

Development of Techniques for Handling Organometallic Compounds

- as with other physical sciences, development is strongly coupled with advances in techniques:

NMR, ir, X-ray crystallography

- Many organometallic are air-sensitive, so technique development in handling compounds in vacuum or an inert atmosphere are central

- Increasing degree of sophistication...exclusion of O₂ and H₂O-

simple funnel with N₂ flow over flask, filter frit, etc.

glove bag

Schlenk tubes*

cannula methods

inert atmosphere boxes (several types)*

high vacuum line(*)

*depends upon purity of inert gas

- Historical:

1900- Alfred Stock developed chemical vacuum line for handling *volatile* non-metal hydrides (boranes, silanes, etc.). Rotary, oil-filled vacuum pump had just been developed...improved vacuum. Used soft glass; not able to withstand thermal shock; Hg pools used on stopcocks. Stock's death was a result of Hg poisoning.

1920- others (Anton Berg; HC Brown) pick up on Stock's design, add their own wrinkles.

Starting about 1960- new materials (silicones, teflon, flexible metal lines and connectors) led to better lines

- Glove bag to vacuum atmosphere's system...

plexiglas, Al, stainless steel

types of procedures for maintaining pure inert gas...gas types (He, N₂, Ar)

Na/K; supported O₂ removing materials

circulating pathways and effectiveness of O₂/H₂O removal

desiccants: LN₂(10⁻²³ Torr); CaH₂; 4Å MS(10⁻³ Torr), Al₂O₃, CaCl₂ (0.2 Torr)

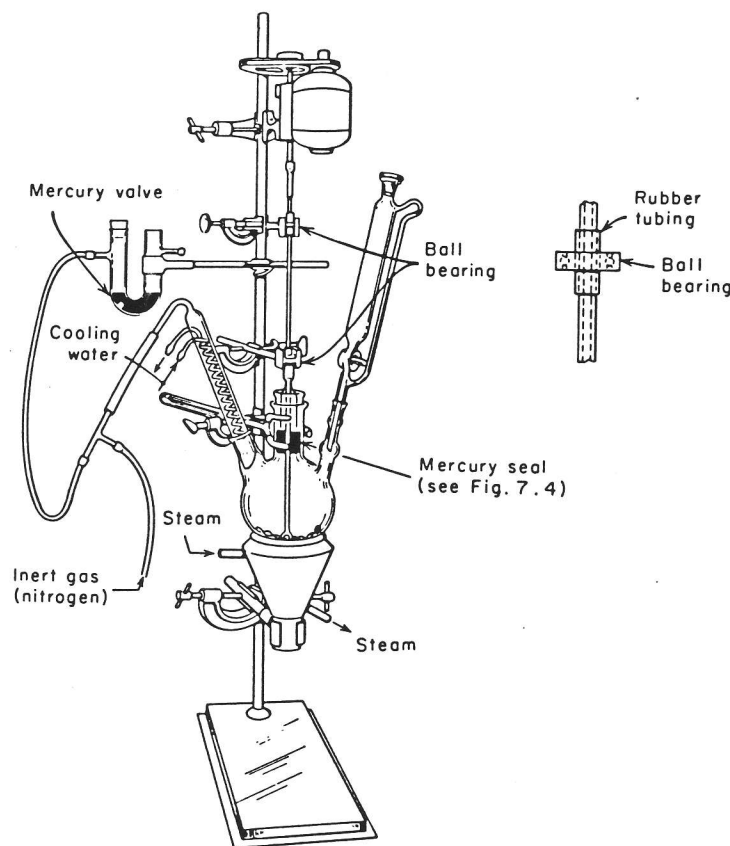


Fig. 7.1. Three-neck reaction flask with dropping funnel, stirrer, and reflux condenser. This type of setup is frequently used for Grignard and similar reactions. (By permission from Fieser and Fieser, "Reagents for Organic Synthesis," John Wiley & Sons, Inc., New York, 1967.)

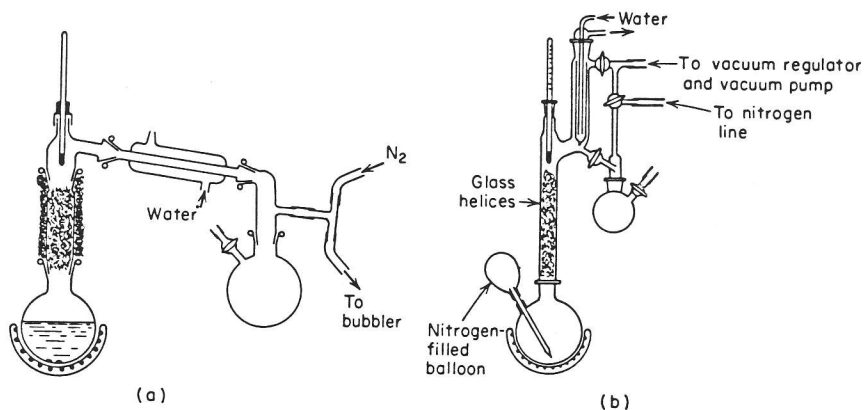


Fig. 7.2. (a) Distillation under inert atmosphere: Note that the complete apparatus should be purged initially, but a flow through the apparatus during distillation reduces the efficiency of separation. The sidearm allows one to pump out the stoppered flask and fill it with nitrogen before it is attached to the still. Also, it allows one to maintain a brisk nitrogen flow over the solvent when it is removed from the still. (b) Distillation at reduced pressure with minimum exposure to the atmosphere: The nitrogen-filled balloon may be replaced by a hose connected to a nitrogen source. During the vacuum distillation the three-way stopcock is turned so the upper and lower sections communicate, but nitrogen does not enter. The other stopcocks are open. The reflux ratio is adjusted by turning the cold finger. When a sample is to be collected, the lower stopcock is closed and the three-way stopcock is turned so that nitrogen is admitted to the receiver, which may then be removed under nitrogen flush. If another fraction is to be collected, the upper stopcock is momentarily closed off, the new receiver is pumped out, and the new fraction is collected.

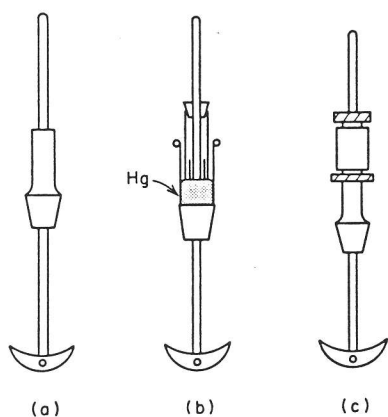


Fig. 7.4. Stirrers. (a) This precision-ground stirrer must be well lubricated with light mineral oil. (b) The mercury-seal stirrer involves a concentric cylinder which is connected to the stirring shaft by means of a one-hole stopper. This cylinder dips into the mercury pool to form the seal. The stirrer shaft is supported as indicated in Fig. 7.1. (c) The upper section of this stirrer is constructed with a stuffing box; another design, which is similar in outward appearance, utilizes an O-ring seal.

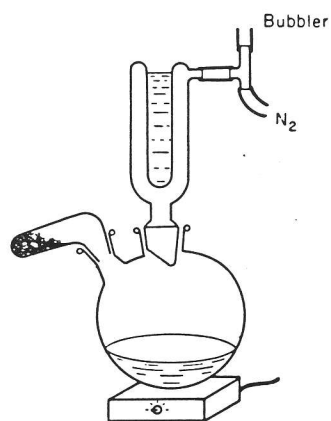


Fig. 7.3. Reaction flask with solids-addition tube, low-temperature reflux condenser, and magnetic stirrer. Solids are introduced by rotating the addition tube and tapping it gently.

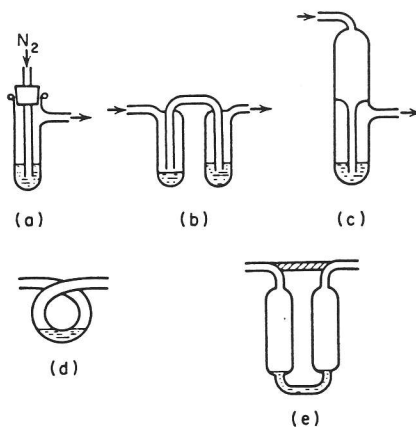


Fig. 7.5. Some common bubbler designs. Examples b through e have the advantage that mineral oil is not sucked into the apparatus if a momentary pressure reversal occurs.

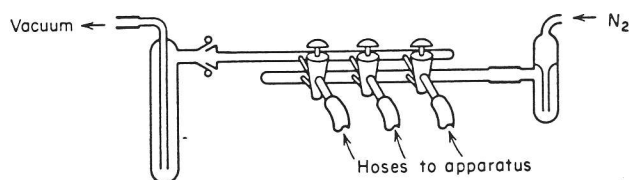


Fig. 7.6. Manifold for medium-vacuum and inert gas. A mineral oil bubbler is included in the nitrogen line, and a low-temperature trap is used in the vacuum line to protect the pump.

Fig 7.8 Schlenk tube. This is useful as a reaction vessel and filtrate receiver. The small glass ears adjacent to the ground glass joint are used to hold the components together with rubber bands or springs. Two sizes are convenient: one with a $\frac{2}{4}$ joint, a length of 250 mm, and diameter of 55 mm; the other with a $\frac{1}{3}$ joint, a length of 160 mm, and 40-mm diameter. Note the gradual flare below the joint, which facilitates removal of solids and liquids.



Fig. 7.9. Fritte or fritted funnel. This is used for filtrations. When stopcock grease is objectionable, Teflon stopcocks may be used. However, these tend to leak and are not suitable for extremely sensitive samples. In this case the lower stopcock may be omitted or the double Schlenk tube (Fig. 7.21) may be used. The fritted funnel is constructed with overall dimensions similar to those of the Schlenk tube.

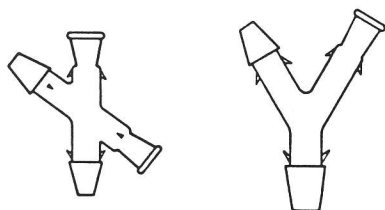
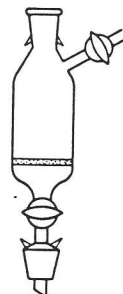


Fig. 7.12. Transfer crosses.

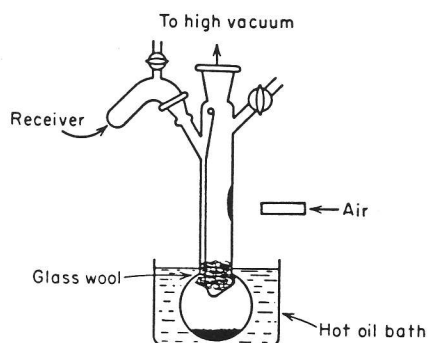


Fig. 7.13. Sublimation apparatus. Typical size: $\frac{1}{3}$ joints; height, 300 mm; diameter, 20 mm.

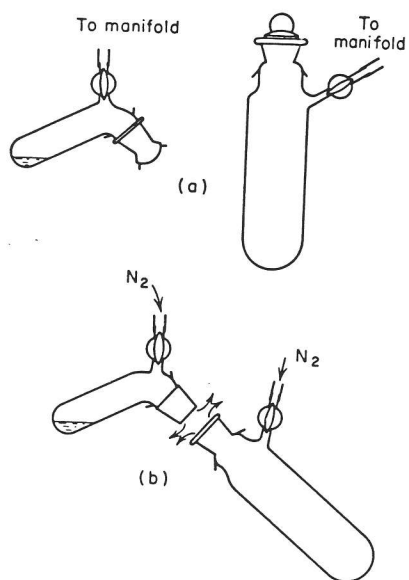


Fig. 7.14. Joining the solids container to a Schlenk tube. (a) Initial purge of tubes; (b) the union of the two parts.

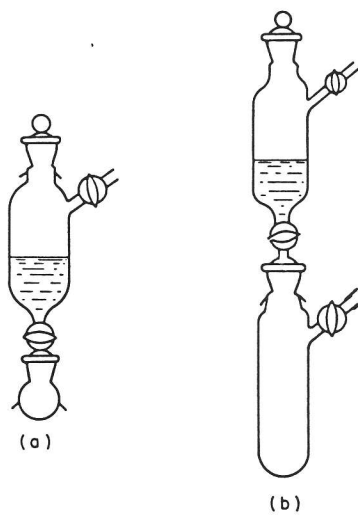


Fig. 7.15. Dropping funnel. (a) The dropping funnel after it has been purged and filled with solvent; (b) dropping funnel attached to a Schlenk tube.

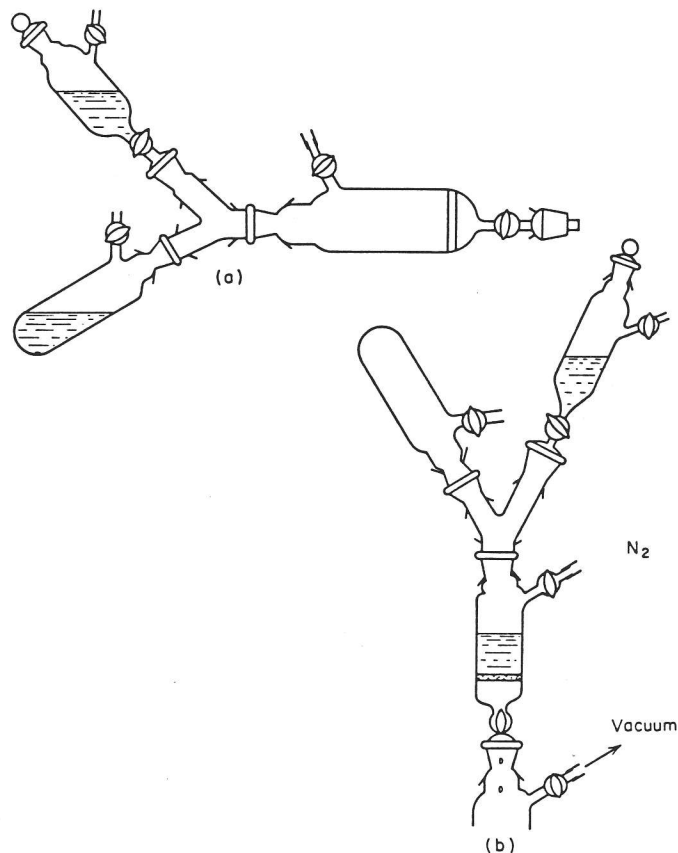


Fig. 7.16. A filtration. (a) Schlenk tube with mother liquor and precipitate and dropping funnel attached to a fritted funnel through a transfer cross. (b) Filtration: The filtrate is being collected in a previously purged Schlenk tube.

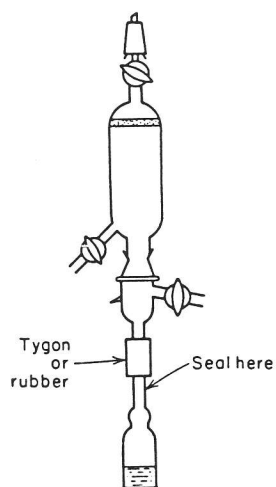


Fig. 7.19. Transfer into a seal-off vial. Note that the vial adapter is designed so it may be purged and then flushed while it is being connected to a fritte (or Schlenk tube). When the solid has been transferred, the system may be evacuated and the vial sealed off. If one wishes to seal off the compound under 1 atm nitrogen, a remote stopcock may be opened to the atmosphere immediately before the seal is to be made. This will avoid the possibility of blowing a bubble in the glass because of excess pressure. The seal is accomplished by heating and then pulling out the glass, which is then sealed in a region of small diameter.

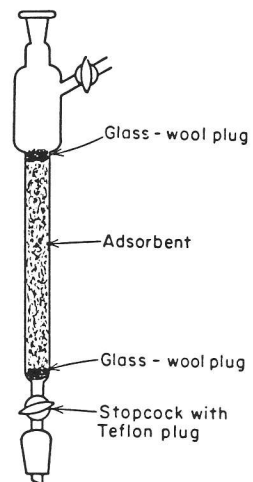


Fig. 7.20. Column for adsorption chromatography. Typical size: $\frac{1}{8}$ inch joints, 45-mm-diameter upper section, 20-mm-diameter column, and a 500-mm column length.

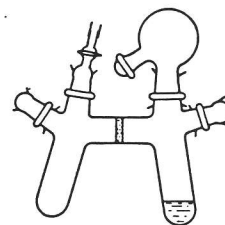


Fig. 7.21. Double Schlenk tube. In this figure a reaction product has been poured into the Schlenk tube. The reaction flask may be removed and the apparatus rotated by about 70° , followed by a slight evacuation of the left chamber to pull the filtrate into the left leg. Typical size: $\frac{2}{4}$ inch joints, 50-mm-diameter legs.

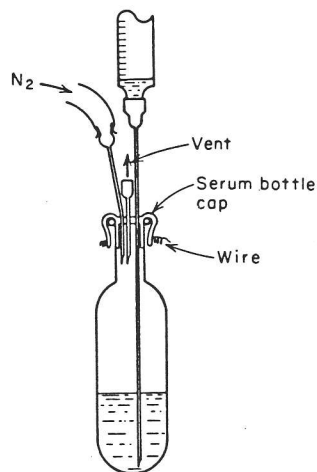


Fig. 7.23. The three-needle technique.

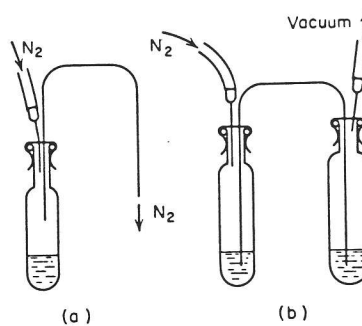


Fig. 7.25. Stainless-steel transfer tube. (a) Initial purge of transfer tube; (b) transfer of liquid through the tube by means of a pressure differential.

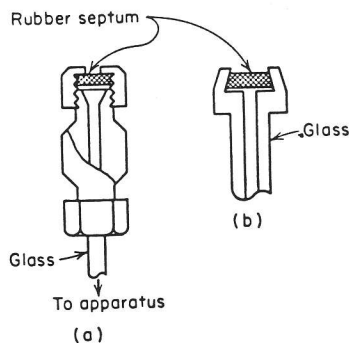


Fig. 7.26. Methods for attaching rubber septa to glass apparatus. (a) A modified Swagelok-type of fitting. The lip on one end of the fitting is turned down on a lathe, and the septum replaces the ferrules. On the other end, a Teflon front ferrule is used to make the connection with a glass tube (see Chap. 4). (b) An all-glass septum holder.

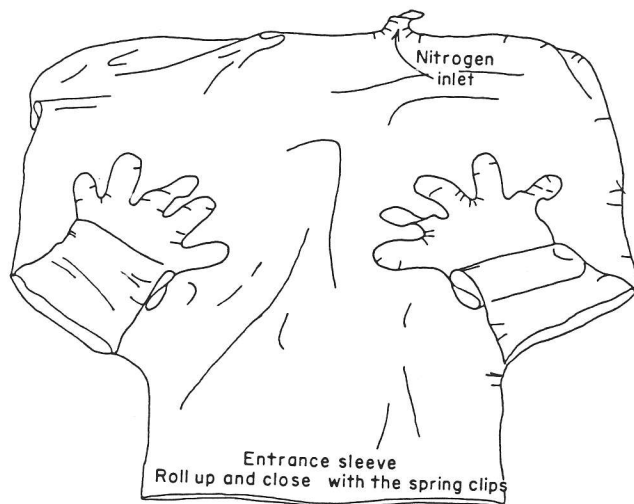


Fig. 8.1. Polyethylene glove bag. (Adapted from Instruments for Research and Industry, Cheltenham, Pa., Glove Bag Brochure.)

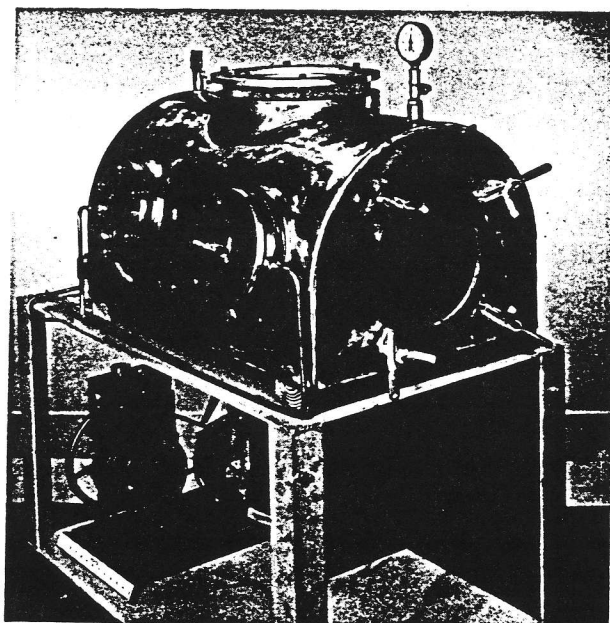
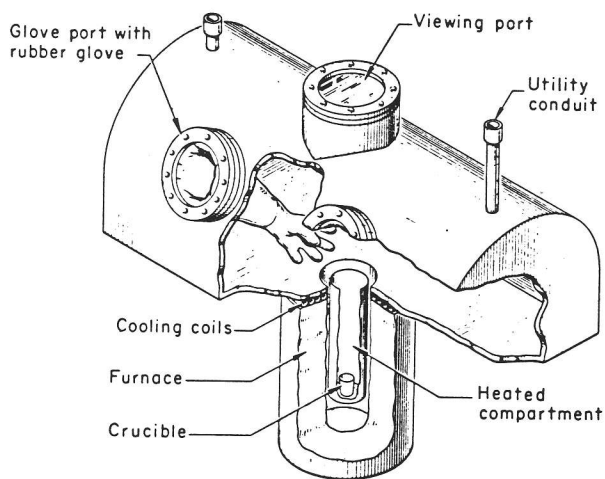


Fig. 8.16. A small evacuable glove box. The furnace arrangement is designed for studies on fused salts. (By permission from C. J. Barton, "Technique of Inorganic Chemistry," H. B. Jonassen and A. Weissberger (eds.), Vol. 3, p. 316, Interscience Publishers, New York, 1963.

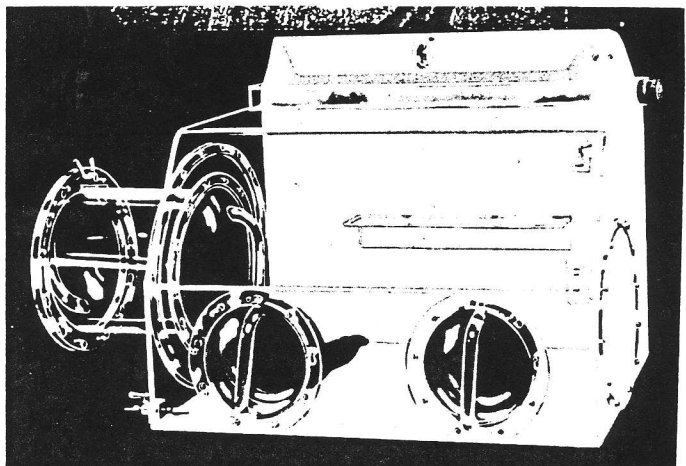


Fig. 8.18. A poly(methacrylate) glove box. (By permission of The Manostat Corp., New York, N.Y., Glove Box Bulletin.)

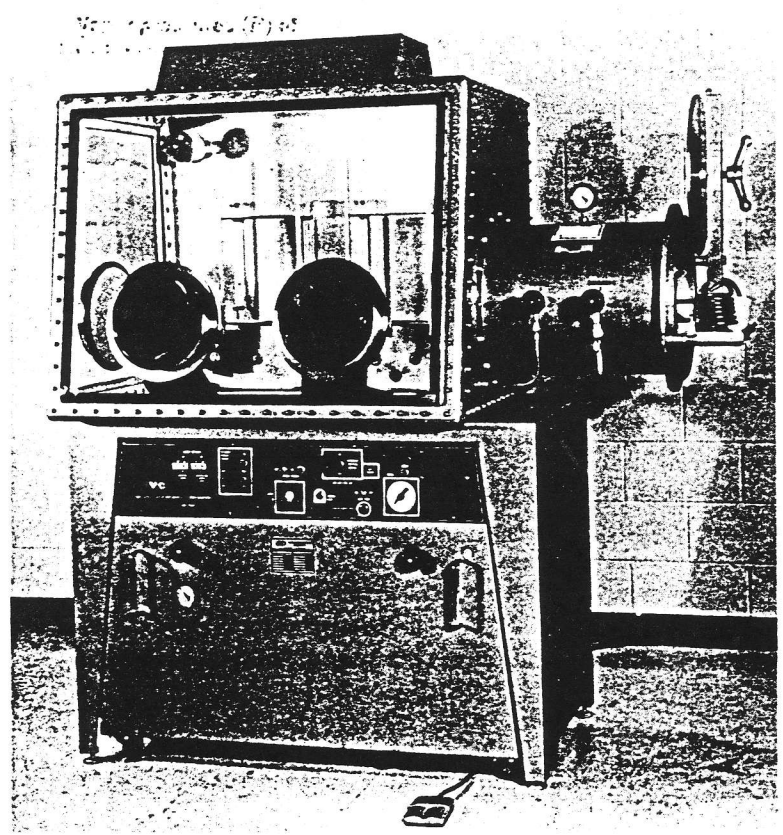


Fig. 8.19. Commercial metal glove box. An aluminum glove box with a recirculating gas-purification system. Note the sliding tray in the ante-chamber for easy transfer of items into the box. (By permission of Vacuum Atmospheres Corp., North Hollywood, Calif., Bulletin B10-64.)

Table 9.4. Miscellaneous dry oxygen scavengers

<i>Agent</i>	<i>Description</i>
Na-K (67–81% K by weight) . . .	Liquid above 0°C and may be used in a U-tube gas bubbler. Removes O ₂ , H ₂ O, and Hg vapor. ^a
Na or K supported on glass wool . .	Similar to above. Supported Na is prepared by embedding the metal chunks in glass wool, evacuating, and heating to 300°C. ^b
CoO	Removes O ₂ at room temperature. Prepared by slowly heating CoCO ₃ to 340°C in vacuo. Not easily regenerated. ^c
Cr ⁺² in silica gel	Prepared by adsorption of a Cr ⁺³ solution on silica gel followed by reduction at 500°C in H ₂ . Efficient, low-capacity O ₂ absorption at room temperature. ^d
Palladized or platinized asbestos (or "Deoxo" unit)	Removes traces of O ₂ from H ₂ at room temperature.
BTS catalyst in reduced form . . .	Similar to above catalysts. 70°C required for removal of O ₂ from an H ₂ stream, 30–40°C required for CO stream.
Ba, Ca, Ca–10% Mg alloy, La, Mg, Th, or Zr	Removal of O ₂ from Ar stream at 300, 650, 475, 500, 600, 400, and 600°C respectively. ^e Also removal of O ₂ and N ₂ at 400, 650, 500, 800, 640, 800, and 1,000°C respectively. ^e
Brass, Cu, Ce, or U	Removal of O ₂ from Ar or N ₂ stream at 500, 600, 300, 200°C respectively. ^e
Li	Similar to Ca. Removes N ₂ and O ₂ but reacts with quartz or glass. ^f

^a E. R. Harrison, *J. Sci. Instr.*, **29**:295 (1952).

^b H. H. Storch, *J. Am. Chem. Soc.*, **56**:374 (1934); E. R. Harrison, *J. Sci. Instr.*, **30**:38 (1953), supported K.

^c H. A. Pagel and E. D. Frank, *J. Am. Chem. Soc.*, **63**:1468 (1941).

^d R. L. Burwell, Jr., private communication.

^e D. S. Gibbs, H. J. Svec, and R. E. Harrington, *Ind. Eng. Chem.*, **48**:289 (1956); note that hot Mg reacts with Vycor or fused silica tubing.

^f P. A. F. White and S. E. Smith, General Reference 9.1, pp. 48, 222.

Table 9.3. Recipes for oxygen-removing solutions

<i>Solution</i>	<i>Preparation</i>
Chromous sulfate (aqueous)	A fresh solution 0.4 <i>M</i> in chrome alum and 0.05 <i>M</i> in sulfuric acid is contacted with lightly amalgamated Zn
Alkaline pyrogallol (aqueous) . . .	15 g pyrogallic acid in 100 ml of 50% aqueous KOH
Sodium hyposulfite (aqueous)	48 g Na ₂ S ₂ O ₄ , 40 g NaOH, and 12 g β-anthraquinone sulfonate in 300 ml H ₂ O
Sodium anthraquinone-β-sulfonate (aqueous)	2% sodium anthraquinone-β-sulfonate in 1.5 <i>M</i> NaOH is contacted with zinc metal
Benzophenone ketyl (oil)	1 g Na dispersed in mineral oil plus 4 g benzophenone in one liter of mineral oil

Table 9.1. Vapor pressures (P) of ice at various temperatures (t)*

$t, ^\circ\text{C}$	P, mm
-90	0.07×10^{-3}
-80	0.40
-70	1.94
-60	8.0 ₈
-50	29.6
-40	96.6
-30	286.
-20	776.
-10	1.95×10^0
0.0	4.58

* After E. W. Washburn, "International Critical Tables," Vol. 3, p. 210.

Table 9.2. Desiccants*

<i>Agent</i>	<i>Equilibrium water vapor pressure, mm</i>	<i>Remarks</i>
CaH ₂	$< 10^{-5}$	Evolves hydrogen; no regeneration; basic
P ₂ O ₅	2×10^{-5}	Capacity limited by formation of a surface film; acidic
Mg(ClO ₄) ₂	5×10^{-4}	Good capacity; regenerate at 250°C in vacuo; dangerous with reducing agents
BaO.....	7×10^{-4}	Small capacity; regeneration is unhandy; basic
Linde Molecular Sieves, 4A or 5A	ca. 1×10^{-3}	Good capacity; regenerate at 400°C in vacuo or in a "dry" gas stream
Alumina (active).....	ca. 1×10^{-3}	Fair capacity; regenerate at 500°C in vacuo or in a "dry" gas stream, or 700°C in air
Silica gel (narrow pore).....	ca. 2×10^{-3}	Fair capacity; regenerate at 300°C
KOH.....	ca. 2×10^{-3}	Small capacity owing to coating of solid with solution; basic
CaO.....	3×10^{-3}	Limited capacity, especially in the presence of CO ₂ ; basic
H ₂ SO ₄ (concentrated).....	ca. 3×10^{-3}	Oxidizing agent; acidic
H ₃ PO ₄ (syrupy).....	ca. 3×10^{-3}	Acidic
CaSO ₄ (Drierite).....	5×10^{-3}	Regenerated at 250°C
CaCl ₂	0.2	Good capacity; slightly acidic

* Adapted in part from R. E. Dodd and P. L. Robinson, "Experimental Inorganic Chemistry," p. 137.

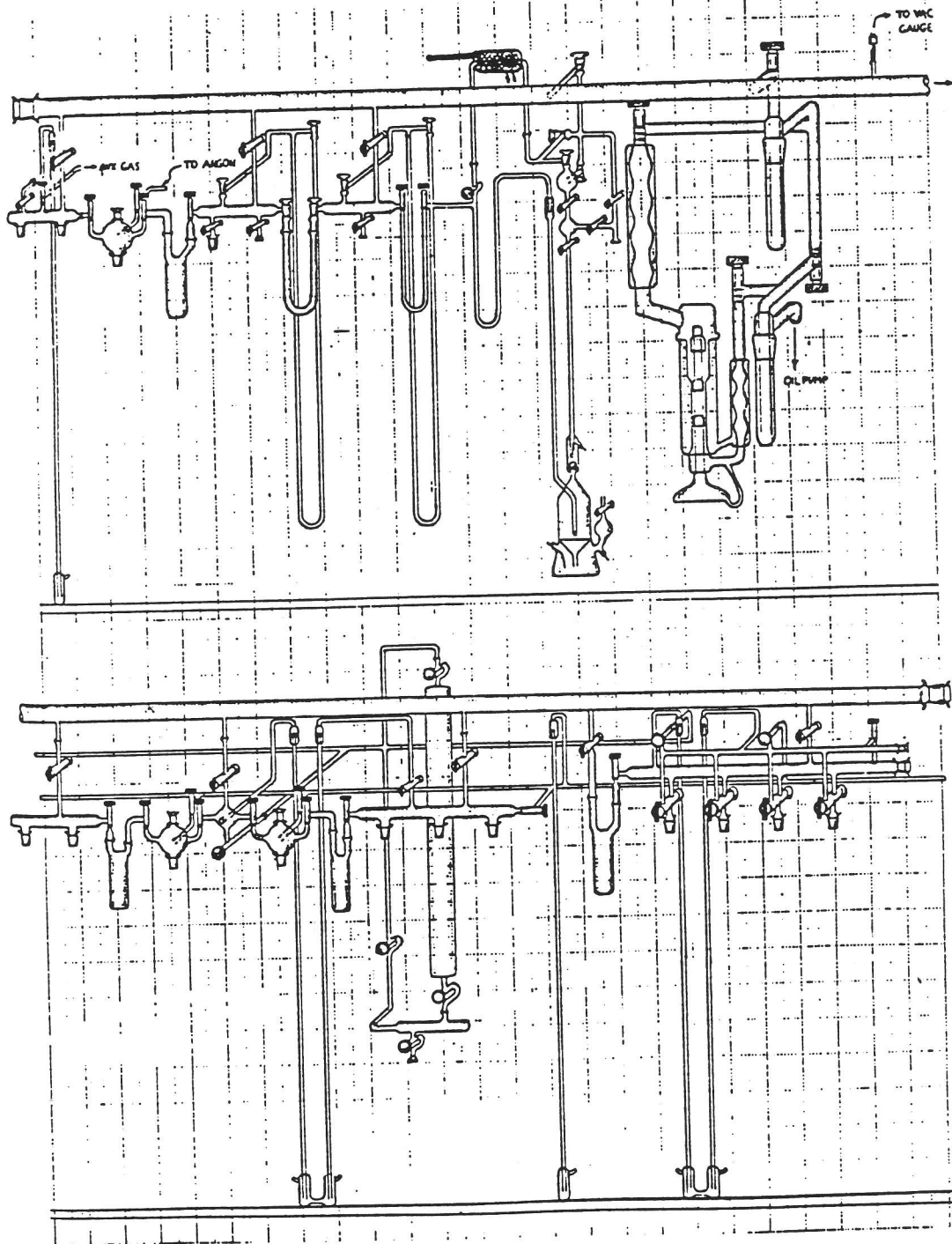


Figure 1. Diagram of a Bercau group vacuum line (courtesy of Dr. Dean M. Roddick).

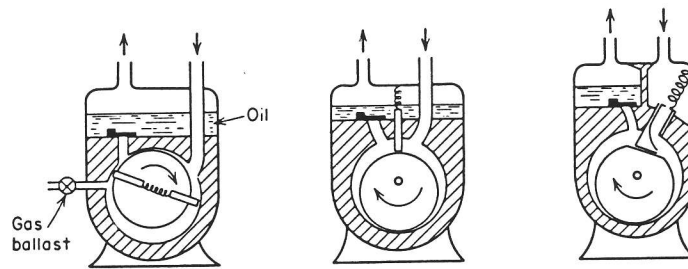


Fig. 2.2. Internal-vane, external-vane, and plunger-type rotary vacuum pumps. It will be noted that the internal-vane pump involves a rotor concentric with the drive shaft, but which is off-center with respect to the stator. By contrast, the external-vane and rotary-plunger pumps have a rotor which is asymmetric with respect to the shaft; however, the shaft is centered in the stator. All three involve close tolerances, so the high-vacuum performance is impaired by particles of dirt or corrosive gases. Some pumps are partially constructed from soft die-cast metal, which is eroded by mercury.

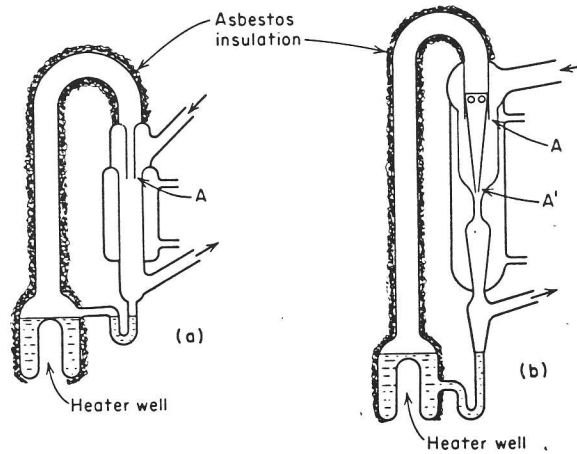


Fig. 2.4. Two simple diffusion pumps. (a) This pump is a single-stage design with a wide annular opening (A) around the jet. Therefore, it will attain a good ultimate vacuum but will not tolerate a high fore pressure. (b) The upper stage in this two-stage pump has a wide annular opening (A); therefore, this pump provides a good ultimate vacuum. The lower stage has a small annular opening (A') so the pump will operate against a high fore pressure.

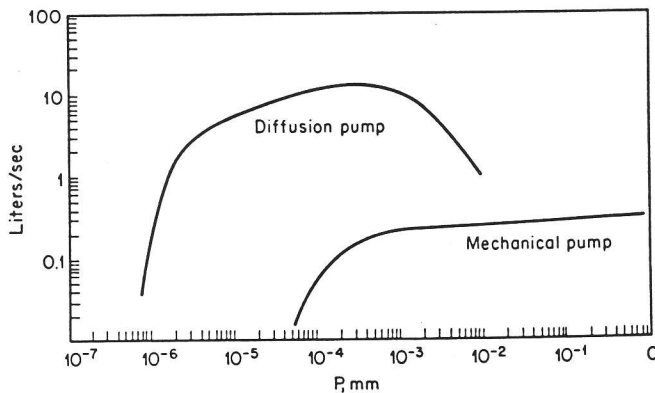


Fig. 2.3. Comparison of the pumping speed of a typical two-stage mechanical pump with a single-stage diffusion pump.

Table 2.1. Comparison of fluids for diffusion pumps

Fluid	Type	Vapor pressure at 25°C (mm)	Resistance to air oxidation
Mercury.....		2×10^{-3}	Best
Apiezon A.....	Hydrocarbon	2×10^{-5}	Poor
Apiezon B.....	Hydrocarbon	4×10^{-7}	Poor
Apiezon C.....	Hydrocarbon	10^{-8}	Poor
Silicone 702.....	Silicone	Better
Silicone 704.....	Silicone	10^{-6} - 10^{-8}	Better
Silicone 705.....	Silicone	10^{-9} - 10^{-10}	Better
Octoil.....	Ester	2×10^{-4}	Fair
Octoil S.....	Ester	2×10^{-5}	Fair

Table 3.3. Vapor pressures of mercury

Temp, °C	Vapor pressure, mm*	Temp, °C	Vapor pressure, mm*
-38.88†	2.191×10^{-6}	180	8.773
-20	2.336×10^{-5}	200	17.27
0	1.996×10^{-4}	220	32.15
20	1.268×10^{-3}	240	56.93
25	1.935×10^{-3}	260	96.40
40	6.340×10^{-3}	280	157.17
60	0.02605	300	247.41
80	0.09095	320	377.32
100	0.2771	340	559.30
120	0.7521	356.58	760.00
140	1.850	360	807.95
160	4.180		

* From -38.88 through 240°C these data are from T. B. Douglas, A. F. Ball, and D. F. Ginnings, *J. Res. Nat. Bur. Std.*, **46**:334 (1951); from 260 through 360°C the data are from F. H. Spedding and J. L. Dye, *J. Phys. Chem.*, **59**:581 (1955).

† Triple point.

Table 4.1. Some common greases for vacuum apparatus

Type and brand	Approximate vapor pressure, mm (room temp.)	Application	Approximate usable range, °C	Resistance to organic solvent vapors	Chemically attacked by
Hydrocarbons:					{ Reactive halides such as BCl_3 , and very strong oxidizing agents such as O_3
Apiezon L.....	10^{-10}	Ground joints	Max 30	Poor	
Apiezon M.....	10^{-7}	Ground joints	Max 30	Poor	
Apiezon N.....	10^{-6}	Stopcocks and joints	Max 30	Poor	
Apiezon T.....	10^{-8}	Stopcocks and joints	Max 110	Poor	
Halocarbon:					Strong reducing agents such as alkali metals, and strong nucleophiles such as alkyl phosphines
Kel-F 90.....	$<10^{-3*}$	Stopcocks and joints	Max ca. 30	Poor	
Silicone:					Tends to cake after long exposure to NH_3 gas Reactive metalloid fluorides like BF_3
Dow Corning HyVac.....	$<10^{-6}$	Stopcocks and joints	ca. -20 to >100	Fair	

* Many samples of this grease contain large quantities of low-molecular-weight volatiles.

Fig. 4.1. Standard taper (♣) and spherical joint (♠). When the joints are lubricated with grease, they must generally be held together. Springs or rubber bands are frequently employed on standard taper joints, while a spring-loaded clamp (illustrated above) or a screw clamp (illustrated in Fig. 4.2) is used with ball joints. The method used for specifying joint sizes in the United States is illustrated, and it is described in detail in National Bureau of Standards, Commercial Standard CS 21-39. British standards are described by Dodd and Robinson, "Experimental Inorganic Chemistry," p. 98, Elsevier Publishing Company, Amsterdam 1954.

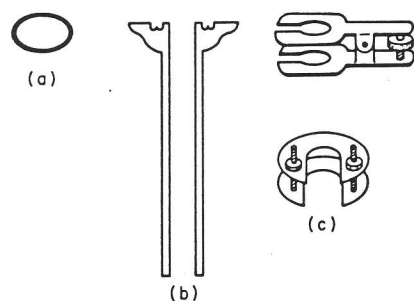
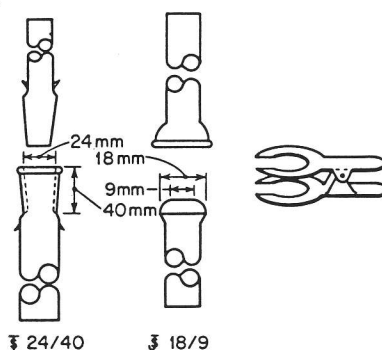


Fig. 4.2. Cross-section of a Urry-type glass O-ring joint and two types of screw clamps. (a) An O-ring. Dimensions and code numbers for standard O-rings are given in Chap. 6. (b) Cross-section of a Urry-type O-joint. Note the ridge which is tooled into the groove. (c) Two types of joint clamps. The upper one is manufactured by A. H. Thomas Co., Philadelphia, Pa.

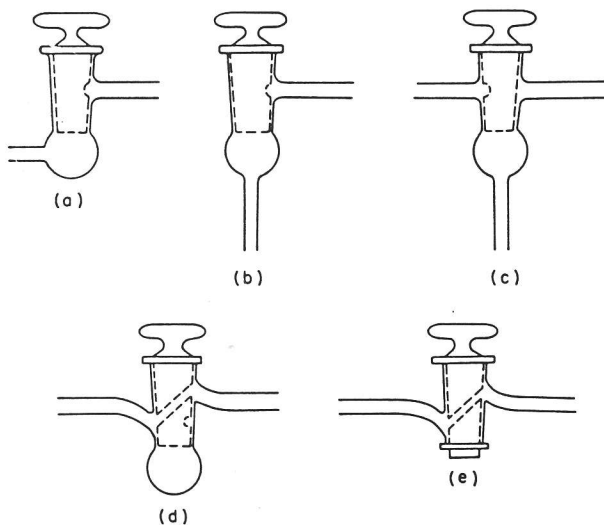


Fig. 4.6. Hollow-plug vacuum stopcocks. The hollow plug is firmly seated in types *a*, *b*, and *c* as long as the lower section is at reduced pressure. Stopcock *d* has a hollow plug with an oblique tube in the center; when the plug is turned by 180°, the lower section may be evacuated. Subsequently, only occasional evacuation of the vacuum cup is necessary. Type *d* does not have provision for evacuation of the plug and is therefore more liable to leak. Prolonged exposure of any of these stopcocks to solvent vapors erodes the grease and introduces leaks. (Adapted from Catalog C-64, Eck and Krebs Co., Long Island City, N.Y.)

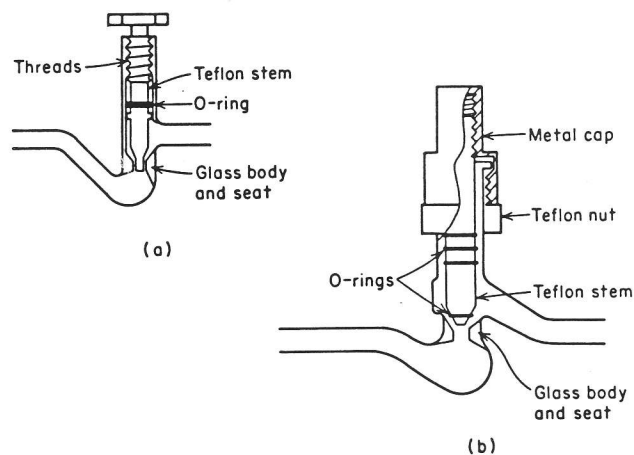


Fig. 4.8. Needle-valve stopcocks. A number of variants of these two basic designs are on the market. In addition to the "straight through" flow pattern shown here, these valves are available in "right-angled" designs. Type *a* uses a threaded Teflon stem working in a threaded glass body. In type *b* the stem does not rotate, so when the cap and Teflon nut are turned, the stem is forced up or down.

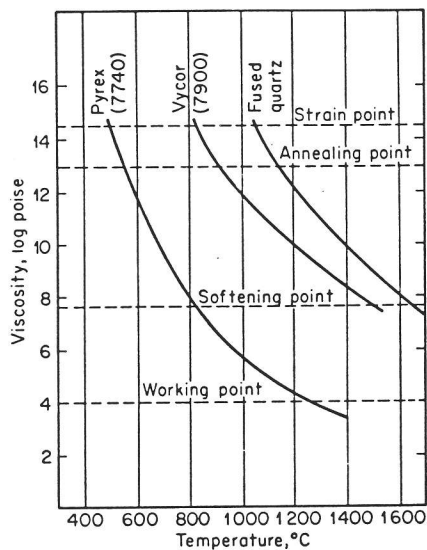


Fig. II.1. Viscosity-temperature curves for some common laboratory glasses. The numbers in parentheses correspond to Corning designations. (Adapted from Corning Glass Works, Corning, N.Y., Bulletin B-83, 1957.)

Table III.1. Permeability Q [in 10^{-10} cm²/sec (cm Hg)] for some common polymeric materials*

Polymer	Temp, °C	H ₂	O ₂	N ₂	CO ₂	H ₂ O
Cellulose and cellulose derivatives:						
Cellophane (cellulose acetate) . . .	25	0.0021	0.0032	0.0047	47-169,000
Cellulose acetate, plasticized . . .	21	4				
	25	11,000-35,000
	30	0.78	0.28	2.38	
Cellulose nitrate	25	1.7	1.95	0.12	2.21	
Ethylcellulose, plasticized	30	26.5	8.4	41.0	
Elastomers:						
Natural rubber, vulcanized	25	48	23	7.9	130	2,300
Butadiene rubber, vulcanized	25	42	19.2	6.5	139	
Buna S, vulcanized	25	40	17	6.2	122	4,400
Buna N, vulcanized	25	16	3.8	1.0	30	6,100
Neoprene, vulcanized (polychloroprene)	25	13	4.0	1.2	25.8	910
Butyl rubber, vulcanized	25	7.2	1.3	3.3	5.1	
Thiokol B, vulcanized	25	1.6	0.3	3.1	65
Silicone rubber	30	91	(270)	460	
Ethylene-propylene rubber	30	(air 11)			
Fluorocarbon polymers:						
Teflon (polytetrafluoroethylene)	20	4.7	
	25	
Teflon FEP (tetrafluoroethylene hexafluoropropene copolymer)	25	4.5	1.9	10	
Kel-F, crystalline (polychlorotrifluoroethylene)	25	0.040	0.005	0.21	<0.3
Polyamides and polyesters:						
Nylon 6 [poly(6-aminocaproic acid)]	20	0.088	
	25	40-4,000
	30	0.038	0.0095	
Mylar [poly(ethylene terephthalate)]	30	0.045	0.011	0.15	
	40	
Polycarbonate poly(4,4'-isopropylidene diphenylene carbonate)	25	1.4	0.3	8.0	
Olefin polymers:						
Polyethylene, low density	~25	(4)	21-66
	30	3.95	1.36	16.7	
Polyethylene, high density	30	0.51	0.18	2.1	
Polypropylene	30	2.3	0.44	9.2	
Polystyrene	25	(15)	920-1,300
	30	1.1	0.29	8.8	
Polyvinyl chloride	25	130-260
	30	0.3	0.11	1.5	
Saran; polyvinylidene chloride Pliofilm, plasticized; rubber hydrochloride	30	5×10^{-3}	9×10^{-4}	0.03	
	25	13-330
	30	0.54	0.14	1.3	
Acrylics:						
Polymethyl methacrylate	25	1,300

* These data are taken from H. Yasuda, "Polymer Handbook," J. Brandrup and E. H. Immergut (eds.), V 13ff, Interscience Publishers, New York, 1966; G. J. van Amerongen, "Elastomers and Plastics," R. Houwink (ed.), vol. 1, pp. 310ff, Elsevier Publishing Company, Amsterdam, 1950; R. P. Bringer, paper presented at Society of Aerospace Materials and Process Engineers, St. Louis, May 7-9, 1962 (fluorocarbon data, in part), Ethylene propylene rubber: "Nordel an Engineering Profile," E. I. du Pont de Nemours and Co., Elastomer Chemical Dept., Wilmington, Del., and Du Pont Bulletin T-3B (Teflon FEP).

Table 1.1 Compounds for cryostatic baths

Temp, °C*	Compound	Temp, °C*	Compound
+6.55	Cyclohexane	-95.0	Toluene
+5.53	Benzene	-96.7	Methylene chloride
0.00	Water†	-111	Trichlorofluoromethane (Freon-11, bp 23.8°C)‡
-8.6	Methyl salicylate	-111.95	Carbon disulfide
-15.2	Benzyl alcohol	-118.9	Ethyl bromide
-22.95	Carbon tetrachloride	-126.59	Methylcyclohexane
-30.82	Bromobenzene	-135	Dichlorofluoromethane (Genetron-21, bp 8.9°C)‡
-37.4	Anisole	-138.3	Ethyl chloride
-45.2	Chlorobenzene		
-51.5	Ethyl malonate	-139	CHCl ₃ , 19.7% by weight C ₂ H ₅ Br, 44.9% trans C ₂ H ₂ Cl ₂ , 13.8% C ₂ HCl ₃ , 21.6%
-57.5	Chloral		
-63.5	Chloroform§		
-83.6	Ethyl acetate	-160.0	isoPentane

* Most of these values are taken from J. Timmermans, "Physico-chemical Constants of Pure Organic Compounds," vols. 1 and 2, Elsevier Publishing Company, Amsterdam, 1950 and 1965.

† Pure crushed ice in contact with distilled water.

‡ These fluorocarbons have low toxicity and are nonflammable. The liquid may be stored for reuse in a stoppered Dewar in a freezer.

§ Reagent-grade chloroform contains a significant quantity of alcohol, which lowers the melting point.

Table 5.2 References to infrared and visible-ultraviolet cells for air-sensitive compounds

Description	Reference
Vacuum-tight infrared cell for liquids.	A. B. Burg and R. Kratzer, <i>Inorg. Chem.</i> , 1 :725 (1962).
Infrared cell for carbon suboxide polymer.	R. N. Smith, D. A. Young, E. N. Smith, and C. C. Carter, <i>Inorg. Chem.</i> , 2 :829 (1963).
Pressure-tight cell for infrared spectra of liquids.	D. C. Smith and E. C. Miller, <i>J. Opt. Soc. Am.</i> , 34 :130 (1944).
Method of attaching dissimilar window materials to a vacuum-tight low-temperature cell.	E. Schwarz, <i>J. Sci. Instr.</i> , 32 :445 (1955). V. Roberts, <i>J. Sci. Instr.</i> , 31 :251 (1954).
Infrared spectra of adsorbed species.	R. P. Eischens, S. A. Francis, and W. A. Pliskin, <i>J. Phys. Chem.</i> , 60 :194 (1956). M. Courtois and S. J. Teichner, <i>J. Catalysis</i> , 1 :121 (1965).
All-glass cell for the infrared <(4.7 μ) spectra of gases.	F. A. Cotton and L. T. Reynolds, <i>J. Am. Chem. Soc.</i> , 80 :269 (1958).
Low-temperature near-infrared and visible cells for liquid ammonia solutions.	M. Gold and W. L. Jolly, <i>Inorg. Chem.</i> , 1 :818 (1962). E. C. Fohn, R. E. Cuthrell, and J. J. Lagowski, <i>Inorg. Chem.</i> , 4 :1002 (1965).
Low-temperature vacuum-tight visible-ultraviolet cell for solutions.	R. Nakane, T. Watanabe, O. Kurihara, and T. Oyama, <i>Bull. Chem. Soc. Japan</i> , 36 :1376 (1963).
Low- and high-temperature gastight visible-ultraviolet cell.	Y. Hirshberg and E. Fischer, <i>Rev. Sci. Instr.</i> , 30 :197 (1959).
Heated infrared cell for solids in a controlled atmosphere.	T. Wydeven and M. Leban, <i>Anal. Chem.</i> , 39 :1673 (1967).
Low-temperature liquid infrared cell.	R. G. Steinhardt, P. A. Staats, and H. W. Morgan, <i>Rev. Sci. Instr.</i> , 38 :975 (1967).
Silver chloride window-to-body seals.	J. F. Harrod and H. A. Poran, <i>Rev. Sci. Instr.</i> , 38 :1105 (1967). A. Guest and C. J. L. Lock, <i>Rev. Sci. Instr.</i> , 39 :780 (1968).
Optical materials and various infrared cell designs.	R. G. J. Miller (ed.), "Laboratory Methods in Infrared Spectroscopy," Heyden and Sons Ltd., London, 1965.
Protective holder for KBr pressed disks.	P. A. Saats and H. W. Morgan, <i>Appl. Spectry.</i> , 22 :576 (1968).