 3-methylbutanoate)	134	—	Dimethyl phthalate	284	—
Ethyl crotonate (ethyl 2-butenolate)	138	—	Diethyl phthalate	298	—
Isopentyl acetate			Methyl cinnamate	—	36
(3-methylbutyl acetate)	142	—	Ethyl 2-furoate	—	36
2-Methoxyethyl acetate	145	—	Methyl stearate	—	39
Ethyl chloroacetate	145	—	Dimethyl itaconate	—	39
Ethyl valerate (ethyl pentanoate)	146	—	Phenyl salicylate	—	42
Ethyl α -chloropropanoate	146	—	Diethyl terephthalate	—	44
Pentyl acetate	147	—	Methyl 4-chlorobenzoate	—	44
Methyl hexanoate	151	—	Ethyl 3-nitrobenzoate	—	47
Ethyl lactate	154	—	Methyl mandelate	—	53
Butyl butyrate	167	—	Ethyl 4-nitrobenzoate	—	56
Ethyl hexanoate	168	—	Dimethyl isophthalate	—	68
Hexyl acetate	169	—	Phenyl benzoate	—	69
Methyl acetoacetate	170	—	Methyl 3-nitrobenzoate	—	78
Methyl heptanoate (methyl enanthlate)	172	—	Methyl 4-bromobenzoate	—	81
Furfuryl acetate	176	—	Ethyl 4-aminobenzoate	—	89
Methyl 2-furoate	181	—	Methyl 4-nitrobenzoate	—	96
Dimethyl malonate	181	—	Dimethyl fumarate	—	102
Ethyl acetoacetate	181	—	Cholesterol acetate	—	114
Diethyl oxalate	185	—	Ethyl 4-hydroxybenzoate	—	116

Procedures for Preparing Derivatives

CAUTION

Some of the chemicals used in preparing derivatives are suspected carcinogens. Before beginning any of these procedures, consult the list of suspected carcinogens on pp. 561–562. Exercise care in handling these substances.

ALDEHYDES AND KETONES

Semicarbazones

Place 0.5 mL of a 2M stock solution of semicarbazide hydrochloride (or 0.5 mL of a solution prepared by dissolving 1.11 g of semicarbazide hydrochloride [$MW = 111.5$] in 5 mL of water) in a small test tube. Add 0.15 g of the unknown compound to the test tube. If the unknown does not dissolve in the solution or if the solution becomes cloudy, add enough methanol (maximum of 2 mL) to dissolve the solid and produce a clear solution. If a solid or cloudiness remains after adding 2 mL of methanol, do not add any more methanol and continue this procedure with the solid present. Using a Pasteur pipet, add 10 drops of pyridine and heat the mixture in a hotwater bath (about 60°C) for about 10–15 minutes. By that time, the product should have begun to crystallize. Collect the product by vacuum filtration. The product can be recrystallized from ethanol if necessary.

**Semicarbazones
(Alternative Method)**

Dissolve 0.25 g of semicarbazide hydrochloride and 0.38 g of sodium acetate in 1.3 mL of water. Then dissolve 0.25 g of the unknown in 2.5 mL of ethanol. Mix the two solutions together in a 25-mL Erlenmeyer flask and heat the mixture to boiling for about 5 minutes. After heating the mixture, place the reaction flask in a beaker of ice and scratch the sides of the flask with a glass rod to induce crystallization of the derivative. Collect the derivative by vacuum filtration and recrystallize it from ethanol.

2, 4-Dinitrophenylhydrazones

Place 10 mL of a solution of 2, 4-dinitrophenylhydrazine (prepared as described for the classification test in Experiment 54D) in a test tube and add 0.15 g of the unknown compound. If the unknown is a solid, it should be dissolved in the minimum amount of 95% ethanol or 1,2-dimethoxyethane before it is added. If crystallization is not immediate, gently warm the solution for a minute in a hotwater bath (90°C) and then set it aside to crystallize. Collect the product by vacuum filtration.

CARBOXYLIC ACIDS

Working in a hood, place 0.50 g of the acid and 2 mL of thionyl chloride into a small round-bottom flask. Add a magnetic stir bar, and attach a water-jacketed condenser and a drying tube packed with calcium chloride to the flask. While stirring, heat the reaction mixture to boiling for 30 minutes on a hot plate. Allow the mixture to cool

to room temperature. Use this mixture to prepare the amide, anilide, or *p*-toluidide derivatives by one of the following three procedures.

Amides

Working in a hood, add the thionyl chloride/carboxylic acid mixture dropwise from a Pasteur pipet into a beaker containing 5 mL of ice-cold concentrated ammonium hydroxide. The reaction is very exothermic. Stir the mixture vigorously after the addition for about 5 minutes. When the reaction is complete, collect the product by vacuum filtration and recrystallize it from water or from water-ethanol, using the mixed-solvents method (See Technique 11, Section 11.10).

Anilides

Dissolve 0.5 g of aniline in 13 mL of methylene chloride in a 50-mL Erlenmeyer flask. Using a Pasteur pipet, carefully add the mixture of thionyl chloride/carboxylic acid to this solution. Warm the mixture for an additional 5 minutes on a hot plate, unless a significant color change occurs. *If a color change occurs*, discontinue heating, add a magnetic stir bar, and stir the mixture for 20 minutes at room temperature. Then transfer the methylene chloride solution to a small separatory funnel and wash it sequentially with 2.5 mL of water, 2.5 mL of 5% hydrochloric acid, 2.5 mL of 5% sodium hydroxide, and a second 2.5-mL portion of water (the methylene chloride solution should be the bottom layer). Dry the methylene chloride layer over a small amount of anhydrous sodium sulfate. Decant the methylene chloride layer away from the drying agent into a small flask and evaporate the methylene chloride on a warm hot plate in the hood. Use a stream of air or nitrogen to speed up the evaporation. Recrystallize the product from water or from ethanol-water, using the mixed-solvents method (See Technique 11, Section 11.10).

p-Toluidides

Use the same procedure as that described in preparing anilides, but substitute *p*-toluidine for aniline.

PHENOLS

α -Naphthylurethanes

Follow the procedure given later for preparing phenylurethanes from alcohols, but substitute α -naphthylisocyanate for phenylisocyanate.

Bromo Derivatives

First, if a stock brominating solution is not available, prepare one by dissolving 0.75 g of potassium bromide in 5 mL of water and adding 0.5 g of bromine. Dissolve 0.1 g of the phenol in 1 mL of methanol or 1,2-dimethoxyethane; then add 1 mL of water. Add 1 mL of the brominating mixture to the phenol solution and swirl the mixture vigorously. Then continue adding the brominating solution dropwise while swirling, until the color of the bromine reagent persists. Finally, add 3–5 mL of water and shake the mixture vigorously. Collect the precipitated product by vacuum filtration and wash it well with water. Recrystallize the derivative from methanol-water, using the mixed-solvents method (See Technique 11, Section 11.10).

AMINES

Acetamides

Place 0.15 g of the amine and 0.5 mL of acetic anhydride in a small Erlenmeyer flask. Heat the mixture for about 5 minutes; then add 5 mL of water and stir the solution vigorously to precipitate the product and hydrolyze the excess acetic anhydride. If the product does not crystallize, it may be necessary to scratch the

walls of the flask with a glass rod. Collect the crystals by vacuum filtration and wash them with several portions of cold 5% hydrochloric acid. Recrystallize the derivative from methanol–water, using the mixed-solvents method (See Technique 11, Section 11.10).

Aromatic amines, or those amines that are not very basic, may require pyridine (2 mL) as a solvent and a catalyst for the reaction. If pyridine is used, a longer period of heating is required (up to 1 hour), and the reaction should be carried out in an apparatus equipped with a reflux condenser. After reflux, the reaction mixture must be extracted with 5–10 mL of 5% sulfuric acid to remove the pyridine.

Benzamides

Using a centrifuge tube, suspend 0.15 g of the amine in 1 mL of 10% sodium hydroxide solution and add 0.5 g of benzoyl chloride. Cap the tube and shake the mixture vigorously for about 10 minutes. After shaking the mixture, add enough dilute hydrochloric acid to bring the pH of the solution to pH 7 or 8. Collect the precipitate by vacuum filtration, wash it thoroughly with cold water, and recrystallize it from ethanol–water, using the mixed-solvents method (See Technique 11, Section 11.10).

Benzamides (Alternative Method)

In a small round-bottom flask, dissolve 0.25 g of the amine in a solution of 1.2 mL of pyridine and 2.5 mL of toluene. Add 0.25 mL of benzoyl chloride to the solution, and heat the mixture under reflux for about 30 minutes. Pour the cooled reaction mixture into 25 mL of water, and stir the mixture vigorously to hydrolyze the excess benzoyl chloride. Separate the toluene layer and wash it, first with 1.5 mL of water, and then with 1.5 mL of 5% sodium carbonate. Dry the toluene over granular anhydrous sodium sulfate, decant the toluene into a small Erlenmeyer flask, and remove the toluene by evaporation on a hot plate in the hood. Use a stream of air or nitrogen to speed up the evaporation. Recrystallize the benzamide from ethanol or ethanol–water, using the mixed-solvents method (See Technique 11, Section 11.10).

Picrates

In an Erlenmeyer flask, dissolve 0.2 g of the unknown in about 5 mL of ethanol and add 5 mL of a saturated solution of picric acid in ethanol. Heat the solution to boiling and then allow it to cool slowly. Collect the product by vacuum filtration and rinse it with a small amount of cold ethanol.

CAUTION



Great care must be taken when working with saturated solutions of picric acid. Picric acid may detonate when heated above 300°C. It is also known to explode when hearted rapidly. For this reason, it is strongly recommended that you check with your instructor before preparing this derivation.

Methiodides

Mix equal-volume quantities of the amine and methyl iodide in a small round-bottom flask (about 0.25 mL is sufficient) and allow the mixture to stand for several minutes. Then heat the mixture gently under reflux for about 5 minutes. The methiodide should crystallize on cooling. If it does not, you can induce crystallization by scratching the walls of the flask with a glass rod. Collect the product by vacuum filtration and recrystallize it from ethanol or ethyl acetate.

ALCOHOLS

3,5-Dinitrobenzoates

Liquid Alcohols

Dissolve 0.25 g of 3,5-dinitrobenzoyl chloride in 0.25 mL of the alcohol and heat the mixture for about 5 minutes¹. Allow the mixture to cool and add 1.5 mL of a 5% sodium carbonate solution and 1 mL of water. Stir the mixture vigorously and crush any solid that forms. Collect the product by vacuum filtration, and wash it with cold water. Recrystallize the derivative from ethanol–water, using the mixed-solvents method (See Technique 11, Section 11.10).

Solid Alcohols

Dissolve 0.25 g of the alcohol in 1.5 mL of dry pyridine and add 0.25 g of 3,5-dinitrobenzoyl chloride. Heat the mixture under reflux for 15 minutes. Pour the cooled reaction mixture into a cold mixture of 2.5 mL of 5% sodium carbonate and 2.5 mL of water. Keep the solution cooled in an ice bath until the product crystallizes, and stir it vigorously during the entire period. Collect the product by vacuum filtration, wash it with cold water, and recrystallize it from ethanol–water, using the mixed-solvents method (See Technique 11, Section 11.10).

Phenylurethanes

Place 0.25 g of the *anhydrous* alcohol in a dry test tube and add 0.25 mL of phenylisocyanate (*α*-naphthylisocyanate for a phenol). If the compound is a phenol, add 1 drop of pyridine to catalyze the reaction. If the reaction is not spontaneous, heat the mixture in a hot-water bath (90°C) for 5–10 minutes. Cool the test tube in a beaker of ice, and scratch the tube with a glass rod to induce crystallization. Decant the liquid from the solid product or, if necessary, collect the product by vacuum filtration. Dissolve the product in 2.5–3 mL of hot ligroin or hexane, and filter the mixture by gravity (preheat funnel) to remove any unwanted and insoluble diphenylurea present. Cool the filtrate to induce crystallization of the urethane. Collect the product by vacuum filtration.

ESTERS

We recommend that esters be characterized by spectroscopic methods whenever possible. A derivative of the alcohol part of an ester can be prepared with the following procedure. For other derivatives, consult a comprehensive textbook. Several are listed in Experiment 55I.

3,5-Dinitrobenzoates

Place 1.0 mL of the ester and 0.75 g of 3,5-dinitrobenzoic acid in a small round-bottom flask. Add 2 drops of concentrated sulfuric acid and a magnetic stir bar to the flask and attach a condenser. If the boiling point of the ester is above 150°C, heat at reflux while stirring for 30–45 minutes. If the boiling point of the ester is above 150°C, heat the mixture at about 150°C for 30–45 minutes. Cool the mixture, and transfer it to a small separatory funnel. Add 10 mL of ether. Extract the ether layer 2 times with 5 mL of 5% aqueous sodium carbonate (save the ether layer). Wash the organic layer with 5 mL of water, and dry the ether solution over magnesium sulfate. Evaporate the ether in a hot-water bath in the hood. Use a stream of air or

¹ 3,5-Dinitrobenzoyl chloride is an acid chloride and hydrolysis readily. The purity of this reagent should be checked before its use by determining its melting point (mp 69–71°C). When the carboxylic acid is present, the melting point will be high.

nitrogen to speed the evaporation. Dissolve the residue, usually an oil, in 2 mL of boiling ethanol and add water dropwise until the mixture becomes cloudy. Cool the solution to induce crystallization of the derivative.

**Preparation of a Solid
Carboxylic Acid from an
Ester.**

An excellent derivative of an ester can be prepared by a basic hydrolysis of an ester when it yields a solid carboxylic acid. A procedure is provided in Experiment 55I. Melting points for solid carboxylic acids are included in the Carboxylic Acids Table in Appendix 1.

Index of Spectra

INFRARED SPECTRA

Anisole 875
Benzaldehyde 274, 878
Benzamide 882
Benzil 276
Benzilic acid 279
Benzocaine 351
Benzoic acid 289, 880
Benzoin 273
Benzonitrile 877
Borneol 261
n-Butyl bromide 179
n-Butylamine 876
Camphor 260, 880
Carbon disulfide 863
Carbon tetrachloride 860
Carvone 109
Chloroform 860
Decane 870
1,2-Dichlorobenzene 874
N,N-Diethyl-*m*-toluamide 363
6-Ethoxycarbonyl-3,5-diphenyl-2-cyclohexenone 325
Isoborneol 261
Isopentyl acetate 92, 881
Limonene 109
Mesityl oxide 879
Methyl benzoate 881
Methyl isopropyl ketone 866
Methyl *m*-nitrobenzoate 344
4-Methylcyclohexanol 185, 875
4-Methylcyclohexene 183, 871
Mineral oil 859
2-Naphthol 874
Nitrobenzene 876
Nonanal 878
cis-Norborene-5,6-*endo*-dicarboxylic anhydride 410, 882
Nujol 859
Paraffin oil 859
t-Pentyl chloride 180
Polystyrene 863
Styrene 872
Sulfanilamide 372
Triphenylmethanol 287

¹H NMR SPECTRA

Benzocaine 352
Benzyl acetate (60-MHz NMR spectrum) 893
Benzyl acetate (300-MHz NMR spectrum) 894
Borneol 262
Camphor 262
Carvone 110
N,N-Diethyl-*m*-toluamide 364
6-Ethoxycarbonyl-3,5-diphenyl-2-cyclohexenone 326
Ethyl 3-hydroxybutanoate 233
1-Hexanol 914
(*E*)-4-(4-Hydroxy-3-methoxyphenyl)-2-buten-1-ol 309
(*R*)-4-Hydroxy-5-methyl-2-hexanone 320
Isoborneol 263
Limonene 110
Phenylacetone 887
1,1,2-Trichloroethane 900
Vegetable oil 217
Vinyl acetate 904

¹³C NMR SPECTRA

Borneol 264
Camphor 263
Carvone 111
Cyclohexanol 929
Cyclohexanone 930
Cyclohexene 930
2,2-Dimethylbutane 928
Ethyl phenylacetate 926
Isoborneol 264
1-Propanol 927
Toluene 931

MASS SPECTRA

Acetophenone 954
Benzaldehyde 953
Bromoethane 945
1-Bromohexane 957
Butane 948
1-Butanol 952
2-Butanone 954
1-Butene 950
Chloroethane 944
Cyclopentane 950
Dopamine 941
Methyl butanoate 956

Propanoic acid 955
Toluene 951
2,2,4-Trimethylpentane 949

ULTRAVIOLET-VISIBLE SPECTRA

Benzophenone 418
Naphthalene 418

MIXTURES

Borneol and isoborneol 265
t-Butyl chloride and *t*-butyl bromide 172
1-Chlorobutane and 1-bromobutane 172
Sonogashira coupling products 299–301

Index

- A**
- Ab initio calculations, 141–148
- Acetamides, 483; preparation, 987–988
- p*-Acetamidobenzenesulfonyl chloride: preparation, 368
- Acetaminophen, 55, 60, 69; experiment, 64–67; preparation, 64
- Acetanilide, 60, 61; chlorosulfonation, 366
- Acetate esters: preparation, 494–497
- Acetic acid: hazards, 559
- Acetone: hazards, 559; HNMR spectrum, 318; reaction with isobutyraldehyde, 313–314; tautomerism, 150–151; wash, 571
- Acetophenone: aldol condensation, 309, 310, 315; condensation with aldehydes, 535; mass spectrum, 955
- Acetyl chloride test, 482, 485
- Acetylation: *p*-aminophenol, 64; aromatic substrates, 508–509; isopentyl alcohol, 88; salicylic acid, 56–57
- (S)-(+)-*O*-Acetylmandelic acid: use as a chiral resolving agent, 248
- Acetylsalicylic acid: preparation, 56–57. *See also* Aspirin
- Acid hydrazides, 491
- Acids: chlorides, 884; concentrations (*see inside front cover*); removal by extraction, 28, 700; solubility, 456–458. *See also* Carboxylic acids
- Acid-washed alumina, 778
- Activated adsorbent, 783
- Activated charcoal, 675
- Actual yield, 567
- Acylglycerol, 183
- Adapter: Neoprene, 577; thermometer, 576, 723; vacuum, 576, 724
- Addition funnel, 615
- Addition methods: liquid reagents, 615–617
- Addition polymers, 372, 373–374, 376
- Addition reactions, 465; bromine to 4-methylcyclohexene, 180; bromine to unknowns, 464; reactivity of carbonyl groups, 154; regioselectivity calculation, 150–151
- Adjustable metal clamps, 609
- Adsorb, 778
- Adsorbents, 778; interactions, 778–779
- Aggregation pheromones, 351
- Air drying, 677
- Air peak, 824
- Alarm pheromones, 351, 354
- Alcohol: derivatives, 488, 983–984, 989; esterification, 494–497; identification tests, 483–488; infrared spectroscopy, 487–488, 874; NMR spectroscopy, 488; oxidation, 541; reaction of ketones to, 240; tables of unknowns, 983–984
- Aldehyde enigma, 520–522
- Aldehydes, 459, 468–474; derivatives, 474, 973–974, 986; identification tests, 446; infrared spectroscopy, 474, 878–879; NMR spectroscopy, 474, 900; tables of unknowns, 973–974
- Aldol condensation, 309–310, 320–321, 535–539; chalcone preparation, 523–527; green chemistry, 313–316
- Aldrich Handbook of Fine Chemicals*, 584–585, 960, 967
- Alembic, 719
- Alkaloid, 74
- Alkanes: infrared spectroscopy, 871
- Alkenes: C–H out-of-plane bending, 871–872; identification tests, 464; infrared spectroscopy, 467, 871–872; NMR spectroscopy, 900, 904
- Alkyl chlorides: preparation, 172–173
- Alkyl halides: identification tests, 459; preparation, 175; reactivities, 158–162. *See also* Halides
- Alkylates, 197
- Alkylation, 197
- Alkylbenzenes, 909
- Alkynes: identification tests, 464; infrared spectroscopy, 873–874; NMR spectroscopy, 467, 900
- 4-Allyloxyanisole: NMR spectrum, 912
- Alumina, 778
- Alumina G, 802
- Aluminum heating block, 602–604
- Ambident nucleophile, 312
- Amide derivatives, 476; preparation, 987
- Amide formation, 64; *p*-aminophenol, 64; 3-nitrophthalic acid, 424; *m*-toluic acid, 359
- Amides: infrared spectroscopy, 883
- Amines: derivatives, 483, 980–982, 987–988; infrared spectroscopy, 482, 876–877; NMR spectroscopy, 482; tables of unknowns, 980–982
- p*-Aminobenzoic acid: action, 364–365; esterification, 347
- p*-Aminophenol, 64; acetylation, 64
- 5-Aminophthalhydrazide: preparation, 424
- Analgesics, 53; composition, 62–63; essay, 60–64; thin-layer chromatography, 69–73
- Analyzer, 839
- Anesthetics: essay, 343–347; preparation, 347; structures, 346
- Anethole: NMR spectrum, 912
- Angina pectoris, 74
- Anhydrides: infrared spectroscopy, 884; ring opening, 391
- Anilide derivatives: preparation, 987
- Anisaldehyde: aldol condensation, 309, 310
- Anisole: infrared spectrum, 876; NMR spectrum, 910
- Anisotropy in NMR spectroscopy, 900–901, 910–911
- Anthracene-9 methanol, 410
- Antihistamines: GC-MS analysis, 516–517
- Anti-inflammatory, 53
- Antiplatelet, 53
- Antipyretic, 53, 60
- APC tablets, 61
- Apparatus assembly, 573; macroscale, 609–610; microscale, 611; securing, 609
- Aqueous-based organozinc reaction, 289–292
- Arachidonic acid, 55
- Aromatic compounds: C–H out-of-plane bending, 874; Friedel-Crafts acylation, 508–515; infrared spectroscopy, 468, 873; nitration, 236; NMR spectroscopy, 468, 897, 905, 908–914
- Aromatic halides: carbonylation, 518–520
- Aromatic rings: compounds, 932–934
- Aromatic substitution: acetanilide, 334; acylation, 508–509; aniline, 334; anisole, 334; chlorosulfonation, 366–367; methyl benzoate, 339; nitration, 339; relative reactivities, 333–335
- Aromaticity: detection, 467–468
- Aspartame, 430, 441; structure, 442
- Aspirator, 638–639; vacuum distillation, 754; removal of noxious gases, 621–622
- Aspirator trap, 639, 639, 752
- Aspirin, 69; baby, 55; buffered, 55; combination tablets, 55; essay, 53–55; preparation, 56–57; tablets, 55
- Atom economy, 222, 236
- Atomic weights. *See inside front cover*
- Automatic pipet, 595
- Azeotropes, 740–748
- Azeotropic distillation: applications, 744–747; microscale apparatus, 746
- B**
- Baby aspirin, 55
- Back extraction, 702
- Backwashing, 702
- Bactericidal, 364
- Bacteriostatic, 364

- Baeyer test, 466–467
 Balance: analytical, 596, 597; draft shield, 596; top-loading, 596
 Banana oil: preparation, 88
 Band, 786
 Barfoed's test, 431, 438–441
 Base peak, 942
 Bases: concentrations (*see inside front cover*); removal by extraction, 28, 700–701
 Basic alumina, 778
 Basic hydrolysis of esters test, 490
 Basis set orbitals, 142–144
 Beakers: measuring volume with, 596
Beilstein Handbuch der Organischen Chemie, 965–968, 970
Beilstein reference, 581
 Beilstein test, 459–460
 Benedict's test, 431, 436, 437
 Benzalacetophenone: preparation, 309, 523
 Benzaldehyde, 315; aldol condensation, 309, 310; benzoin condensation, 266; infrared spectrum, 272, 879; mass spectrum, 954; NMR spectrum, 912
 Benzamide, 483; infrared spectrum, 883; preparation, 988
 Benzene: hazards, 559
 Benzil: infrared spectrum, 274; preparation, 272–274; rearrangement, 275
 Benzoic acid: infrared spectrum, 277; preparation, 274–277; rearrangement, 275
 Benzocaine, 345; infrared spectrum, 349; NMR spectrum, 350; preparation, 347
 Benzoic acid: infrared spectrum, 287, 881; preparation, 281, 286–288; structure, 442
 Benzoin: coenzyme synthesis, 267; condensation, 266; infrared spectrum, 271; oxidation, 272
 Benzotrile: infrared spectrum, 878
 Benzophenone: photoreduction, 411, 412–417; ultraviolet spectrum, 416
 Benzopinacolone: preparation, 419
 Benzpinacol: preparation, 411; rearrangement, 419
 Benzyl acetate: 300-MHz NMR spectrum, 896; NMR spectrum, 894
N-Benzylamides, 491
 Benzyltriphenylphosphonium chloride: preparation, 327
 Beriberi, 266
 Bial's test, 431, 435
 Binding energy, 146
 Biodiesel, 209–211; analysis of, 214–216; coconut oil, 213; experiment, 211–216
 Biofuels, 207–211
 Bio-Gel P, 798
 Bioluminescence, 421
 Biphenyl, 280
 Bisphenol-A, 378, 379
 Bleach: as an oxidizing agent, 222
 Bleed valve, 763
 Boileezer, 615
 Boiling chip, 615
 Boiling point, 709–717; construction of microcapillary bell, 713; finding literature values, 579–586; lower, 745; pressure correction, 709–710; pressure-temperature nomograph, 710
 Boiling point determination, 709–717; experiment, 49–53; by reflux, 711
 Boiling stones, 615
 Boll, Franz, 111
 Bond-density surface, 147
 Bonds: cleavage, 947–948
 Borneol: C-13 NMR spectrum, 262; infrared spectrum, 259; NMR spectrum, 260; oxidation, 251
 Borneol and isoborneol: NMR spectrum of mixture, 263
 Bottles: labels, 559
 Broad absorption peak, 868
 Bromination: acetanilide, 334; aniline, 334; anisole, 334; phenols, 478–479; unsaturated compounds, 182, 464
 Bromine: in carbon tetrachloride test, 182; in methylene chloride, 465–466
 Bromo derivatives of phenols: preparation, 987
 Bromobenzene: Grignard reaction, 280
 Bromoethane: mass spectrum, 946
 1-Bromoethane: mass spectrum, 958
 Bronchodilator, 74
 Bubbler trap, 191
 Büchner funnel, 577, 636, 670
 Buffered aspirin, 55
 Bumping, 614
 Burns, 551
 Butane: mass spectrum, 949; molecular mechanics, 137
 1-Butanol: mass spectrum, 953; nucleophilic substitution, 163, 175
 2-Butanol: nucleophilic substitution, 163, 165–167
 2-Butanone: mass spectrum, 955
 1-Butene: mass spectrum, 951
 2-Butene: molecular mechanics, 140
 Butenes: calculation of heats of formation, 150–151
n-Butyl alcohol: nucleophilic substitution, 163, 175
sec-Butyl alcohol: nucleophilic substitution, 163
t-Butyl alcohol: nucleophilic substitution, 163
n-Butyl bromide: experiment, 175–177; infrared spectrum, 177; preparation, 173
t-Butyl chloride and *t*-butyl bromide: NMR spectrum of mixture, 171
n-Butylamine: infrared spectrum, 877
- C**
 C-4 and C-5 alcohols: esterification, 494–497
CA Online, 968
 Caffeine: essay, 73–77; extraction, 24; isolation from tea, 77–84; structure, 442
 Calculations, 567–570
 Calibrate, 827
 Camphor: C-13 NMR spectrum, 261; infrared spectrum, 258, 881; NMR spectrum, 260; oxidation reduction, 251, 252–254
 Capacity: drying agents, 694
 Caraway oil, 103–104
 Carbanion, 279
 Carbocations: density electrostatic potential maps, 154; heats of formation, 152
 Carbohydrates: identification, 431–432
 Carbon dioxide decaffeination process, 76
 Carbon disulfide: infrared spectrum, 864
 Carbon tetrachloride: hazards, 559; infrared spectrum, 861
 Carbon-13 NMR spectroscopy, 923–940; chemical shifts, 924–925; correlation table, 924–925; coupling constants, 926; equivalent carbons, 930–931; proton-coupled spectra, 925–927; proton-decoupled spectra, 927–929; sample preparation, 923–924
 Carbonyl compounds: computational chemistry, 154; infrared spectroscopy, 476, 491, 878
 Carbonylation: aromatic halides, 518–520
 Carboxylic acids: calculation of acidity, 153; derivatives, 476–477, 976–979, 986–987; identification tests, 475–477; infrared spectroscopy, 881; NMR spectroscopy, 476; preparation, 518, 990; tables of unknowns, 976–979; titration, 476
 Carcinogens, 561–562; definition, 547
 β -Carotene, 116; chromatography, 116; isolation, 116
 Carotenoids, 116
 Carrier gas, 818, 821
 Cartesian coordinates, 142
 Carvone, 103–104; C-13 NMR spectrum, 109; gas chromatography, 103–104; infrared spectrum, 106; isolation, 103; NMR spectrum, 107
 CAS (Chemical Abstract Service) Registry Number, 552, 580
 Celite, 637
 Cellulosic ethanol, 208, 209
 Centrifugation, 642
 Centrifuge tube, 577; use in extraction, 693
 Cerium (IV) test, 478, 484–485
 Chalcones, 309; cyclopropanation, 532–535; green epoxidation, 528–531; Michael

- addition, 320; preparation, 523, 535; synthesis of substituted chalcones, 523–527
- Channeling, 786
- Charcoal: activated, 675; powdered, 675–676
- Chemical Abstract Service (CAS) Registry Number, 552, 580
- Chemical Abstracts*, 967, 970
- Chemical equivalence, 893–896
- Chemical literature, 959–971; advanced laboratory techniques, 963–964; advanced textbooks, 961–962; *Beilstein*, 965–968; *Chemical Abstracts*, 967; computer online searching (*CA Online*), 968; current interest, 970; general synthetic methods, 960; handbooks, 959–960; qualitative organic analysis, 967; *Science Citation Index*, 970; scientific journals, 969; search methods, 960–961; 970–971; specific synthetic methods, 962–963; use of in chalcone preparation, 523–527
- Chemical shift, 892, 924–925; tables (*see inside back cover*)
- Chemical shift reagents, 916
- Chemiluminescence: definition, 421; experiment (luminol), 428
- Chemiluminescent, 423
- Chemotherapy, 363
- Chiral recognition, 103
- Chiral reduction, 226
- Chiral resolving agent, 248
- Chiral shift reagent, 231
- 4-Chlorobenzaldehyde: reaction with base, 520
- 1-Chlorobutane and 1-bromobutane: NMR spectrum of mixture, 169
- Chloroethane: mass spectrum, 945
- Chloroform: hazards, 559–560; infrared spectrum, 861
- Chloroform-*d*, 890
- Chlorophyll: chromatography, 116
- Chlorophyll *a*, 116
- Chlorophyll *b*, 116
- Chloroplasts, 116
- Chlorosulfonation: acetanilide, 366
- Chromatic aberration, 848
- Chromatogram, 824
- Chromatography, 68; column, 36; experiment, 36–37; gas, 817–836; gel, 797–798; HPLC, 812–817; normal-phase, 814; paper, 811; reversed phase, 814; separation, 781–786; thin-layer, 36, 801–812. *See also* Column, Gas, Paper, High performance, and Thin-layer chromatographies
- Chromic acid test, 471–472, 486–487
- Cinnamaldehyde: Wittig reaction, 327
- Claisen head, 576, 751
- Clamp holder, 578, 609
- Clamps, 578, 609
- Classification tests. *See specific functional group*
- Clean Air Act of 1990, 199
- Cleaning solution: preparation and safety precautions, 571–572
- Closed system, 724
- Cloves, 95
- CMR spectroscopy, 923–940; *See also* Carbon-13 NMR spectroscopy
- Cocaine, 343–344
- Coconut oil, 213
- Coenzyme, 267
- Coffee, 73–77; caffeine content, 75
- Cold baths, 607–608
- Cold pressing, 184
- Cold-finger condenser, 768
- Column: chromatographic, 779–781; chromatographic, macroscale, 787–788; chromatographic, microscale, 789; dimensions, 815–816; efficiency, 736; fractionating, 736–738; size, 785; semi-prep, 815; Vigreux, 736–737
- Column chromatography, 36, 777–800; adsorbent quantity, 785–786; adsorbents, 783; collecting fractions, 795; column size, 785–786; depositing the adsorbent, 790–792; experiment 41–44; flash chromatography, 798–800; flow rate, 786; macroscale columns, 787–788; microscale columns, 789; monitoring, 795; packing the column, 786–790; preparing the support base, 787–790; sample application, 792–793; separation of a mixture, 41; solvent reservoirs, 794–795; solvents, 784–785
- Combination pain relievers, 55
- Competitive inhibitor, 365
- Completeness: drying agents, 694
- Computational chemistry, 132, 141–148; acetone tautomerism, 150–151; butane isomers, 150–151; carbocation energies, 153–154; electrostatic potential maps, 148, 154; experiment, 149–150; heats of formation, 145–146; reactivity of carbonyl groups, 154–155; strengths of carboxylic acids, 153
- Condensation polymers, 372, 374–375, 377–379
- Condensation reactions: aldol, 309–310; benzoin, 266; luminal preparation, 424–426; Wittig reaction, 327
- Condenser, 576; cold-finger, 768; water, 612, 723–724
- Cones, 111
- Confirmation, 133–135
- Conical funnel, 577
- Conical vials: measuring volumes with, 596; use in extraction, 688–693
- Connectivity matrix, 142
- Cooling methods, 598–608
- Copolymer, 371
- Corey-Chaykovsky reaction, 532
- Corn, ethanol, 216–220
- Correlation chart: infrared spectroscopy, 868; C-13 NMR spectroscopy, 924–925; NMR spectroscopy, 897
- Correlation table: infrared spectroscopy, 868
- Coupling constant, 903–905
- Cracking, 195, 785
- Craig tube, 640–642, 672; centrifugation, 641
- CRC Handbook of Chemistry and Physics*, 10, 13, 50, 579–581, 672, 673, 959–960, 967
- Cross-linked polymers, 372
- Crystallization, 662–680; acetylsalicylic acid, 57; common solvents, 673; Craig tube, 672; experiment, 16–24; inducing crystal formation, 676–677; macroscale, 662, 665–671, 676–677; microscale, 662, 672, 677; mixed solvents, 679; solvent selection, 662, 673–674, 679; summary of steps, 671; theory of, 664–665
- Cyano groups: detection, 462; spectroscopy, 462
- Cycloaddition. *See* Diels-Alder reaction
- Cyclodextrins, 825
- Cyclohexane: molecular mechanics, 138–140
- Cyclohexanol: carbon-13 NMR spectrum, 930
- Cyclohexene: carbon-13 NMR spectrum, 931
- Cyclo-oxygenase, 55
- Cyclopentadiene: preparation, 405
- Cyclopentane: mass spectrum, 951
- ## D
- DDT, 401–402
- Deactivated adsorbents, 783
- Dean-Stark water separator, 745
- Decaffeinated coffee, 75
- Decane: infrared spectrum, 871
- Decantation, 635, 668
- Decolorization, 675–676; by column chromatography, 796–797; using a column, 676
- Decomposition point, 649–651
- Decoupler, 928
- Decoupling, 928
- Deet, 354
- Degassed, 816
- Dehydration: 4-methylcyclohexanol, 179–183
- Density: determination, 717–718
- Density electrostatic potential maps, 148, 154
- Density-elpot, 148
- Derivative formation: osazones from sugars, 438

- Derivatives: caffeine, 79; methods of preparation, 986–990; tables of, 973–985. *See also specific functional groups*
- Deshielding, 901, 902
- Desiccator, 678
- Desorbing, 779
- Detector, 816
- Detonation, 196
- Deuteriochloroform, 889; hazards, 560
- Deuterium oxide, 891
- Dextrorotatory, 842
- Diamagnetic anisotropy, 900
- Diatomaceous earth, 637
- Dichlorobenzene: NMR spectrum, 933
- 1, 2-Dichlorobenzene: infrared spectrum, 875
- Dichloromethane. *See* Methylene chloride
- Dictionary of Organic Compounds*, 960
- Dicyclopentadiene, 405; cracking, 405
- Diels-Alder reaction, 390; anthracene-9 methanol, 410; conversion to diester, 394–395; cyclopentadiene, 405; essay, 400–405; experiment, 393–394; furan, 390; maleic anhydride, 390, 405
- Diene, 400
- Dienophile, 400
- Diesel fuel, 201
- Diester, 394–395
- Diet soft drinks; HPLC analysis, 441–444
- Diethyl ether, 655, 657, 658, 659; hazards, 560
- N,N*-diethyl-*m*-toluamide, 354–355; infrared spectrum, 361; NMR spectrum, 362; preparation, 358
- 1, 2-Dimethoxyethane: hazards, 560
- 2, 2-Dimethylbutane: carbon-13 NMR spectrum, 929
- 3, 5-Dinitrobenzoates, 479, 488, 492; preparation, 989
- 2, 4-Dinitrophenylhydrazine test, 469–470
- 2, 4-Dinitrophenylhydrazones, 474; preparation, 986
- Dioxane: hazards, 560
- 1, 4-Diphenyl-1, 3-butadiene: preparation, 327–333
- Dipole-dipole interaction, 654
- Direct contact, 76
- Direct method, 773
- Discoloration, 649–651
- Disparlure, 353
- Dispensing pumps, 588–589
- Dispersion forces, 654
- Disposable pipets. *See* Pasteur pipets
- Dissolve, 779
- Distillate, 45, 724
- Distillation, 719–729; bulb-to-bulb, 757–758; equipment evolution, 719–721; simple, 719; steam, 770–777; theory, 721–726; vacuum, 749–763. *See also* Fractional, Simple, Steam, and Vacuum distillations
- Distillation head, 576, 723; Claisen, 576; Hickman, 726
- Distribution, 779
- Distribution coefficient, 682–684
- Diuretic, 74
- Dopamine: mass spectrum, 942
- Double bonds: detection, 464; spectroscopy, 467
- Double refraction, 837
- Doublet, 901
- Downfield, 892
- Drugs: identification, 67–68; TLC analysis, 68, 69–73
- Dry film method, 856
- Dry ice, 607
- Drying agents, 694–699; table, 695
- Drying tube, 578, 617; noxious gases and, 620–621
- Duisberg, Carl, 60–61
- Dynamic, 779
- E**
- E1/E2 Reactions. *See* Elimination reactions
- Ebulliator, 751
- Electron-density surface, 147
- Electron-donating groups, 909–910
- Electron-withdrawing groups, 910–911
- Electrophilic addition, 339
- Electrophilic aromatic substitution reactions. *See* Aromatic substitution
- Elemental analysis, 452
- Elimination reactions: 4-methylcyclohexanol (E1), 179–183
- Elpot map, 148
- Eluates, 780
- Eluents, 780
- Elutants, 780
- Elutes, 780
- Elution techniques, 793–794
- Emulsions, 699
- Enantiomeric excess, 844
- Enantioselective, 226
- Enantiospecific, 226
- Endo, 253
- Energy minimization, 133–135
- Energy transfer, 416–417
- Enol dianion, 423
- Environmental chemistry, 220
- Equal, 430
- Equipment: examples of, 575–578
- Erlenmeyer flasks: crystallization, 668, 670; measuring volumes with, 596
- Essential oils, 91; GC-MS analysis, 502–503; HPLC analysis, 499; isolation, 103–104, 500–502
- Ester, 57
- Esterification: *p*-aminobenzoic acid, 347; C-4 and C-5 alcohols, 494–497; isopentyl alcohol, 88; salicylic acid, 57; vanillin, 539–541
- Esters, 183; derivatives, 491–492, 984–985, 989–990; essay, 84–87; hydrolysis, 490; identification tests 488–492; infrared spectroscopy, 491, 882; NMR spectroscopy, 491; tables of unknowns, 984–985
- Ethanol, 123–125, 208–209; corn, 216–220; hazards, 560; preparation, 126–127
- Ether: hazards, 560. *See also* Diethyl ether
- Ethers, 659; infrared spectroscopy, 875–876; preparation, 320
- Ethyl acetoacetate: chiral reduction, 226–230; condensation, 320
- Ethyl *p*-aminobenzoate. *See* Benzocaine
- Ethyl (S)-hydroxybutanoate: optical purity determination, 230–231; preparation, 226
- Ethyl 3-hydroxybutanoate: NMR spectrum, 231, 232
- Ethyl 4-iodobenzoate: NMR spectrum, 299
- Ethyl phenylacetate: carbon-13 NMR spectrum, 927
- 2-Ethyl-1, 3-hexanediol: oxidation with hypochlorite, 541
- Ethylene glycol dimethyl ether, 560
- Eugenol, 94, 95
- Eutectic, 644
- Eutectic point, 645
- Evaporation to dryness, 625
- Exo, 253
- Experiments, unauthorized, 550
- Extraction, 681–708; caffeine, 24; determining organic layer, 28, 693–694; experiment, 24–32; liquid-liquid, 703–704; screw-cap centrifuge tube, 35; separation of a mixture, 28; solid phase, 704–707; solid-liquid, 702–703; solvent selection, 684–685; use of centrifuge tube, 693; use of conical vial, 688–693; use in purification, 700–702; use of separatory funnel, 686
- Eye safety, 546
- F**
- Fat replacers, 187
- Fats and oils, 183–188; fatty acid composition, 186; unsaturated, 186
- Fatty acids, 184
- trans*-Fatty acids, 186
- Feedstock, 221
- Fermentation: essay, 123–125; ethyl acetoacetate, 226; sucrose, 123, 127
- Ferric chloride test, 57, 58, 473, 478
- Ferric hydroxamate test, 489–490
- Ferrous hydroxide test, 459, 461–462
- FID. *See* Flame-ionization detector
- Filter Aid, 637
- Filter cones, 630
- Filter flask, 577, 636
- Filter paper, 630, 633, 635–636

- Filter tip pipet, 594, 640
 Filtering media, 637–638
 Filtering pipet, 634–635, 670
 Filtrate, 632
 Filtration, 630–642; Craig tube, 640–642;
 filter cones, 630; fluted, 668; methods,
 631; vacuum, 636
 Fireflies: essay, 421–423
 Fires, 546–547
 First Aid, 551
 First order, 908
 Flame front, 196
 Flame-ionization detector (FID), 823
 Flames, 550; heating with, 605–606
 Flash chromatography, 798–800
 Flavors: artificial and synthetic, 84–87
 Fluorene, 37
 Fluorenol, 37; isolation of, 41
 Flourenone, 37
 Fluorescence, 413
 Fluted filters, 630–633, 666, 668
 Food, in laboratory, 550
 Force constant, 133
 Forceps, 578
 Force field, 132
 Forerun, 724
 Fossil fuels, 194–202, 207
 Fraction collectors, 756–757
 Fractional distillation, 722, 729–749;
 apparatus, 738–740; columns, 736–738;
 differences, 730–731; ethanol, 127;
 experiment, 44–49. *See also* Distillation
 Fractionating columns, 729, 731
 Fractions, 724, 730, 795
 Fragment, 941
 Fragment ions, 942–943; peaks, 942
 Fragmentation patterns, 946–948
 Fragrances, 84–87
 Free radical, 947
 Friedel-Crafts reaction, 508–515
 Frontier orbitals, 146
 Frozen core approximation, 145
 Frozen joints, 575
 Fructose, 126
 Funnels: methods of preheating, 668
 Furan, 390
- G**
 Gallic acid, 77
 Gas chromatograph, 818
 Gas chromatography, 45, 817–836;
 advantages of, 822–823; alkyl halides,
 163; analysis of nitro compounds,
 236–239; apparatus, 818; calculation of
 peak areas, 830; carvone, 103, 104;
 collecting the sample, 828–832;
 columns, 819–821; detectors, 823–824;
 gasoline, 203; GC-MS, 835–836; liquid
 phase, 820–821; modern data station
 results, 832–835; peak areas, 824;
 qualitative analysis, 827–828;
 quantitative analysis, 830–832;
 response factors, 831, 833; retention
 time, 824–825; separation, 819;
 stationary phase, 819, 825–826
 Gas chromatography–mass spectrometry
 (GC-MS), 835–836, 948; analysis of
 antihistamine drugs, 516–517;
 essential oils, 502–503
 Gases: collection, 622–623; traps for
 noxious gases, 620
 Gas-liquid phase, 777
 Gasohol, 208
 Gasoline: composition, 194, 195, 206;
 gas chromatography, 203;
 oxygenated, 198; sample gas
 chromatograms, 205
 Gaussian-type orbitals, 143
 GC-MS. *See* Gas chromatography–mass
 spectrometry
 Gel chromatography, 797–798
 Gel-filtration chromatography, 798
 Gel-filtration column, 815
 Gel-permeation chromatography, 798
 Geminal, 253
 General unknowns, 446
 Geometry optimization, 145
 Glassware, 571–580; care and cleaning,
 571–572; drying, 572–573; etching, 575;
 examples, 575–576; frozen joints, 575;
 vacuum distillation, 750
 Global minimum, 133
 Gloves: protective, 551
 Glucose, 124, 126
 Glyceride, 183
 Glyptal, 383, 384
 Gossypure, 353
 Gradient elution systems, 816
 Graduated cylinders, 587–588
 Grain alcohol, 123
 Gravity filtration, 630–635
 Greasing joints, 750
 Green chemistry: aldol condensation
 313–315; application, 226, 236, 240,
 251, 265, 289, 302, 324–325, 410;
 chalcones, 528–531; essay, 220–225;
 twelve principles, 220–221
 Green Chemistry Challenge Award, 221
 Greenhouse effect, 201
 Grignard reactions, 279–280, 289, 518;
 apparatus, 281, 282; starting,
 281–282
 Ground state, 413
 Ground-glass joints, 573
 Grubbs catalyst, 302–303, 390
 Guided-inquiry experiment, 523–527
 Gyplure, 353
- H**
 Halides: detection, 459; elemental analysis,
 464; infrared spectroscopy, 884–885.
See also Alkyl halides
 Halogens: detection by mass spectrometry,
 944–945
 Hamiltonian, 142
 Handbooks, 579; use of, 579–586
 Hartree, 146
 Hazards: solvents, 547–548
 Heat of formation, 145
 Heating mantles, 598–599
 Heating methods, 598–608; aluminum
 block, 602–604; Bunsen burner,
 605–606; evaporation to dryness, 625;
 heating mantles, 598–599; hot plates,
 600; oil bath, 600–604; reflux, 495,
 612–613; sand bath, 604–605; solvents,
 659–660; steam bath, 606–607; water
 bath, 600
 Heating: rate of, 724; under reflux, 612
 Heme, 62
 Hemoglobin, 61
 Hepp, 60
 Herbs: identification of essential oils, 503
 Heteronuclear, 926
 HETP, 738
 Hexane: hazards, 560
 1-Hexanol: NMR spectrum, 917; NMR
 spectrum with shift reagent, 917
 1-Hexyne: infrared spectrum, 299; NMR
 spectrum, 297, 298
 Hickman head, 726, 727, 745
 High performance liquid chromatography
 (HPLC), 812–817; apparatus, 813;
 columns, 813; detectors, 815; diet soft
 drink analysis, 441–444; gradient
 elution, 816; ion-exchange
 chromatography, 815; isocratic, 816;
 mass spectrometry, 836; normal phase,
 814; presentation of data, 817;
 reversed-phase chromatography, 814;
 size-exclusion chromatography, 815;
 solvents, 816
 High pressure liquid chromatography,
 812–817
 Hinsberg test, 482
 Hirsch funnel, 577, 637
 Hit list, 836
 Holdup, 727, 736
 HOMO, 146
 Homonuclear, 925
 Honey, 429
 Hot plate/stirrer, 578
 Hot plates: with oil bath, 600–604; with
 sand bath, 604–605; with water bath,
 600
 Hot pressing, 186
 House vacuum, 754
 HPLC. *See* High performance liquid
 chromatography
 HPLC-MS, 836
 Hybrid-electric, 201–202
 Hydrazides, 491
 Hydrocracking, 197

- Hydrogen generator, 191
 Hydrogenation: methyl oleate, 189; oils, 186
 Hydrolysis: ester unknowns, 490; salicylic acid, 57; sugar, 429
 Hydrolyzed, 77
 Hydrophobic effect, 410
 Hydroxyl group, 57
 Hygroscopic, 677
 Hypochlorite, 251
- I**
- Ibuprofen, 62–63, 69
 Ice bath, 607
 Ice-salt bath, 607
 Ideal solution, 730, 733
 Identification of unknowns, 446–453
 Identity: confirmation, 452
 Ignition test, 467–468
 Immiscible, 24, 653; calculations, 772–773; distillation, 770–772
 In vivo, 363
 Index of refraction, 845
 Inert atmosphere, 617–619; balloon assembly, 618–619
 Inert compound, 458
 Infrared absorption bands: tables. *See inside back cover*
 Infrared spectrophotometer, 867
 Infrared spectroscopy, 851–886; acid chlorides, 884; alcohols, 487–488, 874; aldehydes, 878–879; alkanes, 871; alkene C–H out-of-plane bending, 871–872; alkenes, 871–872; alkynes, 467, 873–874; amides, 883; amines, 482, 876–877; anhydrides, 884; applications, 865; aromatic C–H out-of-plane bending, 874; aromatic compounds, 468, 873; base values, 868, 869; carbonyl base values, 878; carbonyl compounds, 474, 476, 491, 878; carboxylic acids, 881; correlation charts and table, 868(*see also inside back cover*); dry film method, 856; effects of conjugation, 881; effects of ring size, 880; esters, 491, 882; ethers, 875–876; experiment, 49–53; Friedel-Crafts acylation, 509; halides, 884; interpretation, 867–871; KBr pellets, 856–860; ketones, 474, 880; liquid samples, 852–856; neat liquids, 853; nitriles, 878; nitro compounds, 462, 877; Nujol mulls, 859; phenols, 479, 874; recording, 863; salt plates, 852; sample preparation, 852–865; silver chloride cells, 852, 854–856; silver chloride mini-cell, 862; sodium chloride cells, 852–854; solution cell, 862–863; solution spectra, 860–863; solution spectra using salt plates, 860; solvent spectra, 860–863; spectrum calibration, 864–865; survey of functional groups, 871–885; vibrations, 866
 Infrared spectrum, 867
 Injection port, 821
 Insect attractants, 350–356
 Insect repellents, 354–356; preparation, 358–359
 Insecticides: alternatives to, 403–404; essay, 400–405
 Insoluble, 653
 Intensity, 867
 Internal conversion, 416
 Intersystem crossing, 413
 Invert sugar, 429
 Invertase, 126, 429
 1-Iodo-2-methyl-4-nitrobenzene: infrared spectrum, 299; NMR spectrum, 298
 1-Iodo-4-nitrobenzene: NMR spectrum, 297
 4-Iodoacetophenone: NMR spectrum, 298
 Iodoform test, 472–473, 487
 Ionic liquids, 223
 Ionization chamber, 941
 Ionization potential, 941
 Isoamyl acetate. *See* Isopentyl acetate
 Isoborneol, 253; C-13 NMR spectrum, 262; infrared spectrum, 259; NMR spectrum, 261; preparation, 251
 Isobutyraldehyde, 313–314; HNMR spectrum, 318
 Isochractic, 816
 Isolation experiment, 566; caffeine from tea, 77–84; β -carotene from spinach, 116; carvone from caraway and spearmint oils, 103–104; chlorophyll from spinach, 116; essential oils from spices, 95–98
 “Isooctane”, 196
 Isopentyl acetate: esterification, 88; experiment, 88–91; infrared spectrum, 90, 882; preparation, 88
 Isoprene rule, 92
 Isoprene units, 92
- J**
- Joint clips, 751
 Journals, 969
 Juvenile hormones, 403
- K**
- KBr pellets, 856–860
 Ketohexoses: Seliwanoff’s test, 435–437
 Ketones, 468–474; derivatives, 474, 974–976, 986; identification tests, 446; infrared spectroscopy, 474, 880; reduction of, 240–242; tables of unknowns, 974–976
 Ketoprofen, 63
 Kinetics: alkyl chloride hydrolysis, 172–173
 Knocking, 196
- L**
- Labels: commercial bottles, 559; sample, 571
 Labile, 460
 Laboratory notebook, 563–566; format, 564–566; sample pages, 568–569
 Laboratory records, 566–567
 Laboratory safety, 546–562
 Lachrymator, 290
Lange’s Handbook of Chemistry, 581–583, 960, 967
 Lanthanide shift reagent, 916
 LD₅₀, 558
 Leaded gasoline, 197
 Lethal dose, 558
 Levorotatory, 842
 Lewis acid, 334
 Ligroin, 659, 660; hazards, 560
 Limiting reactants, 586
 Limiting reagent, 567
 Limonene: infrared spectrum, 107; NMR spectrum, 108
 Linear combination of atomic orbitals, 142
 Liquid chromatography, 812–817
 Liquid mixture, 721
 Liquid-liquid phase, 777
 Liquids: addition of reagents, 615–617; boiling point determination, 710, 712–715; density determination, 717–718; measurement, 587
 Literature of chemistry, 959–971
 Live steam method, 773
 Local diamagnetic shielding, 900
 Local minimum, 134
 London forces, 654
 Lucas test, 485–486
 Luciferase, 421
 Luciferin, 421
 Lucretius, 98, 101
 Luminol: preparation, 424–426
 LUMO, 146; carbonyl groups, 154–155
- M**
- Macroscale: apparatus assemblies, 609–610; boiling points, 710–712; columns, 787–788; crystallization, 662, 665–671, 676–677; definition, 4, 587; drying procedures, 696–697; equipment, 727–728; steam distillation, 773–775
 Magnetic equivalence, 905–907
 Magnetic spin vanes, 614
 Magnetic stirrers, 614
 Malt, 123
 Maltase, 126
 Maltose, 123
 Manometers, 760–763; connection, 753–754, 762–763; construction and filling, 760
 Mash, 123
 Mass analyzer, 941

- Mass spectrometry, 941–959; analysis of nitro compounds, 236–237; base peak, 942; detection of halogens, 944–945; fragment ion peaks, 942; fragmentation patterns, 946–948; GCMS, 948; *m/e* ratio, 942; *M + 1*, *M + 2* peaks, 942; McLafferty rearrangement, 958; molecular formula determination, 943–944; molecular ion, 942; precise atomic masses, 943; rearrangements, 958
- Mass spectrum, 941–943; interpreted, 948–958
- Mass-to-charge ratio, 941
- Material Safety Data Sheets (MSDS), 552–558; sample pages, 553–558
- Maximum-boiling point-diagram, 740, 742–744
- McClintock effect, 352
- McLafferty rearrangement, 958
- Melting point, 643–652; capillary, 645; corrections, 649–651; decomposition, 649–651; depression, 645; determination, 646–649; discoloration, 649–651; electrical apparatus, 647–649; finding literature values, 579–586; mixture, 646; packing tubes, 646; physical properties, 643; range, 643; shrinkage, 649–651; softening, 649–651; standards, 652; sublimation, 649–651; theory, 644–646
- Melting point tube, 646; sealing methods, 650
- Merck Index*, 10, 13, 21, 24, 50, 583–584, 672, 673, 960
- Mesityl oxide: infrared spectrum, 880
- Methanol: hazards, 553, 560
- Methemoglobinemia, 61
- Methiodides, 483; preparation, 988
- Methyl benzoate: infrared spectrum, 883; nitration, 338–341
- Methyl butanoate: mass spectrum, 957
- Methyl *tert*-butyl ether, 198, 199
- Methyl isopropyl ketone: infrared spectrum, 867
- Methyl *m*-nitrobenzoate: infrared spectrum, 342; preparation, 338
- Methyl oleate: hydrogenation, 189
- Methyl stearate: preparation, 189–194
- 4-Methylcyclohexanol: dehydration, 181; infrared spectrum, 183, 876
- 4-Methylcyclohexene: bromine addition, 180; infrared spectrum, 183, 872; preparation, 179–183
- Methylene chloride: hazards, 560–561
- 2-Methyl-2-propanol: nucleophilic substitution, 163, 168–169
- Mevalonic acid, 92
- Michael addition, 320
- Michael condensation, 535–539
- Micro boiling point determination, 713
- Micro pipet, 803
- Microscale: apparatus assemblies, 611, 768–769; boiling points, 712–715; columns, 789; crystallization, 662, 672, 677; definition, 4, 587; drying procedures, 697–699; equipment, 726–727; steam distillation, 775–776
- Microwave chemistry, 626–628
- Microwave technology, 300
- Midrun, 724
- Mineral oil: infrared spectrum, 859
- Mineral oil bubbler, 191
- Minimization, 133–135
- Minimum-boiling-point diagram, 740–742
- Miscible, 653, 681; distillation, 770–772
- Mixed chemicals, 550
- Mixed solvents, 679
- Mixture melting points, 17
- Mixtures: separation, 700–702; separation by extraction, 28
- Mole fraction, 732
- Mole percentage, 732
- Molecular formula: determination by mass spectrometry, 943–944
- Molecular ion, 942
- Molecular mechanics, 132, 141; butane conformations, 137–138; 2-butene isomers, 140; cyclohexane conformations, 138–139; limitations, 135–136; substituted cyclohexane rings, 139–140
- Molecular modeling, 132–133; aromatic substitution, 341–342; Diels-Alder reaction, 408–409; enolate ions, 311; experiment, 136–140; nitration of anisole, 342; nitration of methyl benzoate, 341–342. *See* Computational chemistry; Molecular mechanics
- Molecular rotation, 840
- Molecular-sieve chromatography, 798
- Molisch's test, 431
- Moncrieff, R.W., 99
- Monoglyme, 560
- Monomers, 371
- Monosaccharides: Barfoed's test, 438–441
- Monosubstituted, 932
- Monoterpenes, 92
- Mother liquor, 16, 641, 664
- Moving gas phase, 818
- MSDS. *See* Material Safety Data Sheets
- Mucic acid test, 439–441, 463
- Multistep reaction sequences, 265–266
- Mydriasis, 344
- N**
- n + 1* rule, 901–903
- NADH. *See* Nicotinamide adenine dinucleotide
- Nanotechnology, 10
- Naphthalene, 194; ultraviolet spectrum, 416
- 2-Naphthol: infrared spectrum, 875
- α -Naphthylurethanes, 479; preparation, 987
- Naproxen, 63
- National Fire Protection (NFPA) rating, 558
- National Energy Act, 208
- Natural products, 681
- Neat liquids, 853
- Neoprene adapter, 577
- Neutral alumina, 778
- Neutralization equivalent, 476
- NFPA rating. *See* National Fire Protection rating
- Nicol prism, 837, 838
- Nicotinamide adenine dinucleotide (NADH), 240
- Nicotine, 404
- Nitration: aromatic compounds, 236–239; methyl benzoate, 338–341
- Nitriles: infrared spectroscopy, 462, 878
- Nitro compounds: detection, 461; infrared spectroscopy, 877
- 3-Nitrobenzaldehyde: aldol condensation, 309, 310
- Nitrobenzene: infrared spectrum, 877
- Nitrogen: elemental analysis, 462; liquid, 608
- 2-Nitrophenol: NMR spectrum, 915
- 5-Nitrophthal-hydrazide: preparation, 424; reduction, 424
- 3-Nitrophthalic acid: amide formation, 424
- Nitrous acid test, 480–481
- NMR spectroscopy, 886–922; absorption of energy, 887; alcohols, 488; aldehydes, 474, 900; alkenes, 467, 900, 905; alkynes, 467, 900; amines, 482; anisotropy, 900–900; aromatic compounds, 468, 897, 905, 908–914; carbon-13, 923–940; carboxylic acids, 476; chemical shift, 892–893; chemical shift ranges, 897; chemical shift reagents, 916–918; chemical shift tables (*see inside back cover*); chemical shifts, 896–897; common splitting patterns, 904; correlation chart, 897; correlation table (*see inside back cover*); coupling constant, 903–905; esters, 491; Friedel-Crafts acylation, 509–510; high-field spectra, 907–908; integrals, 893–896; ketones, 474, 900; *n + 1* rule, 901–903; optical purity determination, 230–231; phenols, 479; protons, 914–916; quantitative use, 171; reference substances, 891–892; ring current, 897; sample preparation, 889–892; shift reagents, 231, 916–918; solvents, 890–891; spin-spin splitting, 901–903
- NOE. *See* Nuclear Overhauser enhancement
- Nonanal: infrared spectrum, 879
- Nonideal solutions, 740
- Nonlimiting, 586

- cis*-Norbornene-5, 6-*endo*-dicarboxylic anhydride: infrared spectrum, 408, 884; preparation, 405
- Norit, 675–676; pelletized, 676
- Normal-phase chromatography, 814
- Notebook, 563–566; format, 564–566
- Novocain, 344
- Noxious gases: capture, 619–622; removal, 619–622
- Nuclear magnetic resonance. *See* NMR spectroscopy
- Nuclear Overhauser effect, 930
- Nuclear Overhauser enhancement (NOE), 930–931
- Nucleophilic substitution: *n*-butyl alcohol (SN2), 175; competing nucleophiles, 163; investigations using 2-Pentanol and 3-Pentanol, 504–508; kinetic study, 172–173; *t*-pentyl alcohol (SN1), 177; preparation of alkyl halides, 172–173, 175; reactivities of alkyl halides, 158–162; SN1 reaction rates, 154; tests for reactivities, 158
- Nujol: infrared spectrum, 860; mull, 859
- Null reading, 841
- NutraSweet, 430
- Nylon: preparation, 385–386
- O**
- Observed rotation, 839
- Octane ratings, 196, 199
- Odor: stereochemical theory, 98–102
- Oil bath, 600–601
- Oil out, 673
- Oils: fatty acid composition, 183–188; isolation of essential, 497–503; vegetable, 186
- Olean, 187
- Olestra, 187
- Online literature search, 523
- Opsin, 111
- Optically activity substance, 839
- Optical purity, 844–845; NMR determination, 230–231
- Organic Syntheses*, 963
- Organoleptic, 84
- Organozinc reactions, 289–292
- Origami, 633
- Ortho, 334
- Oven drying, 677
- Oxidation: alcohols, 487, 541; aldehydes, 470; benzoin, 272; borneol, 251; nitric acid, 273; by sodium hypochlorite, 222
- Oxidation puzzle, 541–543
- Oxides of nitrogen, 199
- Oximes, 474
- Ozone, 199
- P**
- PABA. *See* *p*-Aminobenzoic acid
- Packed, 779
- Palladium catalyst, 292–299
- Palladium on charcoal, 189
- PAN. *See* Peroxyacetyl nitrate
- Paper chromatography, 811
- Paper factor, 404
- Para-disubstituted rings, 911
- Paraffins, 194
- Parameterized, 133
- Partial vapor pressure, 733
- Particulate matter, 199
- Partition coefficient, 682
- Pasteur pipet, 577, 593–594; calibrated, 593; preheated, 668
- PEL. *See* Permissible Exposure Limit
- Penicillin, 364
- Pentane: hazards, 561
- 2-Pentanol, 504
- 3-Pentanol, 504
- Pentoses: Bial's test, 435
- 1-Pentyne; NMR spectrum, 298, 299
- t*-Pentyl alcohol: nucleophilic substitution, 177
- t*-Pentyl chloride: infrared spectrum; preparation, 173
- Percentage yield, 567
- Permissible Exposure Limit (PEL), 552
- Peroxide, 423
- Peroxyacetyl nitrate (PAN), 199–200
- Petroleum: essay, 194–202
- Petroleum ether, 659, 660; hazards, 561
- Phases, 681
- Phenacetin, 60, 67
- Phenols: derivatives, 479–480, 979–980, 987; identification tests, 477–480; infrared spectroscopy, 479, 874; NMR spectroscopy, 479; salicylic acid, 57; tables of unknowns, 979–980
- Phenylacetone: NMR spectrum, 888
- a*-Phenylethylamine: optical purity determination, 242–245, 248; resolution of, 245–248; resolution of enantiomers, 243
- Phenylmagnesium bromide: preparation, 281, 284
- Phenylpropanoids: essay, 91–95
- Phenylurethanes, 488; preparation, 989
- Pheophytin *a*, 117
- Pheophytin *b*, 117
- Pheromones, 87; essay, 350–356; structures, 352; types of, 351
- Phosphorescence, 414
- Photochemistry, 411; essay, 421–423
- Photoreduction: benzophenone, 411, 412–417
- Picrates, 483; preparation, 988
- Pinacol rearrangement, 411, 419
- Piperonaldehyde: aldol condensation, 309, 310
- Pipet: automatic, 595; bulb, 577; disposable, 577; filter tip, 594, 640; filtering, 634–635, 670; graduated, 577, 589–593; Pasteur, 577, 593
- Pipet pump, 590
- Plane-polarized light, 837
- Plastic joint clips, 609
- Plasticizer, 373
- Plastic joint clips, 609
- Plastics: essay, 371; recycling codes, 380
- Polarimeter, 839–840; operation, 841–844
- Polarimeter cell, 840
- Polarimetry, 837–845; carvone, 106; digital polarimeter, 843–844; ethyl (S)-3-hydroxybutanoate, 226; resolution of *a*-phenylethylamine, 243; Zeiss polarimeter, 841
- Polarization sets, 144
- Polarizer, 839
- Pollution: petroleum, 200
- Pollution Prevention Act of 1990, 221
- Polycarbonate, 375, 378
- Polyamide: preparation, 385
- Polyester, 188; preparation, 383–384
- Polymerization, 195
- Polymers, 371–382; chemical structure, 371; infrared spectroscopy, 388; NMR spectrum, 397; preparation, 382–383; recycling codes, 380; synthesizing by Ring-Opening Metathesis Polymerization, 396–398; thermal classification of, 372–373; types of, 372
- Poly-Sep AA, 798
- Polystyrene: infrared spectrum, 864; preparation, 386–387
- Polyunsaturated fats, 186
- Porosity, 635
- Potassium permanganate test, 182, 464, 466
- Precise atomic masses, 943
- Precipitation, 662
- Pregnancy: precautions, 547
- Preparative experiment, 565
- Preparative plates, 807–808
- Pressure tubing, 751
- Primary aromatic, 481
- Primer pheromones, 351
- Product development control, 254
- Product purification: by extraction, 700–702
- Project-based experiments: aldehyde enigma, 520–522; carbonation of aromatic halide, 518–520; chalcone preparation, 523–527; cyclopropanation of chalcones, 532; green epoxidation, 528–531; esterification of vanillin, 539–541; Friedel-Crafts acylation, 508–515; GC-MS analysis of antihistamine drugs, 516–517; GC-MS of essential oils, 502–503; green epoxidation of chalcones, 528–531; investigation of essential oils of herbs and spices, 503; isolation of essential oils from spices, 497–499; isolation of essential oils by steam distillation, 500–502; Michael

- and aldol condensations, 535–539;
nucleophiles, 504–508; oxidation
puzzle, 541–543; preparation of C-4 or
C-5 acetate ester, 494–497
- Prontosil, 363
- Propanoic acid: mass spectrum, 956
- 1-Propanol: carbon-13 NMR spectrum, 928
- Propylure, 353
- Prostaglandins, 54
- Protective groups, 367
- Purification of solids, 662–680
- Purification scheme: experiment, 33–35
- Pyrethrins, 404
- Pyridine: hazards, 561
- Q**
- Qualitative organic analysis, 446, 967
- Quantitative transfer, 587
- Quantum mechanics, 132, 141
- Quadrupole broadening, 916
- Queen substance, 353
- Quenched, 416
- Quencher, 417
- R**
- R_f value, 808–809
- Racemic mixture, 233, 844
- Radiationless transition, 414
- Radical-cation, 941, 946
- Raoult's Law, 732; immiscible liquids,
770–772; miscible liquids, 733–736,
770–772
- Reaction flask, 191
- Reaction methods, 608–629
- Reaction methods: inert atmosphere,
617–619
- Reagents: addition of liquids, 615–617
- Rearrangement: benzilic acid, 275
- Receiving flask, 724, 752
- Recognition pheromones, 351, 354
- Recorder, 941
- Records. *See* Laboratory records
- Recruiting pheromones, 351, 354
- Recrystallization, 58–59, 665
- Recyclable catalyst, 236
- Reducing sugar test, 436
- Reducing sugars, 436
- Reduction: camphor, 252–253; chiral, 226;
ethyl acetoacetate, 226; by ferrous hy-
droxide, 461; fluorenone to fluorenone,
40; by hydrogen, 189; methyl oleate,
189; nitro group, 424, 461; photoreduc-
tion, 411; by sodium borohydride, 251;
by sodium dithionite, 424; by yeast,
226, 228
- Reflux, 612–613
- Reflux apparatus, 612
- Reflux ratio, 739
- Reflux ring, 613
- Reformats, 197
- Reforming, 197
- Reformulated gasoline, 199
- Refractive index, 845–846; temperature
corrections, 850
- Refractometer: apparatus, 846–850;
cleaning, 849
- Refractometry, 845–850; Abbé
refractometer, 846–849; digital
refractometer, 849–850
- Releaser pheromones, 351
- Rendering, 186
- Repellent, 354
- Residue, 724
- Research: simulated laboratory experience,
523–527
- Resolution of enantiomers, 243
- Resolved, 827
- Response factor, 831, 833
- Retention time, 824–825
- Retentivity, 635
- Retinal, 111
- Retinene, 111
- Retort, 719
- Reversed phase chromatography, 814
- Reye's syndrome, 55
- Rhodopsin, 111
- Right-to-know laws, 551
- Ring current in NMR spectroscopy, 900
- Ring-Opening Metathesis Polymerization
(ROMP), 390–393; experiment,
391–392; NMR spectrum for polymer,
397; synthesizing the polymer, 396
- Rods, 111
- Rotary evaporator, 626
- Round-bottom flask, 576
- Rubber septum, 577
- Running, 801; TLC plates, 805–806
- S**
- Saccharine, 430; structure, 442
- Safety, 546–563
- Safety glasses, 546
- Salicylamide, 61, 69
- Salicylic acid: acetylation, 56–57
- Salt, 79
- Salt plates, 852
- SAM. *See* Self-assembled monolayer
- Sample vials, 570–571; labeling, 571
- Sand bath, 604–605
- Schrödinger equation, 142
- Science Citation Index*, 970
- Scientific journals, 969–970
- Scratching: to induce crystallization, 677
- Screw-cap centrifuge tube: experiment, 35
- Secondary containment, 548
- Seed crystals, 677
- Seeding: to induce crystallization, 677
- Seliwanoff's test, 431, 435–437
- Self-assembled monolayer (SAM), 10
- Semicarbazones, 474; preparation, 986
- Semiempirical calculations, 142
- Semiempirical methods, 141–148
- Semi-microscale: column, 788–789; crystal-
lization, 662; distillation, 776–777
- Semiprep column, 815
- Separation methods, 700–702
- Separation scheme, 565, 702; experiment,
33–35
- Separating, 779
- Separatory funnel, 88, 89, 577, 681, 686–688
- Sephadex, 798
- Septum: rubber, 577
- Sex attractants, 351–353, 355
- Sex pheromones, 351
- Sharp absorption peak, 868
- Shielded, 901, 902
- Shielding, 897–900
- Shift reagents, 231, 916–918
- Shikimic acid pathway, 94
- Shrinkage: on melting, 649–651
- Side arm test tube apparatus, 769
- Side products, 564
- Side reactions, 564
- Silica gel, 778
- Silica gel G, 802
- Silver chloride, 852; plates, 854–856
- Silver nitrate, 476; test, 158, 459, 460–461
- Simple distillation, 719–729; apparatus,
723–726; differences, 730–731;
experiment, 44–49. *See also* Distillation
- Single-point calculations, 144
- Single-point energy, 138, 145
- Singlet state, 413
- Size-exclusion chromatography, 798
- Slater-type orbitals (STO), 143
- Slurry, 791–792
- Small-scale: definition, 4
- Smog, 199
- S_N1/S_N2 reactions. *See* Nucleophilic
substitution
- Sodium, 892
- Sodium bicarbonate, 476
- Sodium borohydride, 41, 252
- Sodium chloride, 852; plates, 852–854
- Sodium cyclamate, 430
- Sodium D line, 839, 840
- Sodium fusion tests, 462
- Sodium hydroxide solution, 477
- Sodium hypochlorite: use in oxidation of
alcohols, 541
- Sodium iodide test, 158, 459, 461
- Softening: on melting, 649–651
- Solid-liquid phase, 777
- Solids: measurement, 587; melting point
determination, 643–644; phase
extraction, 704–707; purification by
sublimation, 763; sublimation
behavior, 765–766
- Solubility, 653–661; crystallization, 662–663;
experiment, 6–15; finding literature
values, 579–586; guidelines, 654–656;
predicting behavior, 654–658; tests,
453–458, 653

Soluble, 653
 Solute, 653, 663
 Solution cell: infrared spectroscopy, 862–863; NMR spectroscopy, 890–891
 Solvent, 653, 654
 Solvent disposal, 548–550
 Solvent evaporation: green method, 627; methods, 624–626; reduced pressure, 625; rotary evaporator, 626
 Solvent extraction, 186
 Solvents: abbreviations, 581, 583; boiling points, 660; for crystallization, 673–674; densities, 685; hazards, 547–548; heating methods, 598–599; mixed, 679; organic, 659–660; relative polarities, 654, 657; removing compounds, 785; safety, 547–548; tables (*see inside front cover*); testing for crystallization, 674
 Sonogashira coupling, 292–299
 Soxhlet extractor, 702
 Spatula, 578
 Spearmint oil, 103–104
 Specific rotation, 839
 Specific unknown, 446
 Spectra catalogues, 961
 Spectroscopy, 501; catalogues of spectra, 961; sample preparation, 852–865, 889–892. *See also* Infrared, NMR, and Carbon-13 NMR spectroscopies; Mass spectrometry
 Spices, 95; identification of essential oils, 497–499
 Spin bar, 578
 Spin vane, 614
 Spinach: isolation of pigments, 116
 Spinning-band column, 738
 Spin-spin splitting, 901–903
 Spotting, 801
 Spotting TLC plates, 803–805
 Standard-taper: definition, 573
 Starch, 123
 Starch-iodine test, 59, 439
 Stationary liquid phase, 818
 Stationary phase, 819
 Steam baths, 606–607
 Steam cones, 606–607
 Steam distillation, 770–777; apparatus, 773–774, 775, 776; essential oils, 95–98, 500–502; methods, 773–777; spices, 497–499; trap, 775. *See also* Distillation
 Stem corrections, 715–717
 Steric approach control, 253
 Steric energy, 132
 Stereoisomer, 314–316
 Stirrer, 578
 Stirring bars, 614
 Stirring methods, 614
 STN Easy, 523
 STO. *See* Slater-type orbitals
 STO-3G basis set, 143
 Stone, Edward, 53

Straight-run gasoline, 195
 Strain energy, 132
 Streaming, 786
 Strokes, 195
 Styrene: infrared spectrum, 873
 Sublimation, 763–770; advantages, 766; apparatus, 768–769; caffeine, 78, 81; on melting, 649–651; methods, 767–768; specific directions, 768–769; vacuum, 766
 Substitution reaction, 465
 Sucrose, 126–130, 188; fermentation, 127; hydrolysis, 439
 Suction filtration. *See* Vacuum filtration
 Sugars: identification, 430; reducing, 436
 Sulfa drugs: essay, 363–366; preparation, 366–367; tests on bacteria (*see* Instructor's Manual)
 Sulfamates, 430
 Sulfanilamide, 363; action, 365; preparation, 366–367
 Sulfur: elemental analysis, 463
 Sulfuric acid: solubility, 458
 Supercritical carbon dioxide, 223; state, 76
 Support phase, 819
 Surfaces, 147
 Sweeteners: essay, 428–431
 Syngas, 209
 Syringe, 581, 594–595

T

Tables of unknowns and derivatives, 973–985
 Tailing, 796, 808, 827
 Takeoff, 724; rate of, 739
 Tannins, 77
 TCD. *See* Thermal conductivity detector
 Tea, 73–77; caffeine content, 75; extraction, 77–84
 Terpenes: essay, 91–95
 Territorial pheromones, 351
 Test tube: brush, 578; confused, 182; holder, 578
 Tetracycline, 364
 Tetraethyllead, 197
 Tetrahydrofuran: hazards, 561
 Tetramethylsilane (TMS), 891, 892, 893
 Theobromine, 74
 Theophylline, 74
 Theoretical plates, 736
 Theoretical yield, 567
 Thermal conductivity detector (TCD), 823
 Thermodynamic product, 408
 Thermometer: dial, 602; partial immersion, 715; placement, 751; stem corrections, 715–717; total immersion, 716; types, 715–716
 Thermometer adapter, 576, 723
 Thermometer calibration, 651–652
 Thermoplastic, 372–373
 Thermoset, 373
 Thiamine: catalytic action, 266–268; mechanism of action, 267
 Thiele tube, 646–647
 Thin-layer chromatography, 36, 801–812; analgesics, 69–73; chemical applications, 809–811; commercially prepared plates, 802; development chambers, 805; experiment, 37–39; micropipet preparation, 803; monitoring a reaction, 40–41; preparative, 807–808; R_f values, 808–809; sample application, 803; slide preparation, 802–803; solvent selection, 39–40, 806; spinach, 116
 Thin-layer plate, 801; developing, 805–806; preparation of, 802–803
 Thin-layer slide, 801; preparation of, 802–803
 Three-finger clamp, 578
 Threshold Limit Value (TLV), 552
 Throughput, 739
 TLV. *See* Threshold Limit Value
 TMS. *See* Tetramethylsilane
 Tollens test, 470–471
 Toluene: carbon-13 NMR spectrum, 932; hazards, 561; mass spectrum, 952
m-Toluic acid: amide formation, 359
p-Toluidide derivatives; preparation, 987
 Total energy, 146
 Trail pheromones, 351, 354
 Transesterify, 785
 Trans-fatty acids, 186
 Trap: acidic gases, 165, 175, 368, 512, 620; aspirator, 638–639; manometer, 762; steam distillation, 775; vacuum distillation, 752–753, 762; vacuum pump, 760
 Triangulation of gas chromatography peaks, 830
 1, 1, 2-Trichloroethane: NMR spectrum, 902
 Triglyceride, 184
 2, 2, 4-Trimethylpentane: mass spectrum, 950
 2, 2, 2-Triphenylacetophenone, 419
 Triphenylmethanol: benzoic acid, 278–280; infrared spectrum, 285; preparation, 280–281, 284–286
 Triphenylphosphine: Wittig reaction, 327
 Triphenylpyridine: preparations, 324–326
 Triple bonds: detection, 464; spectroscopy, 467
 Triplet state, 413
 Tubing: pressure, 751; thin-walled and vacuum, 751
 Tylenol, 55

U

Ultraviolet light, 69
 Unisol, 891
 Unknown compounds: identification, 446–453; tables of, 973–985. *See also* specific functional groups

Unleaded gasoline, 197
Unsaturation tests, 182, 464–468
Upfield, 892

V

Vacuum adapter, 576, 724
Vacuum distillation, 749–763; acetophenones, 509; apparatus, 749–750, 756, 757; bulb-to-bulb, 757–758; fraction collectors, 756–757; stepwise directions, 754–756. *See also* Distillation
Vacuum drying, 678
Vacuum filtration, 636–637
Vacuum pump, 758–760; trap, 760
Vacuum traps, 752–753
Vacuum tubing, 751
Valence shell, 142
Van der Waals forces, 654
Vanillin: esterification, 539–541
Vapor phase chromatography. *See* Gas chromatography
Vapor pressure, 764–765
Vaporization, 732
Variation Principle, 141

Vasodilator, 74
Vertical lines, 733
Vigreux column, 736–737
Vinyl acetate: NMR spectrum, 906
Vision: chemistry of, 111–115
Visualization methods, 806
Visualization reagents for TLC, 806–807
Vitamin, 266, 267
Vitamin A, 113, 114
Vitamin B, 266
Volatility, 674
Vomer nasal, 352
Vortex mixer, 693

W

Wash acetone, 571
Waste disposal, 548–550
Water bath, 600
Water condenser, 723–724
Water-jacketed condenser, 612
Water process, 76
Water pump, 638
Water separator: azeotropic, 745
Wavefunction, 142

Wavenumbers, 851
Weighing: liquids, 586; solids, 587
Weight percent recovery, 570
Wintergreen, 91
Wittig reaction, 327
Wooden applicator sticks, 751
Wort, 123

X

Xanthines, 74
Xanthophylls, 116

Y

Yeast: use in fermentation, 127; use as a reducing agent, 226
Yield: calculation, 567–570
Ylide, 267, 327, 328
Ytterbium (III) trifluoromethanesulfonate, 236

Z

Zeiss polarimeter, 841–843
Zymase, 127

Common Organic Solvents

Solvent	Boiling Point (°C)	Density (g/mL)
Acetic acid	118	1.05
Acetic anhydride	140	1.08
Acetone	56	0.79
Benzene*	80	0.88
Carbon tetrachloride*	77	1.59
Chloroform*	61	1.48
Cyclohexane	81	0.78
Dimethylformamide (DMF)	153	0.94
Dimethyl sulfoxide (DMSO)	189	1.10
Ethanol	78	0.80
Ether (diethyl)	35	0.71
Ethyl acetate	77	0.90
Heptane	98	0.68
Hexane	69	0.66
Ligroin	60–90	0.68
Methanol	65	0.79
Methylene chloride	40	1.32
Pentane	36	0.63
Petroleum ether	30–60	0.63
1-Propanol	98	0.80
2-Propanol	82	0.79
Pyridine	115	0.98
Tetrahydrofuran (THF)	65	0.99
Toluene	111	0.87
Xylenes	137–144	0.86

Solvents indicated in boldface type are flammable.

*Suspected carcinogen.

Atomic Mass Values for Selected Elements

Aluminum	26.98
Boron	10.81
Bromine	79.90
Carbon	12.01
Chlorine	35.45
Fluorine	18.99
Hydrogen	1.008
Iodine	126.9
Lithium	6.941
Magnesium	24.30
Nitrogen	14.01
Oxygen	15.99
Phosphorus	30.97
Potassium	39.09
Silicon	28.09
Sodium	22.99
Sulfur	32.07

Concentrated Acids and Bases

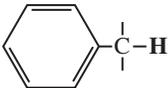
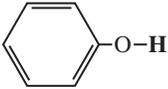
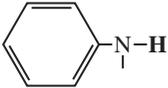
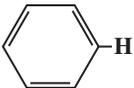
Reagent	HCl	HNO ₃	H ₂ SO ₄	HCOOH	CH ₃ COOH	NH ₃ (NH ₄ OH)
Density (g/mL)	1.18	1.41	1.84	1.20	1.06	0.90
% Acid or base (by weight)	37.3	70.0	96.5	90.0	99.7	29.0
Molecular weight	36.47	63.02	98.08	46.03	60.05	17.03
Molarity of concentrated acid or base	12	16	18	23.4	17.5	15.3
Normality of concentrated acid or base	12	16	36	23.4	17.5	15.3
Volume of concentrated reagent required to prepare 1 L of 1 M solution (ml)	83	64	56	42	58	65
Volume of concentrated reagent required to prepare 1 L of 10% solution (ml)*	227	101	56	93	95	384
Molarity of a 10% solution*	2.74	1.59	1.02	2.17	1.67	5.87

*Percent solutions by weight.

 Infrared Absorption Bands

	Type of Vibration	Frequency (cm ⁻¹)	Intensity
C—H	Alkanes (stretch)	3000–2850	s
	—CH ₃ (bend)	1450 and 1375	m
	—CH ₂ — (bend)	1465	m
	Alkenes (stretch)	3100–3000	m
	(out-of-plane bend)	1000–650	s
	Aromatics (stretch)	3150–3050	s
	(out-of-plane bend)	900–690	s
	Alkyne (stretch)	ca. 3300	s
Aldehyde		2900–2800	w
		2800–2700	w
O—H	Alcohol, phenols		
	Free	3650–3600	m
	H-bonded	3400–3200	m
	Carboxylic acids	3400–2400	m
N—H	Primary and secondary amines and amides		
	(stretch)	3500–3100	m
	(bend)	1640–1550	m–s
C≡C	Alkyne	2250–2100	m–w
C≡N	Nitriles	2260–2240	m
C=C	Alkene	1680–1600	m–w
	Aromatic	1600 and 1475	m–w
N=O	Nitro (R—NO ₂)	1550 and 1350	s
C=O	Aldehyde	1740–1720	s
	Ketone	1725–1705	s
	Carboxylic acid	1725–1700	s
	Ester	1750–1730	s
	Amide	1680–1630	s
	Anhydride	1810 and 1760	s
	Acid chloride	1800	s
C—O	Alcohols, ethers, esters, carboxylic acids, anhydrides	1300–1000	s
C—N	Amines	1350–1000	m–s
C—X	Fluoride	1400–1000	s
	Chloride	785–540	s
	Bromide, iodide	< 667	s

NMR Chemical Shift Ranges (ppm) for Selected Protons

$R-CH_3$		0.7–1.3	$R-\overset{ }{N}-\overset{ }{\underset{ }{C}}-H$	2.2–2.9
$R-CH_2-R$		1.2–1.4	$R-\overset{ }{S}-\overset{ }{\underset{ }{C}}-H$	2.0–3.0
R_3CH		1.4–1.7		
<hr/>				
$R-\overset{ }{C}=\overset{ }{C}-\overset{ }{\underset{ }{C}}-H$		1.6–2.6	$I-\overset{ }{\underset{ }{C}}-H$	2.0–4.0
$R-\overset{O}{\parallel}{C}-\overset{ }{\underset{ }{C}}-H, H-\overset{O}{\parallel}{C}-\overset{ }{\underset{ }{C}}-H$		2.1–2.4	$Br-\overset{ }{\underset{ }{C}}-H$	2.7–4.1
$RO-\overset{O}{\parallel}{C}-\overset{ }{\underset{ }{C}}-H, HO-\overset{O}{\parallel}{C}-\overset{ }{\underset{ }{C}}-H$		2.1–2.5	$Cl-\overset{ }{\underset{ }{C}}-H$	3.1–4.1
$N\equiv C-\overset{ }{\underset{ }{C}}-H$		2.1–3.0	$R-\overset{O}{\parallel}{S}(O)-\overset{ }{\underset{ }{C}}-H$	ca. 3.0
		2.3–2.7	$RO-\overset{ }{\underset{ }{C}}-H, HO-\overset{ }{\underset{ }{C}}-H$	3.2–3.8
$R-C\equiv C-H$		1.7–2.7	$R-\overset{O}{\parallel}{C}-O-\overset{ }{\underset{ }{C}}-H$	3.5–4.8
<hr/>				
$R-S-H$	var	1.0–4.0 ^a	$O_2N-\overset{ }{\underset{ }{C}}-H$	4.1–4.3
$R-\overset{ }{N}-H$	var	0.5–4.0 ^a	$F-\overset{ }{\underset{ }{C}}-H$	4.2–4.8
$R-O-H$	var	0.5–5.0 ^a	<hr/>	
	var	4.0–7.0 ^a	$R-\overset{ }{C}=\overset{ }{C}-H$	4.5–6.5
	var	3.0–5.0 ^a		6.5–8.0
$R-\overset{O}{\parallel}{C}-\overset{ }{N}-H$	var	5.0–9.0 ^a	$R-\overset{O}{\parallel}{C}-H$	9.0–10.0
			$R-\overset{O}{\parallel}{C}-OH$	11.0–12.0

Note: For those hydrogens shown as $-\overset{|}{\underset{|}{C}}-H$, if that hydrogen is part of a methyl group (CH_3), the shift is generally at the low end of the range given; if the hydrogen is in a methylene group ($-CH_2-$), the shift is intermediate; and if the hydrogen is in a methine group ($-CH-$), the shift is typically at the high end of the range given.

^aThe chemical shift of these groups is variable, depending on the chemical environment in the molecule and on concentration, temperature, and solvent.