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Comite Technique Europeen Du Fluor

Working Group Storage, Transport and Safety

HF Handling in Laboratories

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PREFACE

Hydrogen Fluoride (HF) is essential in the chemical industry and there is a need for HF to be produced, transported, stored and used.

The HF industry has a very good safety record ; nevertheless, the European HF producers, acting within CTEF have drawn up this document to promote continuous improvement in the standards of safety associated with HF handling.

This Recommendation is based on the various measures taken by member companies of the CTEF.

It in no way is intended as a substitute for the various national or international regulations, which should be respected in an integral manner.

It results from the understanding and many years experience of the HF producers in their respective countries at the date of issue of this particular document.

Established in good faith, this recommendation should not be used as a standard or a comprehensive specification, but rather as a guide which should, in each particular case, be adapted and utilised in consultation with an HF manufacturer, supplier or user, or other experts in the field.

It has been assumed in the preparation of this publication that the user will ensure that the contents are relevant to the application selected and are correctly applied by appropriately qualified and experienced people for whose guidance it has been prepared.

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The contents of this recommendation are based on the most authoritative information available at the time of writing and on good engineering practice, but it is essential to take account of appropriate subsequent technical developments or legislative changes. It is the intent of the CTEF that this guideline be periodically reviewed and updated to reflect developments in industry practices and evolution of technology. Users of this guideline are urged to use the most recent edition of it, and to consult with an HF manufacturer before implementing it in detail.

This edition of the document has been drawn up by a Working Group "Storage, Transport and Safety" to whom all suggestions concerning possible revision should be addressed through the offices of CTEF. It may not be reproduced in whole or in part without the authorisation of CTEF or of members companies.

1. INTRODUCTION

This guideline has been developed by the Storage, Transport and Safety Group of the Comite Technique Europeen Du Fluor (CTEF). It is intended to cover the safe practices necessary when using HF within laboratory environments. It does not replace or assume any local, national or federal regulations which may govern the specification or use of personal protective equipment.

The guideline has been developed by the European manufacturers association and is based upon many years of practical experience.

It is not intended to be a guideline to which all manufacturers and consumers of HF must adhere nor does it attempt to describe all possible safety considerations in the protection of personnel. This guideline should not be used in isolation to determine suitable protective equipment rather it should be used in conjunction with the supplier(s) of HF and form the basis of a discussion aimed at determining an appropriate level of equipment for given operator duties or activities.

CTEF or its members cannot accept any legal liability or responsibility for the use or misuse of the information contained in this document.

2. OTHER RELEVANT STS DOCUMENTATION

The following STS documents are also relevant when considering the risk assessment necessary when handling HF in laboratories :-

2.1 STS 79/30 - Chemical Safety Data Sheet for Anhydrous Hydrogen Fluoride

2.2 STS 81/37 - Chemical Safety Data Sheet for Aqueous Hydrogen Fluoride

2.3 STS 88/62 - Physical properties of anhydrous HF and hydrofluoric acid solutions

2.4 STS 84/47 - Materials for use with hydrofluoric acid

2.5 STS 94/96 - Advice for Hydrofluoric Acid intoxication

2.6 STS 98/111 - Personal Protective Equipment for use with HF

2.7 STS 99/113 - Training Recommendations

3. SAFE SYSTEMS OF WORK

3.1 Planning

The handling of HF is in itself a hazardous operation and should be completed only in dedicated locations within laboratories which should be clearly labelled as HF Handling areas. This document is intended only for laboratories handling HF and where referred to a 'laboratory' is an HF handling laboratory.

3.2 Alternatives to HF

Before an experiment with HF is started thought should be given to the possibility of using an alternative fluorinating agents. However, it should be remembered that these substitutes are not free of their own hazards and can produce free HF during the course of a reaction.

3.3 Unforeseen HF Formation

It is important to consider the reaction products and the possibility of formation of HF during the reaction, when fluorine-containing compounds are in use e.g. by hydrogenation of halocarbons, reaction of fluorides with acid or thermal breakdown of compounds, the same hazards as a reaction with HF are present and therefore the same precautions should be observed.

3.4 Scale of Experiments

For safety reasons experiments should be performed on as small a scale as possible. Be aware, however, that for continuous experiments, equipment such as pumps and rotameters have lower limits on rates of flow.

4. RISK ASSESSMENT

Each laboratory handling HF should prepare a full Risk Assessment prior to initiating each new experiment or task. The Risk Assessment process should be completed by the laboratory team and should include an independant representative to provide an alternative viewpoint. The Risk Assessment may include the following details :

- Clear identification of the task to be done and the individuals involved.
- Clear identification of the equipment required.
- A review of the competence and understanding of the individuals involved.
- An evaluation of the risks associated with each step of the task.
- Clear identification of the possibility of exposure to all hazards.
- Elimination or mitigation of as many hazards as possible.

- A review of the appropriate level of personal protective equipment required for each step of the task and at what point the personal protective equipment should be worn.
- Clear identification of any health implications associated with the task, the individuals involved in the task and the personal protective equipment to be used.
- Clear identification of the expected duration of the task and any hygiene implications.
- Clear identification of the decontamination procedures required.
- Clear identification of any back up emergency plan, if necessary.

One stipulation of the Risk Assessment shall be that all individuals involved will have undergone specific training in the handling of HF in a laboratory.

5. CALCIUM GLUCONATE GEL

Tubes of calcium gluconate gel to treat HF burns shall be located by the fume cupboard or cubicle where the experiment is being carried out.

There is an expiry date on each tube, the tube should be discarded and a new one obtained before this date. A procedure to ensure that tubes are replaced prior to their expiry date should be in place.

It is suggested that anyone using HF, or who could possibly come in to accidental contact with it (e.g. people working in the same laboratory or regular visitors), shall keep a fresh tube of gel at home at all times. This is for application in the event of any observed delayed effects.

This tube should also have the out of hours medical emergency telephone number denoted on the box. It is suggested that a member of the laboratory team should be designated to order new supplies and re-issuing the gel kept at home.

Note : It is important to begin treatment with gel as soon as possible if HF contamination is suspected. This tube of gel retained in the home is intended for individuals to begin treatment at the earliest opportunity. However, it is important to report any incident, whether actual HF contamination or not, and to receive follow up advice from an occupational health specialist and a fresh tube of gel should be supplied.

Gel is available from a number of suppliers. Refer to STS 94/96 - Advice for Hydrofluoric Acid Intoxication for more information.

6. SIGNS AND LABELLING

It is important that others should be aware of people working with HF in the immediate vicinity. To this end the following signs should be exhibited where HF is in use.

POSITION	SIGN
On the laboratory Door	"HF IN USE"
	"CORROSIVE"
	"TOXIC"
On the office door	List of out of hours telephone numbers of HF users in the lab
Strategic position in the lab	"HF IN USE"
	"CORROSIVE"
	"TOXIC"
	Telephone Number of Emergency Services
Individual fume cupboards (where appropriate)	"HF IN USE"
HF pipework	Should be clearly labelled and colour-coded.

7. PERSONAL PROTECTIVE EQUIPMENT

The correct personal protective equipment should be available within the laboratory. The degree of personal protective equipment worn at any time is dependant upon the nature of the task being performed. For further information refer to STS 98/111 - *Personal Protective Equipment for Use with HF*

Note : In all cases, under normal conditions, personal protective equipment should NOT be relied upon as the primary or only defence mechanism. The primary personal protection should always be the design standards which must eliminate or minimise the risk of exposure to HF wherever possible.

It is highly recommended that an emergency locker containing a full set of the equipment required in case an emergency is situated away from the laboratory handling HF. This will allow easy access to the correct equipment (personal protective equipment, tools, etc.) needed to mitigate an emergency.

Gloves should be tested daily for pin holes by inflating with air, immersing in water and observing any bubbles produced. A glove test chart should be displayed.

Note : It is important to note that "normal" laboratory type gloves may be completely unsuitable for any work with HF and that ALL gloves in use within the laboratory have been shown to be resistant to HF.

8. MATERIALS OF CONSTRUCTION

Examples of common materials compatible with varying strengths of HF at room temperature are tabulated below. Note that their suitability depends greatly upon the temperature and pressure they will be subjected to and the nature of other components present e.g. HCl or organic compounds.

Note : Consultation with a Design Engineer is strongly recommended.

	Grade of HF		
Material	Anhydrous	> 70 %	< 70 %
Mild steel	*	X	X
Stainless steel	*	X	X
PTFE ^	*	*	*
FEP	*	*	*
PFA	*	*	*
Monel	*	*	*
Inconel	*	*	*
Hastelloy	*	*	*
Polyethylene	X	*	*
Polypropylene	X	*	*
PVDF	X	X	X
Glass or glass fibre	X	X	X
Silica containing ceramics	X	X	X
Natural rubber	X	X	X
Silicone rubber	X	X	X
Polyamides e.g. Nylon	X	X	X

"*" - Acceptable
"X" – Not acceptable

Note : PTFE, if filled, must be filled with a suitable material (e.g. CaF_2 but not glass).

Lead washers can be used with the fittings on HF cylinders. It is safe to use in this manner, but should not be considered for rig building.

Note : When considering materials of construction consideration should also be given other components which may be present or which may be formed during the experiment. For example, some of the materials above, though compatible with HF would not be suitable for use with organic components and this loss of containment of HF may not have been considered during the risk assessment. This may be of particular importance when considering the purchase of standard equipment, e.g. peristaltic pumps.

Mechanical properties must also be considered when choosing materials e.g. copper would not be selected for building a rig due to its lack of mechanical strength.

Care should be taken when handling HF around wooden surfaces as the HF can absorb into the surfaces leaving it unsafe to touch. If HF is thought to have come into contact with a wooden surface then it should be scrubbed with a potassium carbonate solution.

It is important to keep PTFE, FEP etc., which are compatible with HF, away from materials like polyamides, e.g. Nylon, which are incompatible with HF. It is recommended that only HF compatible tubing is used and stored in laboratories using HF.

It is also important to keep HF compatible lubricating agents away from lubricants which are incompatible with HF. It is recommended that only HF compatible lubricating agents are used and stored in laboratories using HF. This is particularly important where valves are to be used under severely corroding conditions, they must be supplied lubricated with PTFE / perfluoroether greases instead of the standard silicone grease

Labelling of METAL tubing is essential to avoid confusion where differing materials of similar appearance are used in one rig (e.g. Monel and stainless steel). In cases where confusion arises, an alloy detector should be used to determine the composition of the material of construction.

9. <u>WORKING PRACTICES</u>

i) All work using HF will involve only personnel trained in its use

ii) Lone working with HF is not recommended. Experiments involving HF shall not be left unattended unless the equipment has been designed to be fail-safe and has been subject to a Hazard and Operability (Hazop) study.

iii) All staff starting to work with HF for the first time should undergo a medical examination for lung function capability and base fluoride in urine level. Staff suffering from asthma should not work on jobs involving large amounts of HF due to their increased vulnerability in the event of a major leak.

iv) Other workers in the laboratory or visitors, even if not directly concerned with the HF experiments, shall be informed of its hazards and the precautions necessary to avoid contact with HF. The adequate and correct labelling of equipment, pipework, reactants and products is an important part of this process.

v) It is recommended that permanent members of staff who are not working directly with HF but are working in an "HF Lab" should at least attend an HF awareness course.

vi) Care should be taken to prevent contamination spreading outside the fume cupboard e.g. via equipment and protective equipment. Special care should be taken not to touch equipment whilst wearing HF-contaminated gloves e.g. taps, door handles etc.

vii) Before starting any experimentation potassium carbonate solution and solid should be on hand so HF contamination can be dealt with speedily.

viii) Gloves (or equipment) should be rinsed in soda ash solution immediately after use, followed by rinsing in clean water to avoid the risk of powdery fluoride contamination.

ix) No entry is permitted to offices or control rooms whilst wearing protective equipment, nor are any samples allowed within these areas.

x) Workers involved in maintaining equipment which could be contaminated with HF should have the job fully explained to them. The laboratory staff and the maintenance team should complete a Risk Assessment. If it is deemed necessary a full Permit to Work Certificate may be appropriate.

xi) Local screening of high risk joints / restrictors should be given some thought when designing experimental rigs.

xii) HF shall never be discharged into a fume cupboard, down a drain or into the atmosphere. All HF shall be neutralised prior to disposal.

xiii) Experiments involving complex high pressure rigs using HF shall always undergo a hazard study, the results of which shall be retained. Such hazard studies shall always involve a member of the appropriate laboratory team and will have methods of containment built into them (e.g. dump tanks/scrubbers) to cope with a major release owing to equipment failure.

xiv) When planning an experiment or a series of experiments the inventory of HF that the lab holds should be governed by the frequency that the HF cylinder may need to be changed. It is recommended that cylinders should be sized to last no more than 6 months. It is also thought that if a lab needs HF, it should have its own permanent specifically designed source rather than obtain it from another lab and transport it.

10. <u>FUMECUPBOARD PRACTICES</u>

i) Polycarbonate screens should be fitted to all fumecupboard doors where there is HF in use

ii) The fumecupboards near to laboratory doors should be HF - free zones.

iii) Laboratories shall operate their fumecupboards on an ' all on ' or 'all off ' policy. i.e. if one fumecupboard is faulty in the lab then none of the others can be used until it is fixed. Fumecupboard vents should be designed to ensure that ‘suck back’ from vents into other fume cupboard in-takes is not feasible.

iv) The safe use of fumecupboards is either sash fully down when the lower doors are open or alternatively with the doors closed when the sash is at its maximum operating height.

v) All fume cupboards should be designed and operated to meet the necessary release calculations.

11. HF RIG BUILDING

i) Any rig that is to operate at higher than atmospheric pressure should be referred to a design engineer and the rig under-go a full HAZOP.

ii) The correct materials of construction should be used to prevent corrosion. For anhydrous HF, at relatively low temperature, stainless steel can be used. However, if there is any possibility of more corrosive HF mixtures coming into contact with the material Inconel, Monel or Hastelloy shall be used. Such corrosive mixtures could be HF/water, HF/HCI, etc. The wall thickness of pipework should also be considered.

iii) Pipework should be firmly supported. This is particularly relevant to valves, where there is mechanical stress when opening and closing and to HF manifold lines used to feed more than one rig in a laboratory.

iv) Where there is an HF manifold, a clear line diagram of the apparatus connected to the cylinder and instructions for purging the lines shall be readily available. The laminated diagram and instructions should be clearly visible close to the manifold.

v) HF pipework should be characteristically labelled or colour coded.

vi) Care should be taken in the siting of sample points for streams containing HF. A minimum of two isolation valves is preferred and the sample point should point away from the operator in the event of the sample valves failing.

vii) The sampling procedure for any experimentation should be prepared and included in the Risk Assessment.

viii) Care must be taken to avoid liquid HF being trapped in a closed length of pipe e.g. between two valves. **Under no circumstances should lines suspected to contain trapped HF be heated.**

ix) To protect vulnerable parts of rigs e.g. mass flow controllers or rotameters, from HF vapour that may diffuse back along lines the use sodium fluoride traps should be considered. These consist of a tube packed with sodium fluoride pellets, in the case of HF diffusion the sodium fluoride absorbs the HF and forms sodium bifluoride. **Care must be taken when dismantling these traps as the sodium bifluoride has similar toxicity and corrosion properties to HF, they should be immersed in carbonate solution wearing the appropriate personal protective equipment.**

12 ANHYDROUS HF IN USE

Anhydrous HF is a low boiling liquid (B.pt. 19.5^{o}C) which fumes on contact with air and reacts violently with water and bases, evolving a considerable amount of heat. Due to its low boiling point, its use is limited for liquid phase work at atmospheric pressure.

See in appendix 1 the main physical properties of HF, and STS 88/62 for more details.

Experiments using HF under reflux can be carried out by using PTFE equipment which includes a condenser.

Experiments at reasonably low temperatures (0-15^{o}C) can be carried out using PTFE, PFA or FEP. FEP is particularly suitable due to its translucence, although it is expensive and equipment constructed in this material is not as widely available as PTFE.

12.1 Cylinders

12.1.1 *Inventory & Use Control*

i) Incidents have been reported where cylinders of HF have "exploded" due to overpressurisation because of hydrogen build-up caused by the reaction of HF / water impurity with the metal of the cylinder. This increase in pressure generally takes many years (15 to 20 years) to reach a dangerous level. To prevent such occurrences, an inventory should be held and exhibited of HF cylinders stocked in the workplace. Cylinders over two years old should be checked for excessive pressure build up before use.

ii) Store cylinders in a DRY, COOL, VENTILATED place away from direct sunlight or where there is a risk of fire.

iii) If excessive pressure is found in a cylinder it should be vented through an appropriate scrubbing train with a nitrogen bleed to prevent suck back. For cylinders with dip-pipes it may be necessary to invert the cylinder to ensure that the gaseous head space is vented rather than liquor.

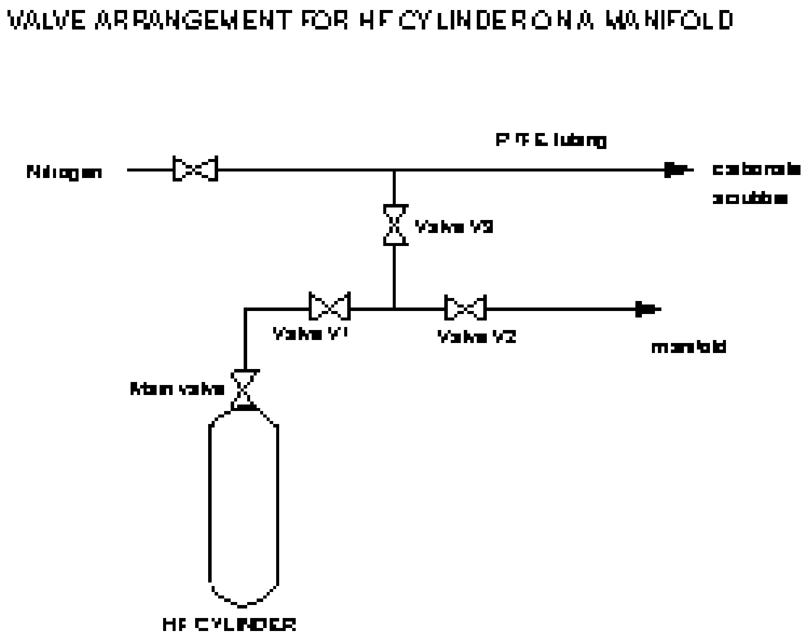
12.1.2 *How to Tell if a Cylinder is Empty*

First check that lack of flow is not due to the cylinder being too cold or that there is a blockage in any line.

HF cylinders should be weighed and compared with tare weights. The empty weight is always stamped on the cylinder, so that it can be weighed to check on the cylinder contents

12.1.3 *Connecting New Cylinder*

Where an HF cylinder is connected to a permanent rig or manifold thought should be given at the design stage to facilitate the changing of the cylinder. A diagram is shown below of a typical system.



Note : HF is invariably trapped in pipework, so never take chances when changing even apparently empty cylinders. When breaking into any line that may have contained liquid HF the correct level of personal protective equipment should be worn.

During normal running the PTFE tubing attached to valve V3 is replaced with a blanking nut.

To change the cylinder :

i) Ensure that the main valve is closed and valves V1, V2 and V3 are also closed.
ii) Remove the blanking nut and replace with PTFE tubing with a T piece and nitrogen purge to a carbonate scrubber.
iii) Carefully open valve V3 and allow any HF into the scrubber.
iv) Gently heat the pipework between valves V1, V2 and V3 with a hair dryer.
v) When the HF has all been released carefully open valve V1 and again allow any HF to the scrubber.
vi) Repeat the gentle heating with the hair dryer for all the pipework between the main cylinder valve and the valve V2 to the manifold.
vii) When the HF has all been released break the line on the swagelock fitting before valve V1, this causes less stress to the system than trying to release the tailpipe on the cylinder.
viii) Ensure that the main cylinder valve is shut on the new cylinder before removing the blanking nut.
ix) Release the tailpipe on the old cylinder and replace the blanking nut.
x) Replace the gasket in the nut before reconnecting the tailpipe to the new cylinder.

xi) Reconnect the pipework, ensure that all valves are closed and carefully open the new cylinder main valve.
xii) Test for any leaks using an ammonia wash bottle.
xiii) Remove the PTFE tubing and replace with a swagelock blanking nut.

Always keep the main valve on HF cylinders closed when not in use. Remember to decontaminate all tools and gloves when finished.

12.1.4 *Cylinders with Seized Valves*

Do not attempt to open seized valves on HF cylinders. Replace the blanking nut and dome, fix a label and arrange for the cylinder to be returned to the supplier.

12.2 Feed Systems

12.2.1 *Atmospheric Pressure HF Feed*

This has many advantages over rotameters, low flows 10 - 20 ml/min can be obtained which are both reproducible and constant.

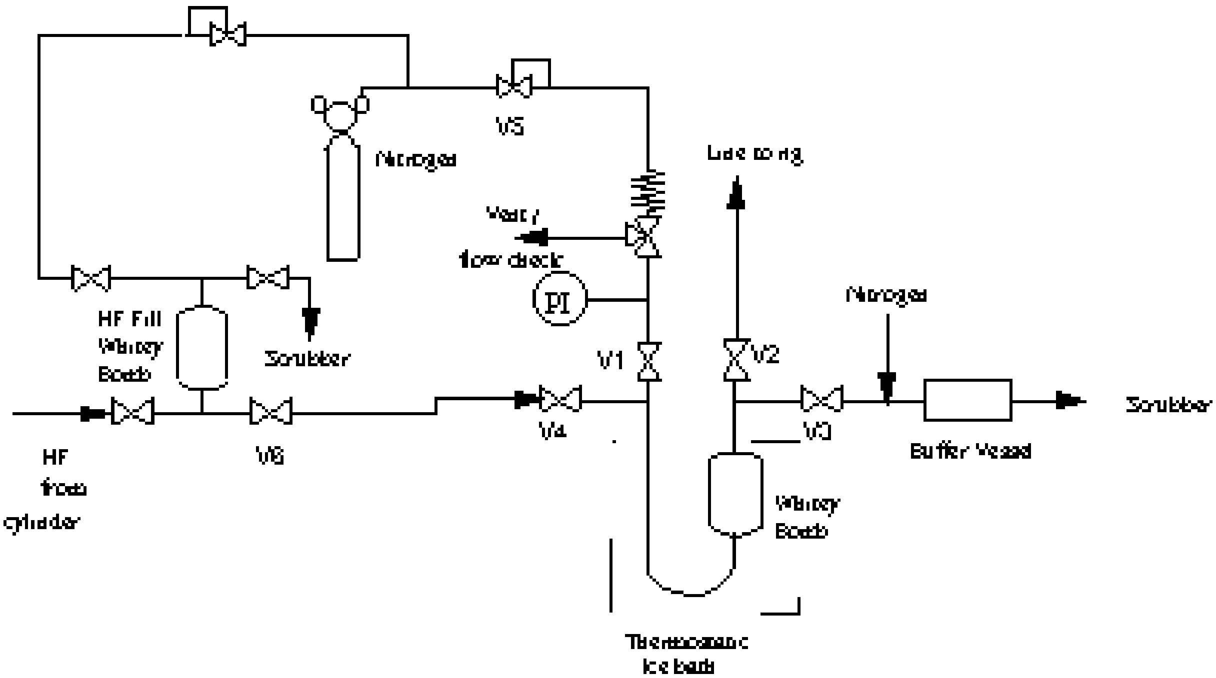
When a gas is sparged through a liquid, the off - gas contains a volume of the liquid dependent on the vapour pressure of the liquid at the temperature and pressure of the liquid. This principle can be used to feed low levels of HF into an experiment where a low volume of diluent such as nitrogen will not affect the experiment. Depending upon the pressure and the rate required, the HF is heated or cooled and the sparge rate varied.

HF at a temperature of 0°C has a vapour pressure of 0.48 bara. Thus 0.52 mls of nitrogen should pick up 0.48 mls HF. However, HF boils in the form of an oligomer and therefore 1 ml of nitrogen will give approximately 2 mls HF.

The feed system can be filled from a weighed HF reservoir at a pressure of ~ 2 barg by closing valves V1, V2 and V3 and opening valves V4 and V6. This would allow the available volume of the sample bomb to be filled before the pressures equalised and the flow ceased. Ensure that there is a nitrogen flow through the buffer vessel and to the scrubber. Close valves V4 and V6, then carefully crack open valve V3 to release the excess pressure to the scrubber. This can then be fully opened whilst the temperatures and pressures equilibrate.

The metered nitrogen feed can be added by opening valve V1 and continuing to feed the off gas to the scrubber. When steady, conditions are obtained, the feed can be diverted to the experiment by opening valve V2 and closing valve V3. The nitrogen purge to the scrubber prevents any suckback of the scrubber liquor.

ATMOSPHERIC HF FEED



The actual rate of HF can be adjusted by altering the nitrogen feed, or the temperature of the water bath and determined by titrating the HF against caustic soda using phenolphthalein.

12.2.2 ***High Pressure HF Feed***

High Flows (up to 7 g/min)

For pressure systems, liquid HF can be fed from a reservoir via an appropriate pump and vaporised by trace heating or an electrically-heated block on route to the reactor. Rate of flow is typically measured by weight change in the reservoir versus time. A double diaphragm pump should be used with a fluorinated fluid inserted between the diaphragms.

Low flows (0.5 - 2 g / min)

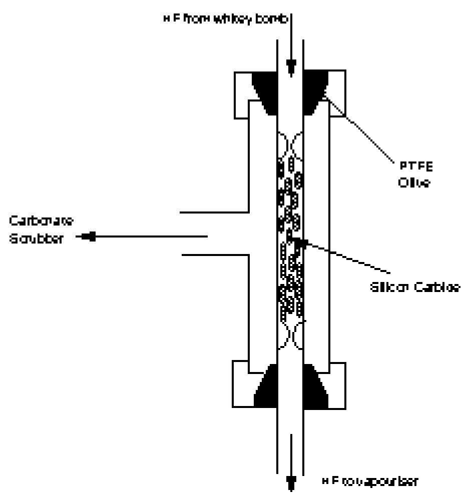
Nitrogen sparge system.

A similar arrangement as the atmospheric sparge system can also be used at pressure. The thermostatic ice bath is substituted by a hot oil bath and the nitrogen is controlled by a mass flow controller protected from HF vapour by a sodium fluoride trap. The temperature of the oil is dependant on the working pressure of the rig. For a pressure of 10 barg. a temperature of 80^{o}C will give a flow of HF equivalent to 0.5 mls for every ml of nitrogen.

Care should be taken on materials of construction and in the operating instructions to prevent hot HF at pressure from being ejected inadvertently to the atmosphere.

Flow Restrictor

A more recent development has been to use flow restrictors and pass liquid HF through a crimped tube packed with silicone carbide powder. Standard practice is to pack a 1/8th inch OD tube which is itself contained in a 1/4 inch tee. The other arm of the tee is linked to a potassium carbonate scrubber in case there is any leak from the crimped tube. The HF is fed from a sample bomb pressurised with nitrogen controlled by a valve. A manifold system of restrictors can be used since the performance of the restrictors does vary with time. The HF can be vaporised downstream from the restrictors using trace heating tape or a heating block. This method has the advantage of no moving parts, is easy to maintain and can, when the restrictors have settled in, give steady HF flows for long periods of time.



13. AQUEOUS HF IN USE

Aqueous HF can be more hazardous than its anhydrous form because of its lower volatility and therefore higher persistence, it does not fume at lower concentrations making it hard to distinguish from other chemicals or water. The burns caused by aqueous solutions can be serious because often they have delayed symptoms which causes late teatment and therefore the effect of a burn may be more pronounced.

Therefore greater thought must be given when ordering aqueous grades. The greatest care must be taken in handling this material, with the appropriate personal protective equipment being worn, as described earlier. The smallest volume required should be ordered to prevent unnecessary disposal problems at the end of an experiment.

A number of concentrations are readily available. For HF concentrations below 40% or between 40 and 60 %, dilute the next highest strength HF to the required strength with water, adding the acid to the water. Always carefully add acid to water possibly as ice in a suitable vessel with stirring and cooling, wearing the appropriate personal protective equipment.

For concentrations between 60 and 100 %, dilution of anhydrous HF with aqueous HF is required. Wear the appropriate personal protective equipment. In a fume cupboard weigh, or measure by volume, HF into an ice-cooled polypropylene measuring cylinder by slowly passing liquid via 1/4 "PTFE or stainless steel tubing connected to a dip-pipe HF cylinder. HF should never be removed from the fumecupboard in a measuring cylinder or any open vessel.

Calculate the amount of 40 % aqueous HF needed to achieve the required strength of HF. e.g. 100 mls of 85 % HF would require 75 g of anhydrous HF plus 25 g of 40 % aqueous HF. Cool the aqueous acid in a plastic container held in ice, then add the anhydrous HF with stirring, at a very slow rate. Reweigh the vessel. A violent reaction occurs with evolution of heat with a loud crackling sound and spitting if the addition is uncontrolled or the temperature is too high. Not only is this potentially hazardous, but the required concentration will not be achieved as HF will be lost as vapour.

The prepared solution should be stored in a properly labelled, polypropylene or FEP bottle - or Monel, Inconel or Hastelloy sample bomb (depending upon the vapour pressure of the prepared solution) in a safe place, e.g. in a bunded container enclosed in a ventilated area.

14. DISPOSAL

The correct level of personal protective equipment should be worn during this procedure.

Solutions containing HF should be neutralised by pouring slowly, with stirring, into a solution of sodium or potassium carbonate (20 % w/w with ice) contained in a plastic vessel.

Carbonate is preferable to hydroxide since the evolution of CO_2 has the advantage of removing heat, although the resultant frothing must be controlled by the rate of addition and constant stirring. After neutralisation (checked by pH paper/indicator), any organic should be separated and disposed of by suitable methods. The aqueous phase, if in the pH range 6-9, may be discarded by pouring slowly down a sink simultaneously with copious amount of cold water.

Anhydrous liquid HF is disposed of by pouring into a solution of ~ 69 % w/w calcium chloride hexahydrate and ice contained in a plastic vessel and sending the product for burial. Approximately 20 % excess of the chloride is used, 5.5 kg of $CaCl_2.6H_20$ with 2.5 kg of ice being used to dispose of every kilogram of anhydrous HF.

Liquid HF must be fed via a pipe into the bottom of the vessel.

If possible, $CaCl_2.6H_20$ should be utilised as soon as possible after mixing so that full use is made of the cooling that has been generated.

HF containing vapour should be neutralised by passing the vapour, diluted with an air or nitrogen stream to prevent suck back, into a static potassium carbonate scrubber using phenolphthalein as indicator. Thought should be given as to the need for an antisuck back device in case there is an interruption in the air or nitrogen supply. Visible traps should be used wherever possible so that any 'suck-back' can be traced visibly. If the carbonate is totally neutralised additional carbonate should be added to the scrubber and then flushed down the drain with copious amounts of water.

15. DECONTAMINATION

It is important that everything that has possibly been in contact with HF should be thoroughly decontaminated after use. HF has a tendency to form a layer on metallic surfaces, it is readily absorbed into wood and will dissolve and form an aqueous layer on moist surfaces. Tools, gloves etc. should be immersed into a sodium or potassium carbonate bath before washing with water.

All personal protective equipment used during an experiment should be assumed to have been contaminated with HF and should be thoroughly decontaminated e.g. by use of a safety shower. Particular care should be taken with removing personal protective equipment to ensure that secondary contamination does not occur. Where personal protective equipment is known to have been contaminated it should be decontaminated and disposed of.

Wooden surfaces should be wiped with carbonate solution. Known spillages of HF onto wooden surfaces should be treated with carbonate solid followed by washing with carbonate solution and finally water.

Appendix 1 : Physical Properties

Table 1 : Physical Properties of Anhydrous Hydrogen Fluoride

Molecular Weight	20.01 (monomeric)
Boiling point	19 .5 ºC at 760 mm Hg
Melting point	-84.0 ºC
Specific Gravity	0.98 at 10 ºC
Vapour Density (Air = 1)	2.4 at 20ºC
Critical Temperature	187.9 ºC
Critical Pressure	64.9 bar
Critical Density	290.0 kg/m^3
Heat of Vaporisation at boiling point	374.5 kJ/kg
Heat of fusion at freezing point	196.9 kJ/kg
Specific heat at constant pressure Liquid at boiling point Vapour at 25 ºC, 760 mm Hg	 2.32 kJ/kg ºC 1.46 kJ/kg ºC*
Viscosity of liquid at 0ºC	0.25 cP
Surface tension at 0ºC	10.27 mN/m
Flash point	Non Flammable
Explosive range	Non Explosive
Solubility in water	Soluble in all ratios
Odour threshold	<1 ppm

Table 2 : Variation of vapour pressure, vapour density and liquid density with temperature.

Temperature ºC	Vapour pressure Bar Abs	Density Saturated Vapour Kg/m^3	Density Liquid Kg/m^3
-10	0.31	-	1,025
0	0.49	2.15	1,002
10	0.71	2.52	980
20	1.03	3.17	968
30	1.46	3.98	945
40	2.02	4.98	928
50	2.76	6.19	908
60	3.7	7.65	888
70	4.9	9.39	867
80	6.4	11.44	844
90	8.25	13.85	820
100	10.52	16.64	796

Table 3 : Variation of Density of Superheated Vapour at 1013 mbar with temperature

Temperature ºC	Vapour Density Kg/m^3
19.5	2.93
30	1.79
40	1.13
50	0.85
60	0.77
70	0.74
80	0.71
90	0.68

Table 4 : Heat of Dilution of Hydrogen Fluoride

Concentration of HF % w/w	Heat of Dilution* KJ/Kg
100	0
90	217
80	380
70	502
60	623
50	736
40	820
30	874
20	912
10	937
0	970

** Heat evolved when 1 kg of anhydrous liquid HF at 25 ºC is diluted to the given concentration*