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with a screw cap thread and a PTFE valve (Wilmad) are convenient for freeze thawing of NMR samples. Alternatively NMR tubes with "J Young" valves (Wilmad) can also be used.

Vacuum-lines, Schlenk and glovebox techniques

Manipulations involving materials sensitive to air or water vapour can be carried out by these procedures. Vacuum-line methods make use of quantitative transfers, and **P**(pressure)-**V**(volume)-**T**(temperature) measurements, of gases, and trap-to-trap separations of volatile substances.
It is usually more convenient to work under an inert-gas atmosphere using **Schlenk** type apparatus. The *principle* of Schlenk methods involve a flask/vessel which has a standard ground-glass joint and a sidearm with a tap. The system can be purged by evacuating and flushing with an inert gas (usually nitrogen, or in some cases, argon), repeating the process until the contaminants in the vapour phases have been diminished to acceptable limits. A large range of Schlenk glassware is commercially available (e.g. see Aldrich Chemical Catalog and the associated technical bulletin AL-166). With these, and tailor-made pieces of glassware, inert atmospheres can be maintained during crystallisation, filtration, sublimation and transfer.
Syringe techniques have been developed for small volumes, while for large volumes or where much manipulation is required, dryboxes (*glove boxes*) or dry chambers should be used.

ABBREVIATIONS

Titles of periodicals are defined as in the Chemical Abstracts Service Source Index (CASSI), except that full stops have been omitted after each abbreviated word. Abbreviations of words in the texts of Chapters 4, 5 and 6 are those in common use and are self evident, e.g. distn, filtd, conc and vac are used for distillation, filtered, concentrated and vacuum.

TABLES

TABLE 1. SOME COMMON IMMISCIBLE OR SLIGHTLY MISCIBLE PAIRS OF SOLVENTS

Carbon tetrachloride with ethanolamine, ethylene glycol, formamide or water.
Dimethyl formamide with cyclohexane or petroleum ether.
Dimethyl sulfoxide with cyclohexane or petroleum ether.
Ethyl ether with ethanolamine, ethylene glycol or water.
Methanol with carbon disulfide, cyclohexane or petroleum ether.
Petroleum ether with aniline, benzyl alcohol, dimethyl formamide, dimethyl sulfoxide, formamide, furfuryl alcohol, phenol or water.
Water with aniline, benzene, benzyl alcohol, carbon disulfide, carbon tetrachloride, chloroform, cyclohexane, cyclohexanol, cyclohexanone, diethyl ether, ethyl acetate, isoamyl alcohol, methyl ethyl ketone, nitromethane, tributyl phosphate or toluene.

TABLE 2. AQUEOUS BUFFERS

Approx. pH	**Composition**
0	2N sulfuric acid or N hydrochloric acid
1	0.1N hydrochloric acid or 0.18N sulfuric acid
2	**Either** 0.01N hydrochloric acid or 0.013N sulfuric acid **Or** 50 mL of 0.1M glycine (also 0.1M NaCl) + 50 mL of 0.1N hydrochloric acid
3	**Either** 20 mL of the 0.2M Na_2HPO_4 + 80 mL of 0.1M citric acid **Or** 50 mL of 0.1M glycine + 22.8 mL of 0.1N hydrochloric acid in 100 mL
4	**Either** 38.5 mL of 0.2M Na_2HPO_4 + 61.5 mL of 0.1M citric acid **Or** 18 mL of 0.2M NaOAc + 82 mL of 0.2M acetic acid
5	**Either** 70 mL of 0.2M NaOAc + 30 mL of 0.2M acetic acid **Or** 51.5 mL of 0.2M Na_2HPO_4 + 48.5 mL of 0.1M citric acid
6	63 mL of 0.2M Na_2HPO_4 + 37 mL of 0.1M citric acid
7	82 mL of M Na_2HPO_4 + 18 mL of 0.1M citric acid
8	**Either** 50 mL of 0.1M Tris buffer + 29 mL of 0.1N hydrochloric acid, in 100 mL **Or** 30 mL of 0.05M borax + 70 mL of 0.2M boric acid
9	80 mL of 0.05M borax + 20 mL of 0.2M boric acid
10	**Either** 25 mL of 0.05M borax + 43 mL of 0.1N NaOH, in 100 mL **Or** 50 mL of 0.1M glycine + 32 mL of 0.1N NaOH, in 100 mL
11	50 mL of 0.15M Na_2HPO_4 + 15 mL of 0.1N NaOH
12	50 mL of 0.15M Na_2HPO_4 + 75 mL of 0.1N NaOH
13	0.1N NaOH or KOH
14	N NaOH or KOH

These buffers are suitable for use in obtaining ultraviolet spectra. Alternatively, for a set of accurate buffers of low, but constant, ionic strength (I = 0.01) *covering a pH range 2.2 to 11.6 at 20°, see* Perrin *Aust J Chem* **16** 572 *1963*. "In 100 mL" means that the solution is made up to 100 mL with pure water.

TABLE 3A. PREDICTED EFFECT OF PRESSURE ON BOILING POINT*

Temperature in degrees Centigrade

760 mmHg	0	20	40	60	80	100	120	140	160	180
0.1	-111	-99	-87	-75	-63	-51	-39	-27	-15	-4
0.2	-105	-93	-81	-69	-56	-44	-32	-19	-7	5
0.4	-100	-87	-74	-62	-49	-36	-24	-11	2	15
0.6	-96	-83	-70	-57	-44	-32	-19	-6	7	20
0.8	-94	-81	-67	-54	-41	-28	-15	-2	11	24
1.0	-92	-78	-65	-52	-39	-25	-12	1	15	28
2.0	-85	-71	-58	-44	-30	-16	-3	11	25	39
4.0	-78	-64	-49	-35	-21	-7	8	22	36	51
6.0	-74	-59	-44	-30	-15	-1	14	29	43	58
8.0	-70	-56	-41	-26	-11	4	19	34	48	63
10.0	-68	-53	-38	-23	-8	7	22	37	53	68
14.0	-64	-48	-33	-23	-2	13	28	44	59	74
16.0	-61	-45	-29	-14	2	17	33	48	64	79
20.0	-59	-44	-28	-12	3	19	35	50	66	82
30.0	-54	-38	-22	-6	10	26	42	58	74	90
40.0	-50	-34	-17	-1	15	32	48	64	81	97
50.0	-47	-30	-14	3	19	36	52	69	86	102
60.0	-44	-28	-11	6	23	40	56	73	86	107
80.0	-40	-23	-6	11	28	45	62	79	97	114
100.0	-37	-19	-2	15	33	50	67	85	102	119
150.0	-30	-12	6	23	41	59	77	95	112	130
200.0	-25	-7	11	29	47	66	84	102	120	138
300.0	-18	1	19	38	57	75	94	113	131	150
400.0	-13	6	25	44	64	83	102	121	140	159
500.0	-8	11	30	50	69	88	108	127	147	166
600.0	-5	15	34	54	74	93	113	133	152	172
700.0	-2	18	38	58	78	98	118	137	157	177
750.0	0	20	40	60	80	100	120	140	160	180
770.0	0	20	40	60	80	100	120	140	160	180
800.0	1	21	41	61	81	101	122	142	162	182

* *How to use the Table*: Take as an example a liquid with a boiling point of 80°C at 760mm Hg. The Table gives values of the boiling points of this liquid at pressures from 0.1 to 800mm Hg. Thus at 50mm Hg this liquid has a boiling point of 19°C, and at 2mm Hg its boiling point would be -30°C.

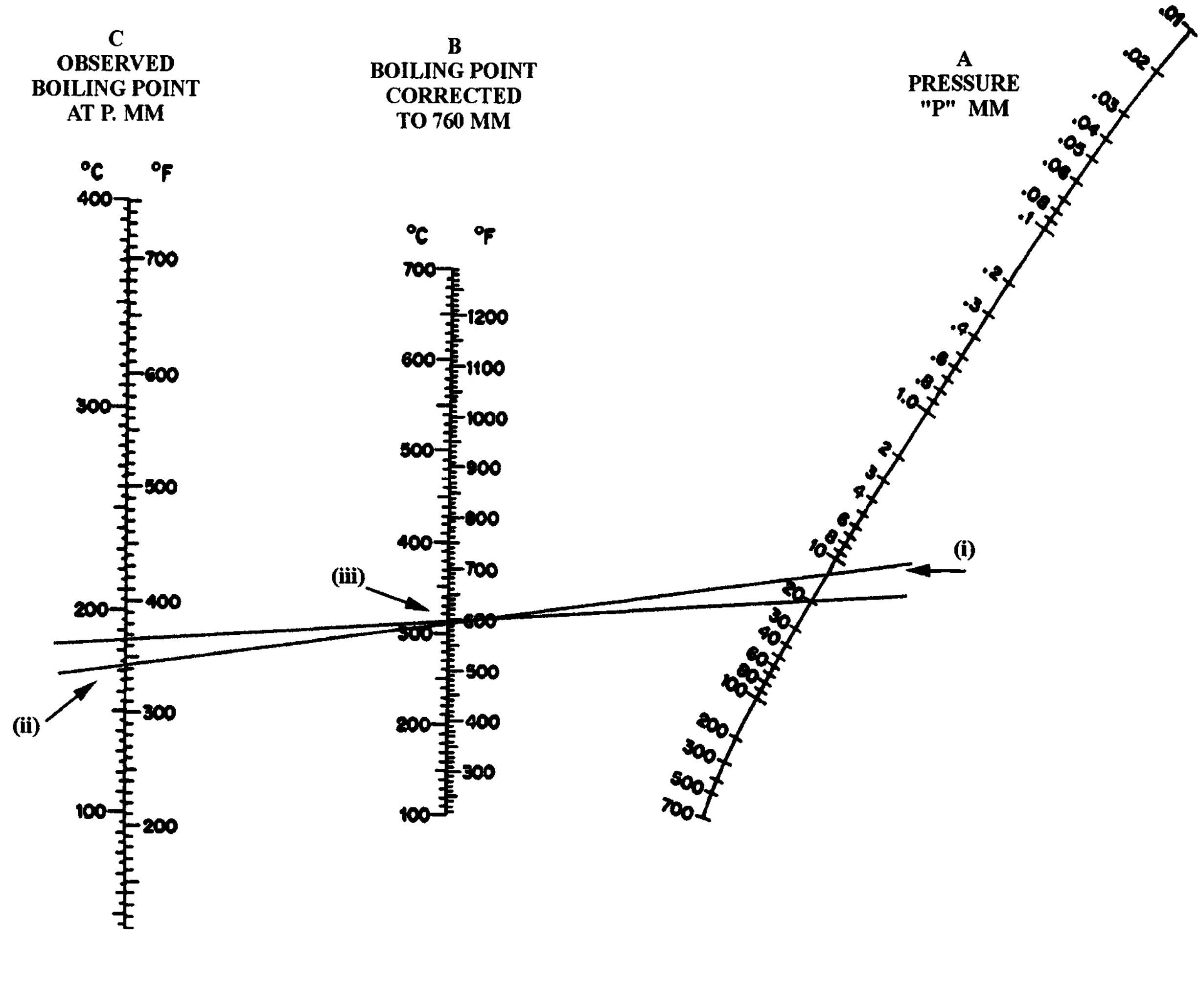
TABLE 3B. PREDICTED EFFECT OF PRESSURE ON BOILING POINT*

Temperature in degrees Centigrade

760mmHg	200	220	240	260	280	300	320	340	360	380	400
0.1	8	20	32	44	56	68	80	92	104	115	127
0.2	17	30	42	54	67	79	91	103	116	128	140
0.4	27	40	53	65	78	91	103	116	129	141	154
0.6	33	40	59	72	85	98	111	124	137	150	163
0.8	38	51	64	77	90	103	116	130	143	156	169
1.0	41	54	68	81	94	108	121	134	147	161	174
2.0	53	66	80	94	108	121	135	149	163	176	190
4.0	65	79	93	108	122	136	151	156	179	193	208
6.0	72	87	102	116	131	146	160	175	189	204	219
8.0	78	93	108	123	137	152	167	182	197	212	227
10.0	83	98	113	128	143	158	173	188	203	218	233
14.0	90	105	120	136	151	166	182	197	212	228	243
18.0	95	111	126	142	157	173	188	204	219	235	251
20.0	97	113	129	144	160	176	191	207	223	238	254
30.0	106	123	139	155	171	187	203	219	235	251	267
40.0	113	130	146	162	179	195	211	228	244	260	277
50.0	119	135	152	168	185	202	218	235	251	268	284
60.0	123	140	157	174	190	207	224	241	257	274	291
80.0	131	148	165	182	199	216	233	250	267	284	301
100.0	137	154	171	189	206	223	241	258	275	293	310
150.0	148	166	184	201	219	237	255	273	290	308	326
200.0	156	174	193	211	229	247	265	283	302	320	338
300.0	169	187	206	225	243	262	281	299	318	337	355
400.0	178	197	216	235	254	273	292	311	330	350	369
500.0	185	205	224	244	263	282	302	321	340	360	379
600.0	192	211	231	251	270	290	310	329	349	368	388
700.0	197	217	237	257	277	296	316	336	356	376	396
750.0	200	220	239	259	279	299	319	339	359	279	399
770.0	200	220	241	261	281	301	321	341	361	381	401
800.0	202	222	242	262	282	302	322	342	262	382	403

* *How to use the Table*: Taking as an example a liquid with a boiling point of 340°C at 760mm Hg, the column headed 340°C gives values of the boiling points of this liquid at each value of pressures from 0.1 to 800mm Hg. Thus, at 100mm Hg its boiling point is 258°C, and at 0.8mm Hg its boiling point will be 130°C.

FIGURE 1: NOMOGRAM



How to use Figure 1:
You can use a nomogram to estimate the boiling points of a substance at a particular pressure. For example, the boiling point of 4-methoxybenzenesulfonyl chloride is 173°C/14mm. Thus to find out what the boiling point of this compound will be at 760mm (atmospheric), draw a point on curve A (pressure) at 14mm (this is shown in (i). Then draw a point on curve C (observed boiling point) corresponding to 173° (or as close as possible). This is shown in (ii). Using a ruler, find the point of intersection on curve B, drawing a line between points (i) and (ii). This is the point (iii) and is the boiling point of 4-methoxybenzenesulfonyl chloride (i.e. approx. 310°C) at atmospheric pressure. If you want to distil 4-methoxybenzenesulfonyl chloride at 20mm, then you will need to draw a point on curve A (at 20mm). Using a ruler, find the point of intersection on curve C drawing through the line intersecting (iii, curve B, i.e. 310°C) and the point in curve A corresponding to 20mm. You should have a value of 185°C, that is, the boiling point of 4-methoxybenzenesulfonyl chloride is estimated to be at 185°C at 20mm.

TABLE 4. HEATING BATHS

Up to 100°	Water baths
-20 to 200°	Glycerol or di-*n*-butyl phthalate
Up to about 200°	Medicinal paraffin
Up to about 250°	Hard hydrogenated cotton-seed oil (**m** 40-60°) or a 1:1 mixture of cotton-seed oil and castor oil containing about 1% of hydroquinone.
-40 to 250° (to 400° under N_2)	D.C. 550 silicone fluid
Up to about 260°	A mixture of 85% orthophosphoric acid (4 parts) and metaphosphoric acid (1 part)
Up to 340°	A mixture of 85% orthophosphoric acid (2 parts) and metaphosphoric acid (1 part)
60 to 500°	Fisher bath wax (highly unsaturated)
73 to 350°	Wood's Metal*
250 to 800°	Solder*
350 to 800°	Lead*

* In using metal baths, the container (usually a metal crucible) should be removed while the metal is still molten.

TABLE 5. WHATMAN FILTER PAPERS

Grade No.	1	2	3	4	5	6	113
Particle size retained (in microns)	11	8	5	12	2.4	2.8	28
Filtration speed*(sec/100mL)	40	55	155	20	<300	125	9

Routine ashless filters

Grade No.	40	41	42	43	44
Particle size retained (in microns)	7.5	12	3	12	4
Filtration speed* (sec/100mL)	68	19	200	38	125

	Hardened			**Hardened ashless**		
Grade No.	50	52	54	540	541	542
Particle size retained(in microns)	3	8	20	9	20	3
Filtration speed* (sec/100mL)	250	55	10	55	12	250

Glass microfilters

Grade No	GF/A	GF/B	GF/C	GF/D	GF/F
Particle size retained (in microns)	1.6	1.0	1.1	2.2	0.8
Filtration speed (sec/100mL)*	8.3	20.0	8.7	5.5	17.2

**Filtration speeds are rough estimates of initial flow rates and should be considered on a relative basis.*

TABLE 6. MICRO FILTERS*

Nucleopore (polycarbonate) Filters

Mean Pore Size (microns)	8.0	2.0	1.0	0.1	0.03	0.015
Av. pores/cm^2	10^5	$2x10^6$	$2x10^7$	$3x10^8$	$6x10^8$	$1\text{-}6x10^9$
Water flow rate(mL/min/cm^2)	2000	2000	300	8	0.03	0.1-0.5

Millipore Filters

Type	*Cellulose ester*		*Teflon*		*Microweb#*	
	MF/SC	MF/VF	LC	LS	WS	WH
Mean Pore Size (microns)	8	0.01	10	5	3	0.45
Water flow rate (mL/min/cm^2)	850	0.2	170	70	155	55

Gelman Membranes

Type	*Cellulose ester*				*Copolymer*	
	GA-1	TCM-450	VM-1	DM-800	AN-200	Tuffryn-450
Mean Pore Size (microns)	5	0.45	5	0.8	0.2	0.45
Water flow rate (mL/min/cm^2)	320	50	700	200	17	50

Sartorius Membrane Filters (SM)

Application	Gravi-metric	Biological clarificatn	Sterili-sation	Particle count in H_2O	For acids & bases
Type No.	11003	11004	11006	11011	12801
Mean PoreSize (microns)	1.2	0.6	0.45	0.01	8
Water flow rate (mL/min/cm^2)	300	150	65	0.6	1100

* Only a few representative filters are tabulated (available ranges are more extensive). # Reinforced nylon.

TABLE 7. COMMON SOLVENTS USED IN RECRYSTALLISATION

Acetic acid (118°)	*Cyclohexane (81°)	*Methanol (64.5°)
*Acetone (56°)	Dichloromethane (41°)	*Methyl ethyl ketone (80°)
Acetylacetone (139°)	*Diethyl ether (34.5°)	Methyl isobutyl ketone (116°)
Acetonitrile (82°C)	Dimethyl formamide (76°/39mm)	Nitrobenzene (210°)
*Benzene (80°)	*Dioxane (101°)	Nitromethane (101°)
Benzyl alcohol (93°/10mm)	*Ethanol (78°)	*Petroleum ether (various)
n-Butanol (118°)	2-Ethoxyethanol (cellosolve 135°)	Pyridine (115.5°)
Butyl acetate (126.5°)	*Ethyl acetate (78°)	Pyridine trihydrate (93°)
n-Butyl ether (142°)	Ethyl benzoate (98°/19mm)	*Tetrahydrofuran (64-66°)
γ-Butyrolactone (206°)	Ethylene glycol (68°/4mm)	Toluene (110°)
Carbon tetrachloride (77°)	Formamide (110°/10mm)	Trimethylene glycol (59°/11mm)
Chlorobenzene (132°)	Glycerol (126°/11mm)	Water (100°)
Chloroform (61°)	Isoamyl alcohol (131°)	Xylenes (*o* 143-145°, *m* 138-139°, *p* 138°)

***Highly flammable, should be heated or evaporated on steam or electrically heated water baths only (preferably under nitrogen). None of these solvents should be heated over a naked flame.**

TABLE 8. PAIRS OF MISCIBLE SOLVENTS

Acetic acid: with chloroform, ethanol, ethyl acetate, acetonitrile, petroleum ether, or water.
Acetone: with benzene, butyl acetate, butyl alcohol, carbon tetrachloride, chloroform, cyclohexane, ethanol, ethyl acetate, methyl acetate, acetonitrile, petroleum ether or water.
Ammonia: with ethanol, methanol, pyridine.
Aniline: with acetone, benzene, carbon tetrachloride, ethyl ether, *n*-heptane, methanol, acetonitrile or nitrobenzene.
Benzene: with acetone, butyl alcohol, carbon tetrachloride, chloroform, cyclohexane, ethanol, acetonitrile, petroleum ether or pyridine.
Butyl alcohol: with acetone or ethyl acetate.
Carbon disulfide: with petroleum ether.
Carbon tetrachloride: with cyclohexane.
Chloroform: with acetic acid, acetone, benzene, ethanol, ethyl acetate, hexane, methanol or pyridine.
Cyclohexane: with acetone, benzene, carbon tetrachloride, ethanol or diethyl ether.
Diethyl ether: with acetone, cyclohexane, ethanol, methanol, methylal (dimethoxymethane), acetonitrile, pentane or petroleum ether.
Dimethyl formamide: with benzene, ethanol or ether.
Dimethyl sulfoxide: with acetone, benzene, chloroform, ethanol, diethyl ether or water.
Dioxane: with benzene, carbon tetrachloride, chloroform, ethanol, diethyl ether, petroleum ether, pyridine or water.
Ethanol: with acetic acid, acetone, benzene, chloroform, cyclohexane, dioxane, ethyl ether, pentane, toluene, water or xylene.
Ethyl acetate: with acetic acid, acetone, butyl alcohol, chloroform, or methanol.
Glycerol: with ethanol, methanol or water.
Hexane: with benzene, chloroform or ethanol.
Methanol: with chloroform, diethyl ether, glycerol or water.
Methylal: with diethyl ether.
Methyl ethyl ketone: with acetic acid, benzene, ethanol or methanol.
Nitrobenzene: with aniline, methanol or acetonitrile.
Pentane: with ethanol or diethyl ether.
Petroleum ether: with acetic acid, acetone, benzene, carbon disulfide or diethyl ether.
Phenol: with carbon tetrachloride, ethanol, diethyl ether or xylene.
Pyridine: with acetone, ammonia, benzene, chloroform, dioxane, petroleum ether, toluene or water.
Toluene: with ethanol, diethyl ether or pyridine.
Water: with acetic acid, acetone, ethanol, methanol, or pyridine.
Xylene: with ethanol or phenol.

TABLE 9. MATERIALS FOR COOLING BATHS

Temperature	Composition	Temperature	Composition
0°	Crushed ice		
-5° to -20°	Ice-salt mixtures	-77°	Solid CO_2 with chloroform or acetone
Up to -20°	Ice-MeOH mixtures	-78°	Solid CO_2 (powdered; CO_2 snow)
-33°	Liquid ammonia		
-40° to -50°	Ice (3.5-4 parts) - $CaCl_2$ $6H_2O$ (5 parts)	-100°	Solid CO_2 with diethyl ether
-72°	Solid CO_2 with ethanol	-196°	liquid nitrogen (see footnote*)

Alternatively, the following liquids can be used, partially frozen, as cryostats, by adding solid CO_2 from time to time to the material in a Dewar-type container and stirring to make a slush:

13°	*p*-Xylene	-55°	Diacetone
12°	Dioxane	-56°	*n*-Octane
6°	Cyclohexane	-60°	Di-isopropyl ether
5°	Benzene	-73°	Trichloroethylene or isopropyl acetate
2°	Formamide	-74°	*o*-Cymene or *p*-cymene
-8.6°	Methyl salicylate	-77°	Butyl acetate
-9°	Hexane-2,5-dione	-79°	Isoamyl acetate
-10.5°	Ethylene glycol	-83°	Propylamine
-11.9°	*tert*-Amyl alcohol	-83.6°	Ethyl acetate
-12°	Cycloheptane or methyl benzoate	-86°	Methyl ethyl ketone
-15°	Benzyl alcohol	-89°	*n*-Butanol
-16.3°	*n*-Octanol	-90°	Nitroethane
-18°	1,2-Dichlorobenzene	-91°	Heptane
-22°	Tetrachloroethylene	-92°	*n*-Propyl acetate
-22.4°	Butyl benzoate	-93°	2-Nitropropane or cyclopentane
-22.8°	Carbon tetrachloride	-94°	Ethyl benzene or hexane
-24.5°	Diethyl sulfate	-94.6°	Acetone
-25°	1,3-Dichlorobenzene	-95.1°	Toluene
-29°	*o*-Xylene or pentachloroethane	-97°	Cumene
-30°	Bromobenzene	-98°	Methanol or methyl acetate
-32°	*m*-Toluidine	-99°	Isobutyl acetate
-32.6°	Dipropyl ketone	-104°	Cyclohexene
-38°	Thiophene	-107°	Isooctane
-41°	Acetonitrile	-108°	1-Nitropropane
-42°	Pyridine or diethyl ketone	-116°	Ethanol or diethyl ether
-44°	Cyclohexyl chloride	-117°	Isoamyl alcohol
-45°	Chlorobenzene	-126°	Methylcyclohexane
-47°	*m*-Xylene	-131°	*n*-Pentane
-50°	Ethyl malonate or *n*-butylamine	-160°	Isopentane
-52°	Benzyl acetate or diethylcarbitol		

For other organic materials used in low temperature slush-baths with liquid nitrogen see R.E.Rondeau [*J Chem Eng Data* **11** 124 *1966*]. ***NOTE**: Use high quality pure nitrogen, do not use liquid air or liquid nitrogen that has been in contact with air for a long period (due to the dissolution of oxygen in it) as this could EXPLODE in contact with organic matter.

TABLE 10. LIQUIDS FOR STATIONARY PHASES IN GAS CHROMATOGRAPHY

Material	Temp.	Retards
Dimethylsulfolane	0-40°	Olefins and aromatic hydrocarbons
Di-*n*-butyl phthalate	0-40°	General purposes
Squalane	0-150°	Volatile hydrocarbons and polar molecules
Silicone oil or grease	0-250°	General purposes
Diglycerol	20-120°	Water, alcohols, amines, esters, and aromatic hydrocarbons
Dinonyl phthalate	20-130°	General purposes
Polydiethylene glycol succinate	50-200°	Aromatic hydrocarbons, alcohols, ketones, esters.
Polyethylene glycol	50-200°	Water, alcohols, amines, esters and aromatic hydrocarbons
Apiezon grease	50-200°	Volatile hydrocarbons and polar molecules
Tricresyl phosphate	50-250°	General purposes

TABLE 11. METHODS OF VISUALISATION OF TLC SPOTS

Reagent	Compound	Preparation	Observations
Iodine	General	Iodine crystals in a closed chamber or spray 1% methanol solution of Iodine	Brown spots which may disappear upon standing. Limited sensitivity.
H_2SO_4	General	50% solution, followed by heating to 150°C	Black or coloured spots
Molybdate	General	5% $(NH_4)_6Mo_7O_{24}$ + 0.2% $Ce(SO_4)_2$ in 5% H_2SO_4, followed by heating to 150°C.	Deep blue spots
Vanillin	General	0.5g vanillin, 0.5 mL H_2SO_4, 9 mL ethanol	various coloured spots
Ammonia	phenols	Ammonia vapour in a closed chamber	various coloured spots
$FeCl_3$	phenols, enolic compounds	1% aqueous $FeCl_3$	various coloured spots
2,4-DNP	aldehydes, ketones	0.5% 2,4-dinitrophenylhydrazine/2M HCl	red to yellow spots
HCl	aromatic acids and amines	HCl vapour in a closed chamber	various coloured spots
Ninhydrin	amino acids, and amines	0.3% ninhydrin in *n*-BuOH with 3% AcOH, followed by heating to 125°C/10 min	blue spots
$PdCl_2$	S and Se compds	0.5% aq. $PdCl_2$ + few drops of conc. HCl	red and yellow spots
Anisaldehyde	carbohydrates	0.5 mL anisaldehyde in 0.5 mL conc H_2SO_4 + 95% EtOH + a few drops of AcOH Heat at 100-110°C for 20-30 minutes	various blue spots

TABLE 12. GRADED ADSORBENTS AND SOLVENTS

Adsorbents (decreasing effectiveness)	**Solvents** (increasing eluting ability)
Fuller's earth (hydrated aluminosilicate)	Petroleum ether, b. 40-60°.
Magnesium oxide	Petroleum ether, b. 60-80°.
Charcoal	Carbon tetrachloride.
Alumina	Cyclohexane.
Magnesium trisilicate	Benzene.
Silica gel	Diethyl ether.
Calcium hydroxide	Chloroform.
Magnesium carbonate	Ethyl acetate.
Calcium phosphate	Acetone.
Calcium carbonate	Ethanol.
Sodium carbonate	Methanol.
Talc	Pyridine.
Inulin	Acetic acid.
Sucrose = starch	

TABLE 13. REPRESENTATIVE ION-EXCHANGE RESINS

Sulfonated polystyrene
Strong-acid cation exchanger
AG 50W-x8
Amberlite IR-120
Dowex 50W-x8
Duolite 225
Permutit RS
Permutite C50D

Carboxylic acid-type
Weak acid cation exchangers
Amberlite IRC-50
Bio-Rex 70
Chelex 100
Duolite 436
Permutit C
Permutits H and H-70

Aliphatic amine-type
weak base anion exchangers
Amberlites IR-45 and IRA-67
Dowex 3-x4A
Permutit E
Permutit A 240A

Strong Base, anion exchangers
AG 2x8
Amberlite IRA-400
Dowex 2-x8
Duolite 113
Permutit ESB
Permutite 330D

TABLE 14. MODIFIED FIBROUS CELLULOSES FOR ION-EXCHANGE

Cation exchange	**Anion exchange**
CM cellulose (carboxymethyl)	DEAE cellulose (diethylaminoethyl)
CM 22, 23 cellulose	DE 22, 23 cellulose
P cellulose (phosphate)	PAB cellulose (*p*-aminobenzyl)
SE cellulose (sulfoethyl)	TEAE cellulose (triethylaminoethyl)
SM cellulose (sulfomethyl)	ECTEOLA cellulose

SE and SM are much stronger acids than CM, whereas P has two ionisable groups (pK 2-3, 6-7), *one of which is stronger, the other weaker, than for CM* (3.5-4.5). For basic strengths, the sequence is: TEAE » DEAE (pK 8-95) > ECTEOLA (pK 5.5-7) > PAB. **Their exchange capacities lie in the range 0.3 to 1.0 mg equiv/g.**

TABLE 15. BEAD FORM ION-EXCHANGE PACKAGINGS[1]

Cation exchange	Capacity (meq/g)	Anion exchange	Capacity (meq/g)
CM-Sephadex C-25, C-50.[2] (weak acid)	4.5±0.5	DEAE-Sephadex A-25, A-50.[7] (weak base)	3.5±0.5
SP-Sephadex C-25, C-50.[3] (strong acid)	2.3±0.3	QAE-Sephadex A-25, A-50.[8] (strong base)	3.0±0.4
CM-Sepharose CL-6B.[4]	0.12±0.02	DEAE-Sepharose CL-6B.[4]	0.13±0.02
		DEAE-Sephacel.[9]	1.4±0.1
Fractogel EMD, CO_2^- (pK ~4.5), SO_3^{2-} (pK ~<1).[5]		Fractogel EMD, DMAE (pK ~9), DEAE (pK ~10.8), TMAE (pK >13).[5]	
CM-32 Cellulose.		DE-32 Cellulose.	
CM-52 Cellulose.[6]		DE-52 Cellulose	

[1] May be sterilised by autoclaving at pH 7 and below 120°. [2] Carboxymethyl. [3] Sulfopropyl. [4] Crosslinked agarose gel, no pre-cycling required, pH range 3-10. [5] Hydrophilic methacrylate polymer with very little volume change on change of pH (equivalent to *Toyopearl*, Sigma), available in superfine 650S, and medium 650M particle sizes. [6] Microgranular, pre-swollen, no pre-cycling required. [7] Diethylaminoethyl. [8] Diethyl(2-hydroxypropyl)aminoethyl. [9] Bead form cellulose, pH range 2-12, no pre-cycling required. Sephadex and Sepharose from Pharmacia-Amersham Biosciences, Fractogel from Merck, Cellulose from Whatman.

TABLE 16. LIQUIDS FOR DRYING PISTOLS

	Boiling points (760mm)		Boiling points (760mm)
Ethyl chloride	12.2°	Toluene	110.5°
Dichloromethane	39.8°	Tetrachloroethylene	121.2°
Acetone	56.1°	Chlorobenzene	132.0°
Chloroform	62.0°	*m*-Xylene	139.3°
Methanol	64.5°	Isoamyl acetate	142.5°
Carbon tetrachloride	76.5°	Tetrachloroethane	146.3°
Ethanol	78.3°	Bromobenzene	155.0°
Benzene	79.8°	*p*-Cymene	176.0°
Trichloroethylene	86.0°	Tetralin	207.0°
Water	100.0°		

TABLE 17. VAPOUR PRESSURES (mm Hg) OF SATURATED AQUEOUS SOLUTIONS IN EQUILIBRIUM WITH SOLID SALTS

Salt	Temperature 10°	15°	20°	25°	30°	% Humidity at 20°
$LiCl.H_2O$			2.6			15
$CaBr_2.6H_2O$	2.1	2.7	3.3	4.0	4.8	19
KOAc			3.5			20
$CaCl_2.6H_2O$	3.5	4.5	5.6	6.9	8.3	20
CrO_3			6.1			32
$Zn(NO_3)_2.6H_2O$			7.4			42
$K_2CO_3.2H_2O$			7.7	10.7		44
KCNS			8.2			47
$Na_2Cr_2O_7.2H_2O$			9.1			52
$Ca(NO_3)_2.4H_2O$	6.0	7.7	9.6	11.9	14.2	55
$Mg(NO_3)_2.6H_2O$			9.8			56
$NaBr.2H_2O$	5.8	7.8	10.3	13.5	17.5	58
$NaNO_2$			11.6			66
$NaClO_3$			13.1			75
NaCl	6.9	9.6	13.2	17.8	21.4	75
NaOAc			13.3			76
NH_4Cl			13.8			79
$(NH_4)_2SO_4$			14.2			81
KBr			14.7			84
$KHSO_4$			15.1			86
KCl			15.1	20.2	27.0	86
K_2CrO_4			15.4			88
$ZnSO_4.7H_2O$			15.8			90
$NH_4.H_2PO_4$			16.3			93
KNO_3			16.7	22.3	29.8	95
$Pb(NO_3)_2$			17.2			98
H_2O	9.21	12.79	17.53	23.76	31.82	100

TABLE 18. DRYING AGENTS FOR CLASSES OF COMPOUNDS

Class	Dried with
Acetals	Potassium carbonate.
Acids (organic)	Calcium sulfate, magnesium sulfate, sodium sulfate.
Acyl halides	Magnesium sulfate, sodium sulfate.
Alcohols	Calcium oxide, calcium sulfate, magnesium sulfate, potassium carbonate, followed by magnesium and iodine.
Aldehydes	Calcium sulfate, magnesium sulfate, sodium sulfate.
Alkyl halides	Calcium chloride, calcium sulfate, magnesium sulfate, phosphorus pentoxide, sodium sulfate.
Amines	Barium oxide, calcium oxide, potassium hydroxide, sodium carbonate, sodium hydroxide.
Aryl halides	Calcium chloride, calcium sulfate, magnesium sulfate, phosphorus pentoxide, sodium sulfate.
Esters	Magnesium sulfate, potassium carbonate, sodium sulfate.
Ethers	Calcium chloride, calcium sulfate, magnesium sulfate, sodium, lithium aluminium hydride.
Heterocyclic bases	Magnesium sulfate, potassium carbonate, sodium hydroxide.
Hydrocarbons	Calcium chloride, calcium sulfate, magnesium sulfate, phosphorus pentoxide, sodium (not for olefins).
Ketones	Calcium sulfate, magnesium sulfate, potassium carbonate, sodium sulfate.
Mercaptans	Magnesium sulfate, sodium sulfate.
Nitro compounds and Nitriles	Calcium chloride, magnesium sulfate, sodium sulfate.
Sulfides	Calcium chloride, calcium sulfate.

TABLE 19. STATIC DRYING FOR SELECTED LIQUIDS (25°C)

Liquid	Water	Linde Type 4 A	Linde Type 5 A	Activated Alumina	Silicic Acid Gel
MeOH	Residual H_2O %	0.54	0.55	—	0.60
	Wt % absorbed	2.50	1.50	—	—
EtOH	Residual H_2O %	0.25	0.25	0.45	0.68
	Wt % absorbed	7.00	6.80	1.50	—
1-Butylamine	Residual H_2O %	1.65	1.31	1.93	2.07
	Wt % absorbed	10.40	18.20	3.40	—
2-Ethyl-hexylamine	Residual H_2O %	0.25	0.08	0.43	0.53
	Wt % absorbed	15.10	21.10	6.10	1.70
Diethyl ether	Residual H_2O %	0.001	0.013	0.16	0.27
	Wt % absorbed	9.50	9.20	6.20	4.30
Amyl acetate	Residual H_2O %	0.002	—	0.33	0.38
	Wt % absorbed	9.30	—	7.40	1.80

TABLE 20. BOILING POINTS OF SOME USEFUL GASES AT 760 mm

Argon	-185.6°	Krypton	-152.3°
Carbon dioxide (sublimes)	-78.5°	Methane	-164.0°
		Neon	-246.0°
Carbon monoxide	-191.3°	Nitrogen	-209.9°
Ethane	-88.6°	Nitrous oxide	-88.5°
Helium	-268.6°	Nitric oxide	-195.8°
Hydrogen	-252.6°	Oxygen	-182.96°

TABLE 21. SOLUBILITIES OF HCl AND NH_3 AT 760mm (g/100g OF SOLUTION)

Gas	Temperature °C	MeOH	EtOH	Et_2O
Hydrogen Chloride*	-10	54.6	—	37.5 (-9.2°)
	0	51.3	45.4	35.6
	20	47.0 (18°)	41.0	24.9
	30	43.0	38.1	19.47
Ammonia	15	21.6 (27.6g/100g MeOH)	13.2 (9.2g/100mL soln)	—
	25	16.5 (19.8g/100g MeOH)	10.0 (6.0g/100mL soln)	—

* Saturated EtOH with HCl is ~ 5.7M at 25°C, i.e. 21.5g/100mL of solution.

TABLE 22. PREFIXES FOR QUANTITIES

Fractional	deci (d) = 10^{-1}	centi (c) = 10^{-2}	milli (m) = 10^{-3}	micro (μ) = 10^{-6}	nano (n) = 10^{-9}	pico (p) = 10^{-12}	femto (f) = 10^{-15}	atto (atto) = 10^{-18}
Multiple	deca (d) = 10^{1}	hecto (h) = 10^{2}	kilo (k) = 10^{3}	mega (M) = 10^{6}	giga (G) = 10^{9}	tera (T) = 10^{12}	penta (P) = 10^{15}	eka (E) = 10^{18}

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