



Safety Risk Assessment for Corrosion Testing in Hydrofluoric Acid

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ABSTRACT

Despite the regular inspections and current contingencies made in the HF plant at Necsa, an isolated case where two mild steel vessels leaked HF during operation occurred without warning. The failure of both these vessels necessitated their premature decommissioning, and showed that better understanding the corrosion of mild steel used in the HF industry was needed. The failure of the mild steel was traced to nitric acid (HNO₃) contamination in the sulphuric acid (H₂SO₄) feedstock which eventually concentrated in the technical grade HF product downstream. A study to simulate the industry corrosion conditions in a laboratory was required to better estimate the service life of the steels used in the HF plant because understanding the effect of HNO₃ contamination on the plant's steels and the determination of corrosion inhibition strategies was essential. Unfortunately, HF is an extremely hazardous chemical and concentrations above 70% HF have a fuming capacity, making it a potentially lethal chemical for corrosion experiments. Therefore, to safely work with HF, the safety risk for working with the chemical first had to be categorised and assessed. This entailed determining the internal hazards of handling HF and its consequences, the probability of exposure and then mitigating actions that would lower the risk to a point that corrosion experiments could safely be conducted. A regular safety risk assessment, a process description, a "what if" analysis, as well as an external hazard and operability study (HAZOP) was completed. This led to the implementation of a specialized experimental setup that proved to be prepared for any HF exposure contingencies while still producing HF corrosion data comparable to literature. Moreover, preventative measures including the use of HF resistant PPE (Personal Protective Equipment), well ventilated facilities and HF resistant Teflon containers were put in place which eventually resulted in the safe execution of corrosion tests completed there.

1. Introduction

Hydrofluoric acid (HF) is a very toxic chemical that causes severe burns when it comes in contact with skin and if swallowed (Goldemberg, 2009; Hathaway and Proctor, 2014). Below 60%, HF releases negligible fumes when opened to the atmosphere or when it is spilled (Tepperman, 1980:691–692). However, an HF concentration of 70% has a fuming capacity (vapour pressure is 20 to 40 kPa at 20°C), producing hydrogen fluoride fumes on spilling (Hathaway and Proctor, 2014). Moreover, HF has a highly polarized nature (highly positive H⁺ atom and very negative F⁻ atom) and HF molecules are heavier than air. This results in a ground hugging effect, which makes the fumes difficult to vent off, and exposure is more probable than for other gasses used in a laboratory environment. Inhalation of HF causes irritation of the respiratory tract, which may lead to pulmonary oedema up to 48 h after exposure which is fatal (Tepperman, 1980:691–692).

Since there were no available data, to understand the effects of the different concentrations and combinations of HF and HNO₃, a viable option was to reproduce the unique corrosive environment in a laboratory. The results from these experiments would then be used to develop a suitable method for predicting the corrosion rates of the steel tanks and components in the plant, which were exposed to HF with higher levels of HNO₃ (0.1 to 1 %). However, there were great safety concerns with doing laboratory corrosion tests with such a toxic chemical.

It was decided that corrosion experiments and examinations be conducted in accordance with the relevant ASTM (2009) standards practices (G31 – 72) and guides (G161 – 00) for corrosion test and examinations (ASTM Standards, 2009; ASTM Standards, 2009a; ASTM Standards, 2009b). However, these procedures assume that all laboratory immersion corrosion experiments can be conducted in glass reactors, using probes and measuring instruments which are completely resistant to chemical attack by the corrosive solutions prepared. Glass and materials reinforced with glass fibres (like fibreglass or fibre-reinforced plastics), as well as materials rich in silica and silicon are not resistant to HF (Dulski, 1996). For lower concentrations of HF (>4% HF), antimony probes can be used (Boyes, 2010).

Consequently, all experiments needed to be conducted in highly HF resistant reactors (Teflon) with examination and handling of corrosion products only conducted in glassware when all the un-reacted HF had been completely washed off the corrosion products. However, the corrosion tests needed to be conducted in corrosive solutions in excess of 40% HF, which ultimately eliminated the possibility of conducting potentiostatic and potentiodynamic polarization measurements without the use of a custom-made and extremely specialized potentiostatic setup. Therefore, only mass loss and visual examinations could be used to quantify the corrosion of mild steel in this study.

From the safety assessment and HAZOP (Rausand, 2011) study conducted beforehand, the inherent hazard of conducting corrosion experiments with HF was identified. Subsequently, the actions to mitigate contact with HF and then to manage the unlikely event of HF exposure were addressed and implemented into the experimental design. The experimental setup was made with the focus on safe execution of a corrosion test and to minimize contact with HF during loading and unloading of the corrosive solution and metal coupons. While attempting to adhere to standard practices identified in the ASTM procedures, modifications needed to be made to the experimental setup to address all hazards identified for working with HF. However, concerns about the quality of the corrosion results were raised, since the external environment was altered to maximize safety. The entire corrosion setup was made to fit into a polypropylene fume cupboard which was open to constant ventilation (0.5 to 1 m/s linear flow speed) and connected to potassium hydroxide (KOH) scrubbers to neutralize HF fumes which might escape during the corrosion tests. This introduced external factors to the corrosion setup, and was specifically identified as a lack of pressure (constant venting and sweeping of HF) in the Teflon corrosion reactor. Moreover, because HF fumes escape the reactor, the volume of corrosive solution in contact with the coupons over the course of a corrosion test would also be affected. To address this, relatively simple corrosion tests on mild steel were conducted in the safety approved experimental setup and related to available corrosion rates from literature. Moreover, fuming tests were conducted to establish exactly how much HF would fume off during a typical corrosion experiment conducted

in the fume cupboard, while a water bath was regulating the temperature.

The first corrosion tests selected to prove that the modified setup would produce relevant corrosion data were the planned interval corrosion tests (PICT), as prescribed by Watchter and Treseder (ASTM Standard, 2009). These relatively simple corrosion tests were necessary to determine the duration of an HF corrosion test in the current setup. The PICTs were initially conducted using lower concentrations HF (40% and 48%), because the fuming risk was greatly diminished, and were thus safer to conduct, according to the original internal safety risk assessment conducted at South African Nuclear Energy Corporation SOC Limited (Necsa). However, it was also essential that laboratory simulated corrosion experiments were conducted in 70% HF concentration to be useful to industry (Valkenburg, 2012). The experimental setup constructed for the PICT led to establishing whether or not implementing mitigating actions for working with safely HF, as established during the safety assessment, impacted the quality of the corrosion data collected.

2. Safety risk assessment

2.1. Background

In the safety risk evaluations, the safety risk category level needs to be determined, and any necessary safety related tasks performed (Louw, 2014). Thus, the project researcher follows progressively more elaborate procedures for experiments that are categorized as having higher safety risk levels.

The safety risk category levels are:

- Negligible/Low: one person can evaluate the safety risk.
- Moderate: More than one person is needed to evaluate the safety risk of the planned action. A detailed process description with sufficient detail for the safety risk evaluation on the planned experiments must be prepared beforehand. A “what if” analysis, described by Flynn and Theodore, (2001) is an internal HAZOP which may be completed by the project manager for use by the relevant safety committee (e.g. Project Safety Committee (PSC)) when ratifying the experiments. The PSC comprises all the significant stakeholders

(e.g. Safety Officer, Radiation Protection Officer, Facility Manager, Laboratory Manager and Line Manager) and decides whether all the safety actions are acceptable for undertaking the experiments safely in the designated laboratories, and if the safety risk needs to be escalated to a higher safety risk level.

- Significant: Here, actions which are not part of normal operations are included and which constitute significant safety risks to applicable operators (and the organisation as a whole). Over and above general requirements for a moderate safety risks, an independent person must be involved in the evaluation of the safety risk and planned tasks. An external HAZOP study is required to be placed before the relevant safety committee (e.g. PSC) when ratifying the decision to handle the safety risk for a significant safety risk level, and to ascertain if it is necessary to escalate to a safety risk to an even higher level.
- High: This is when the planned action does not fall within any of the previous safety risk categories, and it must be escalated to obtain the Safety, Health, Environment and Quality (SHEQ) in the organisation (e.g. Necsa) approval of the project processes (Louw, 2014).

Firstly, the safety risk evaluation process must determine the safety risk level of the actions in the project. This was done by evaluating the risk in accordance with the prescribed criteria, and is a record of the initial evaluation. During the evaluation, different scenarios which can lead to exposure of personnel or equipment hazards of the chemical were identified and assessed, and then subsequently the safety risk level was determined. In the assessment of chemical scenarios, the risk assessment focused on the description of and motivation for the task, quantity description (related to toxicology) and operation description (Flynn and Theodore, 2001). Other crucial safety risk elements also covered were the experimental setup, the facility, and finally physical together with environmental factors.

Experimental setup

Safety risk elements assessed during the experimental setup are:

- The total volume of the chemical used in the experimental setup
- The materials of construction used and their resistance to HF at the working pressure
- The liquid flammability, at the temperatures and volumes being considered (flash point and volume)
- Effluent generation expected and disposal (volumes).

Facilities

Safety risk elements assessed when conducting experiments in the designated work space are:

- Changes and modification to the current ventilation system
- Availability of an adequate scrubber (KOH) for neutralizing of gasses vented off during the experiment.

Physical and Environmental factors

Safety risk elements assessed in terms of the physical environment the researcher needs to work in when conducting the experiments are:

- The influence of the noise levels exposed to in the facility
- The wet bulb globe temperature in the work area
- Exposure to glare or strong light emissions during the experiment
- Segmental and whole body vibrations
- Use of a laser and the classification of the laser
- The degree of ergonomic stress that may be encountered.

Secondly, using the findings from the safety risk assessment, the identified safety related tasks needed to be performed to mitigate the safety risk and effectively put the corrosion experiments in a lower risk category. These tasks included specific actions to be performed and precautions which must be considered when performing the planned experiments.

When the safety risk assessment found that the safety risk was at a significant level, a procedural HAZOP needed to be conducted where the hazard were analysed with respect to the following:

- Causes (e.g. failures)
- Consequences
- Preventative measures to eliminate the causes
- Protective or emergency measures to reduce the consequences
- Recommendations to prevent or protect against its consequences
- Rating the risk (frequency X severity)
- Assigning responsible persons to carry out actions to implement the recommendations.

HAZOP studies are frequently conducted in industry with the procedures and example layouts used to complete this document being generally available (Flynn and Theodore, 2001; Rausand, 2011). Therefore, only the findings from the procedural HAZOP are discussed, as well as the implementation from the recommendations made.

3. Materials and Methods

3.1. Materials

The HF used for the PICT was aqueous hydrogen fluoride (70% Industrial grade) collected from the Pelchem SOC Ltd. fluorochemical plant, situated on the site at Necca. The 70% HF was a high quality product (fluorosilicic acid ≤ 100 ppm, sulphuric acid ≤ 200 ppm and nitric acid < 5 ppm) received in sealed 25 L bottles, which were intended for export and sold by the Protea Chemicals (Inland). The lower concentration HF (40% and 48%) were analytical grade ($[\text{NO}_3^-] < 5$ ppm) solutions purchased from Merck (Pty) Ltd.

3.2. Methods

The following is a summary of the actual process description used when campaigning for consent to conduct the corrosion experiments, using HF in the laboratories. The main focus needed to be on safe handling of the materials and operation of the instruments in a well-ventilated corrosion setup constructed from materials compatible with HF. The process description is therefore more detailed, but serves well as the methodology of the PICTs conducted. To calculate the corrosion rate (CR) in mm/y from metal loss, Equation 1 was used:

$$CR = 87.6 \times (W/\rho At) \quad [1]$$

where W = weight, ρ = density, A = area of the coupons exposed to HF over time (t).

3.3. Process description

Each experiment was conducted in a 500 mL Teflon bottle that was placed in a water bath to maintain the acid at a constant temperature, while corrosion of the coupons was allowed to take place. The coupons were assembled into a cylindrical rack (made of Teflon) which was placed upright in the bottle (Figure 1 b). The HF was then poured into the bottle, until the cylindrical rack holding the coupons (Figure 1 c) had been covered completely. The associated bottle lid, with a 2 mm diameter drilled hole, was then screwed on. This allowed for the release of HF fumes that could have allowed pressure to build up in the Teflon container. The temperature was maintained at 25°C using an immersion cooler attached to the polypropylene lid, custom-made to cover the bath, which allowed the immersion cooler to hang freely in the water while being shielded from HF fumes by a polypropylene cylinder attached to the lid (Figure 1 a). The lid over the water bath had a release valve in the open position, which would release any HF fumes that escaped from the corrosion reaction. The fumes would then exit through the extraction line of the fume cupboard which led out to the KOH scrubber.

PICTs were allowed to run for a period of time that would be experimentally determined by the planned interval test of Wachter and Treseder in ASTM Standard G 31-72 (ASTM Standard, 2009). Four coupons per bottle were introduced to a

maximum of 500 mL HF. Subsequently, one coupon was removed from the corrosion rack at a specified time; this continued over the entire duration of a corrosion experiment, until all coupons had been removed. This was done to establish the metal samples' susceptibility to corrosion, the liquid's corrosiveness and the rate of the corrosion reaction under the simulated conditions (Van der Merwe et al., 2016). Each time a coupon was removed from the bottle, it was rinsed with water and air-dried before being stored in a desiccator. All these actions took place in 40 L spill tray placed in the fume cupboard to ensure safe working conditions. After testing, the corrosion products were removed from the coupons by ultrasonic cleaning for 30 min, followed by mechanical cleaning with a steel brush to ensure complete scale removal from the coupon prior to weighing.

When the last coupon was removed, completing an experiment, the remaining liquid in the bottle with corrosion products were sampled and sent for analyses. The remaining HF-containing effluent was discarded into a 25 L waste bottle. When the waste bottle was at capacity, it was sealed and safely stored in the chemical cage outside of the AC building, then sent to HF plant on site for disposal.

4. Results

4.1. Preventative measures

4.1.1. Safety risk assessment for HF corrosion tests

Work conducted using hydrofluoric acid inherently carries a significant risk level for any safety risk assessments done in any organisation (e.g. Necca). All activities could be maintained at a moderate safety risk, as long as the concentration of HF used in the fume cupboard was not more than 60% concentration in a volume less than 2.5 L. Thus, the initial PICT experiments using 40% and 48% HF could safely be conducted under a moderate risk category, as long as the HF volume was less than 2.5 L at all times. This included the HF feed to the corrosion reactor, products and waste generated after the tests had been completed.

All materials used to contain the corrosion tests were compatible with HF. HF not in use during a

corrosion test was stored in sealed high density polyethylene containers, thus automatically had a low safety risk level. As a safeguard, the experimental design was constructed so that it shielded the metallic and electronic components from the HF and possible fumes during an experiment using the polypropylene lid over the bath (Figure 1 a). The process description and “what if”, described by Flynn and Theodore (2001), were the only requirements for corrosion experiments using 40% and 48% HF as a moderate safety risk was applicable. These experiments could be conducted with no additional recommendations or additional actions.

testing HF in acid concentrations where the water concentration was too high (Van der Merwe, 2016). Therefore, corrosion tests using 70% HF became essential. This meant that the safety risk assessment for fuming HF would escalate the corrosion experiments to a higher safety risk category, which meant that the entire assessment had to be redone.

Thus, as well as the requirements of the safety risk assessment for a moderate safety risk scenario, a HAZOP conducted by an outside specialist was needed before the experiments were allowed. During the external HAZOP, a concern was raised regarding the volume of HF fumes vented off during a corrosion experiment, and their effect on the volume of corrosive liquid available for the corrosion reaction in the 500 mL Teflon reactor. To address this concern, before the publication of the HAZOP findings, 500 mL of 70% HF was placed in the reactor (Figure 1b) and allowed to stand in a fume cupboard for 9 days. It was determined that the 2 mm hole in the reactor lid allowed for a maximum of 22.7 mL (which came to 2.5 mL per day) and would mean that less than 5% of the reactor volume would escape during a 10 day corrosion experiment.



Figure 1: Experimental setup: (a) water bath with immersion cooler setup, (b) corrosion reactor components: Teflon bottle, cylindrical corrosion rack and steel coupons, and (c) complete assembly for safely simulating the corrosive conditions in HF.

Results from the first PICTs indicated that corrosion rates were excessive when

4.1.2. Hazard and operability study for HF corrosion tests

During the HAZOP, the hazards associated with the corrosion testing of materials in 70% HF were identified. A detailed analysis of the hazards, recommended actions and quantified assessments of risks were then conducted and the findings of

that hazard assessment are summarised in Table 1.

4.1.2.1 Accidental release measures

From the safety assessment, accidental release measures were put in place in the unlikely event of HF being released in the lead up to, or disassembling a corrosion experiment. In the event that there was a spillage, it would be contained in the spill trays in which the

experiments and sample preparations took place. At the outset to clean a spill, protective equipment should be worn as previously specified (Table 2) and adequate ventilation provided. The HF may not leave the fume cupboard or be flushed away into the drain. Next, powdered lime (CaO), available on hand (here, right outside the fume cupboard), needed to be applied to the spill area to naturalize the acid. The neutralized products could then be collected in polyethylene containers and saved for disposal (Pelchem, 2015). In the unlikely event that HF did come in contact with the skin, calcium gluconate gel, available on hand, should be massaged into the affected area and medical attention sought as soon as possible.

Table 1: Findings made during the hazard assessment.

No.	Causes	Con-sequences	Preventative/mitigation measures
1	Loading HF into Teflon bottle	Possible spillage of HF to drip tray in fume cupboard	<ul style="list-style-type: none"> • Drip tray in fume cupboard • All materials HF compatible
2	Fumes generated lead to explosive release of pressure	Possible injury to personnel	<ul style="list-style-type: none"> • Hole in top of Teflon bottle • Ventilation connected to fume hood and KOH scrubber • Two people present at all times • No operation when ventilation is off
3	High temperature leads to more HF fumes	Possible injury to personnel	<ul style="list-style-type: none"> • Experiment controlled at 25 °C • Immersion cooler with thermometer controls bath temperature • Built in thermometer in bath • Second cooler available • Scientist checks temperature once a day

4.2. Corrosion tests

A summary of the corrosion data collected from the different HF concentrations is shown in Table 3, and superimposed on to Figure 2. When HF comes in contact with mild steel, a corrosive reaction takes place, whereby a fluoride scale forms on the surface of the steel, and essentially protects the steel from corroding further (Hansen, 1996). This type of pre-passivation is applied in commercial HF production facilities by allowing the HF storage tanks to be attacked by HF and HF fumes for 24 hours prior to commissioning (Jennings, 2007). The coupons in the PICT experiments were pre-passivated in the same manner, so that the mild steel coupons, covered with the fluoride scale, would represent the starting condition of the steel in the corrosion tests and was inherent to mass loss and corrosion rate calculations conducted.

Table 3: Comparison between corrosion rates determined from PICTs and adopted corrosion data determined from literature (Honeywell, 2002)

Weight % HF	Average corrosion rate resolved from literature		Apparent corrosion rate taken on day 8 at 25°C in PICT	
	(MILS per year)	(mm/y)	(MILS per year)	(mm/y)
40	150	3.8	413.7	10.5
48	80	2.0	256.1	6.5
70	40	1.0	67.0	1.7

Honeywell fluorine products (Honeywell International Inc, 2014) is an HF supplier which documented the properties of HF to make technical and safety related literature openly available, to ensure safe usage of their products. From this, corrosion rates for carbon steel against HF concentration was provided and reported in MILS per year (1 MIL = 0.0254 mm). The values for average corrosion of mild steel could be converted and adapted so that apparent corrosion rates from PICT tests in this study could be compared (Figure 2).

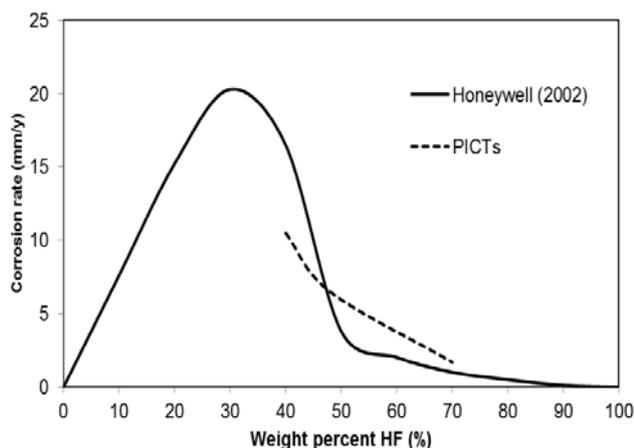


Figure 2: Corrosion rates of mild steel at 25 °C after 8 days from PICTs superimposed onto average corrosion rates of carbon steel vs. HF concentration, adapted from Honeywell (2002).

5. Discussion

5.1. Safety risk assessment

Findings made during the safety meetings were that the setup and ventilation in the laboratories were adequate for handling of the corrosion reactors up to 2.5 L of 70% HF per experiment, and that during the handling (loading and removal of coupons and waste), a second trained chemical worker needed to be present to assist during the experiment. This person was especially tasked with venting off HF fumes to the KOH scrubber during handling of the reactor bottles, or cleaning up any HF spills using powdered lime (CaO).

Recommendations made from the findings of the HAZOP study (Table 1) were that preventative measures were properly implemented. If any significant modifications needed to be made after this study was closed out, a separate procedural HAZOP study would need to be conducted.

5.2. Corrosion tests in HF

The corrosion safety risk assessment for testing in up to 70% HF was approved by the safety committee in November 2015 and registered with a unique document number in the company's archives. PICT tests commenced briefly afterwards, with corrosion data for 70% HF being generated effectively and safely, owing to the additional precautions identified during the safety risk assessment. The experiments were successfully conducted, and the results will be

presented in more detail elsewhere (Van der Merwe, 2016).

The effect of HF concentration on the corrosion rate of mild steel after 8 days was calculated using Equation 1. The results were related to average corrosion rates reported in the Honeywell (2002) special chemical edition where hydrofluoric acid properties were summarised (Table 3). On average, lower HF concentrations (between 5 and 50 % HF) produce significantly higher corrosion rates (>5 mm/y), while higher HF concentrations (>50 % HF) corrodes mild steel significantly slower (<2 mm/y). PICTs safely conducted over the HF range of 40 to 70 % showed a similar trend to Honeywell (2002) (Figure 2). However, corrosion rates differed slightly as the exact corrosive conditions (temperature, pressure and volume of HF used) was not known, and therefore could not entirely be reproduced.

6. Conclusions

- Since hydrofluoric acid is an extremely hazardous chemical which holds a significant safety risk to any operator, a complete safety risk assessment was conducted and all safety concerns associated with using fuming HF in corrosion tests were addressed. The experimental setup, methodology and safety precautions were approved by the PSC and corrosion tests with HF concentrations of no more than 70% was allowed to continue in the designated laboratories at Necs.
- The PICTs were the first corrosion experiments conducted in the experimental setup designed specifically for the safe execution of HF corrosion tests. The results from these tests served to successfully establish that corrosion experiments could safely be conducted in 70% HF for up to 8 days without losing significant quantities of HF (< 5 vol. %) due to strong ventilation.
- Corrosion rates determined for PICTs, for different HF concentrations, were relatable to average corrosion rate trends determined for mild steel over the same range reported by other HF suppliers. This proved that HF can be used safely to do corrosion experiments while producing reliable corrosion data.

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