Comité Technique Européen du Fluor (CTEF)

Working Group on Storage, Transport and Safety (STS)

Group 1

RECOMMENDATION ON BULK TRANSPORT, DISTRIBUTION AND HANDLING OF ANHYDROUS HYDROGEN FLUORIDE (AHF) AND HYDROFLUORIC ACID SOLUTIONS (HF)

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PREFACE

Anhydrous hydrogen fluoride/ hydrofluoric acid (AHF/HF) is essential in the chemical industry and there is a need for HF to be produced, transported, stored and used.

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

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

Each company, based on its individual decision-making process, may decide to apply the present recommendation partly or in full.

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

It results from the understanding and many years of experience of AHF/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 AHF/HF manufacturer, supplier or user, or other expert 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 Eurofluor 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 AHF/HF manufacturer before implementing it in detail.

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

AHF is an acronym for anhydrous hydrogen fluoride.

HF is an acronym for hydrofluoric acid solutions of any concentration below 100%.

1. INTRODUCTION – GENERAL REMARKS

This guideline has been developed by the Storage, Transport and Safety Group of the Comité Technique Européen du Fluor (CTEF). It is intended to offer recommendations required for the transport, the distribution and the handling of anhydrous hydrogen fluoride (AHF) or hydrofluoric acid solutions (HF) at ambient temperatures (from -20°C to +50°C), unless stated otherwise.

This Recommendation - based upon many years of practical experience - concerns the design, testing, construction and use of packages or transporting equipments such as railcars, road tanks, ISO tank-containers, intended for the transport of AHF and HF overland and/or by land-sea.

It is not intended to be a guideline to which all manufacturers and consumers of HF must adhere nor does it attempt to describe any additional design standards other than those associated with AHF/HF. It does not replace or assume any local or national regulations, which may identify other requirements.

All materials of construction, which are mentioned in this document should be double-checked and there should be a search for more information on materials, in our "Recommendation on materials of construction for Anhydrous Hydrogen Fluoride and Hydrofluoric Acid solutions" available from Eurofluor publication webpage www.eurofluor.org.

The following recommendation deals only with the bulk transportation of anhydrous hydrogen fluoride and of hydrofluoric acid solutions.

2. DEFINITIONS

ISO	International Standard Organisation
CSC	International Convention for Safe Containers
ADR	European Agreement Covering the International Carriage of Dangerous Goods by Road
RID	Regulation Covering International Carriage of Dangerous Goods by Rail
UIC	International Union of Railways
IMDG	International Maritime Dangerous Goods Code
DOT	Department of Transportation - USA
CTC	Canadian Transport Commission

3. CONCENTRATIONS AND TYPES OF PACKAGES FOR THE TRANSPORT OF AHF/ HF

Hydrofluoric Acid is commercially used in the following concentrations:

- Anhydrous Hydrogen Fluoride (AHF: UN 1052) and Hydrofluoric Acid aqueous solutions at concentrations more than 85% (UN 1790)
- Hydrofluoric Acid Solutions (HF) with more than 70% and less than 85 % Hydrogen Fluoride (UN 1790);
- Hydrofluoric Acid Solutions with less than 60% Hydrogen Fluoride (UN 1790)

AHF and HF is transported and handled:

- In bulk, in:
  - Railcars
  - ISO Tank Containers
  - Road tankers
- In other type of packages such as cylinders, drums, jerricans, or bottles.

Note: This classification is not necessarily the established by ADR, RID nor IMDG

4. BULK TRANSPORT

4.1. Design specifications by type of transport unit

4.1.1. Railcars

AHF and HF are usually shipped in amounts between 20 and 60 tons, shell full, non-coiled, non-insulated railcars designated in accordance with the UIC and RID standards.

The following parts should be considered in a railcar design:

- The underframe (chassis), designed in accordance with the UIC standards;
- The wagon, designed in accordance with the UIC standards;
- The tank car fasteners, in accordance with the UIC, RID
- The tank, designed as a pressure vessel and in accordance with the UIC and RID standards;
- Painting, in accordance with the UIC and RID standards.

For the materials, construction and additional equipment of the tank, see point 4.2.2 of this document.

4.1.2. ISO-Tank containers

The term “tank-container” used in this document means a type of transport equipment, built to contain liquid and composed of a tank fixed in a frame, designed according to the ISO standards.

Tanks should be designed and constructed as a pressure vessel to meet the design requirements specified by an approved regulation body (e.g. CSC, ADR, RID, UIC, IMDG, DOT or CTC, including a shunt test).

The tank should be mounted in a suitable steel frame rated for 34,000 kg and conforming to ISO 1496, with corner castings to ISO 1161.

Typical general dimensions are for the ISO series 1 freight containers (ISO 668):

Size	20’	30’
Length, mm.	6.058	9.125
Width, mm.	2.438	2.438
Height, mm.	2.438 or 2.591	2.438 or 2.591

For the materials, construction and additional equipment of the tank, see point 4.2.2 of this document.

4.1.3. Road tankers

The maximum size of the road tank is limited by the national and international regulations for the various types of vehicles.

The transport tanks should be designed according to international regulations, ADR and other national regulations as appropriate.

The transport tanks for AHF should be exclusively for this product.

There are two types of road tankers:

- When the tank is attached to the chassis:
  The method of attachment of the tank onto the chassis or frame (bolts, brackets, etc) should be constructed in steel, properly designed for the duty and adequate in particular to avoid any movement of the tank relative to its supports.
- When the tank is without chassis.
  The attachment pieces should not be welded directly onto the tank, but should be continuously welded onto reinforcing plates, which are of an adequate dimension and thickness.

In both cases, the chassis should be designed as a “AT vehicle” specified by ADR.

For the materials, construction and additional equipment of the tank, see point 4.2.2 of this document.

4.2. General design specification

4.2.1. Construction code

In any vehicle (railway tank, ISO tank, road tanker), the chassis and the tank should always be designed to such a standard as to satisfy national or international regulations for such equipment.

In all three cases, the tank for AHF/HF should be designed according to international regulations and other national regulations as appropriate, taking into account the following requirements, bearing in mind that these basically apply to the choice of materials of construction.

The tank should be designed and constructed as a pressure vessel to meet the design requirements specified by an approved regulation body.

The minimal design pressure of the shell is 21 barg for AHF and >85% HF, and between 4 and 10 barg for HF, as requested by RID/ADR depending on the HF concentration.

The 10 barg design pressure of the shell allows a minimum thickness of the shell as a good protection against mechanical impact in case of accident.

The chosen materials of construction should be suitable for use with HF.

The concentration of the acid and the expected ambient temperature must be taken into account. Low concentration and high temperatures increase the rate of corrosion, so lined steel tank should be considered. Low temperatures may lead to embrittlement of the carbon steel.

The minimum number of connections necessary should be provided on the tank to minimise the potential number of leakage points.

All fittings, manhole and openings should be fitted to the top of the tank. All supports for fittings should be welded to the tank. Screwing directly into the tank is prohibited.

All valves, fittings, etc. should be contained in a housing to protect them against damage caused by overturning or impacts and to minimise the effects of weather and give mechanical protection. The protective device must be constructed in such a way that water and other liquids can drain. The valve housing should have a facility for fitting locks, or closures to prevent unauthorised access.

A ladder can be fixed to the frame to give auxiliary access to the top of the tank.

Sufficient walkway area to enable safe access for loading / unloading should be provided. Safety equipment (e.g. handrails) must be provided at the access point (either on vehicle or at loading/unloading station) to minimise the potential for any slips, trips and falls.

Construction of the tank for HF must be carried out by an approved contractor.

4.2.2. Tank Design Materials and construction

Shells shall be made of suitable metallic materials which, unless other temperature ranges are prescribed in the various classes, shall be resistant to brittle fracture and to stress corrosion cracking between -20°C and +50°C.

For welded shells only materials of faultless weldability whose adequate impact strength at an ambient temperature of –20 ºC can be guaranteed, particularly in the weld seams and the zones adjacent thereto, shall be used. If fine-grained steel is used, the guaranteed value of the yield strength be shall not exceed 460 N/mm2 and the guaranteed value of the upper limit of tensile strength Rm shall not exceed 725 N/mm 2 , in accordance with the specifications of the material.

If fine-grained steel is used, the guaranteed value of the yield strength Re shall not exceed 460 N/mm 2 and the guaranteed value of the upper limit of tensile strength Rm shall not exceed 725 N/mm2, in accordance with the specifications of the material.

The elongation for fine-grained steels it shall be not less than 16% in any case, and not less than 20% for other steels.

Carbon steel may suffer hydrogen stress corrosion cracking and hydrogen blistering in contact with HF.

The steel should be tested for resistance to hydrogen assisted stress.

Carbon steel approved for use with HF should be used to construct the tank and internal fittings. Steel sheets should be checked using ultrasonic to ensure they are lamination free.

Wall thickness for AHF and solutions should be considered:

Example of calculation for a life time of 20 years:

Wall thickness for AHF and solutions of more than:	**85% HF**	**70% HF**
Wall thickness according to mechanical strength and design pressure (mm)	x	x
Lower limit of the wall thickness x for atmospheric pressure operation (mm)	6	6
Safety margin recommended by CTEF (mm)	2	2
Corrosion allowance for 20 years 25°C (*) (mm)	20 x 0,1 = 2	20 x 0,4 = 8
Total minimum thickness for a new tank (initial) (mm)	2+ x = **10**	8 + x = **16**

(*) 0.4 mm corrosion per year for HF 70% at 25°C. (Additional thickness to be added for higher temperatures), and:

(*) 0.1 mm corrosion per year for HF 85% at 25°C. (Additional thickness to be added for higher temperatures).

The thickness of the wall must be monitored by ultrasonic tests, according to the RID/ADR regulations.

CTEF strongly recommends never using a tank with a thickness lower than 8 mm for AHF and for HF.

The 'dished ends' of the tank will be hot pressed or dished and spun followed by stress relief.

A 500 mm manhole access opening should be provided on top of the tank. The manhole should not cut through any line of welding.

The tank may be baffled to the extent indicated by ADR or RID. The baffles must be of a dished spherical form and should be attached to backing strips welded to the shell.

All welds should be done using shielded metal arc or submerged arc techniques, and should be 100% radio graphed.

The completed HF tank should be stress relieved after welding has been completed.

The internal surface of the tank, may be lined or not, depending on the concentration of the HF. It may be:

- Carbon steel for AHF and HF solutions with more than 85% hydrofluoric acid;
- Cleaned carbon steel or lined steel, for HF solutions with more than 70% and less than 85% hydrofluoric acid;
- Lined, for HF solutions with 70% or less, hydrofluoric acid.

Criteria for the lining:

Some examples for the internal lining of the iso-tanks for transportation of diluted HF:

HF%	WALL THICKNESS	MATERIAL	MATERIAL MANNU-FACTURING PROCESS	COMMENTS / EXPERIENCES
75%	2 layers 5mm + 5 mm	Iso- butyl	CALENDERING	Calendered product The wall thickness was obtained by calendering and belding basic sheets of 1 mm thick. Blisters between layers appeared
75%	2 layers 5 mm + 5 mm	Iso- butyl	EXTRUSION	Manufactured by extrusion The degree of compactness of the extruded sheets is far superior to that of the calendered ones
<60%	at the responsibility of the ISO-tank supplier	Rubber lined	at the responsibility of the ISO-tank supplier	
40-60%		Halar (E-CTFE) Lining	Spray lining	successful applications for more than 5 years
73%	2 layers 4 mm	Chlorobutyl rubber (CIIR)	CALENDERING	Use for rail tank and an iso container
73%	2 layers 4 mm	Bromobutyl rubber (BIIR)	CALENDERING	Since 2010
30-49-70% HF	2 layers	Chlorobutyl lining (1/4 inch) Natural Rubber (1 mm, to protect the steel)		
>70%	-	-	-	Exclusively fine grain structured steel C355 (10 years lifetime)

HF%	WALL THICKNESS	MATERIAL	MATERIAL MANNU-FACTURING PROCESS	COMMENTS / EXPERIENCES
71-75%				Isotankers: fine grain carbon steel (i.e. EstE355)

Note: It is very important to choose carefully the manufacturer who will prepare and carry out the lining on the metallic wall. The quality of the bonding between the lining material and the tank wall is fundamental because it affects significantly the mechanical resistance and the porosity of the liner, and, consequently, the final resistance of the lined tank against corrosion.

Both the tank and the frame or chassis will be treated to protect against weather.

Reminder: Note that the design of the tank must cover particular circumstances, such as the possibility of resistance under vacuum, due to temperature variation or to handling operation. Such design data should be checked by appropriate thickness tests, when possible (in case of a lined tank, tests cannot be performed).

4.2.3. Additional Equipment Design Standards

4.2.3.1. Manhole for AHF or HF

4.2.3.1.1. General

The safe loading and unloading of anhydrous hydrofluoric acid can be improved by standardizing the sizes and the layout of the valves.

4.2.3.1.2. Manhole

The manhole will be placed on the top of the barrel at the mild point on railcars and at the mild point or at one end on ISO tank containers.

Manhole minimum diameter: 500 mm.

4.2.3.1.3. Layout of valves

- Valves should be angle valves
- Number of valves
    - 1 or 2 for the liquid phase based on local requirements and Risk Assessment.
    - 1 or 2 for the gas phase based on local requirements and Risk Assessment.
- At least one relief valve with bursting disc, if the unit is to be transported by ship as required by IMDG.

The liquid phase valve(s) should be located on the longitudinal axis and the gas phase and the relief valve- if any- on the transversal axis of the barrel.

The manhole flange should be designed to hold an internal shut off valve with an orifice identical to the valve size. It must be protected against the risk of ejection. See figure 1.

The valves must be closed by blind flanges when not in use.

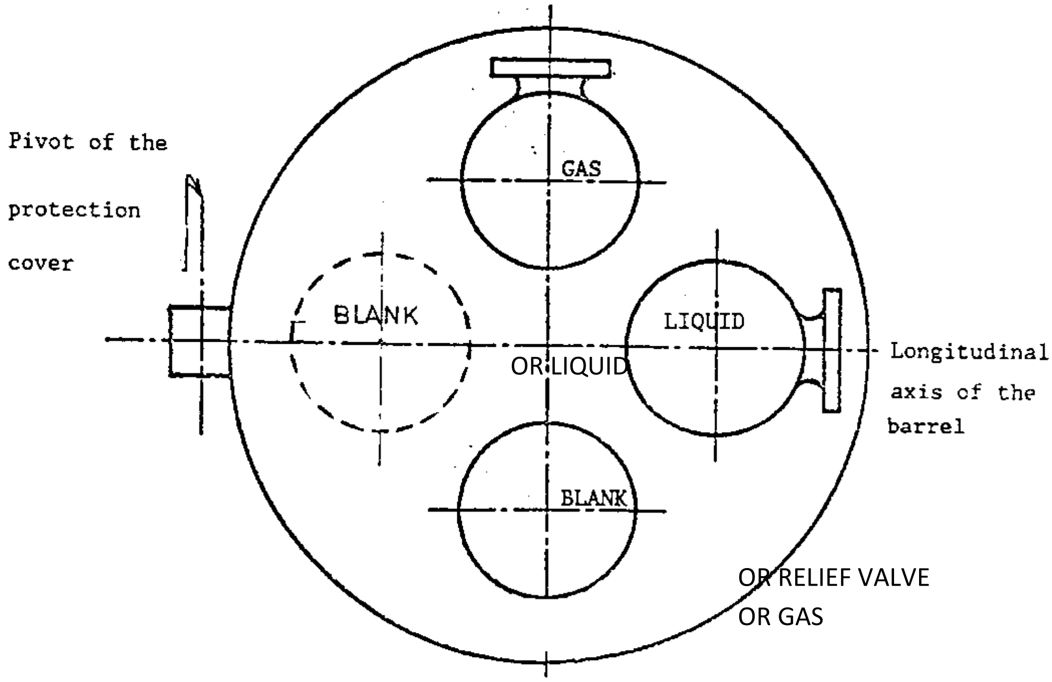


Figure 1

4.2.3.2. Internal shut off valve

It is recommended to include with each service valve an internal shut off valve, so that in case of failure of the service valve, the device must remain shut.

A mechanism must prevent the accidental or unauthorized opening of the shut off valve in the course of transport.

The stem between the 2 halfs of the valve must be at the level of the valve flange.

The top of the disc or ball must be at 2 mm below the level of the valve flange.

4.2.3.3. Service valves

4.2.3.3.1. General

For anhydrous HF, it is recommended the use of pneumatically actuated valves, fitted with an internal stop/shut off valve (see § 4.2.3.2 above), the stem of which should be protected by a bellows, and an outlet valve, size PN25-DN40 RF Form D.

When HF concentration is less than 85%, FEP or PFA (PTFE can also be used) lined plug or ball, pneumatically actuated, valves should be used.

Pneumatically actuated valves, fitted with an internal stop/shut off valve may be used for HF solutions, but special attention should be paid to inspect more frequently the internals of the valves, due to possible higher corrosion.

The valves will be located on top of the tank and housed to be protected against weather or other damages. The valves must be clearly identified as vapour or liquid, either by labelling or colouring.

The liquid valve should have a dip-pipe connected to the check valve body (if welded, it must be stress relieved and radio graphed). The pipe may be in PTFE or PTFE coated, with a thickness of at least 3 mm. The material of the dip-pipe must be consistent with the material of the tank. The dip-pipe will be sized to terminate in a sump, such that the end of the dip-pipe is 10 mm below the bottom of the tank and at least 10 mm above the bottom of the sump. The bottom end of the dip-pipe should be secured in a steady welded to the shell. It should be possible to remove the dip-pipe without having to enter the tank.

According to their type, the valves are provided with a manual override facility.

Individual air actuation points should be provided local to each valve, if pneumatic. Non-self-sealing couplings should be used.

Valves should be checked for signs of leakage prior to each use.

4.2.3.3.2. Valves Flanges lay out

- Flange connected to the barrel (inlet), see figure 2 (spigotted type DN40)

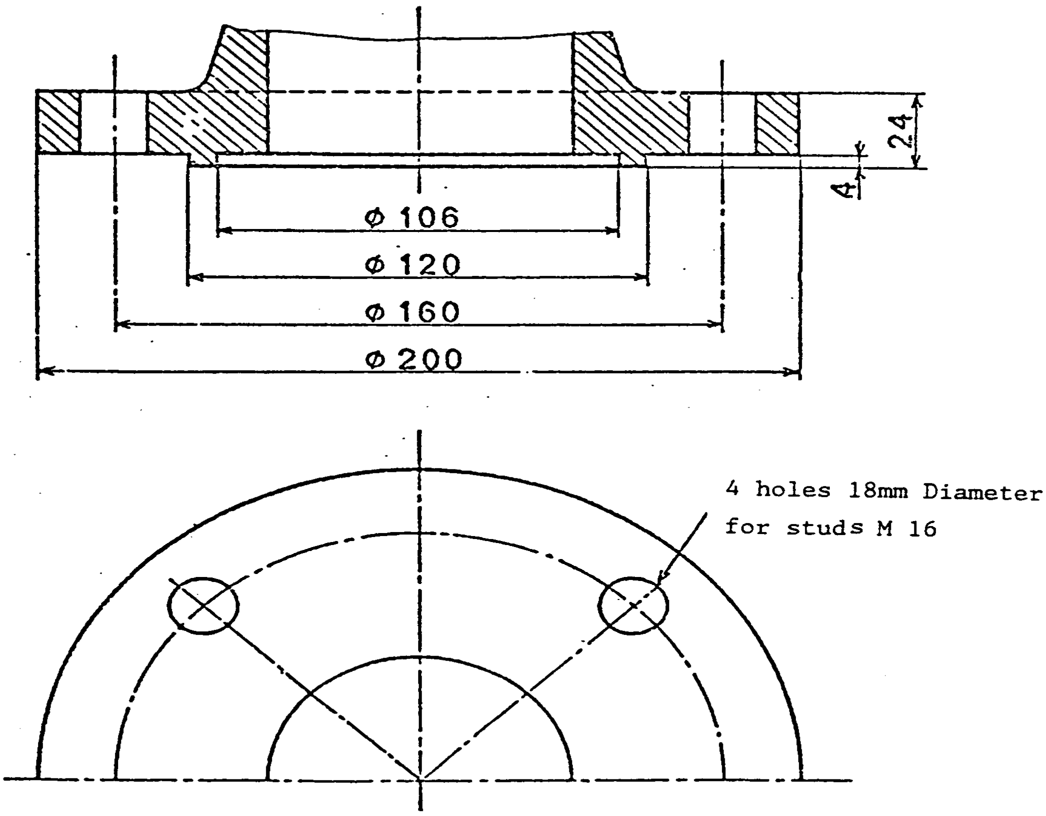


Figure 2

- Flange connected to the flexible (outlet), see fig. 3 hereunder

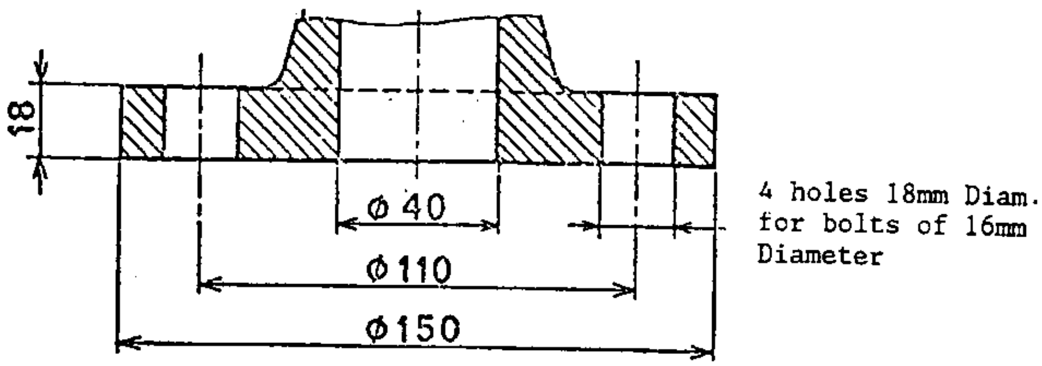


Figure 3

4.2.3.3.3. Pneumatic operation

If any, the following conditions should apply. Layout See fig.4

Each valve must have independent actuation.

Compressed air connections

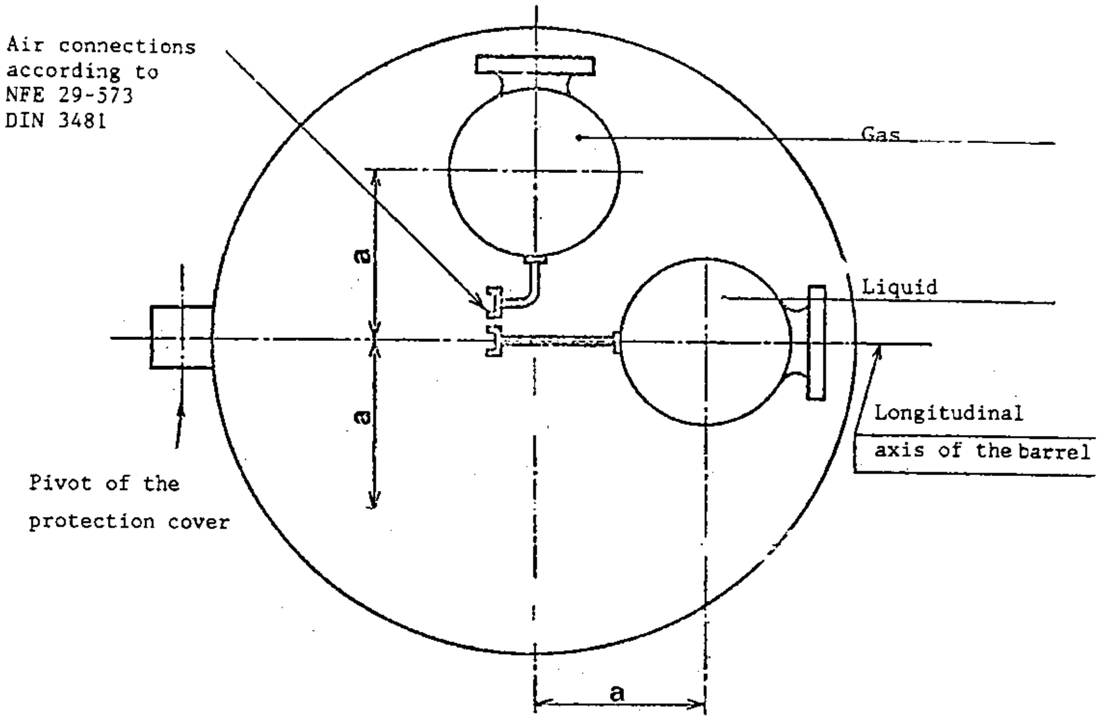
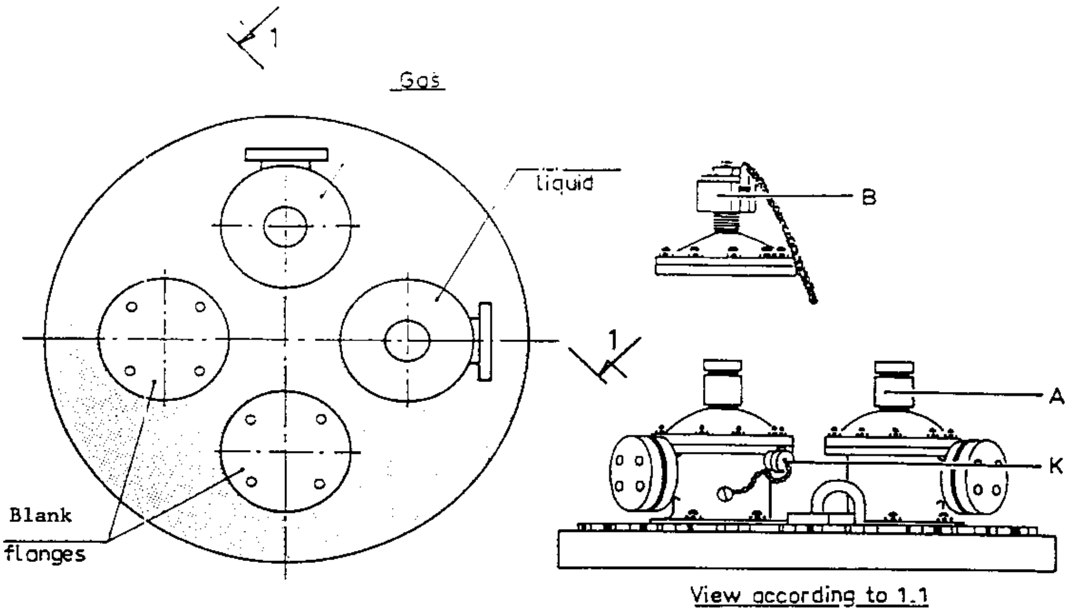


Figure 4

- <u>Procedure in case of compressed air supply failure</u>

Exceptionally, it must be possible to actuate the valves manually and locally.

It leads to an alternate operating procedure, which is described in figure 5.



Operating Instructions

Opening

1 Unscrew locking cap (A) (locking mecanism is now released)
2 Attach compressed-air line to coupling (K) (minimum compressed-air pressure 4 bar, maximum compressed-air pressure 6 bar)
3 Switch on compressed-air (valve opens)

Closing

1 Switch off compressed-air and vent compressed-air line (valve closes)
2 Detach compressed-air line
3 Screw on locking cap (A) and tighten it manually

Quick shutoff in case of danger

Switch off compressed air and vent compressed air line immediately (valve closes)

Emergency operating procedure in case of compressed-air supply failure

Opening

1 Unscrew locking cap (A)
2 Screw on manual operating device (B)
3 Extend release rope (allowing to operate from a safe place) after inserting in Locking device
4 Screw bolt into manual operating device (B) (valve opens)

Closing

1 Unscrew bolt from manual operating device (valve closes)
2 Unscrew manual operating device (B)
3 Screw on locking cap (A) and tighten it manually

Quick shutoff in case of danger

Pull release rope till Locking device disengages

Attention

Valves which are not needed must be locked and fitted with a blank flange (especially if they are connected by a compressed-air ring line)
Angle valve test: Remove the test screw (R) whilst the angle valve is open.
If there is any gas leakage the valve is defective.
CLOSE IT IMMEDIATELY! Personnel who is carrying out the test must be fully protected

<u>Figure 5</u>

4.2.3.4. Bursting Disc & Relief Valve

Relief valves are required by IMDG for ship transport. For land transport, relief valves may not be legally required, and CTEF recommend not installing them to avoid a possible source of leakage.

A flanged outlet should be provided to enable a relief valve arrangement to be fitted. Where necessary a suitable relief valve preceded by a Monel bursting disc should be fitted. The 'tell-tale' hole between the relief valve and the bursting disc should be sealed in such a way as to allow access for inter-trip inspection. A good recommendation is to include a plug valve fitted on the “tell-tale” hole and a pressure gauge.

A threaded blank emergency cap should be ready for use. Threads should be well greased with a product suitable for HF.

4.2.3.5. Nuts & Bolts

All those nuts and bolts on the tank, both on the internal, exposed surfaces and on the external flanges should be:

- Bolts ASTM A193 B7 or ASTM A193 B7M
- Nuts ASTM A194 2H

The above specifications are justified by the fact that high tensile strength steel must be avoided for bolts and nuts in order to be protected from hydrogen stress corrosion. The hardness of the steel should be limited.

4.2.3.6. Gaskets

All gaskets must be made of material resistant to HF, capable of good service proven by experience, especially for flanges in the liquid phase. For example, PTFE, CaF_2 filled PTFE, spirally wound Monel or PTFE with a Monel guard ring.

<u>Note</u>: all gaskets must be changed after each use and replaced with new gaskets each time.

<u>Note</u>: Even during construction and testing the gaskets used must meet this standard. This is to prevent the possibility of wrongly specified gaskets finding their way into HF service.

4.2.3.7. Protective device for valves

<u>4.2.3.7.1. General</u>

This device intends to ensure the safety of the tank during transportation especially the protection of valves, nozzles and branches in case of accident including derailment and overturn.

It is basically a cover that protects the valves situated on the tank and specifically installed on the manhole.

4.2.3.7.2. Conditions of use

The device must protect the valves, nozzles or branches within the range of reference temperatures between -20°C and + 65°C.

It should be strong and stiff enough to withstand the impact of the total weight of the filled tank action on the free end of the device in horizontal direction. The impact must not cause either deformations or cracks resulting in leakage of the cylindrical shell or the valves, nozzles or branches.

The protective device may be an integral part of the tank or a construction, which is connected with the tank in such a way that it cannot be removed without using a force exceeding the total weight of the tank.

4.2.3.7.3. Materials

All parts of the protective device must be made from a material with a resilience and tensile strength sufficient to withstand the conditions of temperature and stability indicated in the previous paragraph. The material of the protective device must be compatible with that of the tank.

4.2.3.7.4. Construction

The protective device must be constructed in such a way that:

- No parts of valves or nozzles rise above the device
- Access to and manipulation of valves and nozzles are not obstructed or impaired
- Water and other liquids can drain
- If the relief valve is fitted in the man hole flange, the protective cover should have an opening with enough free area for the exhaust of the gases when the valve opens

Protective collars out of steel plate of the same thickness as the walls of the tank welded around the valves and nozzles as high as the valves have been found to provide suitable protection on rail, ship, road or tank cars.

Another example of protective device, fulfilling the aforementioned conditions is the framework of an ISO-container according to ISO 1496/III.

The side of opening of the cover should be in accordance with Figure 1.

The cover must be equipped with a safety pin.

An instruction sheet containing operating instructions should preferably be located inside the cover (see figure 5).

4.2.3.7.5. Test on prototype

The protective device must be tested by using a force equivalent to the weight of the tank filled with water, acting on the top of the device in the direction parallel to the longitudinal axis of the tank.

The test must not cause any deformation or crack resulting in leakage of the shell, valves or nozzles.

5. FINISH TREATMENT

5.1. Internal Surfaces

5.1.1. Steel tanks

The internal surfaces should be shot blasting to SA2.5 standard and thoroughly cleaned and dried before final closure.

The tank should be transported under an atmosphere of dry air or nitrogen (to a dew-point of –30°C) at 1.0 barg.

Prior to use for the Anhydrous HF transport, the tank should be 'passivated' or pickled. This is necessary to allow the formation an Iron Fluoride layer on the internal surfaces. This layer prevents corrosion of the metal.

The passivation may be achieved by half filling the tank and allowing the tank to stand at least for 24 hours. During this time the tank should be monitored for evidence of leaks, particularly around the flanges. This HF should then be removed from the tank and returned to the plant for reprocessing.

5.1.2. Lined steel tanks

The internal surfaces should be thoroughly cleaned and dried before final closure.

The tank should be transported under an atmosphere of dry air or nitrogen at 1.0 barg.

5.2. External Surfaces

Both the external surfaces of the tank and the frame should be shot-blasted, thoroughly cleaned and painted with an appropriate heavy duty industrial and sea anti-corrosion coating.

6. TESTING AND CERTIFICATION

A certificate should be provided for <u>each tank</u> quoting at least:

- the serial number
- the maximum operating pressure
- the maximum operating temperature
- the water capacity
- the maximum filling weight in HF
- the date of pressure test
- the test pressure

Tests and certifications should be in accordance with the requirements of an approved regulation body and with the present legal standards. Good complementary maintenance will assure the endurance of the tank in safe conditions.

The Minimum requirements are as follows:

6.1. Pre-Construction

The grade of steel selected should be tested prior to constructing the tank for:

- Thickness.
- Homogeneity.
- Impact Strength.
- Yield stress.
- Permanent elongation at fracture.
- Ultrasonic test, with a minimum square of 200 mm × 200 mm. to ensure it is lamination free.
- Approval of the construction procedure
- Approval of welder's qualification and welding procedure.
- Tests on traction, on bending and on resilience of reference samples and welded samples

6.2. Post-Construction

The completed tank should be uniquely numbered and undergo the following testing:

- All welds should be 100% radio graphed or 100 % ultrasonic combined with 25 % radiographic examinations of the welds, all knots being controlled by radiography prior to stress relieving.
- Physical check against design, both internally and externally.
- Where fitted, each lining will be spark-tested to ensure complete internal coverage.
- The capacity should be accurately determined.
- A hydraulic pressure test according to the regulations and to a minimum of 10 bar (AHF) or 4 bar (Aqueous HF) 4 bar.
- A leakproofness test (effective internal pressure at least equal to the maximum working pressure) and a leakproofness test and a check of satisfactory operation of the equipment

Note: Replacement of gaskets following the hydraulic test is strongly recommended.

Before commissioning, all the valves and accessories should be tested in order to guarantee their leak tightness under operating conditions. An air pressure test must be carried out at a pressure greater than the operating one, and detection of leaks should be performed with soap solution.

The prototype should be submitted to stress test.

The Competent Authority shall witness some or all of these tests prior to issuing test certification. Test certificates shall be available at all times for inspection by regulatory bodies.

6.3. Each Journey

Prior to each journey the tank will undergo the following tests:

- Check the nameplate and the documents that must be updated.
- Visually inspect tank and framework or chassis for damage.
- Wearing appropriate protective clothing, check the condition of the valve protection boxes, operation of valves and signs of leakage. If there are any signs of leakage (such as visible indications of fluorides at the flanges) then the tank must be taken out of service until the joint has been repaired.
- When the tank is fitted with a relief valve, it should be examined for signs of damage or leakage. Wearing appropriate protective clothing, the manometer or the ‘tell-tale’ hole between bursting disc and relief valve should be inspected. In the event of a leak (indicative of a defective disc) the tank must be taken out of service until the device has been repaired.
- Check legibility of markings.
- Prior to operation, a leak test of the piping system connected to the tank should be performed to check the integrity of the valves and of the hose.

Following loading or unloading the integrity of valves should be checked prior to fitting blanking plates.

6.4. Periodic Testing

Shells and their equipment should undergo periodic inspections at fixed intervals. The periodic inspections should include: an external and internal examination and a hydraulic pressure test.

Regarding ADR the following test should be performed:

<table>
<tr><th>PERIODIC TESTING</th><th>TANK</th><th>TANK CONTAINER</th><th>RAIL TANK</th></tr>
<tr><td>- An external and internal examination;
- A leakproofness test (effective internal pressure at least equal to the maximum working pressure)
in accordance of the shell with its equipment and check of the satisfactory operation of all the equipment;
- As a general rule, a hydraulic pressure test.
The test pressure for the hydraulic pressure test depends on the calculation pressure and shall be at least equal to the pressure indicated below:
<table>
<tr><td>Nº ONU</td><td>SUBSTANCE</td><td>Calculation pressure (bar)</td><td>Test Pressure (Bar)</td></tr>
<tr><td>1052</td><td>Hydrogen fluoride, anhydrous</td><td>21</td><td>10</td></tr>
<tr><td>1790</td><td>HF 75%</td><td>10</td><td>4</td></tr>
</table></td><td>6 years</td><td>5 years</td><td>8 years</td></tr>
<tr><td></td><td></td><td></td><td></td></tr>
<tr><td>- An external examination
- Leakproofness test of the shell with its equipment and check of the satisfactory operation of all the equipment, which has to be subjected to an effective intern al pressure at least equal to the 25% of the maximum working pressure and it shall not be less than 20 kPa (0.2 bar) (gauge pressure).</td><td>3 years</td><td>2,5 years</td><td>4 years</td></tr>
</table>

This CTEF Recommendation goes beyond the legal rules according to the safety experience of the HF producers and users. So the recommendations for each inspection, apart from the legal ones are:

6.4.1. Testing on a 2.5 or 3 or 4 year basis

These tests have to be performed according to the type of tank.

- ISO-Tank containers (each 2.5 years)
- Tank vehicles (each 3 years)
- Tank-wagons (each 4 years)

The inspection should be carried out by an independent inspector, covering the following issues:

<u>External inspection</u>:

The frame should be examined in accordance with CSC and RID, ADR or IMDG requirements. The vessel should be inspected for any defects in material or welds, followed by a random ultrasonic thickness on areas of the shell and ends.

With the agreement of the expert and approved by the Competent Authority, an internal inspection may be avoided or replaced by other testing methods to avoid additional inside corrosion after testing.

In lined steel tanks, the internal surface should be examined. In that case, a careful decontamination should be carried out.

Then the tank should be decontaminated at an authorised plant with an adequate scrubber system, taking into account that the water would be HF contaminated, to the extent necessary to verify the valves.

Due to the relative porosity of the plastic material, bubbles can be formed with loosening of the lining. Care must be taken as the bubbles may be filled with liquid acid.

Valves and fittings should be checked for any defect in their materials and in operation. Check actuators and springs, if any, and renew critical parts.

The relief valve should be re-certified as per the regulatory requirements and the manufacturer instructions.

All fittings should be refitted and a leak test using dry air or nitrogen should be completed following final closure.

Check of the satisfactory operation of all the equipment. The tank should be subjected to an effective internal pressure at least equal to the maximum working pressure.

When a gas is used for the leak proof test it should be carried out at a pressure at least equal to 25% of the maximum working pressure. In all cases it should not be less than 20 kPa (0.2 bar) (gauge pressure) (ADR/RID)

The nameplate details and particular documents should be up-dated.

If necessary, the internal tank surfaces should be cleaned and passivated again.

If necessary, the transport unit should be repainted and labelled.

The Competent Authority shall witness some or all of these tests prior to re-issuing test certification. Test certificates shall be available at all times for inspection by regulatory bodies.

6.4.2. Testing on a 5 or 6 or 8 year basis

These tests have to be performed according to the type of tank.

- ISO-Tank containers (each 5 years)
- Tank vehicles (each 6 years)
- Tank-wagons (each 8 years)

The tank should be fully decontaminated; all fittings are removed and decontaminated later. A careful decontamination should be carried out when the tank is rubber lined.

After full decontamination the following checks should be made:

- Remove all fittings and decontaminate them individually.
- Blank off all flanges and carry out a hydraulic test according to the regulations and to a minimum of 10 barg for AHF and 4 barg for Aqueous HF.
- Clean and dry the internal surfaces. Shot blast if it is a steel tank and if condition warrants.
- Carry out a thorough internal and external examination of the vessel and fittings for any defects in operation, material or welds, followed by a random ultrasonic thickness checks on areas of the shell and ends.

Check the satisfactory situation of the dip pipes.

In lined steel tanks, check the internal surface. Due to the relative porosity of the plastic material, bubbles can have formed, with loosening of the lining.

Care must be taken, as the bubble may be filled with liquid acid. If necessary, repair the lining.

- Examine the frame in accordance with RID, ADR or CSC requirements.
- Where fitted the relief valve should be re-certified as per the regulatory requirements and the manufacturers instructions.
- Liquid and vapour line valves should be fully reviewed and if necessary replaced with new or reconditioned ones.
- All bolts and nuts should be replaced with new. (See 4.2.3.5)
- The manlid and fittings should be replaced only after a thorough internal inspection to ensure complete cleanliness of the barrel. All the gaskets must be new (see 4.2.3.6.).
- All fittings should be refitted and a leak test using dry air or nitrogen should be completed following final closure. The test pressure will be at the maximum working pressure.
- If required, the external surface of the tank and frame should be thoroughly cleaned, painted and labelled.
- The nameplate details and documents should be up-dated.

- The operation of the valves and air actuators, if any, to be checked, making sure the universal joints on the actuator shafts are well greased with a product suitable for HF.
- Prior to use in traffic the internal tank steel surfaces should be "passivated" again (see 5.1.1.)

The Competent Authority shall witness some or all of these tests prior to re-issuing test certification. Test certificates shall be available at all times for inspection by regulatory bodies.

Note: this CTEF Recommendation goes beyond the legal rules according to the safety experience of the HF producers and users.

7. LABELLING REQUIREMENTS

All movements of HF beginning or moving through the member states of the European Union are subject to Council Directive 96/35/EC June 1996 on the Appointment and Vocational Qualification of Safety Advisers for the Transport of Dangerous Goods by Road, Rail & Inland Waterways. This Directive requires that all companies loading, transporting and unloading HF within the member states of the European Union must appoint a qualified Dangerous Goods Safety Advisor (DGSA) whose role is to advise on the standards required. These DGSAs advise on the correct current labelling requirements.

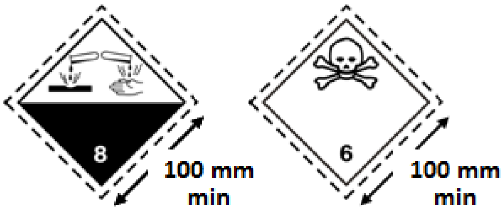
A labelling example is shown below.

Note: ADR / RID / IMDG are periodically reviewed. Therefore, the accuracy of this example should be verified by a qualified DGSA to ensure they are adequate for the product and journey being undertaken.

All data shown is taken from the 2013 version of the ADR transport regulations.

Example 1: ADR and RID Labelling for Hydrofluoric acid solution containing between 60 and 85% Hydrogen Fluoride.

- Marking and labelling of packages :

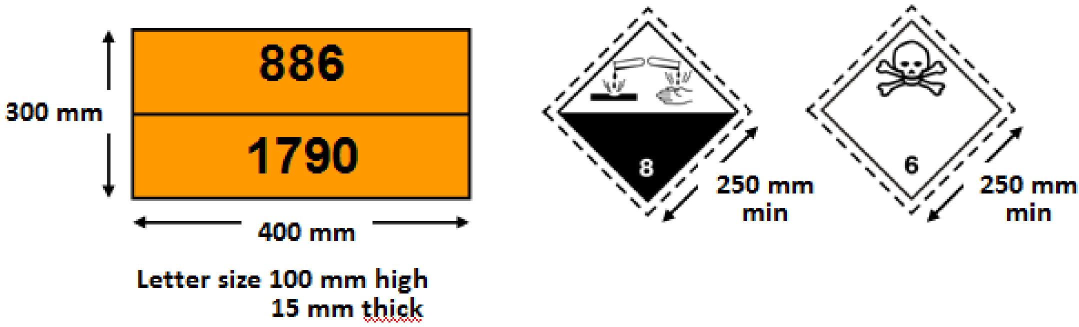


UN 1790 **Letter size 12 mm high**

Note :
only 6 mm high for packages of 30 litres capacity or less (or 30 kg max mass net)

Obligation from the 1st of January 2014

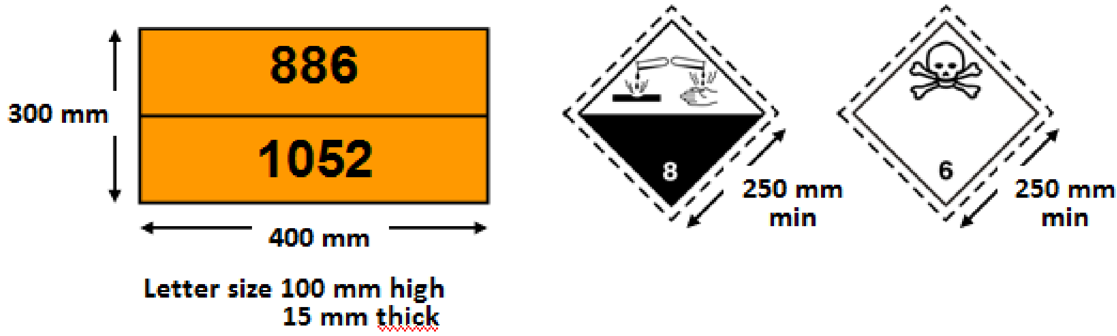
- Placarding and marking of tanks, tank-containers and portable tanks :



- Provisions for labels, placards and marks :
    - The orange-coloured plate marking is displayed on both sides of the tank (or tank-container, or portable tank).
    - The orange-coloured plates shall be reflectorized.
    - The material used shall be weather-resistant and ensure durable marking
    - The plate shall not become detached from its mount in the event of 15 minutes' engulfment in fire.
    - It shall be remain affixe irrespective of the orientation of the vehicle.
    - The placards shall be affixed to both sides and at rear of the tank or the tank-vehicles.
    - The placards shall be affixed to both sides and at each end of the tank-container or portable tank.
    - All labels shall be able to withstand open weather exposure without a substancial reduction in effectiveness
    - When the placarding is affixed to folding panels, they shall be designed and secured so that they cannot unfold or come loose from the holder during the carriage (especially as a result of impacts or unintentional actions)

Example 2: ADR Labelling for Anhydrous Hydrofluoric Acid.

- <u>Placarding and marking of tanks, tank-containers and portable tanks :</u>



- Provisions for labels, placards and marks : see example 1