HF Acid Alkylation Processes: Pump and Mechanical Seal Application and Design Considerations for Increased Reliability

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ABSTRACT

Hydrofluoric (HF) acid is an extremely corrosive solution commonly found in many oil refineries where it is used as a catalyst in the production of high octane gasoline blending stock. HF acid is almost synonymous with the alkylation unit, which is where the processing takes place within the refinery process flow. There are two main alkylation processes available, HF and Sulfuric acid; this tutorial will focus on HF alkylation and the challenges associated with handling this solution and the process streams that come in contact with HF acid. The HF alkylation process uses hydrofluoric acid which is dangerous and requires special treatment, particularly in the area of shaft sealing along with pump design and construction. While each individual facility may have its safety and reliability guidelines in place to manage HF acid and exposure to it, it is universally accepted in industry that minimizing personnel exposure to this fluid is a prime concern. This tutorial will attempt to address several topics centered on reliable operation of pumps in an HF alky unit, including pump and mechanical seal design and construction, along with mechanical seal support system considerations. In covering these topics, the tutorial will draw upon the combined previous experience of the authors in addressing these applications along with accepted good practices from relevant industry standards. The reader should review the content and consult as a reference, keeping in mind that not all of the content is applicable to every application and that each application should always undergo a thorough engineering review.

INTRODUCTION

In modern petroleum refining, the HF Alkylation Unit combines isobutane and butylene in the presence of HF acid and excess isobutane to form iso-octane, or alkylate. The alkylate will have an octane number of 95 or greater and is used as a lead free octane improver for gasoline. There were historically different licensors of the HF Alkylation unit technology, and the most common process flow is represented in Figure 1: note that depending on the unit licensor, the equipment utilized and process flow may differ slightly.

There are a number of different sources of Liquefied Petroleum Gas (LPG) providing feedstock for the alkylation unit in any refinery. The feedstock preparation should be flexible and able to accommodate varying sources, with the most common sources coming from a fluidic catalytic cracking unit (FCCU) or a hydrocracker. The goal of the pre-treatment process is to remove propane, purify butane, and remove water from the system. Beyond the pre-treatment stage, the processes cease being HF acid free environments until the final products are produced. The critical pump and seal applications in the process flow begin to take shape in the process streams of the
mixing nozzle, reactor riser, HF acid recovery, main fractionator top/bottom product, normal butane takeoff, depropanizer, and the acid stripper. The reaction of the HF acid catalyst in the reactor with the dry olefin and unreacted isobutane feed combine to form the alkylate product. In addition to the various fractionation processes utilized to separate the hydrocarbon streams, there are also scrubbing processes with potassium hydroxide (KOH) and sodium hydroxide (NaOH) which are used to remove traces of HF acid and organic fluorides prior to sending specific finished products to storage. In addition to alkylate, common product streams leaving an HF Alkylation Unit are normal butane liquid, LPG propane, and tar/acid soluble oil (ASO).

Figure 1: Common HF Alkylation Unit process flow

Figure 2: Top-top nozzle orientation sketch - vent and drain connections

PUMP TYPES AND CONSTRUCTION

Historically, the major HF Alkylation Unit technology providers have used special variations of API 610 Centrifugal Pumps and more recently, API 685 Sealless Centrifugal Pumps. Centerline mounted, single stage overhung (OH2) pumps and radially split, one and two stage between bearings (BB2) pumps are the most common pumps used in HF acid service. Specific to HF acid process, the suction and discharge nozzle orientation of top suction, top discharge is preferred for the OH2 pumps, but not specifically required by any particular design standard. Orientation of the suction and discharge nozzles in this manner facilitates having bleeders on the suction and discharge piping, eliminating casing vents which would be another connection on the casing where HF acid containing material could collect and cause issues. The orientation of the nozzles in a top-top manner also reduces pipe strain on the suction and discharge nozzles. Figure 2 depicts a recommended pump nozzle configuration that has been used successfully in many HF acid applications.

The materials of construction for a HF acid service pump would be considered a modified S-5 construction (refer to table H.1 in API 610 11th Edition). The S-5 construction utilizes a carbon steel pressure casing with Monel Alloy 400 (UNS N04400) impellers and throat bushings, Monel K500 (UNS N05500) shafts, B7M (ASTM A193 grade) studs with 2HM (ASTM A194 grade) nuts, and wetted hardware of Monel K500. Pure Monel castings (grade M-35-1) can be used but the size of the cast part can become cost and strength prohibitive. Cast Monel also has documented concerns with internal voids and defects showing up after the pump is placed in HF acid service, even if the casting passed a non-destructive testing (NDT) examination including X-Ray. Solid Monel Alloy 400 covers machined from a plate can be used effectively up to a 20” diameter; beyond this size and overlaid or cladded carbon steel is required to achieve the desired material strength for the pressure casing.

The key component associated with many HF acid service pumps is the use of a Monel overlay or cladding that is utilized in all areas of the pump that have fits in contact with the process fluid. Typical overlay areas would include the case and cover gasket areas, case wear ring area, center stage area when applicable, and any case drain threaded area (if allowed by the unit licensor). The overlay/cladding’s
purpose is to aid in disassembly of the pump. Recalling that the pressure casing material of HF acid pumps is carbon steel, when this material is exposed to HF acid over time iron fluorides will form and accumulate within the internal passageways of the pump. Accumulation of iron fluorides in critical fit areas with relatively tight tolerances and clearances between mating surfaces can have detrimental effects in reducing the clearances making disassembly very difficult if not impossible. The overlay/cladding on the other hand does not form iron fluorides, therefore utilization in the critical fit locations of the pump allows for disassembly and preservation of the critical fits as well as future equipment repairs. The use of Monel cladding is essential especially with respect to the case ring and throat bushing locations to avoid collapsing these components due to continual iron fluoride formation.

Monel cladding is accomplished by undercutting the carbon steel and applying a pure nickel butter layer (ERNi-1), followed by applying two passes of Monel Alloy 400 (ERNiCU-7) to provide an after machining thickness of at least 1/8” thick. The Monel cladding seems to retain material integrity better when applied past where it is required, otherwise the cladding can peel off due to corrosion and erosion. The stripping of this protective layer in key areas has been a re-occurring concern in HF acid service pumps for many years. In many cases, premature degradation of pump components has been attributed to high flow velocities and localized impingement in critical areas. While this concern is valid, the application of Monel cladding or overlays in critical fit areas is more crucial and issues with the cladding has been a contributing factor to reduced usability of some components.

As noted, while the base carbon steel material is acceptable for HF use, there is gradual iron fluoride formation over time due to reactions with the process. As scale breaks loose, more scale forms, and these free components can cause localized erosive damage once loose from the base material. Additionally, the iron fluoride formation can force its way under the cladding and actually force the cladding loose from the base metal. To this point, application of the cladding, including blending of the overlay to avoid sharp breaks in material boundaries (base metal and overlay) and extending the cladding beyond the required area are good measures to avoid reduced component life due to corrosive and erosive mechanisms. Figure 3 depicts examples of good Monel cladding applied to an HF acid service pump.

Figure 3: Monel overlay on pump head fit

Although suction and discharge flange cladding isn’t required by specific HF Alkylation unit licensors, it is recommended as the suction and discharge flanges have the most potential to have HF acid and water exposure, which can create diluted acid that is highly corrosive to the flange. In this instance, the overlay/cladding is only required in the gasket sealing area. When repairing Monel cladding/overlay, a hydrogen bake-out must be done before weld overlay is re-applied. Typical hydrogen bake-out takes place after undercutting the area to be overlaid. A hydrogen bake-out procedure is essential to drive out atomic hydrogen that has diffused into the steel casing, as the trapped hydrogen can lead to cracking in a weld or embrittlement of the casing material. Hydrogen bake-out requires heating the steel to an elevated temperature and allowing time for diffusion of the hydrogen out of the material. The hydrogen free material will be weldable at this point and ready to accept the procedure for overlay/cladding in critical areas.

Generally agreed good practices for HF acid pumps requires heating the pump case/cover to 450-600 °F (230 – 316 °C) and holding for a minimum of 2 hours. Once the bake-out has been completed, welding must be started within 1 hr. A local heat treat can be done, but typically results of welding and cladding are improved if the entire pump volute or head is included in the bake-out process. When considering repairs for the pressure containing parts of the pump outside of any overlay/cladding, a recommended practice is to pre-weld bake-out of the complete part being repaired at 450-550 °F (230 – 288 °C) for a minimum of two hours, then furnace cooled to 250 °F (120 °C) at a rate of 100 °F (38 °C) per hour.

It is recommended that the weld repair be performed using ER70S-3 wire using Gas Tungsten Arc Welding (GTAW) methods, maintaining 250 °F (120 °C) maintenance temperature. The use of the ER70S-3 wire is based on experience that some HF Alkylation unit users have documented as concerns with weld repairs of carbon steel cases.
using 7018 welding rods, as the 7018 was found to lift away once the area was exposed to HF acid. In one instance, a pump case repaired with 7018 passed both a helium leak test and inline static pressure test with alkylate seal flush applied to the casing for multiple days, only to develop a leak upon exposure to HF acid. The post-leak tear down and inspection found that the weld repair had separated from the base metal upon exposure to the HF acid in the process. If feasible, it is a good practice to overlay the repaired area with Monel to avoid potential issues in any case.

When speaking specifically about the internal wear rings within the pump, a good practice with regards to clearances is that pump wear ring and throat bushing clearances be maintained the larger of 0.025”-0.030” (0.63 to 0.76 mm) diametrical clearance or what is specified in API 610 11th Edition depending on the associated diameter. The larger clearances are required to avoid potential issues with wear ring or throat bushing collapse over time due to iron fluorides that may build up behind overlay / cladded areas. Metallic wear rings and throat bushings should be made from Monel Alloy 400, with the case rings coated with a coating such as Colmonoy 4, 5, or 6 in order to provide the 100 BHN difference in hardness between the mating wear ring or throat bushing collapse over time due to iron fluorides that may build up behind overlay / cladded areas. Metallic wear rings and throat bushings should be made from Monel Alloy 400, with the case rings coated with a coating such as Colmonoy 4, 5, or 6 in order to provide the 100 BHN difference in hardness between the mating wear ring or shaft/shaft sleeve. In many HF acid pumps, Teflon® PFA fiber reinforced composite materials have shown very good success for case rings and throat bushings although API 610 11th Edition does not address HF Acid applications directly and based on the unique nature of the fluid these applications would fall under the description of ‘Engineered Seal’. In order to accurately assess the application, one needs to consider the actual fluid being sealed in most HF applications as a function of the primary seal flush, which is typically going to be either isobutane, propane, or alkylate. Looking objectively at the fluid being sealed is useful in evaluating potential seal design configurations and it allows API 682 to be referred to for guidance in terms of general design criteria. API 682 would consider these fluids flashing hydrocarbons, in which case a Type A seal is recommended. A Type A seal is a pusher seal utilizing multiple springs and elastomer (O-ring) secondary sealing elements in a rotating or stationary seal head orientation.

Noted in a tutorial from the 32nd Pump Symposium, the requirements for effective sealing of very light hydrocarbons in terms of the mechanical seal is a maximization of both seal face stability and lubrication. Mechanical seals operating in the services described will do so with very little hydrodynamic load support due to the low viscosities in place. It is likely that the seal face in such applications will operate in a solid to mixed friction regime; in these operating regions, the face materials are likely to experience higher wear rates due to increased temperature (from rubbing friction) and potential break down due to hydrostatic loading of the faces themselves, which is a function of the very high pressures typically associated with these applications.

Further, balance ratio, which is dimensionless value associated with closing and opening areas of the seal face geometry, must be optimized to minimize the face generated heat and loading in order for the seal to have a reasonable chance to survive. For Type A mechanical seals, the balance diameter is typically the diameter of the sliding contact surface of the dynamic O-ring. In a Type B and C seal, the balance diameter is the mean effective diameter of the bellows core. Due to the nature of the bellows geometry, the design is considered inherently balanced at low pressure. As pressure increases, the balance diameter decreases to a degree determined by the temperature, material characteristics, plate thickness, and geometry of the core, which leads to a net overall increase in balance ratio and face load (Kalfrin 2016). Figure 4 highlights the balance diameter differences between the seal types.
MECHANICAL SEAL MATERIALS OF CONSTRUCTION

One of the key variables warranting consideration when applying mechanical seals to HF applications are the materials of construction. Critical to the material selection process is whether or not water is present and if it is, in what amount as the corrosive nature of HF varies accordingly. In an Alkylation process unit, fresh HF acid to the unit is anhydrous (no water present) and subsequently the vast majority of equipment and equipment component interactions will be with anhydrous HF acid. It is important to note that HF acid dissolved in water is one of the most difficult acids to handle and highly corrosive in very small concentrations. If HF acid would come into contact with moisture or water at any point during the process, corrosive damage would be substantial.

As noted by Wallace and Middleton, when seeking out reference sources for material compatibility in these services, some of the most reliable information on the corrosion resistance of materials to HF acid in addition to detailed specification requirements is derived from tests in a suitable loop in an actual HF-alkylation unit. The maturation and simplification of material selection in HF services is due in large part to not only advances in the materials themselves but also to well document the performance results of specific materials under service conditions. The unique properties of the process fluid make accurate ‘testing’ very complicated as under typical atmospheric conditions, anhydrous HF is bordering on a gas with a boiling point of 67.1 °F (19.5°C). Corrosion tests with anhydrous HF in a laboratory would require a very elaborate test with specific controls in place for accurate replication of live process conditions (Wallace / Middleton, 1997). There have been instances where end users have opted to submerge test pieces of various materials in live process streams to evaluate corrosion rates and compatibility over a specified period, and the data from such evaluations has been very useful. Tests with HF solutions in water are, on the other hand, comparatively easy based on the nature of the solution. To this point, when laboratory tests results are quoted in support of the HF resistance of particular material for a mechanical seal such as an elastomer or carbon grade it is very important to check whether the test had really carried out on anhydrous HF, otherwise these data should be questioned (Wallace / Middleton, 1997).

Seal Face Materials

As mechanical seal technology and material advancements have progressed over the years, the selection of suitable materials for chemically aggressive services such as HF acid has become more concise and no doubt continued material advancements will likely provide alternative options for various components. For the purpose of this tutorial, the focus will be more on the most common and widely acceptable materials for use in HF acid applications as opposed to archival data. Regarding mechanical seal faces, the most commonly used materials today are carbon-graphite, alpha-sintered silicon carbide, and nickel bound tungsten carbide (to a lesser extent). Carbon would be utilized as the softer of the two material parings in the interface as the primary, or spring loaded member. Based on the volatility of the process in question, there are specific requirements on the type of carbon grade to be used.

Chemically graphite is one of the most inert materials available. It is a good conductor of heat, a natural lubricant and has a laminar grain structure, allowing the individual grains to slide over each other. This laminar structure allows the release of graphite from the surface to be deposited on a counter-face. In the context of a mechanical seal, carbon is the prime material choice due to this very characteristic. During the production process impurities are removed from the raw material and then blended with additives and oxidation inhibitors to enhance the various properties of the base products, referred to as carbon-graphite. Pitch is commonly used as a binder, of which there are numerous chemical formulations. To render mechanical carbon-graphite materials impervious to various process variables, they are impregnated with various substances in order to achieve the required physical and chemical properties. Various additives include resins, waxes, inorganic salts, ceramics and molten metals. Unfilled or non-impregnated
carbon grades have low permeability and resistance to many chemicals and high temperatures. The production process involves repeated infusions with hydrocarbons which are carbonized resulting in dense carbon graphite that is extremely resistant to aggressive chemicals such as HF acid and these specialized grades are the only acceptable carbon face option for these services. While the removal of less chemically resistant additives from the base carbon structure make the material more chemically resistant, there are compromises made in other areas, particularly strength and rigidity that warrant a thorough evaluation of the seal design in these applications if carbon is considered.

Due to the typical high application pressures in many HF acid applications, material strength of a carbon face could be a concern. However, many successful and reliable seal designs in service in these applications have utilized chemically resistant carbon material and overcome the lack of material strength by modifying the primary ring geometry to be more resistant to pressure induced distortion. Aided by more advanced Finite Element Analysis (FEA) modelling techniques, optimized seal face geometry can be designed for the application conditions such that the desirable material properties can be maximized without compromises. Figure 5 is a side by side comparison of like chemically resistant carbon materials simulated at 70 F and 300 PSIG (21 °C and 20.6 BAR), sealing propane, which would be a very typical service condition for a HF acid seal. The modified geometry on the left exhibits an overall net decrease in pressure distortion over the geometry on the right. The modification yields an overall reduction in face temperature, prolonging seal life due to reduced wear and minimizing leakage at the same time.

It is very important that the seal face geometry be reviewed in advance of utilizing an ‘acid-grade’ carbon for these severe duty applications to maximize reliability of the design. Regardless of seal face geometry modifications and specialized material considerations, ultimately there is no carbon grade that is completely HF proof; some specialized grades are more HF resistant than others, but should these faces be exposed or immersed in the HF acid containing process, there will be corrosive wear. This fact ties directly into the importance of the API Plan 32 external flush to HF acid service seals.

Silicon carbide is another option for seal face materials in HF acid applications. Silicon carbide is an advanced ceramic material. The earliest type of silicon carbide available for use in mechanical seals was reaction bonded and developments have made a number of variations available. Silicon carbide is extremely hard, being highly wear resistant and with good mechanical properties. It has high temperature strength and thermal shock resistance, maintaining its high mechanical strength at temperatures as high as 2550 °F (1400 °C). Silicon carbide has higher resistance to chemical corrosion than other ceramics, but the free silicon present in reaction bonded silicon carbide will be attacked by caustics and strong acids, which make it not preferable for HF acid applications.

The only silicon carbide material that can be used in HF acid applications is sintered silicon carbide. Sintered silicon carbide is manufactured by compressing a blend of pure silicon carbide powder, with non-oxide sintering aids. Subsequently sintered using an inert atmosphere at temperatures around 3630 °F (2000°C). Sintered silicon carbide (also referred to as pressure-less sintered), has no free silicon present. Two grain structures are used in production, Alpha (hexagonal) and Beta (cubic), both being almost chemically inert to process chemicals, including aggressive acids such as HF acid. Figure 6 highlights the differences between the two silicon carbide materials in the finished surface and material structure. Usage of silicon carbide face materials may require additional design considerations into
the method of ensuring fluid film lubrication at the faces in seal flush fluids in HF acid services. Such modifications may be recesses, grooves, or other micro-surface treatments design to enhance lubrication and minimize frictional heat generation, which is recommended in hard face on hard face pairings.

Although less popular, tungsten carbide can be considered a hard face material option. Cemented tungsten carbides are derived from a high percentage of tungsten carbide particles bonded together by a ductile metal. The common binders used for seal faces are nickel and cobalt. The resultant properties are dependent upon the tungsten matrix and percentage of binder (typically 6 to 12% by weight per volume). Tungsten carbides have extremely high wear resistance and are very robust materials in general. Corrosion mechanisms give rise to surface depletion of the binder phase, allowing the carbide grains to become detached by wear processes. To increase corrosion resistance, the levels of nickel (Ni), chromium (Cr), and molybdenum (Mo) are increased, with the highest corrosion resistance obtained from TiC-Ni grades (titanium carbide – nickel). These materials can have lower strength and reduced thermal conductivity when compared to more traditional cemented carbide grades and silicon carbide as well. In many cases, the balance of the ability to transfer heat along with good chemical resistance often make silicon carbide materials more preferred options for HF acid services.

Metallurgy

As with mechanical seal faces, the material selection process with regards to seal metallurgy for HF acid services has become rather simplified over the years. Mechanical seal components differ from larger pressure containing pieces such as pump cases and vessels in that the allowable corrosion rates are significantly less by necessity. Loss of metal in seal glands and sleeves through corrosion mechanisms will at the very least make these components unrepairable, increasing overall costs. In addition, the corrosion of internal fits and support surfaces can cause seal faces to track improperly and ultimately the seal becomes compromised. For these reasons, mechanical seal metallurgy selection will typically default to much higher alloy metals with higher corrosion resistance. In the vast majority of HF acid applications, Monel Alloy 400 (UNS N04400) is used extensively for gland plates, collars and most adaptive hardware components.

One of Monel’s drawbacks is that it is comparatively soft when evaluated against more traditional seal metallurgy, so the use of Alloy 400 in thicker cross-section components like gland plates is ideal as it will be more resistant to deviations incurred from normal wear in operation. When considering other seal components, such as sleeves, drive or anti-rotation pins, and fasteners, alternative materials with increased hardness values would be desirable. In the case of the mechanical seal sleeve, there are typically thinner cross-section areas in contact with bushings and other contact surfaces where increased hardness and durability are required. In this case, Monel K-500 (UNS N05500) is an alternative option as the increased hardness over Alloy 400 makes the material more wear resistant and robust, especially in reduced cross-section components. The hardness increase is especially ideal in fasteners, especially seal drive collar set screws as the drive collar screw needs sufficient hardness differential (10 Rc typically) over the pump shaft material to effectively ‘bite’ into the shaft and transmit torque to the seal. As mentioned, the concern with softer alloy materials in seal sleeve construction can be distortion, especially if fasteners used to transmit torque to internal seal components are engaged over top of reduced cross section areas. Distortion or dimpling of the sleeve due to fastener engagement can actually impede cartridge seal installation onto the pump shaft. One design variation that has been successful has been to utilize keys instead of set screws for this purpose, avoiding the potential dimpling or distortion of the sleeve. Figure 7 displays an example of key driven internal seal heads in relation to the mounting sleeve in a cartridge seal design.

Secondary Sealing Elements

Considering Type A seal designs for these services, the majority of the secondary sealing elements in the mechanical seal design will be elastomeric components. There are very specific elastomer material grades that have shown to exhibit
good chemical resistance when in contact with HF acid; it is important to review elastomer selections as many generic grades will swell and degrade with prolonged exposure to the process. As in the case of most materials utilized in these services, it is beneficial to review an experience list of documented installations as test data outside of live HF alky unit can be questionable.

Speaking in generic terms, current and legacy HF alky unit design standards accept the use of perfluoroelastomer secondary sealing elements for mechanical seals. Perfluoroelastomers are the most chemically resistant elastomer available, combining the chemical and thermal resistance of polytetrafluoroethylene (PTFE) with the elastomeric properties of fluoroelastomers (FKM), becoming a fully fluorinated high performance polymer. Various compounds are available which are compatible with a wide chemical base and cover a temperature range 0 °F to 600 °F (-18 °C to 316 °C). For a time, specifically cured fluoroelastomers were being utilized in HF acid applications and they did exhibit good chemical resistance. However, the specific lead-oxide curing of the material and subsequent cost and environmental impacts have made these grades not viable in these applications any more.

MECHANICAL SEAL ARRANGEMENTS

Encompassing all local specifications and regulations regarding the use of various different seal configurations in HF acid service is well beyond the scope of this tutorial. It is beneficial to note key points from international specifications and recommended practices regarding pumps and mechanical seals in these services. For example, the API Recommended Practice for Safe Operation of Hydrofluoric Acid Alkylation Units (API RP 751) states that pumps in HF service should preferably have dual seals or should be of sealless design.

Single Seals

When discussing single seals in HF acid service, the best practice initially is for plant management to do a risk analysis and determine if the environmental controls in place are sufficient to mitigate potential effects associated with various failure scenarios. Fundamentally, all mechanical seals must ‘leak’ as a function of adequate seal face lubrication to mitigate wear and dissipate heat. In a single seal arrangement (one set of seal faces), the leakage from the process will move to lower or atmospheric pressure regions within the seal. At this point one must consider the nature of the leakage in this location, whether it is toxic or hazardous, and how more significant levels of leakage such as in a failure event are managed.

In HF acid applications, the potential impact associated with these considerations can be that much more significant, which is why single seal usage in HF acid service can be a very challenging topic to address. Since all HF alky unit design standards call for a API Plan 32 flush with no HF to always be used the only outside implication should be the external hydrocarbon fluid leaking to the atmosphere. While not ideal, provided the external flush is maintained there should be minimal implications to the outside environment, i.e. no HF acid containing material released.

In many early HF acid applications, the original seal configuration was a single seal with an external flush injection, or API Plan 32. The intent of the external flush injection is that during normal operation the mechanical seal faces are cooled and lubricated by a liquid that is “HF free”. This liquid is typically supplied from the unit, with isobutane and propane being popular choices. Aside from being not contaminated with HF acid, the fluid must be compatible with the process as the flow path into the mechanical seal will lead to injection to the process side of the pump. The operating principles of the single seal and external flush injection have typically been very reliable and all seal leakage would be of relatively safe fluid (compared to the process).

What is detrimental to the performance of single seals in these services are instances when there is an interruption or loss of the external flush, at which point the mechanical seal components are exposed to the process fluid containing the hazardous acid and subsequently corrosive attack of some or all of the seal components is inevitable. This scenario highlights the other purpose of the API Plan 32 injection in HF acid streams outside of lubrication – to serve as another layer of insulation between the hazardous process and mechanical seal components. It cannot be overstated that a reliable source of external flush fluid is critical to the success of a single seal and remains as such even when discussing more complex seal arrangements such as those discussed later in this tutorial. A typical Plan 32 piping example is diagramed in Figure 8.

Figure 8: API Plan 32 example piping diagram

Single seals have advantages in the form of reduced cost over a dual seal based on the relatively simple design and they also utilize a simplified support system. If considering the use of a
single seal in these services, it is recommended that the design incorporate additional features to enhance the performance. Such features might include a distributed flush injection for symmetrical face cooling and purging of the seal face area of vapor bubble formations, a segmented throttle bushing on the atmospheric side of the seal for additional protection in the event of a seal failure, and a reduced clearance throat bushing in the bottom of the seal chamber to restrict ingress of HF acid into the chamber in upset conditions and further maintain the insulation of the seal components from the process.

The throat bushing clearance should be sized for a flow velocity suitable for process exclusion and representative of a laminar flow regime. A good practice to adopt is to include the throat bushing as an integral component to a cartridge seal assembly so that the bushing replacement is guaranteed with each seal change. What hinders external flush effectiveness and leads to excess external flush fluid loss to process is worn throat bushing clearances; incorporation of the throat bushing into the seal cartridge helps ensure these clearances remain intact. Figure 9 is an example of a single seal design in an HF acid service that incorporates the integral throat bushing feature.

Even if the external flush fluid source is reliable, the cleanliness of the fluid must not be in question as fouled external flush piping can lead to degradation of the fluid flow rate and potential contamination of the seal components as well. Many times, the isobutane flush stream utilized in many HF alkylation plants tends to be more prone to fouling when compared to propane for example, but it really depends on the operational parameters of the unit in general.

External flush fluid screens and filters must have regular preventive maintenance (PM) intervals to minimize potential impacts to the seal. Figure 10 highlights external flush fluid contaminants and a carbon seal face after exposure to a trace HF acid stream that was the result of a loss of clean external flush. In many pre-existing installations, the contaminants are iron fluoride scale due to prolonged exposure of carbon steel hard-piping to the process conditions. In more recent installations and where practical, Monel tubing has been utilized for external seal flush interconnecting piping downstream of a filtration element to help mitigate this issue. If Monel tubing is utilized, it is recommended to be replaced each time it is disconnected (use once, then replace). Monel tubing, when exposed to HF acid and oxygen, is prone to stress cracking. The problem usually occurs after the tubing has been in service then opened up and re-connected; the dual exposure (HF and oxygen) and subsequent tightening of the tubing adds additional stress. This risk can be mitigated somewhat in seal flush tubing by using higher alloy isolation valves (Hastalloy C-276 for example) to minimize the length of tubing runs that would require replacement during normal maintenance.

Despite some noted advantages, the criticality of the API Plan 32 availability at all times, even before startup, at the correct pressure and flow rate can be significant detriments to its success. Once the API Plan 32 fluid injection is lost, HF acid will enter the mechanical seal area and could even enter the injection and any leakage past the seal faces at this point would contain HF acid. There may be a temptation to propose a single seal configuration for a service that is classified as containing ‘trace HF acid’, as the connotation of ‘trace’ suggests less severity. It is important to realize that in these streams the HF acid is immiscible with the hydrocarbon and the ‘trace’ of HF acid that is present will be a 100% concentration and not diluted. Additionally, actual percentages associated with ‘trace’ HF acid services vary from as low as 1% to greater than 6%, so clarification must be sought when reviewing applications with this designation.

Even if the exposure risk associated with the presence of HF acid was removed from the equation, a single seal in these applications would be required to seal a very high pressure, low viscosity, volatile light hydrocarbon with minimal normal leakage expectations. When the duty conditions are considered along with HF acid exposure risks, it is understandable to see why many end users opt for additional layers of leakage management and safety associated with multiple seal arrangements. Unless specific parameters have been met,
including a detailed risk assessment, single mechanical seals are not recommended for HF acid services.

**Dual Unpressurized – Wet Containment**

The natural progression beyond a single mechanical seal is to a dual mechanical seal. A dual mechanical seal is an assembly comprised of two sets of seal faces in which the orientation of the assemblies within the housing creates a cavity between the two seals. The cavity between the two seals can be wet or dry and either maintained at a lower pressure than the process pressure or at a higher pressure than the process pressure. Regardless of the configuration, the purpose of a multiple seal arrangement is management of leakage, and with an unpressurized seal the second seal is in place to capture inner seal leakage and aid in diverting this leakage to a safe location for disposal.

In an dual unpressurized seal with wet containment, the inner seal sees the higher duty as it must seal the differential between process pressure and the pressure within the containment cavity, which is slightly above atmospheric pressure and typically operating at the flare or vapor recovery system pressure. The outer, or containment seal is only sealing the differential pressure between the containment cavity and atmospheric. In this configuration it is important to understand that the inner seal is essentially acting as a single seal and so the heat generated by the inner seal must be removed and these faces lubricated by some means. This requires the use of an inner seal flush of sufficient lubricating properties injected at a pre-determined flow rate for the particular application. The requirement of a suitable flush for the inner mechanical seal in the context of an HF acid application defaults back to a reliable API Plan 32 system.

The outer or containment seal in this configuration is supported by an API Plan 52. API Plan 52 uses an external reservoir to provide buffer fluid for the outer seal of an unpressurized dual seal arrangement. During normal operation, circulation is maintained by an internal pumping ring. The reservoir is usually continuously vented to a vapor recovery system and is maintained at a pressure less than the pressure in the seal chamber. While this piping plan has been used successfully in many applications, the nature of the process fluid being sealed in this case makes the selection of this support system less desirable. While the inner seal does contain the higher pressure fluid and should be insulated from HF acid components by the external flush injection, should the inner seal be exposed to HF acid through an upset condition the possibility exists that the API Plan 52 system will be exposed to the same contaminants as they pass through to the buffer fluid system.

Incompatibility of the buffer liquid with the inner seal leakage flow is of a particular concern as contamination over time leads to the liquid buffer becoming an emulsion, typically losing its lubricating properties resulting in the outer seal performance to degrade. Once the buffer fluid is contaminated, it must be replaced and disposed of which requires maintenance intervention and the potential for personnel exposure to HF acid containing material. In fact, regular maintenance of the buffer fluid is required just to maintain a satisfactory level of performance in which case continued personnel exposure becomes a real concern. Buffer fluid compatibility and ideal fluid properties is a topic that has been addressed in many technical documents so it does not require repeating in this tutorial; however, in the context of HF acid services the use of Automatic Transmission Fluid (ATF) should be strictly avoided as the additives in the fluid will break down and polymerization occurs upon contact with inner seal leakage and hazardous constituents.

In addition to the fluid compatibility concerns, the overall system itself needs to be designed for compatibility with HF acid since the exposure risk is a legitimate concern. This requires a fluid reservoir constructed from Killed Carbon Steel or Monel for corrosion resistance along with the associated instrument connections, valve bodies, and trim components designed for exposure to HF acid as well. These material considerations alone will increase overall costs of the system aside from any additional testing requirements for the pressure vessel as mandated by local or industry specifications. The marginal reliability advantages in an HF acid application are outweighed by the significant potential disadvantages and for this reason liquid lubricated dual unpressurized seals are not commonly used in HF acid applications and are not recommended.

**Dual Unpressurized – Dry Containment**

In a dual unpressurized seal design with dry containment, the buffer fluid as described in the previous section is a gas and not a liquid. These dry containment seals are specialized configurations separated into two categories: contacting dry running and non-contacting dry running. In either case, the inner seal remains a wet contacting design that requires lubrication by a clean external flush similar to the single and dual unpressurized wetted seal. The contacting configuration is designed with special grades of carbon and engineered spring loads so that face wear is minimized.

The specialized carbon grade running against a corresponding silicon carbide face has a very low coefficient of friction and in conjunction with a modified spring load it can achieve long life. However, the same grade of carbon has a very limited corrosion resistance and potential exposure to HF acid is a prime concern. Additionally, while the coefficient of friction and wear is low with these seals, there is still wear and a finite life to consider and assurance of the containment seals ability to isolate more significant levels of inner seal leakage requires a regular testing interval for these seal types. A more comprehensive overview of
Dry running, contacting containment seals and subsequent testing protocols can be found in the proceedings from the 31st International Pump Users Symposium (Kalfrin / Gonzalez, 2015). The limited corrosion resistance of the carbon and the testing requirements make dry running, contacting containment seals less desirable options in HF acid applications.

Dry running, non-contacting containment seals do have significant benefits and have been supplied successfully in HF acid configurations with some modifications to the standard configuration. In a non-contacting containment seal, the faces utilize engineered recesses or grooves to generate hydrodynamic lift and subsequently create face separation. In this configuration, the non-contacting containment seal shares the same benefits as the contacting containment seal as the support system is greatly simplified and the need for an external reservoir as in the dual unpressurized wetted configuration is eliminated. In addition, the non-contacting design will generate no frictional heat in operation and experience near zero wear. This characteristic becomes advantageous in the event of an inner seal failure as the containment seal faces will positively seal off, isolating more significant levels of leakage.

Both the contacting and non-contacting arrangements are aided by the support of a continuous purge of the containment cavity (API Plan 72), which aids in forcing non-condensable leakage from the inner seal to flare (API Plan 76). The drawback with a conventional non-contacting containment seal is that with the hydrodynamic lift features being on the containment cavity side, the potential for significant leakage to the atmosphere in the event of a failure is increased as the features may ‘pump’ liquid from higher to lower pressure regions. Typical API Plan 72 and Plan 76 piping diagrams are outlined in Figure 11.

One adaptation to the dual unpressurized non-contacting containment seal that has been used successfully in many HF acid applications over the past seventeen years has been to orient the hydrodynamic lift generating face features of the containment seal on the face inner diameter, away from the process. This seal configuration utilizes a wet contacting seal as the primary seal, a non-contacting containment seal with active lift features on the face inner diameter, and a segmented carbon throttle bushing as a final seal between the containment seal and the atmosphere. The leakage past the inner seal is sealed by the dry running non-contacting seal that is designed to compress moisture-free nitrogen gas from the inside diameter to the outside diameter into the containment cavity.

The mixture of the nitrogen with the vaporized flush leakage is then vented to the flare or vapor recovery system. Nitrogen is supplied outboard of the dry tandem seal between the segmented bushing which restricts its flow to the atmosphere; the dry running seal faces incorporate a series of active lift grooves, which are designed to operate on a thin film of gas. The grooves are configured to pump from inside diameter (ID) to the outer diameter (OD) of the seal faces. This nitrogen quench supply ensures an inert gas film, of which an extremely small amount is vented to the flare or vapor recovery system. This initial concept and subsequent testing was discussed in detail in the proceedings from the 20th International Pump Users Symposium (Wasser, et al 2003). An example of the modified dual unpressurized design described is depicted in Figure 12.
Figure 12: Alternative wet / dry containment with active lift and temperature monitoring

The adaptation of the active lift grooves and low pressure nitrogen make the utility requirements of this configuration minimal and nitrogen consumption rates would be significantly reduced over a conventional API Plan 72 buffer gas injection. The injection of the nitrogen gas to the inner diameter of the containment seal would make the piping plan more of a quench than a purge, although the function of the containment seal design to move the gas to the containment cavity could add confusion when reviewing the piping plans associated with the design. API 682 does make provisions for modified piping plans such as this, allowing for coverage under Plan 99 designation. A Plan 99 is simply an engineered piping plan that is not defined by any of the existing plans in the standard, fitting as HF acid services would usually fall under ‘engineered seal’ applications.

API 682 does not have any specifications for a Plan 99, but it does state that the Plan 99 description and requirements must be clearly defined in specifications outside of the standard. It is not sufficient to indicate “Plan 99” on a seal data sheet or even on a seal layout drawing as a lone descriptor. A drawing of the Plan 99 and notes about its operation should be supplied. A good practice when adopting this designation would be to include a descriptor along with the “99” designation to provide clarity if the proposed piping is a variation of an existing piping plan, such as Plan 72 (99), for example.

The advantages of the design shown in Figure 12 are noticeable in terms of reduced complexity in the overall support system design and enhancements to the monitoring of the containment seal condition in terms of both pressure in the containment cavity and temperature of the containment seal faces. More recently, additional treatments to the inner seal faces in the form of micro-surface structures to reduce interface frictional generated heat and minimize leakage have been implemented to enhance the performance of the seal faces in this configuration. As noted, the basic sealing challenge to overcome with these services is the sealing of poor lubricating fluid, so generating sufficient fluid film support is fundamental to a reliable seal design in these applications. Description of one such treatment that has been incorporated successfully to HF acid applications was first described in the proceedings from the Eleventh International Pump Users Symposium (Wallace / Muller, 1994).

**Dual Pressurized**

Dual pressurized seal configurations utilize two sets of seal faces contained within one housing where the cavity between the two seals is maintained at a pressure higher than that of the fluid within the pump (process pressure). In this configuration, both sets of seal faces are lubricated by the fluid between the two sets of faces, which is referred to as a barrier fluid. Dual pressurized seals, or API 682 Arrangement 3 designs, have distinct advantages in hazardous applications such as HF acid service in relation to not only safety aspects but the ability to seal a difficult or poor lubricating fluid.

From a safety perspective, the leakage past both sets of seal faces would be that of the barrier fluid, so provided the barrier fluid is not a Volatile Organic Compound (VOC), the leakage to the atmosphere will be an inert, non-toxic substance. Similarly, leakage to the process side will be that of the barrier fluid as well, which isolates the pumped product completely from the atmosphere. As the barrier fluid pressure is maintained higher than the process pressure, the lubrication concerns associated with sealing light hydrocarbons are usually eliminated as the base criteria for barrier fluid selection be good lubricity and sealing properties.

Dual pressurized seals are commonly available in either Face-to-Back (FB), Back-to-Back (BB), or Face-to-Face (FF) configurations (see Figure 13). While all three configurations have benefits, speaking specifically in the context of HF acid applications, the BB or FF configuration would be recommended. This recommendation stems from several driving factors, one of which being that based on the higher pressures typically associated with the process streams and the need to pressurize the barrier fluid above these sealed pressure values, a FB configuration would have higher pressure at the inner seal face inner diameter, loading the face materials in tension. This could compromise the face materials from not only a stress perspective, but also from a fluid film lubrication standpoint where the inner seal interface becomes isolated from the barrier fluid due to combined thermal and pressure distortion of the face components (both forces acting in the direction of the applied pressure from the inner diameter). This phenomenon is more prevalent with hard face material combinations, which are likely to be utilized in HF acid applications for chemical compatibility purposes as previously discussed. Another area of concern with the FB configuration is that there is increased potential for thinner cross-section components to be exposed to the corrosive process; this concern is minimized with a BB or FF seal configuration.
The BB or FF dual pressurized configuration is advantageous as the barrier fluid is circulated around the outer diameter of both sets of seal faces. This logic allows for better circulation of the barrier fluid and more effective cooling and lubrication of the inner seal when compared to the FB configuration. The injection of the higher pressure barrier fluid around the outer diameter of the seal faces loads the components compressively as well, allowing for better net overall distortion resistance. Even with a pressurized barrier fluid arrangement, the addition of an external API Plan 32 is recommended to serve as an additional ‘barrier’ between the HF acid containing process fluid and the seal components.

A circulation of a clean external fluid between the seal components and the process also mitigates the formation of iron fluoride scale in these critical areas that could cause potential hang-up of the faces. While the inclusion of an API Plan 32 is recommended with a dual pressurized arrangement, the seal is not dependent on the external fluid injection for face lubrication as that is handled by the barrier fluid. To this point, the seal is less susceptible to wear as a result of interruptions in the external flush supply, unlike the single seal or dual unpressurized designs.

The typical support systems utilized with dual pressurized seals in HF acid services have traditionally been either API Plan 53A, B, or Plan 54. Unlike API Plan 53A that incorporates a pressurized reservoir within the circulation loop, API Plan 53B has only piping and an air or water cooled heat exchanger within the closed loop circuit. Some installations have used finned tubing as the “heat exchanger”. Liquid replenishment to this circuit is provided by a pre-pressurized bladder accumulator. The basic setup is comprised of two parts: the closed loop circulating system and the bladder accumulator. Seal performance is monitored by pressure decrease and not by barrier liquid volume as in API Plan 53A. Flow in the circulating system is induced by an internal pumping device or by circulating pump in the associated piping in some cases.

API Plan 53B is advantageous in that the barrier fluid is not in contact with the pressurized gas, so there is no concern over gas entrainment in the barrier fluid that can then come out of solution at the seal faces. API 682 cautions against using gas pressurization in direct contact with the barrier fluid when the reservoir pressure is above 150 psig (10 barg); user installation experience and independent mechanical seal manufacturer testing has shown that this value can be increased to 300 psig (21 barg) as long as the barrier fluid temperature is less than 250 F (120°C). Some typical API Plan 53A and Plan 53B piping configurations are outlined in Figure 14.
In either support system, the means of pressurizing the barrier fluid requires either a reliable high pressure nitrogen supply or a supplemental booster system to increase available nitrogen pressure to the values dictated by the application conditions. In pressurized arrangements, the desired barrier pressure set point should be the greater of either 30 PSIG (2 barg) or 10% above the seal cavity pressure, which in this case becomes a function of the external flush supply pressure acting upon the throat bushing within the seal chamber. In HF acid applications, it is important to understand potential variations in all system pressures so that the barrier fluid set point can be accurately established to minimize the potential for loss of process containment and HF acid exposure to the atmosphere. As identified with a dual unpressurized wetted system, the material compatibility concerns associated with the support piping and instrumentation with the pressurized wetted systems are of primary importance, and can increase overall system cost and complexity as result.

Additionally, with the barrier fluid pressure being maintained at a higher value than the seal chamber pressure, there will be a need over time to replenish fluid lost through the inner seal just as a function of normal seal leakage. Replenishment of barrier fluid in a pressurized system requires careful consideration to minimize personnel exposure to not only higher pressures but also a hazardous process in this instance. Many pressurized systems have been equipped with automated top-up or filling units that function to replenish the lost barrier fluid without the intervention of operations or maintenance personnel. When considering an API Plan 53A or 53B system with automated make-up, the complexity and operability of the system needs to be carefully reviewed to ensure all process hazard analysis (PHA) scenarios are being addressed.

API Plan 54 utilizes an external source to provide a clean pressurized barrier fluid to a dual pressurized seal. The API Plan 54 “system” supplying the barrier fluid can range from a process pump in the unit providing clean cool lubricant under pressure to a simple lubrication system with minimal components to an elaborate large system with many ancillary components and redundant systems to safeguard and alarm against malfunctions and process upsets to a controlled process stream. The designation of API Plan 54 only means that the dual seal is supplied with pressurized barrier from an external source and does not describe any specific system details. A generic depiction of API Plan 54 is shown in Figure 15.

While there have been stand-alone lubrication systems utilized in HF acid applications, what is more commonly seen in many HF alky units is a dedicated barrier fluid loop where the barrier fluid is pressurized by dedicated process pumps with distribution to multiple pumps and seals within the HF alky unit. In one particular example, the seal support system utilized a combination of an API Plan 32 using isobutane with a pressurized barrier fluid of alkylate in the API Plan 54 loop. The isobutane injection was supplied to the cavity between the inner seal faces and the process fluid to keep HF acid material away from the seal components; the pressure of the isobutane injection was in the range of 290 PSIG (20 barg). The alkylate barrier fluid was taken from the Iso-stripper tower at a pressure of 150 PSIG (10 barg), which was suction pressure for two dedicated flush pumps. The pumps increased the alkylate pressure to 350 PSIG (24 barg) where it was then filtered and supplied to a distribution header that fed multiple pumps in the unit, with the outlet from each mechanical seal routed to a return header back to the suction source of the flush pumps. Figure 16 is an example of the localized barrier fluid piping at each seal in this particular configuration, which is a common design certain legacy HF Alkylation unit licensors, especially in high acid containing pumps.

Figure 16: API Plan 54 piping example

Flow rate is controlled with the globe valve downstream of each seal, with the local flow meters register flush flow in and
out of each seal; a significant difference in flow rates would be a first sign indicator of potential inner seal leakage. The excess flow valve was designed with the intent to close off Alkylate barrier fluid flow in the case of a catastrophic seal failure while the inclusion of the restriction orifice was designed to limit excess flow of alkylate and maintain back pressure on the seal. In the instance of a unit-wide barrier fluid system such as this, the user must accept barrier fluid leakage into the process stream and focus on monitoring flow rates on a macro-scale initially, then isolate troubleshooting to localized areas to determine which seals may be leaking more than predicted or acceptable. In such an arrangement, it is also useful to monitor header pressure in several locations around the piping loop to make sure system integrity is maintained and no loss of containment goes un-noticed. A suggested check list when considering a piping arrangement such as this one would be as follows:

- Monitor total barrier flow rate from supply pump discharge – trend to DCS for increases in flow (flow meter and transmitter).
- Utilize local vortex meters and flow transmitters at each mechanical seal – trend inlet and outlet flow to DCS; 2 GPM decrease across the seal as an indicator of a first sign of trouble.
- Monitor barrier fluid header pressure at several points – provide indication of loss of pressure, i.e. busted pipe, leaking seal, etc.
- Consider low pressure alarm on the barrier fluid header – low alarm set point would be dictated by the downstream pump and subsequent seal arrangement with the highest pressure requirement.
- API Plan 32 and Plan 54 components mounted with sufficient space for equipment access; consider unitized mounting on a panel for a cleaner installation (see Figure 17).

An example mechanical seal configuration utilized with the API Plan 32 and 54 piping configuration is shown in Figure 18. In this example, the mechanical seal is oriented back-to-back with an integral throat bushing supplied to the seal cartridge downstream of the API Plan 32 injection connection. The advantages of mounting the throat bushing in the seal gland were discussed in the single seal section and would be applicable in this case as well as a good practice. Note that in this particular configuration, no internal circulation device is required based on the forced barrier fluid circulation by the external pumps.

Figure 17: API Plan 54 – panel mounted components

Figure 18: Dual pressurized with API Plan 32 / 54

CONCLUSIONS

There is an extensive list of end user applications in HF Alkylation Unit technology and through the application of industry accepted standards for pumps and mechanical seals, along with specific HF Unit licensor guidelines, sound and reliable solutions to these critical services can be applied. There are many older legacy installations that may benefit from some of the technologies described in this tutorial as the benefits have been well documented. The key statement with these hazardous applications is just that – documentation. While no doubt there will continue to be advances in materials and technologies associated with handling these processes, it is important to remember the criticality of the equipment handling this process and be mindful that any new technology or material be evaluated thoroughly and ideally supported with a well-documented history of success in other HF Alkylation Unit services. The intent of this tutorial was to provide an overview to those individuals less familiar with the application of pumps and mechanical seals in HF Alkylation Unit services and is not intended to be a comprehensive design guide, but rather serve as a supplemental aid in concert with specific design standards focused on this processing technology.
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ACKNOWLEDGEMENTS

The authors would like to thank John Crane and Monroe Energy LLC for support in the development of this tutorial.