Neutralising amine selection for crude units

Making a proper selection of neutralising amine chemistry is a challenging task

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Even though crude units have been around for a long time, maintaining long term reliable operation continues to be a challenge. The unit design, operating conditions, feedstocks, and corrosion control programme must all work together to achieve reliable operation (see **Figure 1**). If any part of the system is out of sync, the whole unit will suffer. With constantly changing feedstocks and operating conditions, as well as some unique challenges posed by many opportunity crudes, it can be a challenge to keep everything running properly.

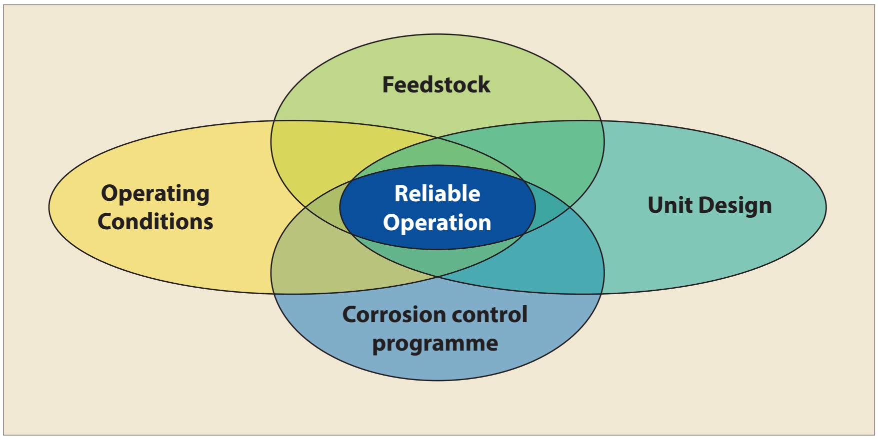

Figure 1 Factors influencing crude unit overhead corrosion and their interaction

The neutraliser system is a critical part of the corrosion control programme on crude tower overhead systems. This article will focus on understanding and applying some basic concepts to the design of neutraliser systems and on some more subtle aspects of neutraliser selection that are often overlooked.

Corrosion control basics

Some of the residual salts remaining in desalted crude hydrolyse to form HCl in the crude furnace. The HCl then ends up in the overhead system of the atmospheric tower. There can also be light organic acids in the crude oil as well as light organic acids formed from the thermal degradation of naphthenic acids in high TAN crudes, which also put additional acids into the overhead system. Neutraliser demand can easily increase by 50-100% when switching to a high TAN crude slate. Opportunity crudes often are difficult to desalt, which increases the amount of HCl formed. They may also contain tramp amine byproducts of H_2S scavengers.

If nothing is done to neutralise these acids, the condensation of steam in the overhead system will produce a highly corrosive, very low pH steam condensate stream. To control pH and reduce corrosion, bases such as ammonia or amines (referred to commonly as neutraliser or neutralising amine) are injected into the overhead system. If these neutralising compounds are improperly employed, they can also lead to fouling and/or corrosion through the formation of ammonium chloride or amine hydrochloride salts.

To prevent formation of salts and to quickly dilute any acids present, a water wash stream is often injected into the overhead system. The water wash, which is typically water recycled from the overhead accumulator, can be injected into the overhead line or into the inlets of heat exchangers. The net water from the overhead accumulator contains a mixture of organic and inorganic acids and bases. This water is commonly used as wash water in the desalter, which provides a potential route for these acids and bases to get back to the crude tower. This recycling of amines will be discussed in more detail in the desalter section.

Neutraliser selection primer

Neutralising amine selection is typically based on a few key parameters. Basically, the neutralising amine must be a strong enough base to raise the pH at the water dew point enough to control corrosion at acceptable levels. It also must not form salts ahead of the initial point where free water is present, either from condensation or injection of water wash. It should be readily available at a reasonable price. Petersen, Lordo, and McAteer[1] go into more depth on this subject.

The nature of any neutraliser salts that might form is an important consideration in neutraliser selection. Some amines, such as ethylene diamine (EDA), will form a solid salt which is a fouling and under deposit corrosion concern. Other amines, such as monoethanol amine (MEA), form liquid salts at typical process conditions. The liquid salts tend to corrode more aggressively than solid salts. Liquid salts tend to be more of a corrosion concern with some fouling potential from the corrosion products that form, whereas solid salts tend to be more of a fouling concern with some level of under deposit corrosion also occurring.

The salt point, that is the temperature where the chloride salt of the amine begins to form as a free phase, is another important consideration in neutraliser selection. The goal is to select an amine neutraliser that does not begin to form a salt ahead of the water dew point. After the bulk water dew point is reached, the water solubility of the salts precludes formation of a separate salt phase. This approach to neutraliser selection provides the pH control desired without the formation of corrosion and fouling inducing salts. In the absence of a water wash it can be challenging to find an amine that will actually accomplish its mission without forming salts.

Because the amines tend to behave independently of each other in forming salts, amine blends are sometimes used to reduce the salt formation temperature. For instance, a 50/50 molar blend of two different amines would cut the amine partial pressure in half compared to the use of 100% of either amine. Some suppliers may use five or more amines blended into a neutraliser product.

For any given amine, the salt point or sublimation point is defined by this equation:

$$K_p = P_{amine} * P_{HCl} \qquad [1]$$

K_p is the dissociation constant for the salt, which is a strong function of temperature. P_{amine} and P_{HCl} are the partial pressures of the amine and HCl respectively. The value of K_p defines the maximum amount of amine and HCl that can exist in the vapour phase at that temperature. If the product of the partial pressures of the amine and chloride present is greater than the dissociation constant K_p then salts will form. If the temperature where the salts form is ahead of the bulk water dew point then a corrosive salt phase will form. Ironically, the product used to control corrosion can help cause corrosion if misapplied.

The following discussion covers a number of different crude tower design and operating parameters that can impact neutraliser selection:

- Overhead water wash vs no water wash
- Tramp amines
- Overhead temperature
- Desalting

Water wash vs no water wash

The use of an overhead water wash typically increases the flexibility the refinery has when it comes to selecting a neutraliser. The water wash forces an immediate water dew point, which greatly reduces the risk of salt formation that could otherwise occur during the more gradual cooling that happens in overhead condensers. With no perceived risk of salt formation, refiners and chemical suppliers are typically happy to use a stronger base with higher salt forming tendencies because these neutralisers are cheap and effective for the intended purpose of neutralising HCl. Water wash also reduces the risks associated with changing process conditions such as temperature and chloride concentrations, assuming that the water wash rate is adjusted to account for upward shifts in overhead temperature. When water wash is injected close to the neutraliser injection point, there is practically speaking no amount of neutraliser that could be added which would lead directly to salt formation in the overhead system.

There is a risk, however, that is often overlooked. This is the risk of salt formation in the atmospheric tower when overhead water is recycled back to the desalter. Amines are organic bases, and as such they have at least some affinity for the hydrocarbon phase. How much of the amine partitions into the crude oil rather than the water is primarily a function of the distribution coefficient and the relative rates of crude oil and wash water. Water entrainment from the desalter also increases the effective amount of partitioning to the desalted crude.

The distribution coefficient, D, is the ratio of the solute concentration in octanol to the solute concentration in water:[1]

$$\log D_{\mathrm{oct/wat}} = \log \left(\frac{[\mathrm{solute}]_{\mathrm{octanol}}}{[\mathrm{solute}]^{\mathrm{ionized}}_{\mathrm{water}} + [\mathrm{solute}]^{\mathrm{neutral}}_{\mathrm{water}}} \right) \qquad [2]$$

The distribution coefficient is sometimes also called the partitioning coefficient, although strictly speaking the two are not the same. The partitioning coefficient calculation is the same as the distribution coefficient except that it does not include ionised forms of the solute in the calculation.

If enough amine partitions to the desalted crude, then it is possible to form an amine chloride salt in the atmospheric tower. Amines which have a high partitioning coefficient, and/or those with higher natural salt points, increase the probability of having salt formation via this recycle route. The effect of desalter operation on amine distribution is discussed later.

The different risk related to water wash is the effect of water carry-over from the overhead accumulator back to the atmospheric tower via the reflux. This risk is especially a concern when water wash is started for the first time or the rate is substantially increased in an existing unit. A trend in recent years at many US refineries has been to process increasing volumes of very light shale crudes and condensates. Increased overhead and wash water rates that result from these feedstock changes will increase the load on the overhead accumulator, which can lead to increased water carry-over.

Entrained water from the accumulator carries dissolved salts with it. When the salts are carried back to the tower with wet reflux, they tend to deposit on the trays as the water boils off. Neutraliser selection can impact how the resulting problems manifest themselves (corrosion or fouling). Changing neutraliser will not typically undo any fouling that has occurred prior to the change, although it can stop it from getting worse.

For units with no water wash the risks are a bit different. The primary risk of salt formation is in the overhead system itself. Without a water wash to force the dew point, the salt forming characteristics in the overhead system are closely linked to the type and amount of neutraliser used as well as the amount of chlorides present. As these values go up and down, the corrosion risk is constantly changing as well.

From a practical standpoint, there should never be more neutraliser recycling back to the crude tower than is present in the overhead system, so the highest risk for salt formation is in the overhead system and not the tower when no water wash is employed. When trying to use an ionic model to estimate the salt point, great care must be taken to use the correct quantity of amine.

One approach is to measure the actual amine concentration in the overhead accumulator boot water, but most refineries do not have the capability of doing that analysis. If significant salt is forming in the overhead system, the overhead water, however, will not reflect the total amine present because some of it is depositing as salts in the condensers. A second approach is to use the measured chlorides and the actual neutraliser injection rate. This approach, however, does not take into account the recycle of neutraliser back to the crude tower via the desalter or reflux (either via entrained water or neutraliser solubility in hydrocarbon). For units that use an alkanol amine (such as MEA) and have desalters that operate with an acidic pH, this second approach may be reasonable. For units that have desalters operating at high pH and/or that use alkyl amines in their neutraliser, this approach can lead to under-prediction of the salt point and the resulting overhead corrosion risk. In this case, a third approach is to use the actual neutraliser injection rate plus an estimate for the amount of amine recycle based on desalter operating conditions and/or estimated recycle via reflux. The best (most conservative) overall approach is to use the larger overhead amine rate as estimated from both the actual injection plus estimated recycle and amine rate estimated from the boot water concentration plus estimated recycle.

This latter approach should provide the most realistic estimate of the real salt formation potential of a given neutraliser. Any neutralising amine recycle reduces the amount of neutraliser needed, so in the case where neutraliser demand is being estimated based on the amount of acids present, such as in a chemical bid process, the amine recycle usually does not need to be accounted for except to the extent that hydrocarbon solubility renders the amine unavailable to neutralise acids in the water phase.

Tramp amines

Tramp amines can make it into the crude unit from four primary sources – slop, make-up water to the desalter, neutralisers in the steam supply, and H_2S scavenger treated crude. Slop systems are most likely to pick up the amines used in the refinery treating systems – typically MEA, DEA or MDEA. Similar to slop, treating amines can end up in the sour water system and make it to the crude unit when stripped sour water is used as desalter wash water. Neutralisers are amines added to steam to prevent acidic condensate formation. For best control and troubleshooting, different amines should be used in the steam and crude overhead neutralisers.

Crude oil that has been treated with triazine will contain either MEA or methyl amine depending on which amine was used to manufacture the product. MEA is by far the most common amine found in H_2S scavenger treated crude oil today. Triazine is also sometimes used to reduce the H_2S load to the flare stack. Great care should be taken to ensure that the MEA byproduct of flare system scavenging cannot end up back in the crude unit.

Amines mostly behave independently of each other when calculating salt points, so with regards to neutraliser selection the primary concern is when the tramp amine is the same as one of the amines in the neutraliser. In that situation, the amine sources can become additive in nature, thus increasing the salt formation risk. For instance, in a crude unit that uses a neutraliser containing MEA, the MEA partial pressure is calculated based on the MEA in the neutraliser plus the MEA entering the crude tower via tramp sources.

MEA is the primary amine that is common as both a tramp amine and a neutralising amine. Refineries that use MEA in their amine treating system or which have problems with H_2S scavengers in their crude supply, should avoid the use of a neutraliser product containing MEA. In addition to the salt formation risks mentioned previously, having multiple sources of MEA can make the challenge of troubleshooting MEA related contamination problems difficult.

Overhead temperature

Overhead temperature plays a dual role in impacting amine behaviour in a crude column. Firstly, the amount of amine that can be present in the overhead system before salts form is a strong function of temperature. Secondly, the temperature can have a significant impact on the amount of hydrocarbon leaving the top of the column.

K_p is a function of temperature for ammonia.[2] Over the temperature range from 200 to 300°F where most crude tower overheads typically operate, the value for K_p increases by roughly an order of magnitude for every 30°F increase in temperature. For constant pressure, vapour rate, and chloride content, this change in K_p with temperature means that every 30°F increase in overhead temperature would allow a 10x increase in ammonia before solid ammonium chloride would start to form. While the absolute values of K_p will be different for amines, K_p would still be expected to undergo similar relative changes with increasing temperature. Ionic modelling can be used to quantify this effect.

As the crude tower overhead temperature increases, the total volume of hydrocarbon going overhead would be expected to increase at a constant crude charge rate and composition. The increase in overhead hydrocarbon decreases the partial pressure of any amines and HCl present, which has the effect of decreasing the potential for salt formation. The effect on salt formation potential of increased hydrocarbon decreasing contaminant partial pressures is normally much

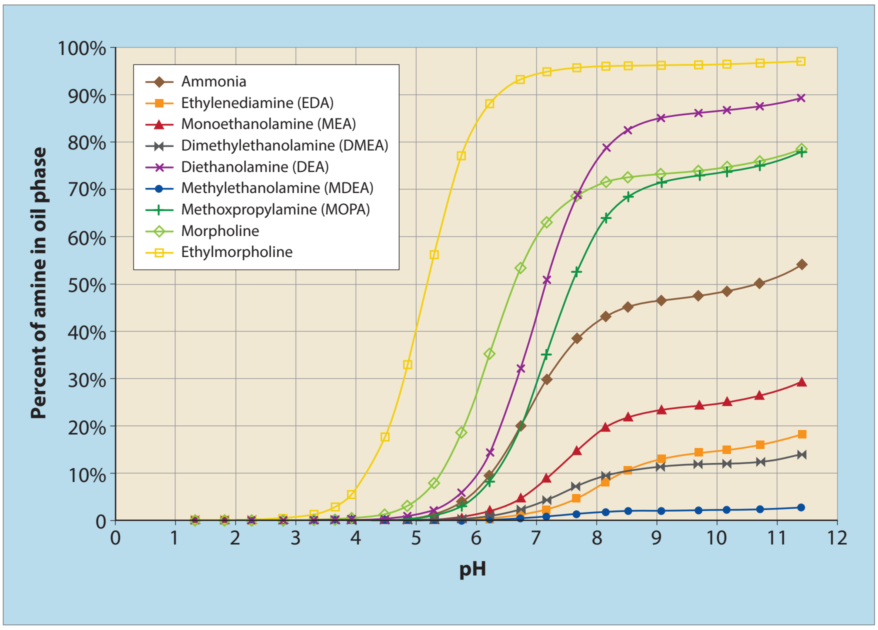

Figure 2 Desalter partitioning characteristics of some common amines
Taken from An In-Depth Look at Amine Behavior in Crude Units Using Electrolyte-Based Simulation, NACE Paper No. 05570.3

smaller than the impact of increasing temperature on K_p. The inverse, however, is also true. Decreasing the overhead temperature decreases the hydrocarbon partial pressure (increases P_{HCl} and P_{amine}) and at the same time decreases the value of K_p (decreases the amount of allowable HCl and amine before salts start to form).

In terms of neutraliser selection, the lower the overhead temperature is, the less flexibility there is in choosing a neutralising amine. For a low crude tower overhead temperature, a neutraliser with a strong affinity for the aqueous phase in the desalter and a low salt formation potential (high value of K_p) is required. Often times in such systems these dual objectives cannot be met with a single amine so the chemical supplier is forced to use a blend of amines to minimise salt formation risk in the crude column. Blended amine products are typically significantly more expensive than single amine products, but the costs are normally easy to justify based on the improved operating flexibility. Blended amine neutralisers are especially valuable to the refiner trying to maximise diesel production, which normally involves operating the crude tower overhead temperature as low as possible to minimise naphtha yield. Some refineries will use a lower cost neutralising amine when the refinery is operating in a maximum gasoline mode and an amine blend when in a maximum diesel mode to lower neutraliser cost without substantially increasing corrosion risk.

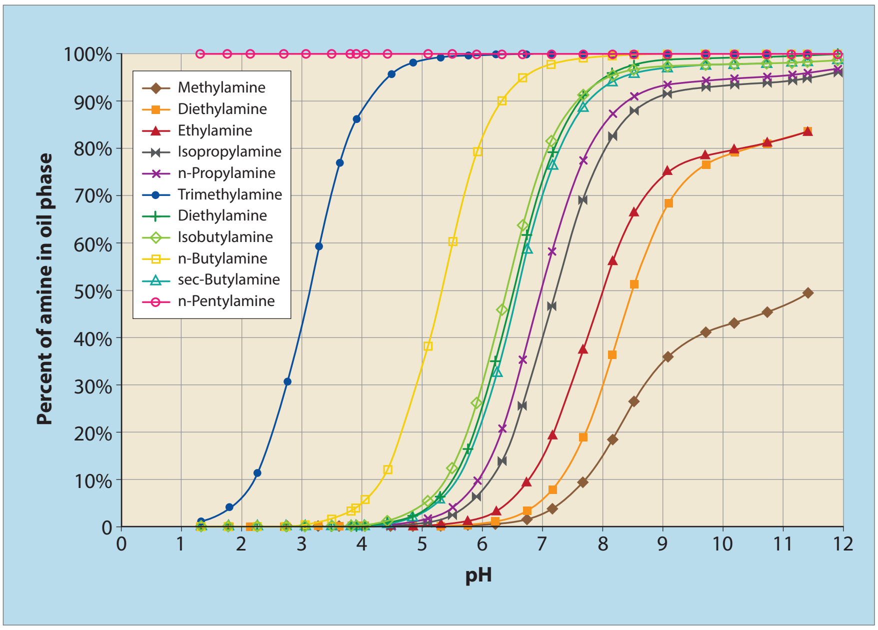

Figure 3 Desalter partitioning characteristics of common alkyl amines
Taken from An In-Depth Look at Amine Behavior in Crude Units Using Electrolyte-Based Simulation, NACE Paper No. 05570.3

Desalting

As mentioned previously, when atmospheric tower overhead water is used as desalter wash water, some of the amine will partition to the crude oil phase in the desalter and end up in the atmospheric tower. Routing the overhead water to the sour water stripper (SWS) can greatly reduce the risk through dilution, but it will also consume additional energy and SWS capacity. If stripped sour water (SSW) is then used as desalter wash water there will still be neutralising amines in the SSW, but at a lower concentration than if the overhead water were routed directly to the desalter. The neutralising amines will have been diluted by other water sources feeding the SWS.

Figures 2 and **3** show the typical partitioning behaviour of a variety of amines in a desalter.[3] The actual percentage of a given amine that goes out with the desalted crude will depend on the amine, the percentage of wash water used, the temperature in the desalter, and the pH of the desalter water. The crude oil used may potentially have a small impact as well. These figures are indicative only because they were generated for a system with a specific hydrocarbon composition, percentage of wash water, and temperature.

These figures clearly show that both the pH and nature of the amine compound play critical roles in determining how the amine will behave in a desalter. Alkyl amines tend to have a much stronger affinity for hydrocarbon than do the alkanol amines. The oxygen functionality in alkanol amines greatly enhances their affinity for the aqueous phase in a desalter. When selecting the neutralising amine, it is important to understand where the crude tower overhead water is going to be routed and the overall quality of the wash water (pH) used in the desalter. Routing overhead water directly to the desalter and running the desalter with high pH wash water both tend to reduce the flexibility in neutralising amine selection. Routing overhead water to the SWS and maintaining low desalter wash water pH allow greater flexibility in neutraliser selection at the cost of creating additional SWS feed, and potentially the need to acidify the desalter wash water.

Some refineries will inject an acid into the desalter to lower the wash water pH and reduce the partitioning of amines into crude oil. Acids commonly used for this purpose are citric, acetic, and hydroxy acids such as glycolic acid. Besides adding substantially to the refinery chemical costs, the acids can each create their own set of negative side effects.[4] Desalter acidification can lead to overhead corrosion, increased neutraliser consumption, fouling, and increased BOD/COD loading on the wastewater system. While use of acids to lower desalter wash water pH can be beneficial, it is critical that the refiner makes sure that the cure is not worse than the problem being addressed.

More recently, Dorf Ketal has introduced an aldehyde based reactive adjunct chemistry into the market. The chemistry converts the amine to a non-salt forming, water soluble imine. The amine conversion has the beneficial side effect of lowering the pH of the water in the desalter without the need for acid injection and its negative side effects. Initial trials of the new chemistry have been promising, but more time is required to fully evaluate the impact of this new chemistry on refinery operation.

When deciding how to design and operate the crude unit, the operating objectives can sometimes be in conflict with each other, thus requiring costs and benefits to be weighed very carefully. To minimise the amount of neutraliser that goes to the desalter, it would be desirable to route the overhead water to the sour water stripper first instead of going directly to the desalter. Sour water strippers, however, often inject caustic into the feed to enhance ammonia stripping, resulting in relatively high pH stripped sour water. The benefit of reduced amine to the desalter must be weighed against any negative impacts on partitioning behaviour due to higher pH of stripped sour water feed to the desalter instead of tower overhead water. Also, the impact on sour water stripper capacity utilisation and operating cost need to be factored into the analysis.

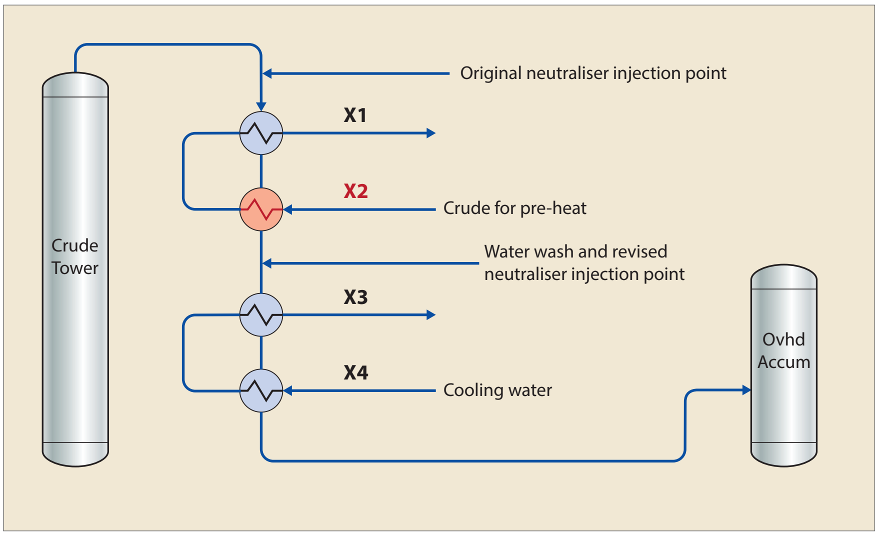

Figure 4 Overhead system for Case 1

With these key considerations in neutraliser selection in mind, let us look at some basic principles in the design of overhead systems and some examples of how to apply this information.

Design principles for overhead corrosion control

Some basic principles in the design of the corrosion control system for crude tower overheads are:

- Inject the neutralising amine ahead of the first point where it is possible to form liquid water in the system.
- Select a neutralising amine that does not form a separate salt phase ahead of the water dew point
- If the second criteria cannot be met a water wash is needed to prevent corrosion and fouling.

There are also a number of additional design considerations that are beyond the scope of this article, such as filmer injection and water wash system design.

Other factors such as shock condensation, neutraliser injection location, and variable operating conditions can also impact neutraliser selection. Failure to properly consider the actual conditions that can exist either in normal operation or during abnormal operational periods can result in corrosion from improper neutraliser selection or through injecting the neutraliser at the wrong location.

These issues can best be illustrated through the use of some real life case studies.

Case 1

The refinery in Case 1 has a crude tower overhead system (see **Figure 4**). The unit processes a high salt content crude in a single stage desalter, and overhead chlorides average 150 ppm. The overhead temperature at the time when the problems occurred ranged between 280°F and 300°F (138-149°C). The first two exchangers in the overhead system, X1 and X2, are raw crude vs overhead exchangers with titanium bundles and carbon steel shells.

To maximise crude preheat and to reduce the quantity of wash water required, the wash water is injected

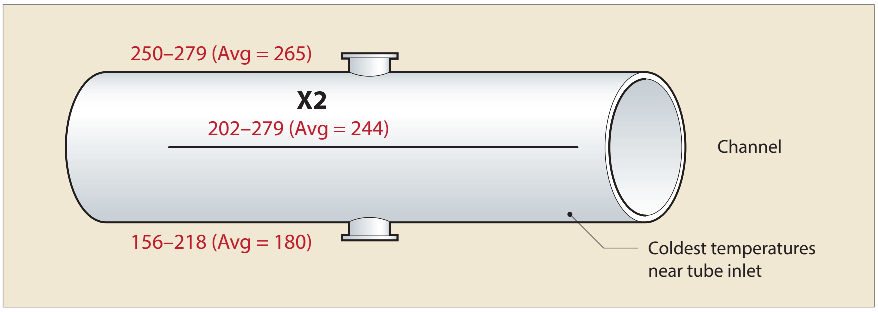

Figure 5 Skin temperature profile on X-2

between the crude vs overhead exchangers and the water coolers.

Historically, the neutralising amine had been injected ahead of the first exchanger. Because of the high chlorides, amine chloride salts formed in the first two exchangers, and they suffered high, chronic corrosion rates of 0.75-2 mm/y on the shells and outlet piping. The titanium bundles did not corrode, but the neutraliser formed liquid amine chloride salts which flowed off of the bundles and onto the CS shells and outlet piping where aggressive corrosion occurred. The chemical supplier modelled the system and determined that the water dew point would not occur in the first two exchangers so that, if no neutraliser were present, the corrosion should go away.

Two months after the neutraliser injection was moved to the water wash downstream of the first two exchangers, X-1 and X-2 were taken out of service during a planned shutdown to replace X-1's shell. During that downtime, corrosion was discovered on the X-2 shell and corroded areas were built up with weld overlay to full shell thickness of 0.28 in. Six weeks after the exchangers were put back in service, the X-2 shell holed through.

During the root cause analysis that followed the event, several interesting contributing factors came into play. Firstly, the chemical supplier based water dew point calculations on normal operation of the tower overhead system. With more detailed analysis of the operating data, it was determined that about 2% of the time the water dew point would actually be expected to occur in X-2 based on the vendor's predicted minimum tube wall temperature in X-2.

The second finding was that temperatures in the exchangers were significantly cooler than the chemical supplier had predicted. Their calculations had predicted a minimum tube wall temperature of 214°F (101°C) and a water dew point of 204°F (96°C, from which the conclusion was that shock condensation of water was not a concern.

In reality, though, the lowest tube wall temperature had to be much lower than predicted as evidenced by the temperature survey results shown in **Figure 5**. During a time period when the insulation was off X-2 for an automated ultrasonic thickness (AUT) inspection, an infrared temperature survey of the shell was also performed. The most interesting thing to note was that the temperature along the bottom of the shell averaged only 180°F (82°C) compared to a measured outlet temperature of over 260°F (127°C). This temperature data indicates that the liquid which condensed on the upper tubes in the bundle continued to subcool as it flowed down the bundle and that it did not mix uniformly with the rest of the vapour in the exchanger.

This temperature along the bottom of the exchanger is representative of the liquid film that forms on the tube surface, which is significantly different from the bulk temperature that a process simulator would calculate or the temperature indicator in the outlet line would measure.

One approach to estimating the coldest tube wall temperature is to take the average of the cold stream inlet temperature and the hot stream outlet temperature. In this example, if the outlet temperature is 260°F (127°C) and the crude inlet temperature is 80°F (27°C), this is first exchanger coming in from the tank farm, the coldest tube wall temperature would be estimated at 170°F (77°C), which is reasonably close to the temperature measured along the bottom of the shell in this case.

In this case, when the neutraliser injection was relocated, the neutraliser was no longer being injected ahead of the water dew point, and extremely aggressive corrosion occurred. In many systems without a water wash, the second objective cannot be met and corrosion will occur due to salt formation, which was the case in this system before the neutraliser relocation was done. In some systems, there is a lot of naturally occurring ammonia or there are tramp amines present, and

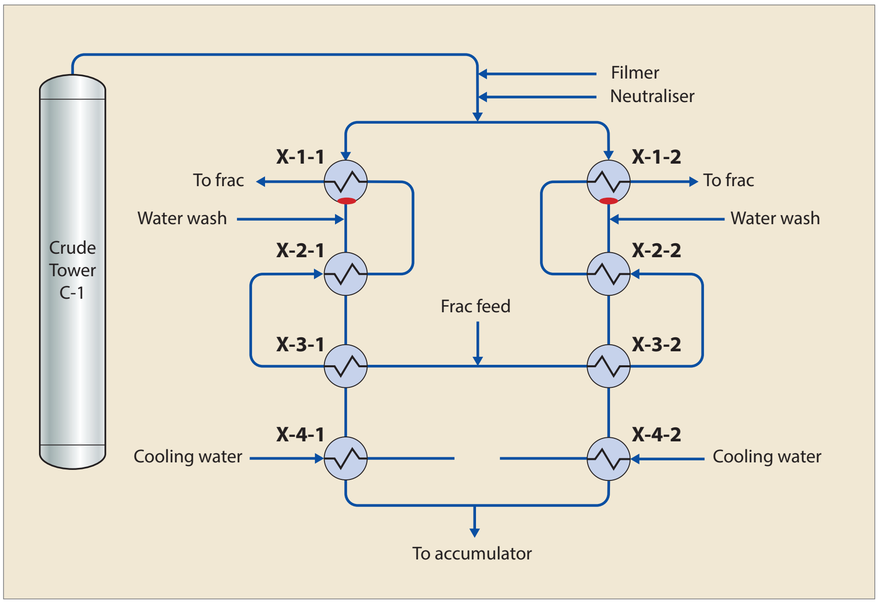

Figure 6 Overhead system for Case 2

a salt point is reached ahead of the neutraliser salt point. In such systems, a water wash is the only practical approach to preventing salt formation.

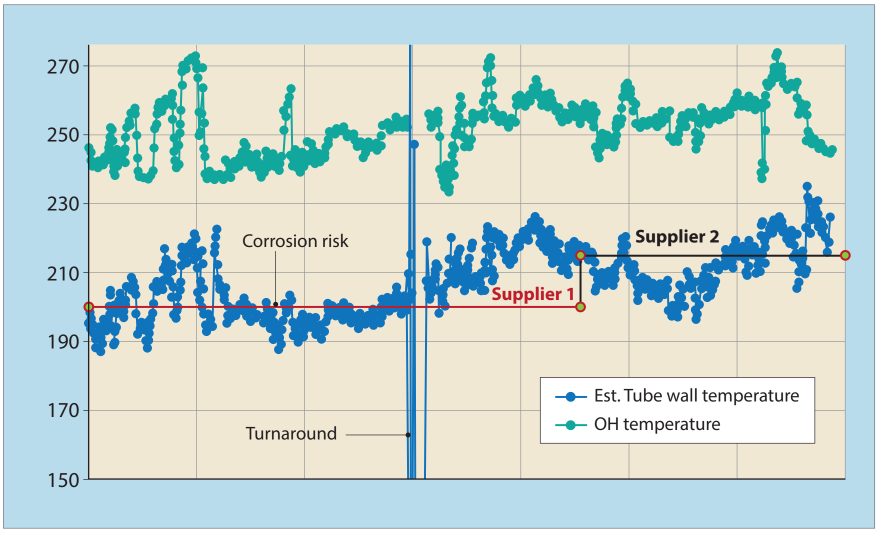

Figure 7 This graph indicates that after the neutraliser change there was an extended period of time when the estimated salt point was significantly higher than the tube wall temperature

Case 2

This crude unit had two banks of exchangers that used crude tower overhead to preheat feed to another column (see **Figure 6**). Water wash was injected between the first and second exchanger in each bank. The exchangers had titanium bundles and carbon steel shells, and the top exchangers in each bank (X-1-1 and X-1-2) had long term corrosion rates of approximately 0.38 mm/y.

A routine inspection a year after a turnaround revealed that the average corrosion rate since the previous turnaround had accelerated to over 3.8 mm/y in the area highlighted in red in **Figure 6**. This change in corrosion triggered much more frequent inspection, which soon determined that the real corrosion rate was more like 12.5 mm/y.

A number of possible causes were explored, but the final conclusion was that a neutraliser change that had occurred when the refinery changed chemical suppliers caused the corrosion in these exchangers. Neither supplier had been routinely tracking salt points in the overhead system, which made the analysis more difficult. During the time frame in question, there did not appear to be any change in overhead chlorides or other key variables that could be linked to corrosion in these exchangers.

After the turnaround, the overhead temperature averaged 8°F hotter than it had before the turnaround, which normally would reduce corrosion risk, although the change in estimated tube wall temperature was small (see **Figure 7**). Supplier 1 had been using a three-amine blend (amines A, B and C). Supplier 2 elected to use a single amine product that used only amine A. As part of supplier 2's troubleshooting effort, they did several salt point calculations that showed a typical salt point of about 215°F (102°C) for neutralising amine A. It was estimated that the lower concentration of amine A in supplier 1's product reduced the salt point of A by about 15°F.

Those approximate salt points are plotted along with the estimated minimum tube wall temperature in **Figure 7**. In the time before the turnaround, the minimum wall temperature and the salt point were very close to each other. When supplier 2 took over and switched neutraliser products, the salt point became significantly higher than the minimum tube wall temperature, which significantly increased the amount of salt that would have formed and thus the amount of corrosion.

As an interim measure, the chemical supplier switched to a multiple amine blended neutraliser product to at least get back to historic corrosion rates. Ultimately, the refinery decided to upgrade the metallurgy on the shells to eliminate the chronic corrosion problems and to increase operating flexibility.

Conclusions

Making the proper selection of neutralising amine chemistry is a challenging task at many refineries, especially with the wide range of opportunity crudes available in the marketplace. Besides traditional considerations like cost and base strength, a number of other factors need also to be considered. Making good decisions also requires a detailed understanding of the full range of process conditions and actual local conditions in the piping and exchangers. Simply relying on a bulk temperature predicted from a simulation or temperature indicator output may not accurately represent the actual conditions when corrosion may occur. Additionally, the presence of tramp amines in the unit and the potential to recycle amines to the crude tower via the desalter can further complicate the neutraliser selection process. If the refiner and chemical supplier make the effort to consider all of these factors when selecting their neutralising amine, a lot of future problems can be avoided.

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