

INSTRUCTIONS
FOR
HF-REACTION APPARATUS
TYPE I

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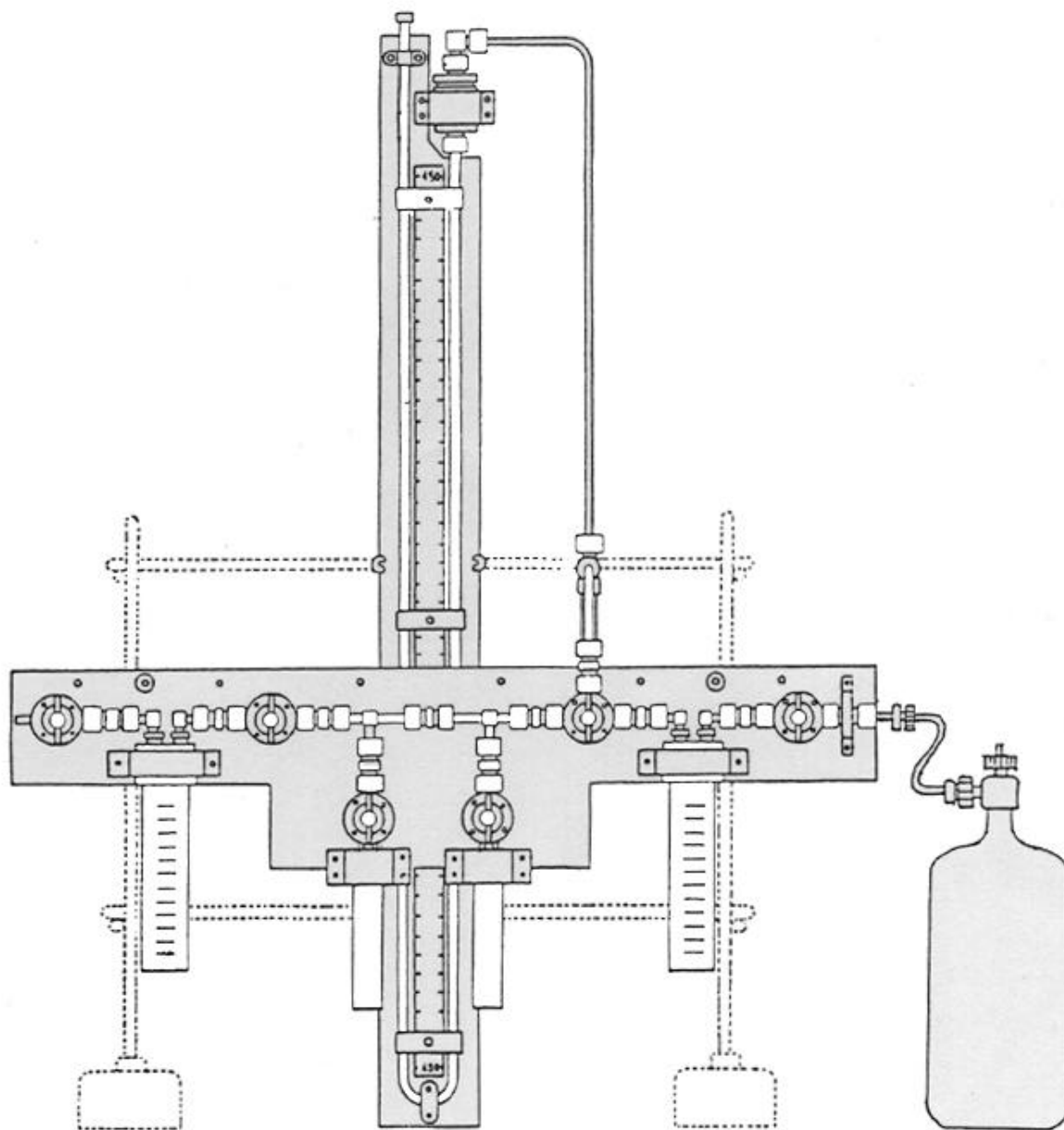


Figure 1 HF- Reaction Apparatus Type I

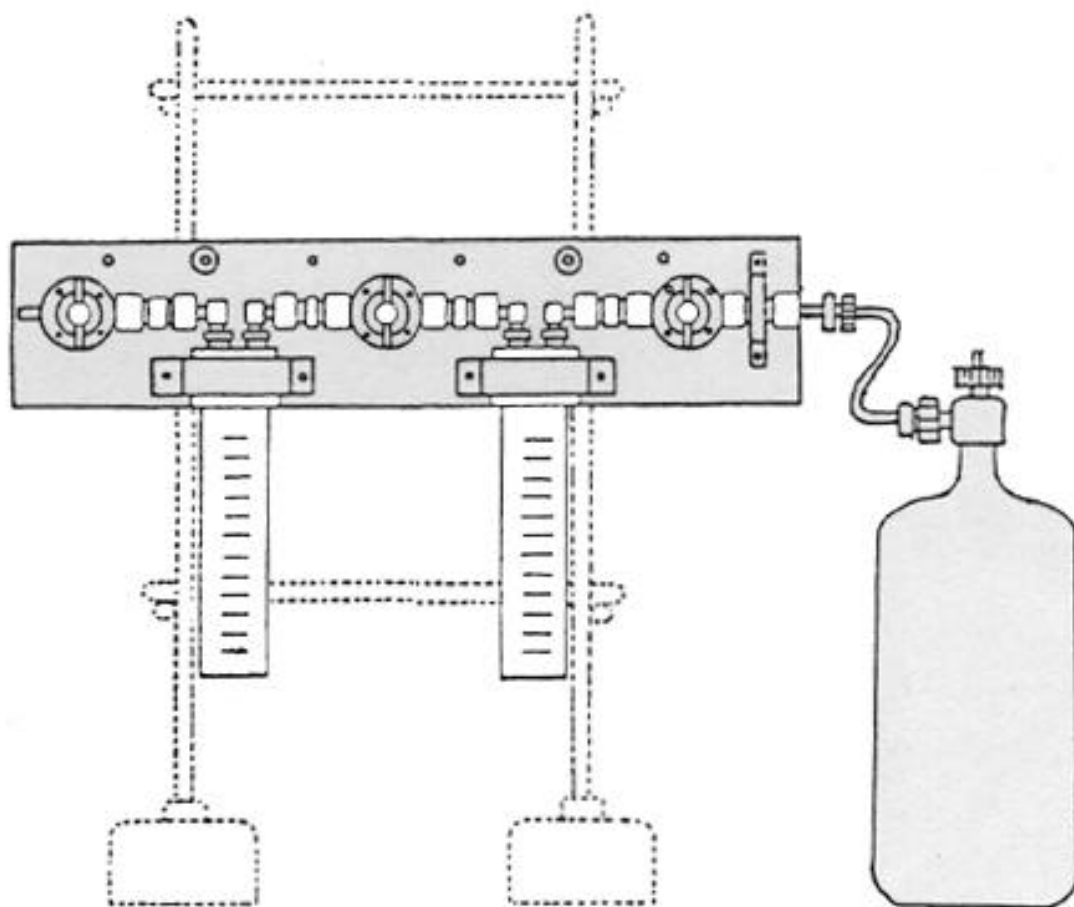


Figure 2 HF-Reaction Apparatus Type II

ASSEMBLY AND USE OF THE HF-REACTION APPARATUS*

This apparatus is made primarily of Daiflon (Poly-trifluoromonoethylen), Which is resistant to almost all chemicals. The apparatus is designed to withstand 3 Atmospheres of pressure. Liquid HF can Be handled safely at temperatures below 50 °C. (See Fig. 3)

The HF apparatus must be used in an Efficient fume hood. A supporting stand such as shown in Fig. 4 can be used. It is preferable to mount the apparatus solidly against the rear wall of the hood.

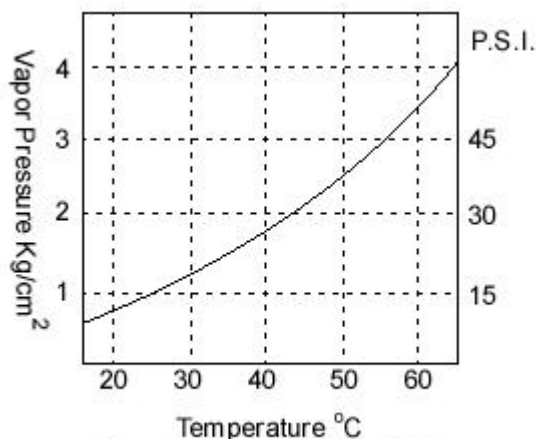


Fig. 3 Vapor Pressure of HF

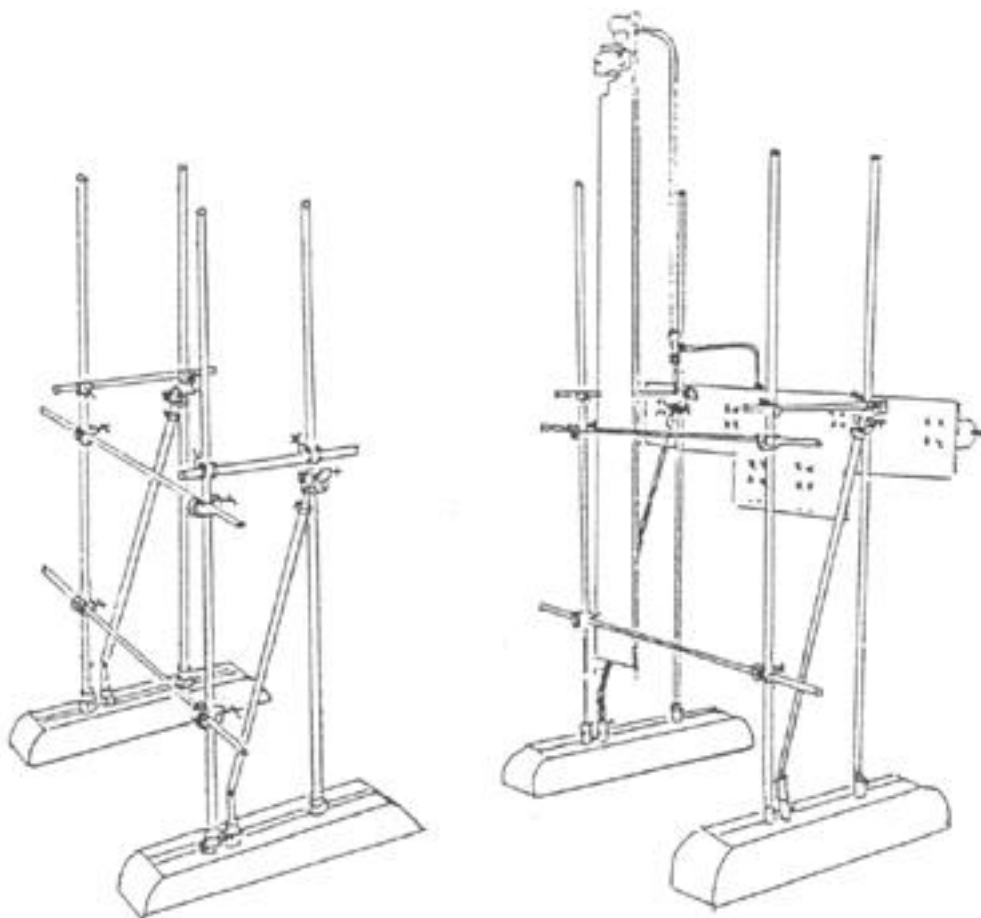


Figure 4 Supporting Stand for HF Reaction Apparatus (Not included)

* See parts catalog for listing showing names and codes for individual parts.

To set up your HF line, the following procedure is recommended:

1. The Valve-Panel and the Manometer-Panel are fixed to the stand using Holders (603) as shown in Figs. 4 and 5.

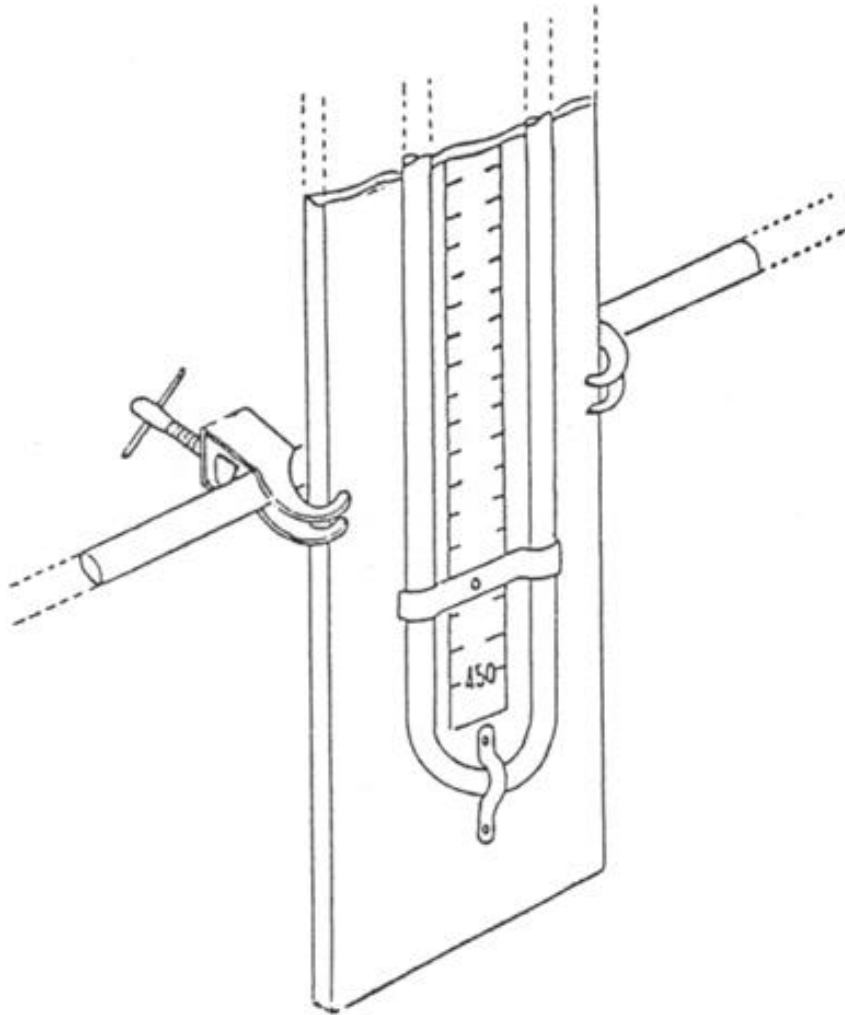


Figure 5 Manometer Mounting

2. Fix an L-Type Tube Connector (304) on the top of the manometer and join a longer L-Shaped Tube (701), Mercury Trap (305 + 704) and a shorter L-Shaped Tube (702) as shown in Figure 6 on page 5. Finally, the Manometer and the vacuum line are connected, using an I-Type Connector (301). Figure 7 on Page 5 illustrates this procedure.

Any Tubes with O.D. 10 mm can be connected readily by inserting one end of the tube into the connector and tightening the cap by hand. It is not necessary to apply much force to obtain a vacuum-tight seal as long as the O-rings are in good condition.

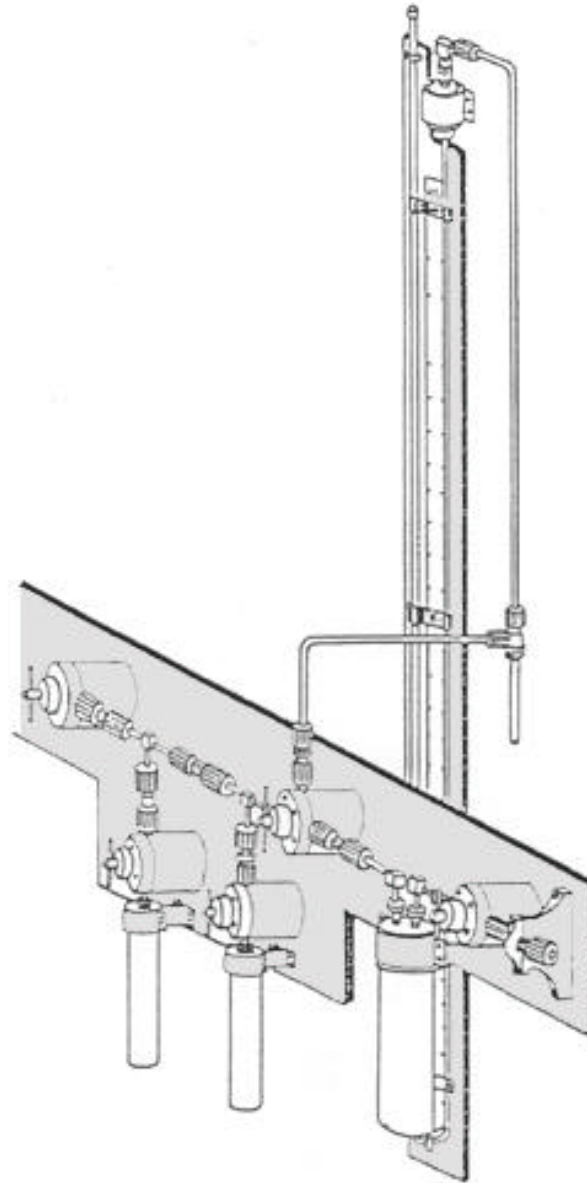


Figure 6 Tube Connection between Valve-Panel and Manometer-Panel

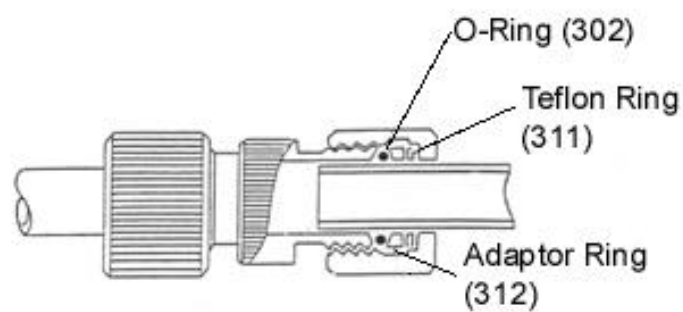


Figure 7 I-Type Connector (301) using Perflon O-Rings

3. The HF cylinder and the HF apparatus are connected with a flexible connecting tube, which is assembled with 908, 909, 910, and 913. (See page 9 for assembly of the flexible connecting tube). The Monel Flanged Adaptor (910) is fixed to the HF cylinder using the Flare Nut (913) through gasket (911 or 912). The Connector Adaptor (908) is inserted into the right terminal I-Type Connector (303), which is tightened by a Teflon core ring s shown in Figure 8.

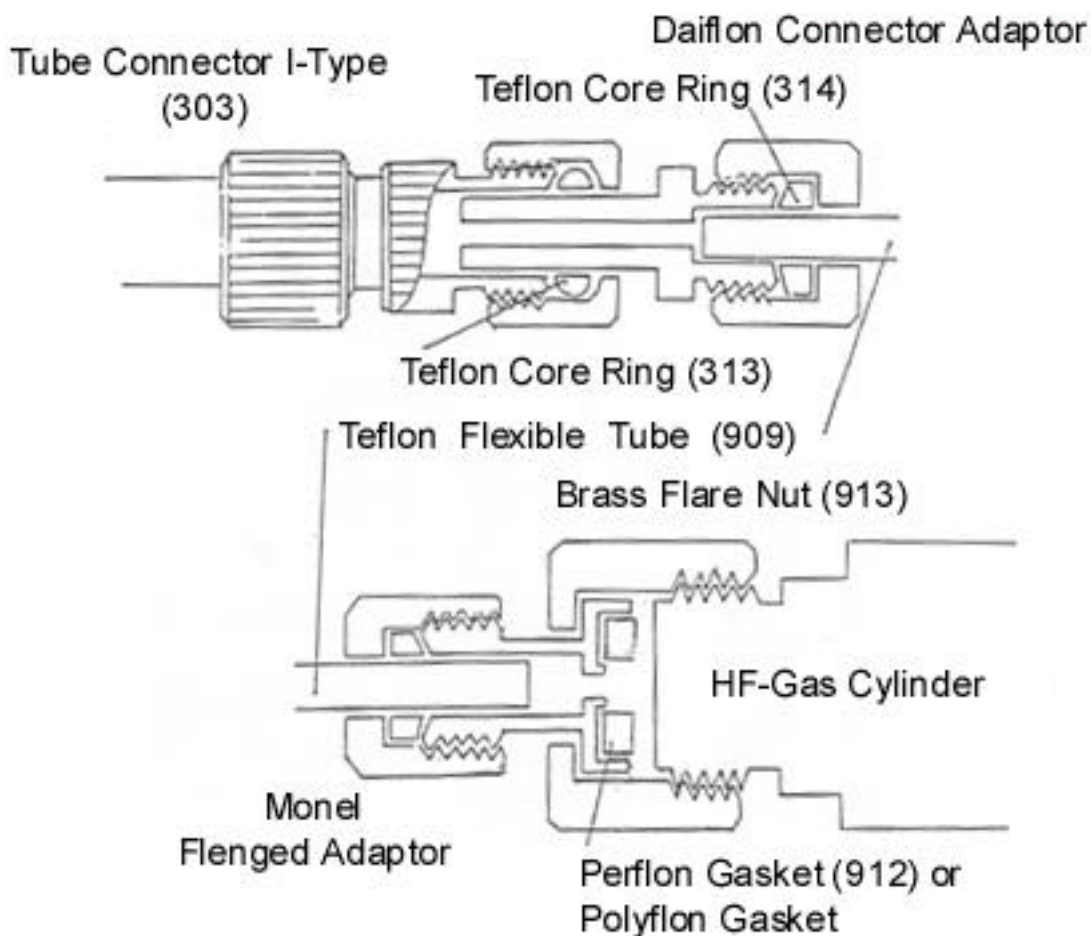


Figure 8 Section of Flexible Connector

4. Remove the Mercury Stopper (602) from the top of the manometer, pour about 34 mL of dry mercury into the U-Tube until the meniscus rises to the zero line. Add about 1 mL of Dialfloil (604) to the mercury and reposition the Mercury Stopper. (Dialfloil retards reaction of the mercury with the HF).

5. Insert the vessels (205, 405) into their sockets, using the proper O-rings and making sure that each O-ring is positioned properly in each socket. Don't apply much force when installing each vessel. A vacuum-tight seal is obtained readily if the O-ring is in good condition.
6. Test the vacuum line as follows: Connect a vacuum pump to the valve at the far left of the line and open all the valves except that of the HF cylinder. Evacuate the vacuum line using the pump, then close the left-hand valve. The mercury meniscus should rise toward the top of the right side of the manometer. If the mercury level falls rapidly, it is most likely that an O-ring is improperly positioned. If it is possible to locate the leak by watching the mercury level while opening and closing valves one by one. A stable mercury level over a period of several hours indicates that the apparatus is properly adjusted.
7. If a mechanical pump is used for the evacuation of the HF line, we recommend the use of an HF-Absorption Cylinder (013). If an HF-Absorption Cylinder is not available, the last vessel must be cooled by liquid nitrogen to trap HF in the exhaust. If evacuation is done by a water pump, a pump of stainless steel or polypropylene is preferable. With the water pump method, dilute HF is run into the waste water system. A change in water flow could permit counterflow of water into the line. This must be carefully avoided.
8. After testing the vacuum line to assure that there is no leakage; the reservoir vessel is chilled to -70°C or lower using liquid nitrogen or methanol-dry ice bath, keeping the line pressure below 20 mm Hg. Slowly open the valve of the HF cylinder⁶; HF will slowly accumulate in the reservoir vessel. During distillation, the vacuum pump and mercury manometer must be isolated from the system by closing the 3-way Valve (103) to the manometer. Generally, contact of HF and mercury must be kept to a minimum in order to keep the mercury clean for a long period of time.

If the line is functioning properly, 50 ml of HF will accumulate in the reservoir vessel within 30 minutes at ordinary room temperature. If the distillation speed is slower than 50 ml/30 min., close the HF cylinder valve, evacuate the vacuum line for a minute by opening the valve (103), again close the valve (103) and continue the distillation. Sometimes, air remaining in the HF cylinder interferes with the vacuum distillation, and such an intermittent evacuation is effective in speeding up the distillation. Don't open the valve for an unnecessarily long time, since a considerable amount of HF is wasted during the process. CoF_3 can be used in the reservoir vessels to remove possible traces of water in the HF. We have found that this is not usually necessary. However, if used, it must be well stirred to prevent caking and rupture of the reservoir by expansion.

The accumulated HF can be redistilled into a reaction vessel in the same manner; i.e., a vessel of water at 50°C or less is raised so as to immerse the reservoir vessel. The reaction vessel is cooled with liquid nitrogen or methanol-dry ice. Distillation of used HF into the last vessel should be done in the same manner. The accumulated HF in the last vessel is discarded slowly through a water pump or by converting it to CaF_2 in the HF Absorption Cylinder (013). Never disconnect a vessel when it contains liquid HF. An accident with liquid HF can be disastrous.

The last vessel may be used for larger scale HF reaction.

9. To wash the line, any part can be removed without interference with other parts. Colored materials which cannot be removed by the ordinary washing process may be removed by putting those parts in concentrated HNO_3 or in aqua regia. After rinsing with water, parts must be dried at 50°C or lower. Higher temperatures may cause deformation. We

recommend replacement of the O-rings during re-assembly, since they tend to deform in time and cause leaks.

10. Be very cautious in handling HF. HF is a corrosive and extremely toxic material. Always use rubber gloves and protective glasses when handling HF. For your safety, we strongly urge that you study thoroughly the references in the following list before commencing work with HF.

References

1. J.H. Simons, in "Fluorine Chemistry" Vol.1, (J.H. Simons, ed.), pp.225-292, Academic, New York, (1950); H.C.Hodge and F.A. Smith, Vol.4, (1965).
2. M. Hudlicky, in "Chemistry of Organic Fluorine Compounds" Pergamon, Oxford, (1961).
3. H.H. Hyman and J.J. Katz, in "Non-Aqueous Solvent Systems" (T.C. Waddington ed.), pp. 47-81, Academic, London, (1965).
4. M. Kilpatrick and J.G. Jones, in "The Chemistry of Non-Aqueous Solvent Systems, Vol. 2, (J.J. Lagowski, ed.),pp. 43-99, Academic, New York, (1967).
5. S. Sakakibara, in "Chemistry and Biochemistry of Amino Acids, Peptides and Proteins" Vol.1, (B. Weinstein, ed.) pp.51-85 Marcel Dekker, New York.
6. If the HF cylinder has not been previously used, it is often wise to open it slightly and release a little HF into an operating and efficient hood, taking extreme care that no HF gas contacts eyes or skin. Some HF cylinders when purchased contain enough permanent gases to blow mercury from the manometer into the hood.

FLEXIBLE CONNECTING TUBE ASSEMBLY

The flexible connecting Tube Assembly consists of the Daiflon Connector Adapter (908), Teflon Flexible Tube (909), and Monel Flanged Adapter (910). Cross-sections of the adapters are shown in Fig.8. To fasten the Teflon tube to each adapter, follow the procedure below.

1. Insert the end of the Teflon Tube (909) into either Adapter (908 or 910).
2. Tighten the Flare Nut quite firmly by hand.
3. Tighten the Flare Nut an additional $\frac{1}{4}$ to $\frac{1}{2}$ turn with a wrench. Don't turn the nut more than $\frac{1}{2}$ turn or the Daiflon Flare Nut may break.
4. Test the connection by pulling on the Flexible Tube by hand. If the tube does not come out easily, a good seal has probably been made. If the tube does come out, the end portion must be removed because it is likely that the Teflon tubing has been drawn to a smaller diameter by passage through the hole smaller than its external diameter. The result of such drawing is shown in Fig.10. Once the external diameter has been reduced, the tube cannot be properly secured by the Teflon Core Ring (314), even by the application of much stronger force. Therefore, if the tube does slip out of the connector upon pulling, the slimmed part of the Teflon tube must be removed with a fine-toothed saw or a single-edged razor blade. The slightly shortened Teflon tube can then be reassembled by the above procedure, applying a little more force for sealing.
5. When the proper force is used, the Core Ring deforms and holds the Teflon tube firmly, as shown in Fig.11.

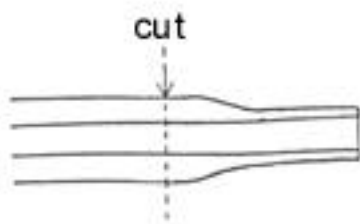


Figure 10

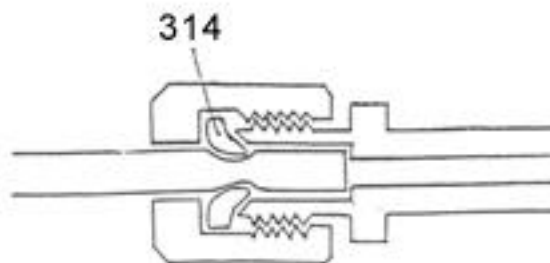
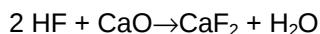


Figure 11

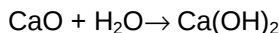
The flexible Connector assembly will then withstand 3 atmospheres of pressure. If the assembly is not properly made, the flexible tube may slip out upon opening the valve of the HF cylinder. This could be very dangerous!

USE OF THE HF-ABSORPTION CYLINDER (013)

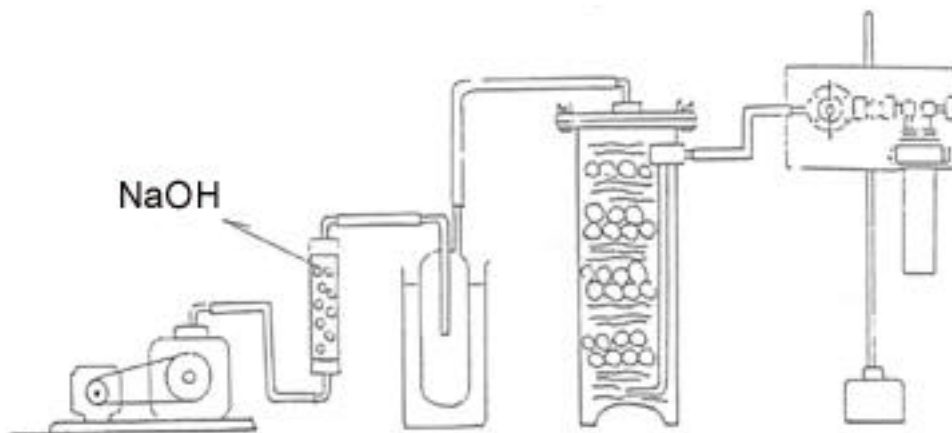
This cylinder is used for the absorption of HF gas in the exhaust of the HF Reaction Apparatus using CaO as an absorbent. HF is converted to CaF_2 , which is insoluble in water.



According to this equation, one mole of water is formed for each 2 moles of HF absorbed. Some of this water is trapped according to the following equation:



Any water escaping absorption can be trapped by an ordinary glass vacuum trap chilled in a dry ice-methanol bath. A typical arrangement of traps between the HF line and the pump is shown below.



Ordinary heavy-wall plastic tubing (polyethylene, polypropylene, or polyvinylchloride) may be used for connecting these traps. A water pump attached directly to the Absorption Cylinder can be used for the evacuation. A water trap should be inserted between the water pump and the Absorption Cylinder to prevent water backing up into the cylinder.

1. To charge the cylinder, first fill about one-fifth of the cylinder with Teflon shavings. Then add alternate layers of CaO and Teflon shavings, using a total of 4 kg of CaO about 1 cm in diameter. Load the cylinder until the top of the packing comes about 5 cm below the top of the cylinder. Wipe the O-ring area clean, apply the O-ring, and place the cover on the cylinder. Fasten the top with four screws. Do not apply much force on the screws; air pressure will insure a good seal during evacuation. If necessary apply a small amount of Fluorogrease for stop leaking. Fluorogrease is available from Peptides International, Inc.

Catalog #	Description	Price
HFI-0424	Fluorogrease	\$50.00

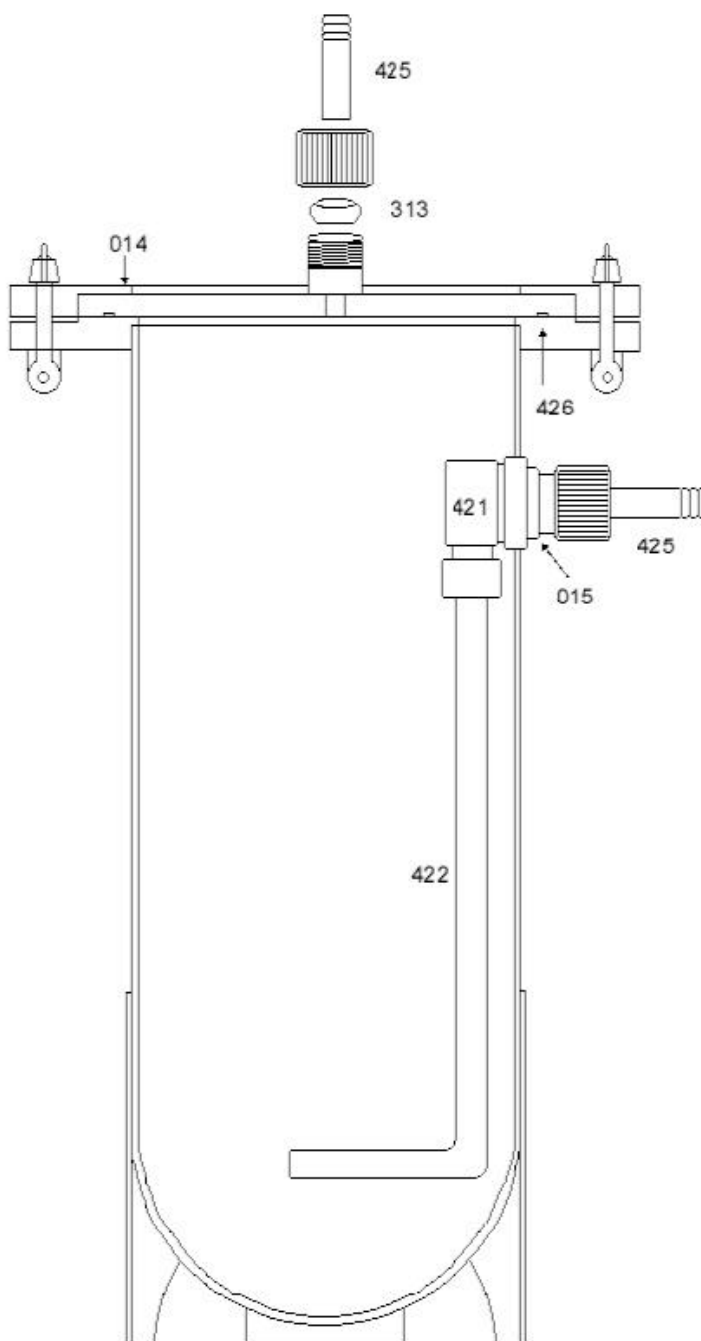
2. During the absorption of HF gas in the cylinder, heat of neutralization is released. Generally, it is not necessary to cool the cylinder if the HF absorbed is less than 50 ml at one time, since the heat capacity of this cylinder is substantial. When more than 50 ml of HF is absorbed in a short-time, the Absorption Cylinder must be chilled in a bucket of ice

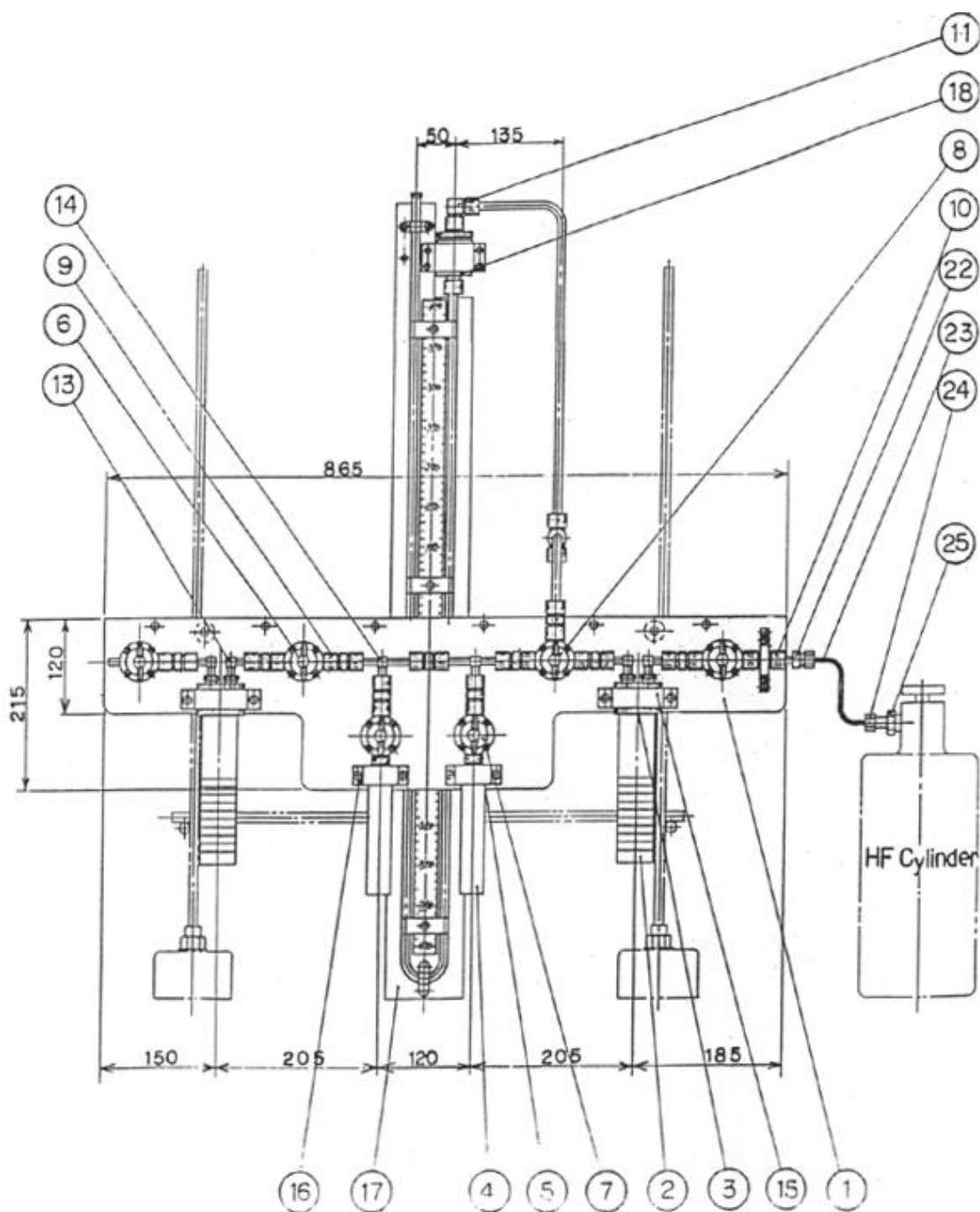
water or in cold running water. Under these conditions, up to 1000 ml of HF can be absorbed with 4 kg of CaO, but the distillation rate must be less than 200 ml per hour.

4 kg of CaO is sufficient to neutralize 1000 ml of HF. Record the amount of HF absorbed each time. When the total reaches 1000 ml, the absorbent must be replaced. Slowly mix

3. The used CaO with 30 liners or more of water in a polyethylene bucket. The mixture must be allowed to stand overnight before dumping.
4. For safety, gas evacuated from the mechanical pump must not be released into the laboratory but should be discharged into a functioning hood.
5. Teflon shavings are available from Peptides International, Inc.

Catalog #	Description	Price
HFI-0423	Teflon Shavings for 013	\$19.00





No	Description	Code
1	Panel (PVC)	
2	Reservoir Vessel	205
3	Cylinder Socket	201
4	Reaction Vessel	405
5	Cylinder Socket	409
6	2-Way Valve	101
7	2-Way Valve	101
8	3-Way Valve	103
9	Tube Connector I Type	301
10	Tube Connector I Type	303
11	Tube Connector L Type	304
12	Tube Connector T Type	305
13	Connecting Tube L-Shape	501
14	Connecting Tube T-Shape	502
15	Socket Holder for 201 (Metal)	803
16	Socket Holder for 409 (Metal)	804
17	Manometer U-Tube and PVC Panel	601
18	Mercury Stopper with Metal Holder	602
19	L-Shaped Tube	701
20	L-Shaped Tube	702
21	One End-Sealed Tube	704
22	Connector Adapter	908
23	Flexible Tube (Teflon Heavy Wall)	909
24	Flanged Adapter (Monel)	910
25	Flare Nut (Brass)	913

