



# **NAPHTHA HYDROTREATING PROCESS**

## **GENERAL OPERATING MANUAL**

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# **UOP NAPHTHA HYDROTREATING PROCESS**

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## I. INTRODUCTION

The UOP Naphtha Hydrotreating Process is a catalytic refining process employing a select catalyst and a hydrogen-rich gas stream to decompose organic sulfur, oxygen and nitrogen compounds contained in hydrocarbon fractions. In addition, hydrotreating removes organo-metallic compounds and saturates olefinic compounds.

The hydrotreating process is commonly used to remove Platforming catalyst poisons from straight run or cracked naphthas prior to charging to the Platforming Process Unit. The catalyst used in the Naphtha Hydrotreating Process is composed of an alumina base impregnated with compounds of cobalt or nickel and molybdenum. The feed source and the type of feed contaminants present determine the catalyst type and the operating parameters. This is important to realize when processing non-design type feeds. Volumetric recoveries of products depend on the sulfur and olefin contents, but usually are 100%  $\pm$ 2%.

Organo-metallic compounds, notably arsenic and lead compounds, are known to be permanent poisons to platinum containing catalyst. The complete removal of these materials by hydrotreating will give longer ultimate catalyst life in the Platforming Unit. Sulfur is a temporary poison to Platforming catalysts and causes an unfavorable change in the product distribution and increase coke laydown. Organic nitrogen is also a temporary poison to Platforming catalyst. It is an extremely potent one, however, and a relatively small concentration of nitrogen in the Platforming Unit feed will cause a large activity offset as well as deposit ammonium chloride salts in the Platforming Unit cold sections.

Oxygen compounds are detrimental to the operation of a Platforming Unit. Any oxygen compounds which are not removed in the hydrotreater will be converted to water in the Platforming Unit, thus affecting the water/chloride balance of the Platforming catalyst. Olefins can polymerize at Platforming Unit operating conditions which can result in exchanger and reactor fouling.

The Naphtha Hydrotreating Process makes a major contribution to the ease of operation and economy of Platforming. Much greater flexibility is afforded in choice of allowable charge stocks to the Platforming Unit. Because this unit protects the Platforming catalyst, it is important to maintain consistently good operation in the Hydrotreating Unit.

In addition to treating naphtha for Platforming feed, there are uses for the UOP Naphtha Hydrotreating Process in other areas. Naphthas produced from thermal processes, such as delayed coking, FCC, thermal cracking, and visbreaking, are usually high in olefinic content and other contaminants, and may not be stable in storage. These naphthas may be hydrotreated to remove the olefins and reduce organic and metallic contaminants, providing a marketable product.

It can be seen that the primary function of the UOP Naphtha Hydrotreating Process can be characterized as a "clean-up" operation. As such, the unit is critical to refinery down stream operation.

**NOTE:** THIS MANUAL IS GENERAL IN NATURE AND CANNOT COVER EVERY POSSIBLE PROCESS OR MECHANICAL VARIATION. ALTHOUGH CARE HAS BEEN TAKEN TO MAKE THIS MANUAL COMPLETE, MANY ITEMS INCLUDING INSTRUMENTATION AND DETAILED PROCEDURES HAVE NOT BEEN GIVEN. THE PURPOSE OF THIS MANUAL IS TO PROVIDE GUIDELINES SO THAT THE REFINER CAN PREPARE A MORE DETAILED OPERATIONS HANDBOOK.

## II. PROCESS PRINCIPLES

The main purpose of the UOP Naphtha Hydrotreating Process is to "clean-up" a naphtha fraction so that it is suitable as charge to a Platforming Unit. There are six basic types of reactions that occur in the hydrotreating unit.

### A. REACTIONS

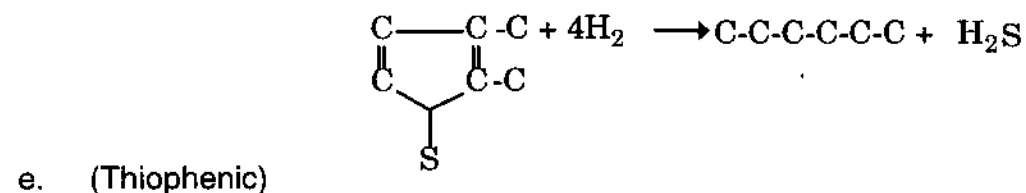
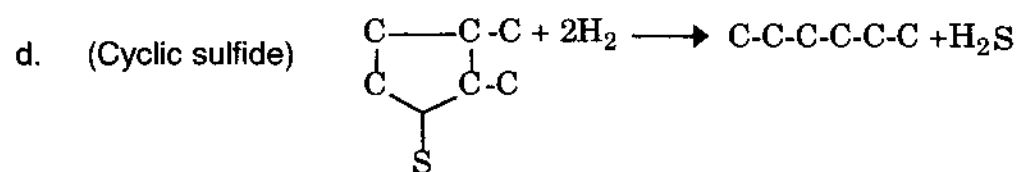
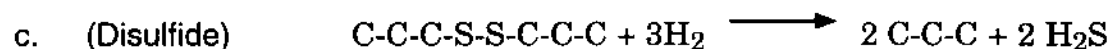
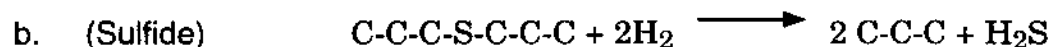
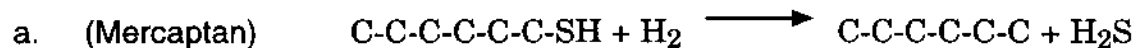
1. Conversion of organic sulfur compounds to hydrogen sulfide
2. Conversion of organic nitrogen compounds to ammonia
3. Conversion of organic oxygen compounds to water
4. Saturation of olefins
5. Conversion of organic halides to hydrogen halides
6. Removal of organo-metallic compounds

### B. DISCUSSION

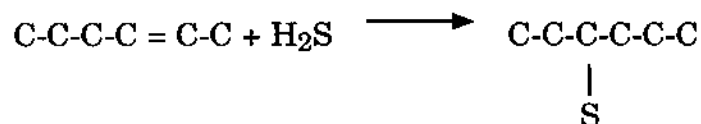
#### 1. Sulfur Removal

For bimetallic Platforming catalyst, the feed naphtha must contain less than 0.5 weight ppm sulfur to optimize the selectivity and stability characteristics of the catalyst. In general, sulfur removal in the hydrotreating process is relatively easy, and for the best operation of a Platforming Unit, the hydrotreated naphtha sulfur content should be maintained well below the 0.5 weight ppm maximum. Commercial operation at 0.2 weight ppm sulfur or less in the hydrotreater product naphtha is common. For higher severity Platforming Units, mainly found in CCR applications, the feed sulfur level is maintained between 0.15 - 0.5 weight ppm. If the sulfur level is below 0.15 weight ppm, then the Platforming feed sulfur content can be increased with the sulfur injection facility located in the Platforming Unit.

Typical sulfur removal reactions are shown below.



It is possible, however, to operate at too high a temperature for maximum sulfur removal. Recombination of hydrogen sulfide with small amounts of olefins or olefin intermediates can then result, producing mercaptans in the product.



If this reaction is occurring, the reactor temperature must be lowered. Generally, operation at 315-340°C (600-645°F) average reactor temperature will give acceptable rates of the desired hydrogenation reactions and will not result in a significant amount of olefin/hydrogen sulfide recombination. The sulfur recombination reaction typically occurs at temperatures greater than 340°C (645°F). This temperature is dependent upon feedstock composition, operating pressure,

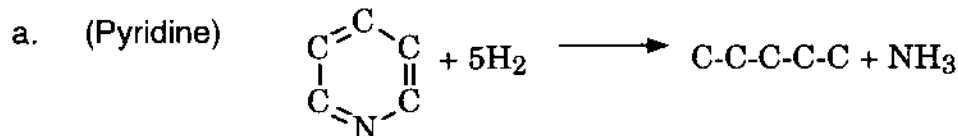
and LHSV. Also, this temperature can be achieved within the reactor due to temperature rise from the saturation of olefins, if present.

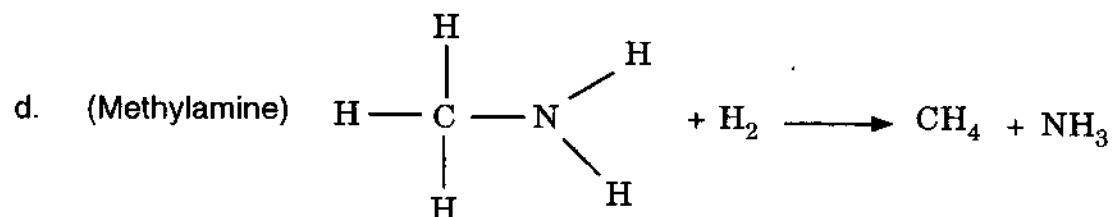
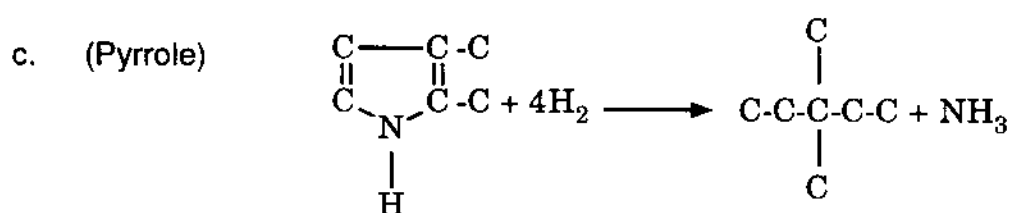
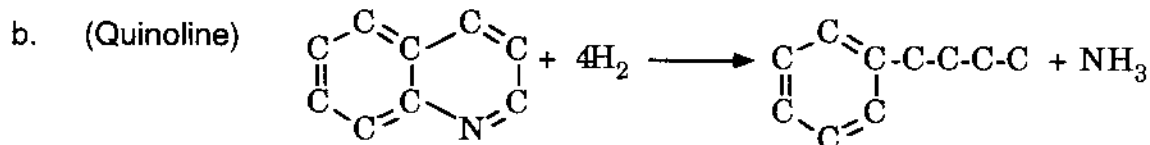
## 2. Nitrogen Removal

Nitrogen removal is considerably more difficult than sulfur removal in naphtha hydrotreating. The rate of denitrification is only about one-fifth the rate of desulfurization. Most straight run naphthas contain much less nitrogen than sulfur, but attention must be given to ensure that the feed naphtha to Platforming catalyst contains a maximum of 0.5 weight ppm nitrogen and normally much less.

Any organic nitrogen that does enter the Platforming Unit will react to ammonia and further with the chloride in the recycle gas to form ammonium chloride. Ammonium chloride will deposit in the recycle gas circuit or stabilizer overhead system. Ammonium chloride salts can be removed by water washing, but will result in downtime or product to slop. Ammonium chloride salts can be minimized by maximizing nitrogen removal in the Naphtha Hydrotreating Unit. Nitrogen removal is much more important when a Naphtha Hydrotreating Unit processes thermally derived naphtha, as these feedstocks normally contain much more nitrogen than a straight run naphtha.

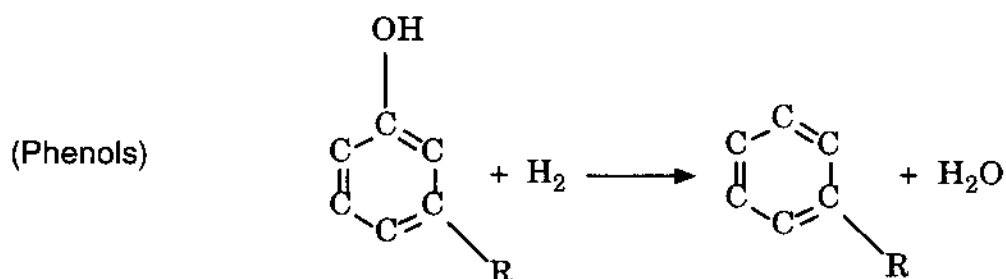
Denitrification is favored more by pressure than temperature and thus unit design is important. If a Naphtha Hydrotreating Unit designed for straight-run naphtha starts processing non straight-run naphtha (except hydrocracked naphtha), there may be incomplete removal of nitrogen. There can be some improvement, usually not a large change, in denitrification with increasing temperature. Equipment design will limit the amount that the pressure can be increased. The ammonia formed in the denitrification reactions, detailed below, is subsequently removed in the hydrotreater reactor effluent wash water.





### 3. Oxygen Removal

Organically combined oxygen, such as a phenol or alcohol, is removed in the Naphtha Hydrotreating Unit by hydrogenation of the carbon-hydroxyl bond, forming water and the corresponding hydrocarbon. The reaction is detailed below. Oxygenates are typically not present in naphtha, but when present they are in very low concentrations. Any oxygenates in the product will quantitatively convert to water in the Platforming Unit. It is important that the hydrotreater product oxygenate level be reduced sufficiently.



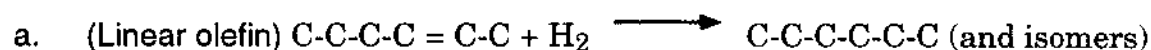
Oxygenate removal is as difficult, if not more, than nitrogen removal. The specific organic oxygen species impacts ease or difficulty of removal. Units normally not designed for oxygen removal may find it difficult to get adequate product quality. Oxygenate removal is favored by high pressure and high temperatures. For high feed concentrations, lower liquid space velocities are required. Processing of such compounds should be done with care. Complete oxygen removal is not normally expected and may only be 50%. However, MTBE has been shown to be essentially removed, but not completely, depending on the feed concentrations.

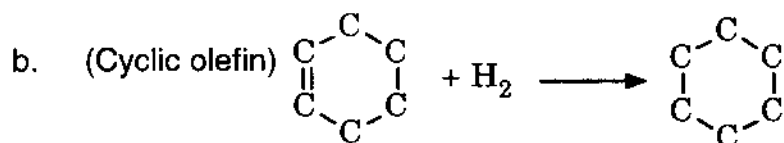
#### 4. Olefin Saturation

Hydrogenation of olefins is necessary to prevent fouling or coke deposits in downstream units. Olefins can polymerize at the Platforming combined feed exchanger and thus cause fouling. These olefins will also polymerize upstream of the naphtha hydrotreating reactor and cause heat transfer problems.

Olefin saturation is almost as rapid as desulfurization. Most straight run naphthas contain only trace amounts of olefins, but cracked naphthas usually have high olefin concentrations. Processing high concentrations of olefins in a Naphtha Hydrotreating Unit must be approached with care because of the high exothermic heat of reaction associated with the saturation reaction.

The increased temperature, from processing relatively high amounts of olefins, across the catalyst bed can be sufficient enough to cause sulfur recombination. The olefin reaction is detailed below.



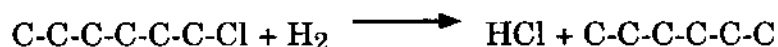


## 5. Halide Removal

Organic halides can be decomposed in the Naphtha Hydrotreating Unit to the corresponding hydrogen halide, which is either absorbed in the reactor effluent water wash or taken overhead in the stripper gas. Decomposition of organic halides is much more difficult than desulfurization. Maximum organic halide removal is thought to be about 90 percent, but is much less at operating conditions set forth for sulfur and nitrogen removal only. For this reason, periodic analysis of the hydrotreated naphtha for chloride content should be made, since this chloride level must be used to set the proper Platforming Unit chloride injection rate.

High feed concentrations of chloride can result in corrosion downstream of the reactor. Chloride corrosion control is described in the Process Flow - Wash Water section of this manual.

A typical organic chloride decomposition reaction is shown below.



## 6. Metal Removal

Normally the metallic impurities in the naphtha feeds are in the part per billion (ppb) range and these can be completely removed. The UOP Hydrotreating catalysts are capable of removing these compounds at fairly high concentrations, up to 5 weight ppm or more, on an intermittent basis at normal operating conditions. The maximum feed concentration for complete removal is dependent on the metal species and operating conditions. The metallic impurities remain on the Hydrotreating catalyst when removed from the naphtha. Some commonly detected components found on used Hydrotreating Hydrobon<sup>®</sup> catalyst are arsenic, iron, calcium, magnesium, phosphorous, lead, silicon, copper, and sodium.

Removal of metals from the feed normally occurs in plug flow with respect to the catalyst bed. Iron is found concentrated at the top of catalyst beds as iron sulfides. Arsenic, even though it is rarely found in excess of 1 weight ppb in straight run naphthas, is of major importance, because it is a potent Platforming catalyst poison. Arsenic levels of 3 weight percent and higher have been detected on used Hydrotreating catalysts. This arsenic loaded catalyst retained its activity for sulfur removal. Contamination of storage facilities by leaded gasolines and reprocessing of leaded gasolines in crude towers are the common sources of lead on used Hydrotreating catalysts. Sodium, calcium and magnesium are apparently due to contact of the feed with salt water or additives. Improper use of additives to protect fractionator overhead systems from corrosion or to control foaming, such as in Coker Units, account for the presence of phosphorus and silicon, respectively.

Removal of metals is essentially complete, at temperatures above 315°C (600°F), up to a metal loading of about 2-3 weight percent of the total catalyst. Some Hydrotreating catalysts have increased capability to remove Silicon, up to 7-8 wt% of the total catalyst. Above the maximum levels, the catalyst begins approaching the equilibrium saturation level rapidly, and metal breakthrough is likely to occur. In this regard, mechanical problems inside the reactor, such as channeling, are especially bad since this results in a substantial overload on a small portion of the catalyst in the reactor.

### C. REACTION RATES AND HEATS OF REACTION

The approximate relative reaction rates for the three major reaction types are:

|                   |         |
|-------------------|---------|
| Desulfurization   | 80-100* |
| Olefin Saturation | 80-100* |
| Denitrification   | 20      |

\*range dependent on specific species.

The approximate heats of reaction (in kJ per kg of feed per cubic meter of hydrogen consumed) and relative heats of reaction are:

---

|                   | <b>Heat of Reaction</b> | <b>Relative<br/>Heat of Reaction</b> |
|-------------------|-------------------------|--------------------------------------|
| Desulfurization   | 8.1                     | 1                                    |
| Olefin Saturation | 40.6                    | 5                                    |
| Denitrification   | 0.8                     | 0.1                                  |

As can be seen from the above summary, desulfurization is the most rapid reaction taking place, but it is the saturation of olefins which generates the greatest amount of heat. Certainly, as the feed sulfur level increases, the heat of reaction also increases. However, for most of the feedstocks processed, the heat of reaction will just about balance the reactor heat loss, such that the naphtha hydrotreating reactor inlet and outlet temperatures are essentially equal. Conversion of organic chlorides and oxygenated compounds are about as difficult as denitrification. Consequently, more severe operating conditions must be used when these compounds are present.

The following table summarizes the physical properties of UOP Hydrotreating catalysts. Refer to section XIV for material data safety sheets on these catalysts.

**TABLE II-1**  
**UOP HYDROBON® CATALYSTS**  
**FOR**  
**NAPHTHA HYDROTREATING SERVICE**

| <u>Designator</u> | <u>Base</u> | <u>Form</u> | <u>Size</u><br>in * | <u>ABD</u><br>lb/ft <sup>3</sup> ** | <u>Metals</u> | <u>Regeneration</u> |
|-------------------|-------------|-------------|---------------------|-------------------------------------|---------------|---------------------|
| S-6               | Alumina     | Sphere      | 1/16                | 36                                  | Ni/Mo/Co      | Steam/Air           |
| S-9               | Alumina     | Sphere      | 1/16                | 38                                  | Mo/Co         | Steam/Air           |
| S-12              | Alumina     | Extrudate   | 1/16                | 45                                  | Mo/Co         | Inert Gas           |
| S-15              | Alumina     | Extrudate   | 1/16                | 45                                  | Ni/Mo         | Inert Gas           |
| S-16              | Alumina     | Extrudate   | 1/16                | 45                                  | Ni/Mo         | Inert Gas           |
| S-18              | Alumina     | Sphere      | 1/16                | 45                                  | Mo/Co         | Inert Gas           |
| S-19              | Alumina     | Extrudate   | 1/18 – 1/16         | 41-45                               | Ni/Mo         | Inert Gas           |
| S-120             | Alumina     | Cylinder    | 1/16                | 47                                  | Mo/Co         | Inert Gas           |
| N-108             | Alumina     | Quadlobe    |                     | 40                                  | Mo/Co         | Inert Gas           |
| N-204             | Alumina     | Extrudate   | 1/20                | 46                                  | Ni/Mo         | Inert Gas           |
| HC-K              | Alumina     | Quadlobe    | 1/20                | 57                                  | Ni/Mo         | Inert Gas           |

\* Sizes may vary

\*\* Sock loaded

### III. PROCESS VARIABLES

#### A. REACTOR PRESSURE

The unit pressure is dependent on catalyst life required and feedstock properties. At higher reactor pressures, the catalyst is generally effective for a longer time and reactions are brought to a greater degree of completion. For straight run naphtha desulfurization, 20 to 35 kg/cm<sup>2</sup>g (300 to 500 psig) reactor pressure is normally used, although design pressure can be higher if feed nitrogen and/or sulfur contents are higher than normal. Cracked naphthas contain substantially more nitrogen and sulfur than straight run naphthas and consequently require higher processing pressures, up to 55 kg/cm<sup>2</sup>g (800 psig). Similarly, higher operating pressures are necessary to completely remove organic halides. Halide contamination of naphtha is usually sporadic in occurrence and is normally due to contamination by crude oil well operators.

The selection of the operating pressure is influenced to a degree by the hydrogen to feed ratio set in the design, since both of these parameters determine the hydrogen partial pressure in the reactor. The hydrogen partial pressure can be increased by operation at a higher ratio of gas to feed at the reactor inlet. The extent of substitution is limited by economic considerations.

Most units have been designed so that the desulfurization and denitrification reactions go substantially to completion well below the design temperature of the reactors, for the design feedstock. Small variations in pressure or hydrogen gas rate in the unit will not cause changes sufficiently to be reflected by significant differences in product quality. This is especially true for denitrification reactions, which are more dependent on the pressure than the desulfurization reactions. Thus, units not designed for nitrogen in the feedstock will have difficulty meeting the Platforming Unit feed requirements.

## B. TEMPERATURE

Temperature has a significant effect in promoting hydrotreating reactions. Its effect, however, is slightly different for each of the reactions that occur. Desulfurization increases as temperature is raised. The desulfurization reaction begins to take place at temperatures as low as 230°C (450°F) with the rate of reaction increasing markedly with temperature. Above 340°C (650°F) there are only slight increases in further removal of sulfur compounds due to temperature.

For higher severity Platforming Units, mainly found in CCR applications, the feed sulfur level is maintained between 0.15 - 0.5 weight ppm. If the sulfur level is below 0.15 weight ppm, then the Platforming feed sulfur content can be increased with the sulfur injection facility located in the Platforming Unit. The hydrotreater reactor temperature should be set to completely hydrotreat the naphtha feed and the secondary "fine" sulfur adjustments are made in the Platforming Unit.

The decomposition of chloride compounds in low concentrations (<10 weight ppm) will occur at about the same temperature as sulfur compound decomposition.

Olefin saturation behaves somewhat similarly to the desulfurization reaction with respect to temperature, except that olefin removal may level off at a somewhat higher temperature. Because this reaction is very exothermic, the olefin content of the feed must be monitored and in some cases limited to keep reactor peak temperature within an acceptable temperature range. At elevated temperatures, an apparent equilibrium condition limits the degree of olefin saturation. This may even cause the residual olefins in the product to be greater at higher temperatures than would be the case at lower operating temperatures. Also, the H<sub>2</sub>S present can react with these olefins to form mercaptans. In such a case, lowering the reactor temperature can eliminate residual olefins and thus mercaptan formation. With typical olefin concentrations this recombination reaction may occur around 650°F (343°C).

Decomposition of oxygen and nitrogen compounds requires a somewhat higher temperature than desulfurization or olefin saturation. The removal of these compounds does not appear to level off at elevated temperatures. Units with

significant levels of nitrogen or oxygen must be designed for high pressure and low liquid hourly space velocity (LHSV) to ensure complete conversion.

The demetalization reactions require a minimum temperature of 315°C (600°F). Above 315°C (600°F), metals removal is essentially complete. Below this temperature, there may be some cases where all the metals will not be removed. However, a lower temperature may be acceptable for certain metals. Due to the permanent poisoning of Platforming catalyst, extreme care and monitoring should be taken if adjusting the temperature below 315°C (600°F).

The recommended minimum reactor inlet temperature to ensure a properly prepared Platforming Unit feed is 315°C (600°F). There are two factors which are important in determining this minimum temperature: First, below the minimum temperature, reaction rates for contaminant removal may be too low. Second, the temperature must be maintained high enough to ensure that the combined feed (recycle or once-through gas plus naphtha) to the charge heater is all vapor.

Normal Reactor design temperatures for both straight run (SRN) and cracked naphthas are 399°C (750°F) maximum. Actual operating temperatures will vary, depending upon the feed type, from 285°C (550°F) to 385°C (650°F). Cracked stocks may require processing at higher temperatures because of the higher sulfur, nitrogen, and olefin contents. For these feeds, the reactor delta T will be in the range of 10-55°C (20-100°F).

As the catalyst ages, the product quality may degenerate, which may be corrected by increasing reactor inlet temperature. If increasing the temperature does not improve the product quality, a regeneration or change of catalyst will be required, depending on the history of the operation and catalyst state.

In addition to catalyst deterioration, scale and/or polymer formation at the top of the catalyst bed may cause high reactor pressure drops which may result in reactor channeling. This can be corrected by skimming the top of the catalyst bed; and/or unloading, screening and reloading. Higher pressure drop problems should be corrected as soon as possible to minimize the risk of equipment damage and

degradation of product quality. Pressure drop is further discussed at the end of this section.

### C. FEED QUALITY

For normal operation, daily changes in hydrotreater reactor inlet temperature to accommodate changes in feed quality should not be necessary. However, in some cases, such as when a refinery is purchasing outside crude from widely different sources, the naphtha quality may change significantly, and adjustment of reactor inlet temperature may be necessary. Changes in the feed olefin content will also affect the heat of reaction and adjustments to the heat balance of the unit may also be required.

The final selection of reactor temperature should be based upon product quality. The above relations of feed quality and temperature assume operation within the normal temperature operating ranges given in the preceding section.

For units that operate with sweet feed, a minimum sulfur is required to maintain the metals in their proper sulfided state. Sulfur will be desorbed off the catalyst if there is low  $H_2S$  in the recycle gas. This will allow the metal to reduce to its metal state, which is not conducive to hydrotreating reactions. This reaction is partially reversible. If the sulfur level decreases below 15 wt-ppm sulfur, then sulfur should be injected into the feed. The same compounds used for fresh catalyst sulfiding can be used for this operation.

### D. HYDROGEN TO HYDROCARBON RATIO

The minimum hydrogen to feed ratio ( $nm^3/m^3$  or SCFB) is dependent on hydrogen consumption, feed characteristics, and desired product quality.

For straight run naphthas of moderate sulfur content, 40-75  $nm^3/m^3$  (250-400 SCFB) is normally required. Cracked naphthas must be processed at higher  $H_2$

ratios [up to 500 nm<sup>3</sup>/m<sup>3</sup> (3000 SCFB)]. As feedstocks vary between these limits, the hydrogen to feed ratio is proportioned between the extremes.

Ratios above 500 nm<sup>3</sup>/m<sup>3</sup> (3000 SCFB) do not contribute to the rate of reactions. The use of low purity hydrogen as makeup gas is limited by economical operation of the recycle compressor. Recycle gas with hydrogen sulfide contents up to 10% and with large quantities of carbon monoxide and nitrogen are not harmful to the catalyst, again when reasonable desulfurization is the only criterion. For nitrogen removal or complete sulfur removal, high hydrogen purity (70% minimum) is necessary, and CO may act as a temporary catalyst poison. The prevention of excessive carbon accumulation on the catalyst requires maintenance of a minimum H<sub>2</sub> partial pressure, so impurities present in the makeup gas require higher operating pressures.

Lower hydrogen to hydrocarbon ratios can be compensated for by increasing reactor inlet temperature. The approximate relation for these variables is 10°C (18°F) higher reactor temperature requirement for a halving of the hydrogen/feed ratio. This rule assumes operation above the minimum values of 315°C (600°F) reactor inlet temperature and 40 nm<sup>3</sup>/m<sup>3</sup> (250 SCFB) hydrogen ratio. This relation is approximate, and it should again be pointed out that the product quality should dictate the actual reactor temperature utilized.

## E. SPACE VELOCITY

The quantity of catalyst per unit of feed will depend upon feedstock properties, operating conditions, and product quality required. The liquid hourly space velocity (LHSV) is defined as follows:

$$\text{LHSV} = \frac{\text{volume of charge per hour}}{\text{volume of catalyst}}$$

With most charge stocks and product objectives, a simplified kinetic expression based on sulfur and/or nitrogen removal determines the initial liquid hourly space velocity. This initial value may be modified due to other considerations, such as size

of unit, extended first cycle catalyst service, abnormal levels of feed metals and requirements of other processing units in the refinery flow scheme. Relative ease of conversion for Hydrobon<sup>®</sup> catalysts indicate that olefins react most easily, sulfur compounds next, then nitrogen and oxygen compounds. There is considerable overlap with several reactions occurring simultaneously and to different degrees. Charge stock variability is so large that only approximate ranges of space velocities can be indicated for the various feed types. SRN is processed at 4-12 LHSV and cracked naphtha at 2-8 LHSV.

For daily changes in the LHSV, inlet temperature on the naphtha hydrotreating reactor may be adjusted according to the equation below:

$$T_2 = T_1 - 45 \ln \frac{\text{LHSV}_1}{\text{LHSV}_2} \quad (\text{for } ^\circ\text{F})$$

$$T_2 = T_1 - 25 \ln \frac{\text{LHSV}_1}{\text{LHSV}_2} \quad (\text{for } ^\circ\text{C})$$

where  $T_1$  = required inlet reactor temperature at  $\text{LHSV}_1$

$T_2$  = required inlet reactor temperature at  $\text{LHSV}_2$

The above relation assumes operation between 4 and 12 LHSV and assumes that reactor temperatures are within the limits discussed in Section II.

## F. CATALYST PROTECTION, AGING, AND POISONS

The process variables employed affect the catalyst life by their effect on the rate of carbon deposition on the catalyst. There is a moderate buildup of carbon on the catalyst during the initial days of operation, but the rate of increase in carbon level soon drops to a very low figure under normal processing conditions. This desirable control of the carbon-forming reactions is obtained by maintaining the proper hydrogen to hydrocarbon ratio and by keeping the catalyst temperature at the proper level.

Temperature is a minor factor in respect to the hydrotreating catalyst life. A higher catalyst temperature increases the rate of the carbon-forming reactions, other factors being equal. It must be remembered that a combination of high catalyst temperature and inadequate hydrogen is very injurious to the catalyst activity.

Catalyst deactivation is measured by the decrease in relative effectiveness of the catalyst at fixed processing conditions after a period of catalyst use.

The primary causes of catalyst deactivation are: (1) accumulation of coke on the active sites, and, (2) chemical combination of contaminants from the feedstock with the catalyst components. In normal operation, a carbon level above 5 wt-% may be tolerated without a significant decrease in desulfurization although nitrogen removal ability can be decreased.

Permanent loss of activity requiring catalyst replacement is usually caused by the gradual accumulation of inorganic species picked up from the charge stock, makeup hydrogen or effluent wash water. Examples of such contaminants are arsenic, lead, calcium, sodium, silicon and phosphorus. Very low concentrations of these species, ppm and/or ppb, will cause deactivation over a long period of service because buildup of deposits depends on the integrated effect of both temperature and time. This effect is important when processing Platforming Unit feed.

Hydrobon® catalysts exhibit a high tolerance for metals such as arsenic and lead. Total metals content as high as 2 to 3 wt-% of the catalyst have been observed with the catalyst still effective. However, if the calculated metals content of the catalyst is 0.5 wt-%, the frequency of product analyses should be increased to prevent metal breakthrough to the Platforming catalyst. Organic lead compounds are decomposed by Hydrobon® catalysts and for the most part deposit in the upper portion of the catalyst bed as lead sulfide. Metals are not removed from the catalyst during a regeneration. When the total metals content, other than silicon, of the catalyst approaches 1 to 2 wt-%, consideration should be given to replacing the catalyst.

The only certain method of minimizing the effect of trace metal contaminants on the catalyst is to limit their entry to the system. This is done by careful, conscientious feed analysis and correcting the source of, or conditions, causing the presence of the metal contaminant.

Apparent catalyst deactivation may be caused by the accumulation of deposit on top of the catalyst bed. This is seen by increased pressure drop across the reactor. The flow pattern through the balance of the bed is disturbed and product quality is diminished. This condition is easily remedied by skimming a portion of the catalyst, screening and reloading, or replacing with fresh catalyst. The procedure for this is described in Section XIII of this manual. The deposits are generally iron sulfide.

The maximum pressure drop that can be sustained is a function of outlet basket design and the product quality. The outlet basket allowable pressure drop ranges from 60-100 psig (4.2 – 7.0 kg/cm<sup>2</sup>), depending on the design. This can be used as a "general" guideline for when to skim the reactor. Normally the entire measured pressure drop is not taken across the outlet basket, since material deposits are on top of the catalyst bed. The product quality and, in some cases, the recycle gas flow rate may be effected at the higher pressure drop. For hydrogen once-through units the flow rate is even more affected and the allowable pressure drop may be less than units with recycle gas compressors. These changes, along with product quality, need to be considered for all units in determining when to alleviate the pressure drop.

Dissolved oxygen, though not a catalyst poison, should be eliminated from the feed. With oxygen in the feed, especially in the presence of olefins, excessive fouling of equipment, particularly the feed-effluent exchangers, can occur. There are anti-fouling agents or dispersents that can be injected to the feed to minimize the effects. Removing the oxygen is the preferred choice.

## IV. PROCESS FLOW AND CONTROL

A typical Naphtha Hydrotreating Unit processing a straight run naphtha for Platforming Unit feed will have a reactor section and a stripper section. In addition, some units have a prefractionation section upstream of the reactor section. A naphtha splitter may also be included, downstream of the stripper section, for units that do not process straight run material. A typical Naphtha Hydrotreating Unit with recycle gas is depicted in Figure IV-1, and a once-through hydrogen unit is depicted in Figure IV-2.

### A. PREFRACTIONATION SECTION

In some special applications, it is desirable to produce a narrow boiling range naphtha cut for feed to the Platforming Unit. An example of this would be an operation aimed at making aromatics, where the end point of the feed to the Platforming Unit is limited to about 160°C (325°F) to concentrate aromatic precursors in the feed. A full boiling range naphtha cut from the crude unit could be processed through a prefractionation section to accomplish this task.

The prefractionation section typically consists of two fractionation columns in series. The first column is the prefractionator and the second column is the rerun. Usually, the feed to the prefractionator will be heat exchanged with rerun column bottoms, and a steam heater can be used to provide the remaining heat that is required. The overhead of the second (rerun) column becomes the heartcut for processing in the reactor section of the hydrotreater. The heartcut boiling range is controlled by the amount of light naphtha taken overhead in the prefractionation column and by the amount of heartcut taken overhead in the rerun column. The initial boiling point (IBP) of the heartcut is adjusted in the prefractionator and the final boiling point is adjusted in the rerun column.

In the prefractionator, the overhead temperature controller directly sets the amount of overhead liquid product, light naphtha, by controlling net overhead liquid control valve. Increasing this overhead temperature will increase quantity of the overhead product and the increase the endpoint of the overhead product. This in turn controls the initial boiling point of the heartcut. For example, if a 38-204°C (100-400°F) boiling range naphtha is charged to a prefractionation section, the light naphtha is

sent overhead and the prefractionator bottoms product now has 82-204°C (180-400°F) boiling range.

The overhead reflux rate is controlled by the prefractionator overhead receiver level controller. As the receiver level increases, the reflux rate increases. For example, when the prefractionator overhead temperature increases above its set point, the net overhead liquid valve closes, thus increasing the overhead receiver level. The high receiver level in turn increases the reflux rate, which decreases the overhead temperature back to its set point.

The prefractionator column bottoms are pumped directly to the second (rerun) column without any reheat. The flow rate is set by the prefractionator bottoms level controller. The desired product is taken overhead in the rerun column. The rerun column is also controlled by an overhead temperature controller. Increasing the overhead temperature will increase the amount of material taken overhead and will increase its endpoint. Thus, if a heartcut of 82-160°C (180-320°F) is desired, it can be obtained by adjusting the rerun column overhead temperature to set the endpoint.

The rerun overhead reflux rate is controlled by the rerun overhead receiver level controller. As the receiver level increases the reflux rate increases. Both columns have reboilers to provide the heat necessary for vaporization of naphtha so that sufficient reflux can be maintained. The overhead product from the prefractionator and the rerun bottoms product are sent to storage for blending or further processing downstream units. A typical prefractionation flow scheme is depicted in Figure IV-3.

## **B. REACTOR SECTION**

The reactor section can be divided into four systems; feed, reactor, wash water, and separator systems.

### **1. Feed System**

Naphtha feed, or feeds, can enter the unit either from intermediate storage or from another process unit. In the case of feed from storage, the tank must be properly

gas blanketed to prevent oxygen from being dissolved in the naphtha. Even trace quantities of oxygen and/or olefin in the feed can cause polymerization of olefins in the storage tank when stored for long periods or in the combined feed/reactor effluent exchangers if the feed is not prestripped. This results in fouling and a loss of heat transfer efficiency.

The feed chloride content should also be monitored. This is important for proper corrosion control, which is described in the wash water section.

Typically, the feed(s) are collected in the feed surge drum where the rates are levelled out in the surge capacity of this drum. The feed surge drum is also provided with a water boot to help remove any free water that comes in with the feed. The removal of the sour water, typically to a sour water header, is a manual operation based on an interface level indicator.

The feed surge drum pressure is controlled by a split range controller to maintain the pressure some quantity above the bubble point of the naphtha. On a low pressure signal, hydrogen or fuel gas will be added to the drum by opening that control valve. On a high pressure signal, the hydrogen or fuel gas valve will close and the vent valve to the fuel gas header or relief header will open. At steady state, both valves should be closed.

Naphtha is routed out the feed surge drum bottom to the charge pumps. The level of the feed surge is typically not controlled and is allowed to fluctuate. There is a level indicator on this vessel. At the suction of the charge pumps there is a sulfur injection connection, which is for the sulfiding of the catalyst during the initial startup. For units with very low feed sulfur contents, there may be a normal sulfide injection pump. The sulfide injection rate is set to maintain at least 15-20 weight ppm. This is required to keep the catalyst metals in their optimum state.

There is a minimum flow spillback line from the charge pump discharge back to the feed surge drum to protect this pump from damage. The flow rate to the reactor is set by a flow indicating controller. Low flow will shutdown the feed inlet and combined-feed exchanger control valve to prevent depressuring of the unit.

## 2. Reactor System

Naphtha feed from the charge pump combines with a hydrogen-rich gas stream, and this combined feed enters the combined feed exchangers, usually on the shell side, where it is heated. The combined feed leaving the exchanger is all vapor, and flows to the charge heater where it is heated to the required reaction temperature. The amount of fuel burned in the heater is controlled by the temperature of the combined feed leaving the charge heater and flowing to the reactor. The temperature controller resets the charge heater fuel gas pressure controller. In some cases a slip stream of combined feed by-passes the combined feed exchanger. This is done to improve the heater firing control by slightly cooling the total combined feed to the charge heater.

The combined feed enters the reactor and flows down through the catalyst bed. When processing straight run naphtha, there is generally very little change in the temperature across the catalyst bed. The reactor effluent enters the combined feed/reactor effluent exchangers, usually on the tube side, where it is cooled. The reactor effluent is then further cooled at the product condenser, in preparation for gas-liquid separation. A wash water injection point is provided in the reactor effluent line to the product condenser to dilute any hydrogen chloride present and to prevent salt buildup in the line or the condenser.

## 3. Wash Water System

Water wash injection points are provided to three different locations in the reactor effluent line. The first two are at the combined feed exchanger and the other is just upstream of the product condenser. The wash water is used to dilute any hydrogen chloride that might be present and so that any salt buildup in the combined feed exchangers, process lines or condenser may be washed out. The typical wash water injection point is just after the last combined feed exchanger bundle, but this should be verified by calculating the dew point and the ammonium chloride desublimation temperature. This water injection should be on a continuous basis. The wash water injection pump injects enough fresh water, typically 3 liquid volume percent of the charge rate, via the flow indicating controller to the system. This

amount is sufficient to prevent salt buildup and dilute hydrochloric acid when processing feeds that contain some organic chloride, typically less than 20 weight ppm. If the feed chlorides are high, then alternative chloride corrosion control is required. The wash water tank is supplied from the cold condensate on level control.

The separator sour water should be monitored regularly, per the analytical schedule in Section IX, to insure that proper corrosion control is occurring. The goal is to keep the separator sour water between 5.5 – 6.5 pH. Failure to do so can result in corrosion, and possible line rupture, in reactor effluent piping and equipment as the process stream cools. Achieving the proper pH is normally not difficult when the feed chloride levels are less than 20 weight ppm. Some adjustment to the wash water injection rate can be made to further dilute the hydrogen chloride. However, the rate should not be decreased below 3 liquid volume percent of the feed rate. If the injection point is changed to a "hotter" location then the rate will need to be increased. It is important that at least 25% of the water injected remains in the liquid phase. If further information on chloride corrosion control is required, please contact UOP.

The reactor effluent and injected water flows to a Product Condenser and into the Separator. The product separator is provided with a water boot to collect the water injected. This water is usually pressured, via interface level control, to a sour water stripper for disposal. The waste water quality should be monitored at this point.

#### **4. Separator System**

Reactor effluent and injected water flows out of the product condenser at a low enough temperature to ensure complete recovery of the naphtha and enters the Separator. A mesh blanket coalescer is provided in the separator to ensure complete separation of gas, hydrocarbon liquid, and water.

##### *Pressure Control*

The reactor circuit pressure is controlled at the Separator by the pressure indicating controller. There are two scenarios, which are discussed, for which the make-up gas is brought into the Naphtha Hydrotreating Unit. The differences are dependent on the pressure of the makeup gas. When the pressure of the make-up gas is higher

than the separator pressure, this pressure controller directly sets the rate of the make-up hydrogen into the unit to replenish the hydrogen consumed by the reactions and keeps the pressure constant (Figure IV-1). Typically the make-up gas comes from the Net Gas Chloride Treaters of the Platforming Unit and is introduced in the reactor effluent line just upstream of the Product Condenser. The separator also has a hand-controlled valve on the gas effluent line, which is normally closed, that can be used to depressure the unit to the relief header in case of emergency.

For units where the make-up hydrogen is at a pressure lower than the separator, the gas must be increased in pressure via a make-up compressor. The Platforming Unit operates at a substantially lower pressure than the Naphtha Hydrotreating Unit and thus the make-up hydrogen must be increased in pressure. The make-up hydrogen is also introduced into the reactor effluent line just upstream of the Product Condenser. The make-up hydrogen is brought in from the Net Gas Chloride Treaters of the Platforming Unit through the Make-up Gas Compressor Drum and Make-up Gas Compressor. The Make-up Gas Compressor Suction Drum contains a monel mesh blanket to remove any entrained liquid droplets before entering the reciprocating compressors. The Separator pressure and Make-up Gas Compressor Drum pressure send a signal to the low signal selector. The low signal selector then controls the spillback valves of the Make-up Gas Compressor. As the signal decreases, the spillback control valve closes and allows more make-up hydrogen to enter the Naphtha Hydrotreating Unit. For example, the Separator pressure becomes too high, then the controller will open the spillback control valves to reduce the make-up hydrogen flow rate to the unit. There is a water cooled exchanger in the spillback line to prevent overheating of the Make-up Gas Compressor.

### *Recycle Gas*

There are alternate methods for providing the required hydrogen-rich gas to the reactor. Most common is a Recycle Gas Compressor taking suction from the top of the Product Separator with the discharge joining the naphtha feed upstream of the combined feed/reactor effluent exchanger. This flow scheme is depicted in Figure IV-1. The gas leaves through the top of the Separator and goes into a Recycle Gas Compressor Suction Drum and on to the Recycle Gas Compressor. The Recycle Gas Compressor Suction Drum contains a monel mesh blanket to remove any entrained liquid droplets before entering the reciprocating compressors. This drum

also is equipped with two trays and connections for water addition. These features are to be used during catalyst regeneration. In normal operation, any condensed liquid is manually routed to the Stripper Column in a batchwise fashion. There are typically two single-stage recycle gas reciprocating compressors that can operate between 50-100% of the design recycle gas flow rate.

#### *Once-Through Gas*

In some units, rather than having a Recycle Gas Compressor, a comparable amount of a hydrogen-rich gas stream is brought into the unit on flow control, and flows on a once-through basis through the reactor section to the Product Separator where it is vented on pressure control. This flow scheme is depicted in Figure IV-2.

The choice between these flow schemes is made during the design of each unit based upon the availability of a high pressure hydrogen-rich gas stream, and the cost of compression for each stream.

### **C. STRIPPING SECTION**

The liquid hydrocarbon in the separator is pressured on level control through the stripper feed/bottoms exchanger, and the heated material enters near the top of the stripper. A reboiler, normally a fired heater, is provided to supply the required heat input for generating vapor. This vapor strips hydrogen sulfide, water, light hydrocarbons and dissolved hydrogen from the feed to the stripper, which then passes overhead to the overhead condenser and to the overhead receiver. Normally, no net overhead liquid product is produced, and all of the liquid in the receiver is pumped back to the stripper as reflux. A reflux/feed mole ratio of approximately 0.25 is sufficient to strip the light ends and water from the tower. The reflux is pumped into the stripper on receiver level control. To increase the amount of reflux, the reboiler heat input must be increased to provide more overhead material. The reboiler firing is controlled by the reboiler stream pressure differential controller, to set the amount of vaporization of the bottom stream. A temperature controller is not used since there is typically little temperature change in

vaporization. The net overhead gas leaves the receiver on pressure control, usually to amine scrubbing and then to fuel gas. The flow scheme is shown in Figure IV-4.

The stripper overhead system is equipped with inhibitor addition facilities to prevent corrosion of the process lines and equipment by hydrogen sulfide in the overhead vapor. The corrosion inhibitor is pumped directly from a drum, diluted with a small slipstream of reflux, and injected directly into the overhead vapor line at the top of the stripper.

The stripper bottoms material is pumped through the feed/bottoms exchanger and is usually charged directly to the Platforming Unit. On many units, a small slipstream of stripper bottoms is further cooled in a trim cooler and sent to storage for later use as sweet startup naphtha.

The dry, stripped Naphtha Hydrotreating Unit product must meet the following specifications to be acceptable as Platforming Unit feed:

|                                |          |
|--------------------------------|----------|
| Total Sulfur, wt-ppm           | <0.5     |
| Total Nitrogen, wt-ppm         | <0.5     |
| Chlorides, wt-ppm              | <0.5     |
| EP, °F                         | 400 max. |
| *Lead, wt-ppb                  | <20 max. |
| *Arsenic, wt-ppb               | 1 max.   |
| *Iron + Chloride, wt-ppm       | 1 max.   |
| *Copper + Heavy Metals, wt-ppb | <25 max. |

Additionally, water plus total oxygen must be low enough to produce less than 5 mole ppm water in the Platforming Unit recycle gas with no water injection to that unit.

\* Lower limit of detection of the test method.

#### D. SPLITTER SECTION

In some special applications, the Stripper bottoms material contains C5 and minus compounds and it will be necessary to fractionate the hydrotreated naphtha before sending to the Platforming Unit. The hydrotreated naphtha is fractionated in the Naphtha Splitter. Light naphtha is typically sent to gasoline blending. The heavy naphtha is sent to the Platforming Unit and should meet the specifications outlined in the previous section.

The splitter is designed to split the C5 and C6 components. The light naphtha product is mostly a C5 fraction, and the heavy naphtha is a C6+ fraction. The C5 fraction is not desired in the Platforming Unit. For greater flexibility, the splitter may also be designed to provide a split between C6 and C7 components. A refiner may want to limit the amount of benzene, methyl-cyclopentane and/or cyclohexane in the heavy naphtha product. The amount of C7+ material can also be limited for the light naphtha product.

Typically, the Naphtha Splitter feed is preheated by the stripper bottoms material in the stripper feed-bottoms exchanger. The splitter feed is pressured on level control into the splitter. A reboiler, usually steam, is provided to supply the required heat input for the column. The heat input is controlled by the steam condensate flow. The overhead vapor is condensed in an air cooled condenser and trim condenser, and liquid collects in the splitter receiver. The receiver level is controlled by a total net overhead flow controller. This controller regulates the amount of reflux back to the column. The light naphtha product flow is cascaded to a temperature controller at a top tray of the column. The flow scheme for the splitter is shown in Figure IV-5.

The splitter pressure is controlled by a pressure controller on the overhead line. Any off gas or non-condensibles that build in the receiver can be vented to a relief header with a hand control valve. The heavy naphtha product is pumped on level control through the stripper feed-splitter bottoms exchanger to the Platforming Unit. If necessary the heavy naphtha can also be sent to tankage after first being cooled. The heavy naphtha usually passes through a heavy naphtha air cooler and a trim cooler before that material can be safely sent to tankage.

## **E. ALTERNATE OPERATIONS**

The hydrotreating columns can also be used for alternate operations when the reactor section is not processing sour naphtha. The columns were designed specifically for two operations. They are 1) to stabilize unstabilized naphtha from storage and 2) to strip any water from the sweet naphtha from storage that will be charged to the Platforming Unit.

### **1. Stabilizing Naphtha**

Unstabilized naphtha is charged to the feed surge drum. From the drum the naphtha is pumped to the stripper column on flow (FRC) control. The naphtha bypasses the reactor section and also the stripper cold feed exchanger. The stripper column, which will run at a lower pressure than design, will remove the proper amount of light ends to achieve the RVP specification. The stabilized naphtha is pressured from the bottom of the column through the stripper hot feed exchanger and the "naphtha to storage" cooler and then to the stabilized naphtha storage tanks.

### **2. Stripping Sweet Naphtha**

Sweet naphtha from storage is pumped to the stripper or naphtha splitter. This flow is controlled by the level in the bottom of the column. The stripper or splitter will run with total reflux. The stripper column removes the water in the overhead receiver water boot. The splitter column removes water out the overhead receiver off-gas line. The splitter overhead receiver usually does not have a water boot. The dry, sweet naphtha is then pumped directly to the Platforming Unit.

**FIGURE IV-1**  
**TYPICAL NAPHTHA HYDROTREATING UNIT**  
**REACTOR SECTION WITH RECYCLE GAS**

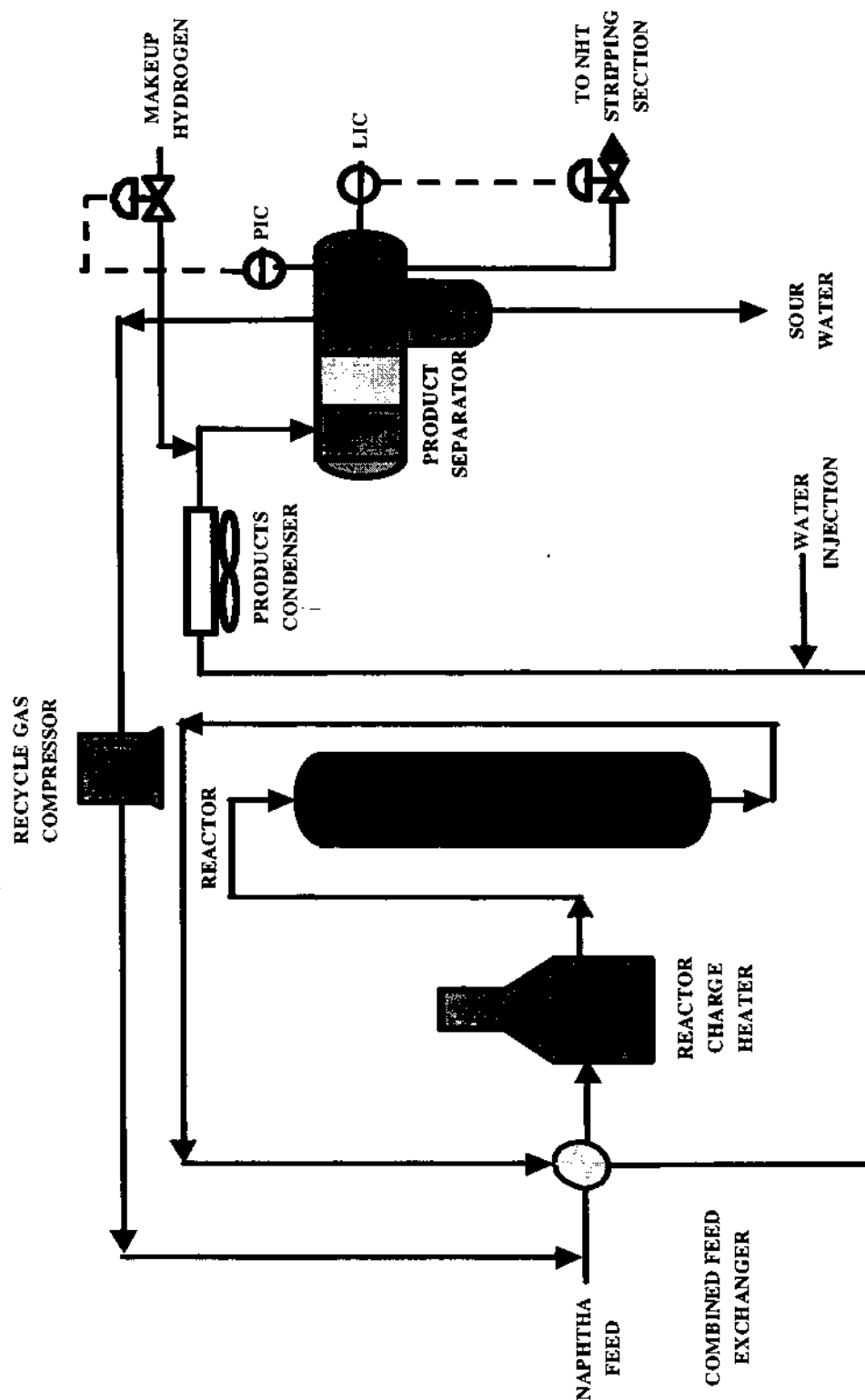


FIGURE IV-2  
TYPICAL NAPHTHA HYDROTREATING UNIT  
REACTOR SECTION WITH ONCE-THROUGH GAS

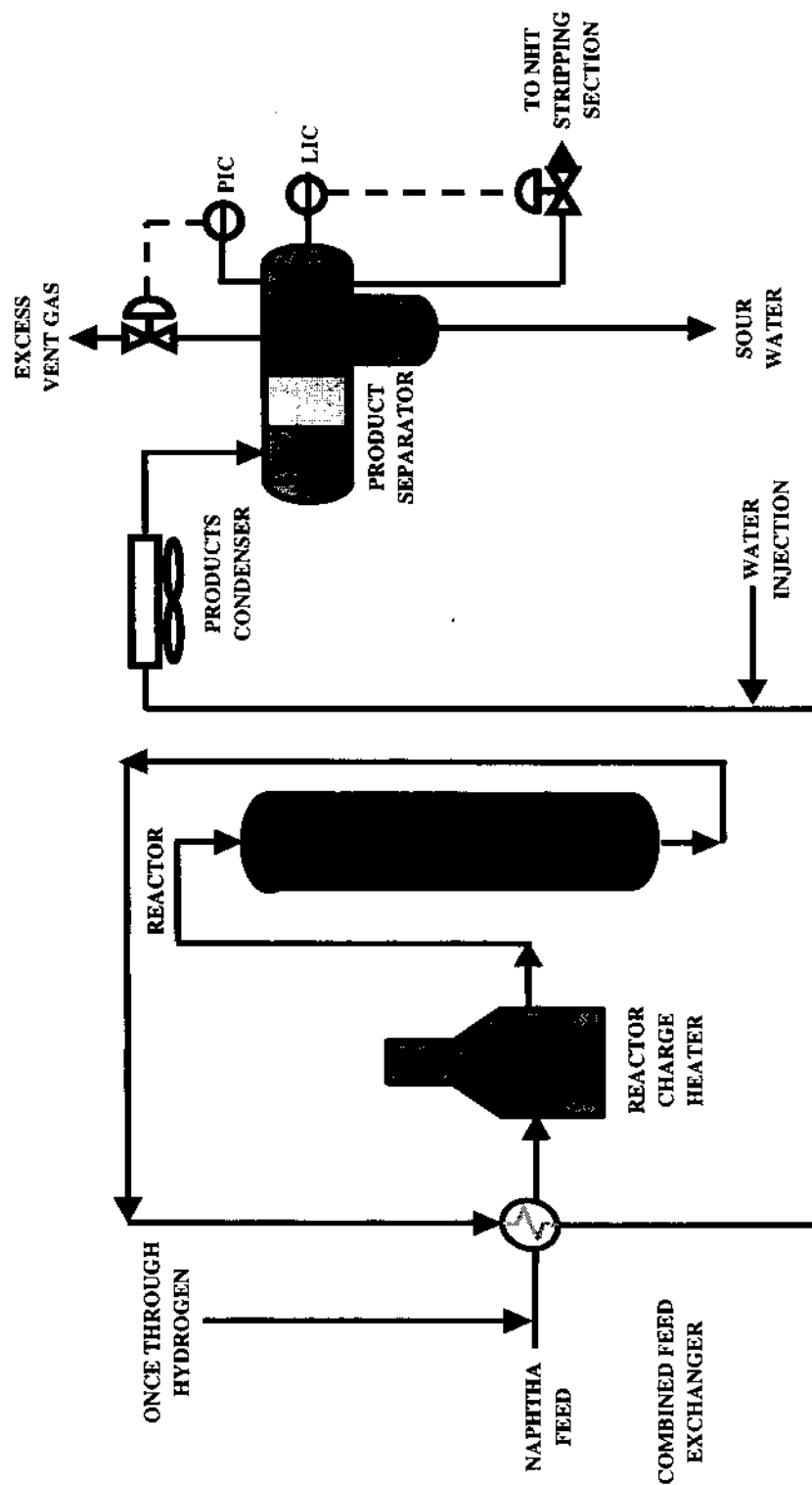
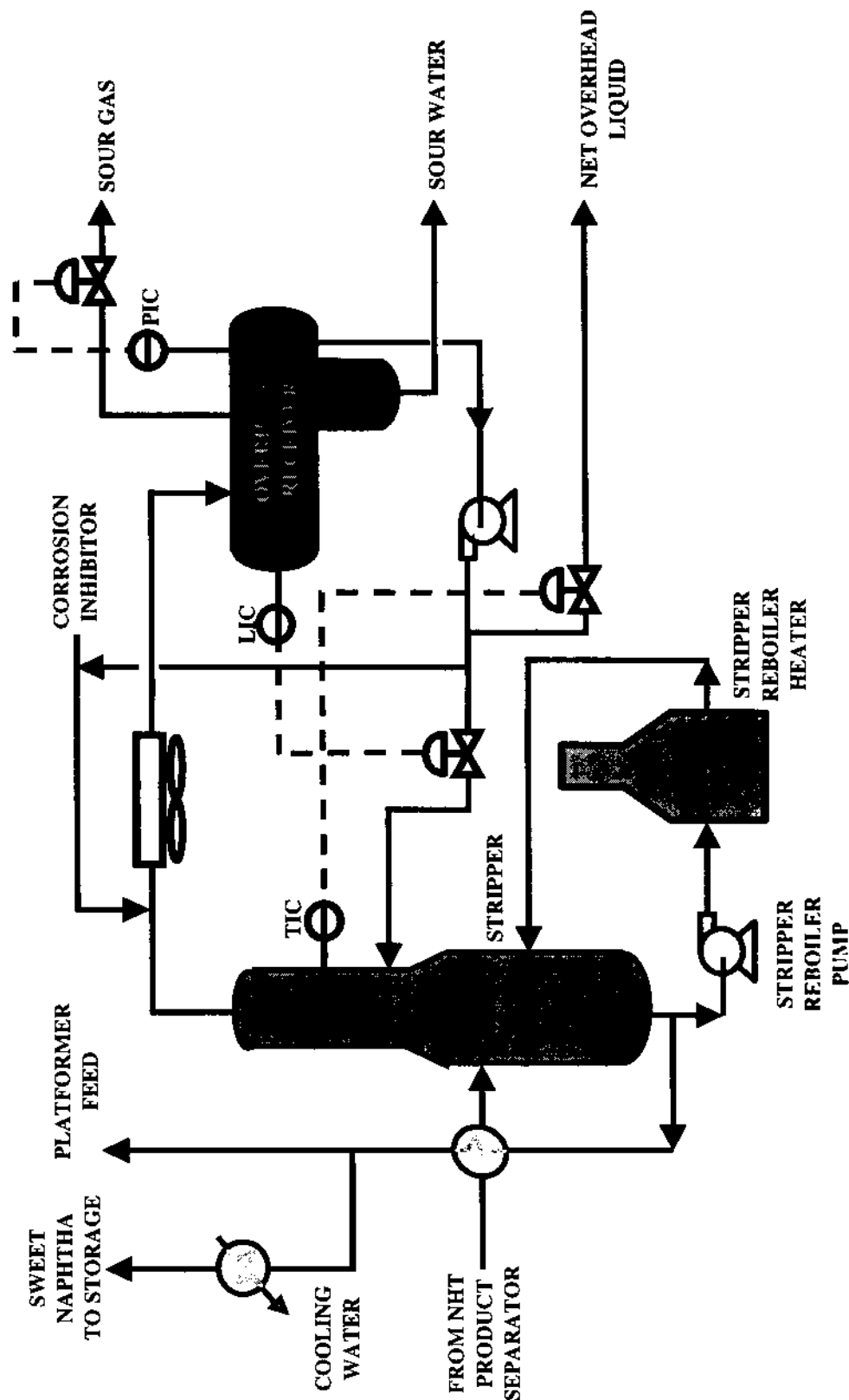
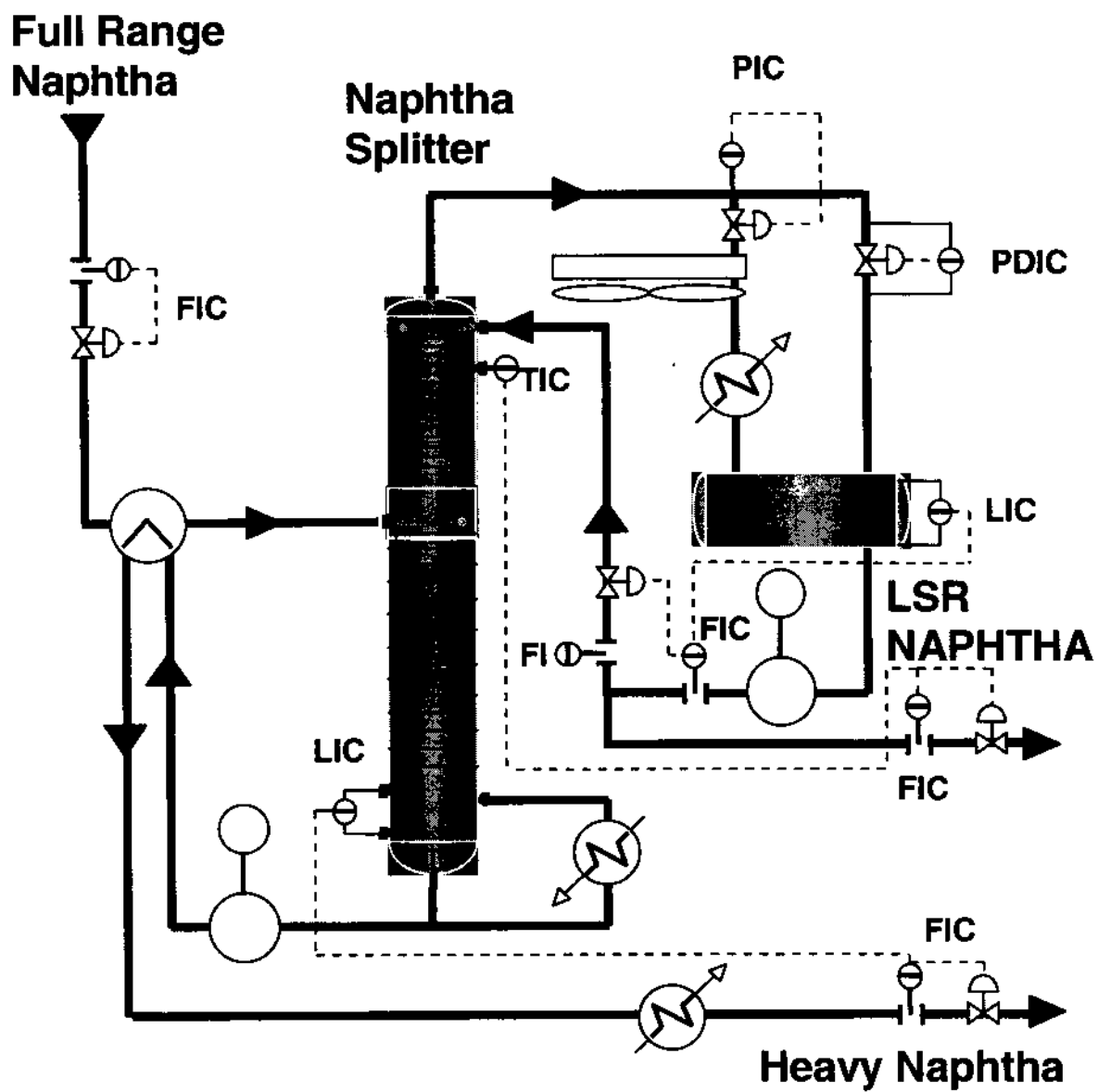




FIGURE IV-4  
TYPICAL NAPHTHA HYDROTREATING UNIT  
STRIPPING SECTION



**FIGURE IV-5**  
**TYPICAL NAPHTHA HYDROTREATING UNIT**  
**SPLITTER SECTION**



## V. PROCESS EQUIPMENT

### A. REACTORS

The UOP Naphtha Hydrotreating Unit utilizes downflow reactors. Typically this consists of one reactor, but for certain feedstocks two reactors in series are required. In general, the purpose of the hydrotreating reactors is to allow the feed to contact the catalyst at reaction conditions while not allowing the catalyst to leave with the product. Catalyst containment is one of the goals of the design. Process vapors enter through the top of the reactor, via an inlet distributor, and flow down through the catalyst bed and out the bottom of reactor.

Typically the naphtha hydrotreating reactor is constructed of killed carbon steel with an alloy lining. The inlet distributor located at the top of the reactor prevents the vapor from disturbing the catalyst bed and enhances the flow distribution through the catalyst. Usually there are two layers of graded bed material on top of the catalyst bed. This aids in flow distribution and minimizes the pressure drop across the reactor. The depth of each layer is a function of the reactor dimensions and the feed types. The top layer is typically 4 to 6 inches deep (100 mm to 150 mm) and consists of specially shaped inert ceramic material used to filter larger particles from the feed. The second layer ranges from 12 to 24 inches (300 mm to 600 mm) in depth and is another specially shaped material, but includes active metals.

At the bottom of each reactor are ceramic support material (balls) of different diameters which help in the flow distribution of the reactor effluent out of the reactor. The varying diameters of the support material are utilized to prevent catalyst migration. An outlet basket prevents the ceramic support material from leaving the reactor.

### B. HEATERS

A Charge Heater is used to supply sufficient heat to the combined feed so that the desired reactions can be obtained with the hydrotreating catalyst in the reactors. The Charge Heater is typically a radiant-convection type with one firing zone, with fuel gas-fired burners located on the floor of the heater box. It is normally a cylindrical updraft type having vertical tubes in the radiant section and sometimes horizontal tubes in the convection section. The combined feed will first flow through the convection section and be preheated. There are a number of passes in the radiant section and each pass contains skin thermocouples. These thermocouples can warn of tube plugging from two-phase flow, mainly during startup. The skin temperature of each pass should be relatively the same.

A snuffing steam connection is provided for purging out any combustible gases from the firebox before lighting pilots during startup.

The firing pattern of the burners should be closely observed, and adjusted if necessary. As in all heaters, flames impinging on the tubes should be avoided. A slightly negative pressure at the bridgewall should be present to provide adequate draft at the burners. If inadequate draft is available at the burners, insufficient air may be available through the burner to complete combustion. This could cause a loss of efficiency, ballooning flame dimensions, or after-burning. As excess air to a burner declines below acceptable levels, flame dimensions increase; unburned hydrocarbon will travel a greater distance to come in contact with oxygen and ignite. There is an oxygen analyzer to monitor the excess oxygen content in the flue gas.

Ballooning flame dimensions can cause a maldistribution of heat or flame impingement. A further decrease in available air may result in incomplete combustion. Unburned fuel is useless and lowers efficiency. Unburned fuel can also ignite in other than burner areas where air can enter the furnace (i.e., tube sheets, inspection doors). This is known as after-burning and can cause tube damage (if ignition occurs in tube areas), refractory damage or structural damage.

Dampers located in the stack above the convection section control draft through the heater. Draft gauges (vacuum gauges) are installed in the radiant sections, convection inlets, and before and after the damper to monitor draft through the heater. A negative pressure must be maintained for safe, efficient heater operation.

### **C. HEAT EXCHANGERS**

Heat exchangers are used to heat and cool many streams in the Naphtha Hydrotreating Unit. The shell and tube combined feed exchangers (CFE) allow the hot reactor effluent to add heat to the hydrotreating feed before the Charge Heater. The reactor effluent is then cooled further so that hydrogen can be separated from the unit product. The total reactor effluent is condensed by an air cooler and trim cooler.

Heat exchangers are used for the reboilers of the Stripper and Splitter Columns. Steam can be used for the Stripper and Splitter Columns.

### **D. RECYCLE COMPRESSORS**

The Naphtha Hydrotreating Unit has one or two reciprocating, motor-driven recycle compressors. The recycle compressors circulate hydrogen-rich gas through the hydrotreating reactor circuit. Without hydrogen circulation, large amounts of coke will form on the catalyst that will prevent the desired catalytic reactions. It is critical to maintain recycle gas flow when feed is being charged to the unit.

### **E. PUMPS**

There are many types of pumps used in the Naphtha Hydrotreating Unit. A high-head multi-stage pump is usually used to supply feed to the reactor section that is at much higher pressure than the Feed Surge Drum. Proportioning pumps are used for chemical injection, such as inhibitor or condensate.

### **F. FEED SURGE DRUM**

The Feed Surge Drum is a pressurized, horizontal killed carbon steel vessel. The naphtha hydrotreating feeds enter through a baffle distributor located at the bottom of the Feed Surge Drum and leaves at the opposite end. A level indicator and level

glass show the hydrocarbon level. Maintaining a liquid seal in the bottom of the drum is important. The liquid outlet line has a vortex breaker. The Feed Surge Drum has a water boot to collect and remove any free water that might be present.

## **G. SEPARATOR**

The Separator is designed primarily to separate hydrogen from hydrocarbon. The Separator is a horizontal killed carbon steel vessel lined with an alloy, and occasionally concrete, for corrosion protection. The cooled reactor effluent enters through a slot type distributor at one end of the vessel to permit proper mixed phase distribution. The hydrogen and liquid separate and both pass through a vertical monel mesh blanket. The mesh blanket is used as a demister pad to coalesce, or help remove, entrained hydrocarbon droplets from the gas stream. A level indicator shows the hydrocarbon level and a level controller controls the flow of hydrocarbon from the separator to the Stripper. Maintaining a liquid seal in the bottom of the separator is important. The liquid outlet line has a vortex breaker.

There is also a water-boot to remove the injected water. A level indicator shows the water level and a level controller controls the flow of water from the Separator. Regular sampling of this water should be performed to verify proper corrosion control.

## **H. OVERHEAD RECEIVERS**

The Stripper and Splitter columns have receivers to collect condensed overhead vapors. The Stripper receiver inlet, has a slotted distributor to permit proper mixed phase distribution. A water boot collects any free water that might be present. There is a level glass and a level control bridle nozzle for the hydrocarbon phase and a level indicator for the water phase. A gas outlet nozzle permits non-condensable gas to go overhead. This valve also acts as the column pressure controller. The liquid outlet lines have a vortex breaker.

The Splitter receiver is basically the same as the Stripper receiver with no water boot. A gas outlet nozzle allows off-gas to go to a relief header. This is controlled

by a hand control valve. A total overhead flow controller typically controls the receiver level.

The typical material of construction of the Splitter receiver is the same as would be used on the Splitter column, which is carbon steel. The Stripper column and receiver are constructed of killed carbon steel. The overhead receiver design temperature is much higher than its operating temperature. The receiver is designed to withstand temperatures that may develop if the overhead condenser should fail.

#### **I. RECYCLE COMPRESSOR SUCTION DRUM**

The Recycle Compressor Suction Drum is a small vertical vessel designed to remove condensable material from the recycle compressor suction stream and thus protect the compressor. The gas stream from the Separator enters the vessel from the side and travels out the top. A partial (monel) mesh blanket is installed to remove entrained liquid. The 2 bubble cap trays are used during regeneration only.

There is a level glass for the liquid hydrocarbon phase. The liquid that is knocked out can be drained manually to the Stripper column.

#### **J. STRIPPER COLUMN**

The stripper column is used to remove light ends, H<sub>2</sub>S and water from the light naphtha product stream. The stripper is typically fabricated out of killed carbon steel with carbon steel or stainless steel valve trays. The top part of the column is narrower than the bottom due to the lower volumes of liquid and vapor in the top section of the column.

#### **K. SPLITTER COLUMN**

The Splitter is used to separate the light naphtha from the heavy naphtha product. The hexane (C6) components and heavier will be taken out the bottoms and sent to the Platforming Unit, tankage or blending system. The pentane (C5) components and lighter will go overhead where they are condensed and the net liquid will be sent to the tankage or blending. The Splitter is typically fabricated out of carbon steel with carbon steel valve trays.

## **VI. COMMISSIONING**

### **A. PRECOMMISSIONING**

#### **PLANT INSPECTION**

Sections of the unit should be checked out by both refinery and UOP personnel as soon as the contractor completes work in those areas. Immediately following inspection of those areas, punch lists which indicate the deviations from the UOP design specifications should be written and distributed to the contractor. In this manner mistakes in construction can be found and corrected early.

Inspection of the plant can be basically divided into the following areas:

1. Vessels
2. Piping
3. Heaters
4. Exchangers
5. Pumps
6. Compressors
7. Instrumentation
8. Catalyst/Chemical Inventory

A discussion and lists of the major points which must be examined in the inspection of these areas follows:

#### **1. Vessels**

The actual installations must be compared against the UOP drawings to assure that the vessels will function as intended. The reactor internals must conform exactly to the UOP design specifications if good distribution is to be attained and catalyst migration is to be avoided. Particular attention must be paid to the following details:

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## Specification Check

1. Review UOP design specifications with the vendor drawings to verify agreement on:
  - a. Pressure, temperature, and vacuum ratings.
  - b. Shell metallurgy, thickness, and corrosion allowance.
  - c. Nozzle size and orientation; flange rating, type and finish.
  - d. Type of lining, thickness and material.
  - e. Stress relieving and/or heat treatment.
  - f. Foundation design for full water weight.
2. Confirm that the vessel has been hydrostatically tested.
3. Verify that all code plate information on the vessel is correct.

## Internal Inspection

### a. Reactors

1. Inlet distributors, quench distributors: metallurgy, type, size, opening sizes, freedom to expand.
2. Vapor/liquid distribution trays: metallurgy, vapor pipe dimensions, orientation, and opening sizes; packing; supports; welding; levelness.
3. Catalyst support grids: metallurgy; grid type and dimensions; screen type and size; supports; welding.
4. Catalyst unloading nozzles: metallurgy, orientation, length.
5. Outlet stools: metallurgy and dimensions.
6. Distributor baskets and support rings: metallurgy; screen type and size; dimensions; quantities.

7. Thermowells: orientation, length, and metallurgy.

**b. Other Vessels**

1. Vessel trays: spacing; levelness, orientation and dimensions of weirs, downcomers, accumulators, draw-off and trap trays, seal pans, distributors, baffles, nozzles, tray contact devices; metallurgy of trays, contact devices, clips, bolts, nuts and gaskets; freedom of movement of valve caps or other contact devices; number, size, and distribution of tray contact devices or perforated plate holes; proper fit of all internals and proper welding of support rings or other support devices; liquid tightness of drawoff trays, seal pans and accumulators, all bolting and clips tightened.
2. Mesh blankets and outlet screens: size; location; levelness; goodness of fit (no bypassing allowable); and metallurgy of blanket, support, tie wires, and grids.
3. Vortex breakers: type, size, and orientation.
4. Baffles: type, orientation, levelness.
5. Instrument nozzles: location, orientation, cleanliness, thermowell length and metallurgy, baffle size and type.
6. Inlet distributors: type, size, orientation, levelness, freedom to expand.
7. Non-fired reboilers: location, orientation, proper supports.
8. Packing: type, size, support, installation.

9. Internal ladders and other devices: location, size, orientation, properly secured.
10. Lining and refractory.
  - a. Hex-steel for concrete lining: clean and properly secured. Lumnite or other specified cement applied according to UOP specifications, with no holes or gaps in the applications.
  - b. Metal linings in good condition. Weld overlays have no gaps or holes in the application.
  - c. Lining is of the proper thickness and covers the required portion of the vessel.
  - d. Other refractory installed correctly with no gaps or holes in the application.

**c. General**

The vessel should be clean (free from trash) and should not have excessive mill scale.

**External Inspection**

1. Manways and nozzles: location, size, flange rating and finish, metallurgy, with proper gaskets, nuts and bolts.
2. Ladders and platforms: correctly positioned, secure and free to expand.
3. Insulation and steam tracing: provided as specified and has expansion joints as required.
4. Vessel grounded correctly.

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5. Correct vessel elevation.
  6. Valves and instrumentation: easily accessible from grade or platform.
  7. Piping:
    - a. Adequate supports and guides for all connecting lines.
    - b. Level and pressure instrument connections drain to a safe location.
    - c. Vents to atmosphere or blowdown provided as specified.
    - d. Relief valves have been bench tested.
    - e. Check valves exist on utility line connections where hydrocarbon backup could occur.
    - f. Connections available for steaming/purging of the vessel.
  8. Fireproofing of structure and supports is complete.
  9. Instrumentation:
    - a. Level glass floats center positioned correctly with respect to vessel tangent line, and are readable from grade or platform.
    - b. Through-view level glasses have rear light for illumination.
    - c. Flange ratings, metallurgy, size, etc. are all correct.

## 2. Piping

The unit must be constructed in accordance with UOP Piping and Instrumentation Diagrams (P&ID's), including all details, elevations, dimensions, arrangements, and other notes on the P&ID's. One must be able to startup, shutdown and conduct normal operations on the unit as envisioned in the UOP design. Also, piping for special procedures such as dry-out, special materials preparation, regeneration and/or alternative flow schemes may have been incorporated into the unit's design, and the unit should be able to operate in all of these modes with piping as designed and constructed.

If the unit is connected to other process facilities, adequate means must be provided to receive feed from or send products to these facilities without contamination of these streams. Minimize as much as possible the effects of upsets of other process units on the operation of the Naphtha Hydrotreating Unit, especially where contamination of feed or product stream might occur. Check all tankage interconnections to minimize the possibility of stream contamination outside of the battery limits.

Check that adequate means of measuring flows, pressures, and temperatures, and sampling of all process streams has been provided.

The following items must be checked to ensure conformity to the UOP design specifications:

- a. Flanges: rating, facing, and metallurgy; type (typically, 2" and smaller are socket weld, 2-1/2" and larger are weld neck flanges).
- b. Gaskets: type; metallurgy (materials or retainer, jackets, winding, filler, etc.); thickness, ring size, etc.
- c. Fittings, connections and couplings: rating and metallurgy.
- d. Valves: rating and metallurgy (body, trim, seats, etc.); packing; seat inserts; bonnet gaskets; grease seals; socket-weld or flange type, rating

and facing; installed in correct direction of flow; lubricant provisions; gear operators; extended bonnets; stops; ease of operation.

- e. Bolting: stud or machine bolts; bolt and nut metallurgy; bolt size.
- f. Pipe: metallurgy, thickness; seamed or seamless; lining.
- g. Tubing: size and thickness; metallurgy; seamed or seamless.
- h. Gauge glasses:
  - (1) Through-view types should have rear-mounted lights.
  - (2) Design pressure and temperature.
  - (3) Special materials of construction.
  - (4) Drains to safe location.
  - (5) Visible from grade (or platform, if required).
- i. Pressure relief valves:
  - (1) Size and style.
  - (2) Lever requirement.
  - (3) Inlet/outlet flange material, facing and rating.
  - (4) Set pressure – must be bench tested.
  - (5) Metallurgy of nozzle, disc, spring, etc.
  - (6) Type (pilot operated, balanced, etc.).
  - (7) Inlet/outlet block valves car-sealed open; valve stems installed in horizontal or below.

j. General:

- (1) Utility systems within the battery limit should follow all relevant pipe class specifications in the same detail required for process lines.
- (2) Package systems (skid-mounted units, etc.) shown on the UOP P&ID should follow all relevant pipe class specifications in the same detail required for other process lines.
- (3) Expansion: review the physical installation to insure that no expansion problems will occur when the unit gets hot and that:
  - (a) Column overhead, reflux, feed and other lines are free to expand.
  - (b) Rotating equipment will not be pulled out of alignment.
  - (c) Sufficient expansion loops have been provided on long hot lines.
  - (d) Pipe shoes are free to move in one direction, and are resting on supports of sufficient size that the shoe will not fall off the support.
- (4) High point vents and low point drains should be installed where necessary.
- (5) Spectacle blinds should be provided where required.
- (6) Car-sealed valves should be locked in proper position.
- (7) Spring hangers should have locking pins removed (after hydrotesting) and necessary adjustments should be made for hot/cold position after startup.

### **3. Fired Heaters**

The heaters must be inspected to ensure that they can be operated in a safe and efficient manner and that the required heat duty needed for the process can be provided. After all, it is important that the possibility of a tube rupture or other heater mishap is minimized.

In particular the following items must be checked:

#### **Specification Check**

All UOP design specifications should be reviewed with vendor drawings to verify agreement on:

1. Conformity to process requirements.
2. Heater type.
3. Tube arrangement, metallurgy, size, and thickness (note that tube metallurgy may be different for radiant, convection, and convection shield tubes).
4. Instrumentation connections.
5. Tube supports and support metallurgy.
6. Refractory.
7. Access doors, observations ports, steam smothering connections, and explosion doors.
8. Stack arrangement.

## **Internal Inspection**

### **a. Radiant Section**

1. Arrangement and symmetry of tube coil with respect to heater wall, burner rings, and tube spacing.
2. Vertical length of tube coil with respect to supports and guides.
3. Fuel gas, fuel oil and pilot burner tips are clean and oriented properly. Burners are properly mounted with clearance for firing and removal. Castable refractory has not been used for burner blocks. Fuel oil tip sizing is adequate with respect to actual fuel oil viscosity.
4. Tubeskin thermocouples, if required, are located properly and installed so that they have good contact with the tubeskin.
5. Refractory is in good condition before and after refractory dry-out. No refractory is resting on tubes.
6. Heater shell expansion joints are packed with asbestos wool and clean.
7. Adequate space for tube expansion.
8. Heater shell is sealed to prevent escape of hot gases and entrance of atmospheric moisture during shutdown.
9. Smothering steam and instrumentation connections are not covered by refractory.
10. Heater is clean and free from debris.
11. Heater instrument connections are open – not filled or covered with refractory.

**b. Convection Section and Stack**

1. If extended surface elements are allowed, the bottom three more rows of convection tubes must be bare.
2. No refractory is on the tubes.
3. Expansion provisions are adequate.
4. Damper is free to move fully open and closed; its position indicator is correct both at the stack and at the damper control; damper is weighted to fail open; the damper, support pipe and bolting are all of the correct metallurgy.
5. Sootblowers, if specified, are provided with provision for inspecting the sootblowing operation.
6. Other checks should be conducted as in the Radiant Section inspection.

**External Inspection**

1. Location with respect to process equipment.
2. Platforms for access to all observation ports, instrumentation, sample connections, sootblowers, and damper connections.
3. Adequate number and arrangement of observation ports to permit visual inspection of the entire length of all wall, hip and shock/shield tubes, and the burner blocks.
4. Hand firing equipment located adjacent to an observation port from which that burner can be viewed.
5. Explosion doors located such that heater gases will not flow towards process equipment and platforms.

6. Explosion doors located such that doors can open completely.
7. Symmetry of external piping and crossovers.
8. Instrumentation and sampling connections.
9. Damper position indicator visible from damper control; damper control functioning properly.
10. Pocketed crossover connections have flanged drains.
11. Decoking connections as specified.
12. Sufficient smothering steam connections into heater firebox. Box valves on smothering steam are located remote from the heater, with drain valves and/or steam traps upstream of final block valve for condensate removal. Weep holes provided in smothering steam lines at low points.

### **Fuel Systems**

1. Fuel lines have battery limit block valves which are remote from the heater and easily accessible. Fuel oil piping and its steam tracing are arranged such that no dead legs or pockets are formed. Fuel lines to burners can be easily disconnected from burners for burner removal. All fuel lines have been leak tested.
2. Fuel oil lines at burner valves are correctly piped with steam crossovers. All steam lines have adequate traps and condensate drains.
3. Shutdown solenoids for fuel shutoff valves have been set properly.
4. Fuel oil circulating lines are provided.

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## Heater Instrumentation

1. All draft gauge, pyrometer and analyzer connections are as specified.
2. All heater TRC's fail upscale during power failure or open circuit.

## 4. Heat Exchangers

### Specification Check

The UOP design specifications should be reviewed with the vendor drawings to check:

1. Metallurgy of shell, tubes, tubesheet, channel cover, baffle, header box, etc.
2. Tube size and thickness: number of shell and tube passes and direction of flowing streams; max/min allowable velocities.
3. Design temperature, pressure and pressure-drop ratings.
4. Nozzle size, flange type, rating, facing and metallurgy; vent and drain connections.
5. Design differential pressure between shell and tube sides of the exchanger.

### Field Inspection

In the field the following items should be checked.

1. Name plate verifies UOP specifications.

2. Flange size, rating, facing and gaskets.
3. Insulation for heat retention and personnel protection.
4. Exchanger properly grounded.
5. Tubular exchangers:
  - a. Elevation
  - b. Slot length of sliding plates adequate for expansion. Exchanger should not be tied down at both ends. Check that sliding ends of multi-shell exchangers make sense with regard to expansion of exchangers and connecting pipe.
  - c. Piping symmetry for parallel exchangers.
  - d. Non-condensable vents in steam service or in totally condensing systems.
  - e. Water coolers; inlet at bottom of exchangers; inlet/outlet block valves with a thermal relief valve inside the outlet block valve; vent and drain connections inside the block valves.
  - f. Witness a shell/tube differential pressure test, if possible (especially important in feed/effluent exchangers). When leak testing piping and equipment, ensure that the design shell/tube differential pressure is not exceeded.
6. Air-cooled exchangers:
  - a. "Auto-variable" or "standard-pitch" fans, as specified.
  - b. Motor switches accessible from grade and located near the exchanger.

- c. Fan pitch set correctly as determined by fan amperage draw.
  - d. Vibration switch on each fan.
  - e. Proper motor/fan rotation. Motors properly grounded.
  - f. Proper elevation and distances from connecting equipment.
  - g. Belt tension on motor drive pulleys is equal and correct.
  - h. Motor amperage can be easily checked.
  - i. Exchanger free to expand.
  - j. Manifold piping arrangements as shown on UOP P&ID.
  - k. Split header design where specified.
  - l. Free draining requirements, as shown on P&ID.
  - m. Tube fin surfaces are in good condition with no construction debris on top of the fins.
7. Adequate space has been provided for pulling tube bundles.

## **5. Pumps**

### **Centrifugal Pumps**

#### **1. Specification Check**

The UOP design specifications should be reviewed against the pump curves and data given by the vendor to confirm agreement on the following:

- a. Head and capacity.
- b. Pressure and temperature rating.
- c. Speed.
- d. NPSH requirement.
- e. Pump type, materials of construction, flange ratings, seals, bearings, number of stages, lubrication and cooling systems, etc.
- f. Type of driver.
- g. Balancing lines for multistage pumps must have flanged joints (not unions).

#### **2. Field Inspection**

The following items should be checked in the field:

- a. Sight flow indicators, inlet/outlet shutoff valves on cooling water lines.
- b. Thermometers/pressure gauges for gland seal and flushing oil manifolds.
- c. Restriction orifices (if required) present in seal flush manifold piping.

- d. Pedestals on pumps operating over 500°F (260°C) should be water cooled.
- e. Cooling water to mechanical seals on pumps operating over 250°F (120°C).
- f. Proper direction of rotation.

### **Reciprocating Pumps**

The vendor information should be checked against the UOP specifications to verify agreement on the following:

- a. Head and capacity (minimum, normal, maximum).
- b. Materials of construction (body/glands, plungers, diaphragms, packing, internal check valves).
- c. Cooling/lubrication systems.
- d. Pressure, temperature ratings.
- e. Relief valve setting must be bench tested.
- f. Pump speed and stroke.
- g. Pulsation suppression devices, if required.

Means for calibrating the pump flow rate should be investigated.

## General

The following items should be checked for all pumps:

- a. Piping to be arranged to permit removal or replacement of pump and driver.
- b. Piping independently supported from pump; pump will not be pulled out of alignment when lines get hot; no vapor pockets in piping.
- c. Suction strainer easily removed for cleaning; strainers fit well so no bypassing can occur; strainers have been installed.
- d. Discharge pressure gauge readable from discharge block valve.
- e. Suction/discharge valves easily accessible and operable, and near to the pump. Accessibility of auxiliary piping and controls.
- f. Check that NPSH requirements have been met.
- g. Warm-up lines provided across discharge check valve when pumping hot fluids.
- h. Base plate grouting complete.
- i. Steam tracing and insulation provided on suction/discharge lines, pump casing, and process seal flush lines, as required.
- j. Minimum flow bypasses (with restriction orifice), if required.
- k. All seal oil, warmup, etc. lines have flanged connections and valves to permit removal of pump.
- l. Lubrication and cooling systems operate correctly.

- m. Adequate means for venting and draining the pump casing are available.
- n. Vacuum service pumps must have a discharge vent back to the system to allow filling the pump with liquid.
- o. Pumps and drivers are aligned correctly.
- p. Check valves are of proper type and installed in the correct direction.
- q. All drains from pumps and associated piping and instrumentation should be routed to a safe location.

## **6. Compressors**

### **Centrifugal Compressors**

#### **a. Specification Check**

The UOP design specifications should be reviewed against the vendor information and drawings to ensure agreement on the following:

1. Design capacity, temperature, pressure and specific gravity.
2. Compression ratio, number of stages.
3. Type of compressor, materials of construction, flange type, rating and facing.
4. Lube and seal oil systems, and estimated seal oil leakage.
5. Instrumentation, as supplied, must be in accordance with the UOP design specifications.
6. Piping furnished with the compressor must conform to the same pipe class as connecting lines.

**b. Field Inspection**

The following items should be checked in the field.

1. Materials of construction; flange type, rating and facing; conformity of compressor piping to applicable pipe specification.
2. Instrumentation for conformity to UOP design specifications, location, operability and access.
3. Lube/seal oil system; operability of all sight flow indicators, instrumentation, pumps (including auto starts), filters, coolers, compressor trips, reservoir, seal oil pot, etc. Check that addition and withdrawal of seal/lube oil to/from the reservoir can be performed easily.
4. General cleanliness of all process and lube/seal oil systems, and of the general compressor area.
5. Insulation as required for heat retention and personnel protection.
6. Check for access to sour oil and compressor casing drains, and that those drains are routed to a safe location.
7. Proper supports on suction/discharge piping.

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## **Reciprocating Compressors**

### **a. Specification Check**

The vendor information and drawings should be compared with the UOP design specifications to verify conformity on the following items:

1. Type of compressor, materials of construction, flange type, rating and facing.
2. Number of stages, compression ratio for each stage.
3. Design capacity, pressure, temperature and specific gravity for each cylinder.
4. Compressor speed; piston speed.
5. Lubricated or non-lubricated.
6. Single or double acting; balanced and opposed or dummy crosshead; fixed or variable clearance pockets; auto or manual suction valve unloading.
7. Cooling to cylinder, packing, gearbox, lube oil.
8. Lube oil system operation.
9. Pulsation suppression devices; distance piece design; packing and distance piece vent piping.

**b. Field Inspection**

The following items should be checked in the field:

1. Materials of construction; flange type, rating, finish, gasketing.
2. Instrumentation for conformity to UOP design specifications, location, operability and access.
3. Lube oil system/cooling water piping, including sight flow indicators.
4. Distance piece/packing vents piped correctly.
5. Adequate and accessible drains which are routed to a safe location.
6. Single strand steam tracing on the bottom of suction lines and snubbers, as specified; insulation as specified for heat retention and personnel protection.
7. Automatic suction valve unloader operation.
8. Two compartment distance pieces, if required ( $>30$  mol-%  $H_2$  in process gas).
9. Acidizing requirements have been met and acidized piping is not in contact with the atmosphere (under nitrogen blanket), includes both process and lube oil piping.
10. Provision for suction strainers, if required.

## 7. Instrumentation

All personnel on site should check to ensure that the instrumentation is provided as specified by UOP; that it is functional; and that a minimum of instrumentation problems will occur when the unit is commissioned. Some of the basic items which must be checked include the following:

- a. Instrument type, range, and size.
- b. Materials of construction and rating of instrument, accessories, and connecting piping, flanges, and valves.
- c. Accessibility of instrument for routine checks and maintenance; rigidly mounted.
- d. Installation according to correct UOP drawing details.
- e. Accessories (pulsation dampeners, filter/regulators, diaphragm seals, excess flow checks, seal pots).
- f. Location of local indicators so they are readable from grade platform or controller assembly, as required.
- g. Process requirements of flow, temperature, pressure, differential pressure, specific gravity, etc.
- h. Controller type, number of modes, chart type, range, cascades.
- i. Power requirements of voltage, frequency; emergency power supply and connections.
- j. Calibration of controllers, transmitters, analyzers, special instrumentation.
- k. Control valve, block and bypass valve sizes for control valve assemblies.

## **8. Catalyst/Chemical Inventory**

### **Catalyst**

- a. It must be verified that sufficient quantities of catalyst, catalyst support material, and other materials (such as asbestos rope, etc.) are on site, are in good condition, and are properly stored (for example, in drums, indoors, and on pallets to prevent contact with moisture).
- b. It must be verified that all equipment required to load the catalyst is on site and in good condition.

### **Chemicals**

It must be verified that the proper type and quantity of chemicals (such as inhibitors, demulsifiers, soda ash, caustic, etc.) are on site and stored properly.

## **B. PRELIMINARY OPERATIONS**

Prior to the commissioning of the plant there are several operations that must be conducted by contractor and refinery personnel to prepare the plant for the actual startup; these are:

1. Commissioning of utilities
2. Final inspection of vessels
3. Pressure test equipment
4. Acid cleaning of compressor lines
5. Wash out lines and equipment and break-in pumps
6. Break in compressors
7. Service and calibrate instruments
8. Dry out fired heaters
9. Reactor circuit dry out
10. Catalyst loading
11. Purge and gas blanketing

It is important that these operations be carried out as thoroughly and as well as possible to help achieve a smooth and trouble-free startup and later steady normal operation. A discussion detailing the major items to monitor in each of these operations follows.

The above outline may be expanded somewhat as follows:

### **1. Commissioning of Utilities**

The various utility lines should be tested and placed into service as soon as the construction schedule allows. Pressure tests should be carried out on all steam condensate, air, fuel gas, fuel oil, flare, and nitrogen lines as is done on all process lines.

The steam lines should be warmed up gradually to prevent damage by water hammer. At the same time, all steam traps and condensate lines should be placed into service. All turbine inlet and outlet flanges should be blinded off at this time. Scale and construction debris can be conveniently removed from the steam lines by blowing them down as long as necessary with steam. To gauge the effectiveness of the steam blowing (and the amount of scale left in the lines), target plates should be installed at the blowdown points. The lines should be repeatedly blown down until virtually unmarked target plates are obtained. Condensate lines should be continually checked and traps removed and cleaned if plugged.

The other utility lines can be cleaned by blowing with steam or air, or by water flushing if possible.

### **2. Final Inspection of Vessels**

All vessels should be inspected before final closing and any loose scale, dirt, etc., should be removed. Any line coming directly off of the bottom of a dirty vessel should be removed.

It is very important that the internals of the hydrotreating reactor be inspected very carefully. The hydrotreating reactor internals should be checked for holes and/or

damage and repaired as required. The catalyst support basket and unloading sleeve should be checked to ensure correct fit in the nozzles.

The product separator should be checked carefully to be sure the cement lining is installed well and that the mesh blanket is securely fastened to the support ring. There should be no gaps in the mesh blanket.

### **3. Pressure Test Equipment**

It is normally the contractor's responsibility to hydrostatically pressure test the unit during construction. The following suggestions are made to help the refiner during this stage of startup activity.

Before any vessel is filled with water, the foundation design must be checked to see if it is rated for this load.

Screens should be placed in the lines before the unit is pressure tested so that the test water can be pumped through the lines for the purpose of washing them.

Screens should be placed in a flange between the suction valve and the pump so that the screen may be removed without depressuring any vessels. The flow through the screen should preferably be downward or horizontal. Precautions should be taken to place the screen in a location where the dirt particles will not drop into an inaccessible place in the line when the flow through the pump stops. If this should happen, it would not be possible to remove the dirt upon removal of the screen.

An air pressure test can be placed on the sections of the unit prior to a water test so that any open lines or flanges may be discovered and taken care of before liquid is admitted. It should be remembered that in pressure testing vessels, the test gauge should be placed at the bottom of the vessel so that the liquid head will be taken into account. Before draining any liquid from a vessel, a vent must be opened on top of the vessel to prevent a vacuum from pulling in the vessel sides.

In pressure testing equipment, particularly in cold weather, care should be taken that the testing of the vessels is not carried out at temperature levels so low that the metal becomes brittle. As metal temperatures decrease, the tendency for brittleness increases. Temperatures above 17°C (60°F) are considered satisfactory for testing to eliminate the possibility of cold fracturing of equipment. Such temperatures can be attained by warming the testing medium.

If the unit contains any austenitic stainless steel, the chloride content of the test water must be less than 50 wt-ppm. If this is not possible, the test water should have a maximum of 0.5 wt-% sodium nitrate added to it.

It will not be practical to test all of the equipment together. Thus, the unit will be divided into sections as governed by the location of the various items of equipment and the test pressures to which each item will be subjected. Suitable blanks must be made up for insertion on nozzles and between flanges to isolate the various sections of equipment as required. Normally, the exchangers, receivers, etc., for the various towers will be tested together with the main vessels. Test pressures will be determined from the pressure vessel summary for the unit. During pressure testing, all safety valves must be blinded off since their normal relieving pressure will be exceeded.

It may be convenient to test the heaters and reactors in one group. A field hydrostatic test on the gas compressor after installation could result in damage to the internals, so the compressors must be isolated from the reactor system. As the heaters are normally tested at a higher pressure than the reactors, it would be simplest to blind off the heaters and test them first and then test the entire system at the reactor test pressure. Blanks can be provided with connections for introduction of water for testing and for venting of air as the system is filled with water. It may be necessary to use thermowell connections and pressure taps for additional vents in the reactor system. At the completion of the hydrostatic test, all water should be removed from the equipment. Where necessary, flanges may be broken to drain low points and the equipment air blown to remove as much water as possible before flanging up.

After hydrostatic pressure testing, a tightness test must be conducted to check all flanges and fittings, especially the ones opened during hydrotesting. This final tightness test must be witnessed by UOP representatives and is normally done just prior to startup.

#### **4. Acid Cleaning of Compressor Lines**

Mill scale, dirt, heavy greases, and other foreign materials that could enter the compressor and result in operating and maintenance problems must be removed from the make-up compressor system. The following items must be acid cleaned:

- a. All make-up gas piping including spillback lines
- b. Make-up compressor suction drums
- c. Make-up gas coolers and intercoolers

The exact procedure to be followed should be supplied by the cleaning contractor, who must accept the responsibility of proposing and carrying out an acceptable and proven procedure for the entire cleaning operation. A discussion and general outline for a typical acid-cleaning operation follows:

##### **Preparation**

- 1.a. A list of metals, alloys, and non-metallic materials in the sections to be cleaned, including block valve trims, gaskets, valve packings, nuts, exchanger tubing, as well as major equipment and piping must be made.
- b. Assurance must be obtained from the cleaning contractor that the chemicals and chemical solutions used in the operation will not be injurious to these materials.

- 2.a. A list must be made of the safe operating pressures of all components in the sections to be cleaned.
- b. Assurance must be obtained by the cleaning contractor that these pressures will not be exceeded (especially if the safety valves in these sections are going to be blinded off; in this case the cleaning contractor should provide safety valves with his equipment).
3. Spool pieces must be made and substituted for turbine meters and for any valves that must be protected from any chemical solutions. Valves which are removed should be cleaned separately and their openings sealed off.
4. Orifice plates must be removed from the lines.
5. All instrument taps in the system must be disconnected or blocked off. Drain points must be provided in the taps to drain off solution, and all instrument drain valves should be opened.
6. All externally mounted liquid level instruments, such as displacement type level transmitters and gauge glasses, should have all block valves adjacent to the vessel closed and all drain valves opened.
7. Pressure gauges and thermowells should not be in place and their connections should be blocked off.
8. All piping strainer screens must be removed.
9. All high points must be provided with vent valves. These vent valves should be opened periodically during the cleaning operations.
10. Major items of equipment such as compressors, pulsation dampeners, etc., must be blinded off.

11. Cleaning circulation circuits must be determined. For a three stage make-up compressor system, this might mean four separate circulation circuits, one for each stage and one for the incoming fresh hydrogen line.

### The Acid-Cleaning Operation

The acid-cleaning operations can be generally divided into the following steps:

1. **Flushing:** All sections should be water flushed to remove all loose dirt, debris, and other foreign material in the lines. It should be noted that process pumps must not be used to circulate any of the flushing, rinsing, or chemical solutions. All transfer and circulating pumps for handling these solutions must be furnished by the chemical cleaning contractor.
2. **Degreasing:** All sections should be flushed with a degreasing solution (generally an alkaline solution such as a soda ash solution) to remove all grease or oil that may have been applied to the lines and vessels as a rust preventative measure. The cleaning contractor should specify the type and concentration of the solution to be used.

During this and other phases of the operation, the contractor may want to heat the circulating solutions. In doing so, reboilers or exchangers must not be used as a means of heating them. All heating is to be external to the systems being cleaned and by equipment furnished by the chemical cleaning contractor.

After this step, all sections should be rinsed with water.

3. **Chemical Cleaning:** All sections must be treated with an acid solution to remove all rust and scale from the metal surface. There are several types of cleaning solutions that can be used to do this step (such as inhibited hydrochloric acid or inhibited phosphoric acid); it is the responsibility of the cleaning contractor to select one which has been proven by experience. A suitable inhibitor must also be chosen to reduce the attack on metal. The contractor should specify the concentration to be used and the percentage of

metal components (such as iron) to be allowed in solution. Afterwards, the acid circulation should be followed by a water rinse.

4. **Neutralizing:** All sections must be flushed with a neutralizing solution (perhaps a soda ash solution) to neutralize all traces of acid left in the system. The cleaning contractor should specify the type and concentration of the solution to be used. After this step, all sections should be rinsed with water.
5. **Passivating:** In order to form an anti-rust skin, a solution with a passivating agent must be circulated through each section. Afterwards, each system is allowed to dry. Note that any passivating agent used must meet with UOP's approval and must be flushed from the system prior to startup.

After completing the cleaning operation, the vessels and lines should be inspected to determine the quality of the cleaning. Treated surfaces should be clean, rust-free, and dull gray in color. In-line turbine meters, valves, strainers, and all other equipment which was removed must be installed. Afterwards, the make-up system must be nitrogen purged and left under nitrogen pressure until the startup.

## 5. Wash Out Equipment and Break In Pumps

After pressure test has been completed on any vessel with its connected piping, receivers, exchangers, etc., required blanks are pulled and water is circulated for the purpose of removing any dirt, scale, etc. Much of the dirt is picked up in the pump screens where it is taken from the system by removing and cleaning the screen.

All possible lines and pumps should be used during the washing procedure for complete cleanout of the system. Of course, no water circulation should be carried out in the gas sections of the unit.

### a. Vessels and Lines Flushing

All towers and drums should be manually cleaned before flushing. The fire water system should be flushed first and can be used to supply water for flushing the rest of the plant.

Before flushing, open overhead vents on vessels (to avoid vacuum), disconnect pump suction and discharges, cover pump nozzles, and "drop out" or "roll" control valves and orifice plates. Open compressor headers and blank off compressors.

Fill vessels with water and flush lines away from vessels or drums, especially if equipped with internals that could be fouled. All lines not flushed by vessel drainage must be flushed independently.

Lines connected to exchangers should not be flushed into exchangers but the joint should be disconnected and the exchanger flange covered with a piece of sheet metal.

After sufficient flushing, the line can be reconnected and water flushed through the exchanger to the next section of the line.

Reconnect pump suction lines after initial flushing and insert 1 mm (20 mesh) screen linings in pump strainer and continue flushing, changing to spare pump and cleaning strainers when plugged. This operation should continue until no debris is collected on the strainers.

Any equipment that has had water flushed into it should be opened and cleaned manually. Block valves or other valves not "rolled" or "dropped out" should be checked for closure or rolled out for cleaning as required.

All equipment blinds not necessary during startup should be removed during or after the flushing operation.

A mechanical flow diagram should be used as a cleaning "checkoff" list.

**b. Inspection and Running In of Pumps**

Prior to unit startup, all centrifugal pumps should be thoroughly checked and run in properly (after pressure testing and water flushing) as indicated in the following outline:

**CAUTION:** Many high head pumps are not designed to pump water. To do so can result in damage to the pump internals. Check the vendor's specifications before attempting to run in pumps with water.

1. Check to see that all necessary water piping has been made to stuffing boxes, bearing jackets, pedestals and quench glands. Make sure that all necessary lube oil piping is installed, and that this piping is not mistakenly connected to the water system.
2. Check arrangements to vent the pump for priming if the pump is not self-venting. See that special connections such as bleeds and drains are properly installed.
3. Check strainers in pump suction lines. Strainers must be installed before aligning pumps. A 4 mm (three to five mesh) strainer is provided for each pump suction line during startup. To avoid pump damage during flushing with water, the strainers should temporarily be lined with 1 mm (20 mesh) screen.

Remove this screen after water flushing is completed. All strainers should be flagged, and a list similar to the blind list should be kept, so as to prevent a "lost" screen from plugging and upsetting unit operation later on.

4. Check that power or steam is available for running in the pump. Check that pressure gauges and any special instrumentation are in working order.

5. Water circulation on motor driven hydrocarbon pumps can result in motor overloading if the full pumping capacity is used. In this type of equipment, the capacity must be reduced by throttling the discharge during such periods. An ammeter can be used to determine the required throttling.
6. Before lubricating oil-lubricated bearings, check bearing chamber in pumps to see that no slushing compounds or shipping grease is left in the chamber.
7. Mechanical-type pumps should be flushed with water prior to pump operation so no dirt gets into the seal and scores the seal faces.
8. It is extremely important that the proper type and viscosity oil and proper grade of grease is used to lubricate the equipment. Refer to manufacturer's instructions and refinery lubricating schedule for this information.
9. See that the driver rotates the pump in the direction indicated by the arrow on the pump casing. Rotate the pump by hand to see that it is clear before starting.
10. Couple up and align the pumps, then check for cooling water availability and start flow of cooling water to the pumps requiring external cooling, before they are run in.
11. Open pump suction valve and close discharge valve (crack discharge valve for high capacity, high head pumps). Make sure the pump is full of liquid.
12. Start the pump. As the pump is motor driven, the pump will come up to speed. Immediately check discharge pressure gauge. If no pressure is shown, stop the pump and find the cause. If the discharge pressure is satisfactory, slowly open the discharge valve and give the desired flow rate. Check the amperage of the motor. Do not run the pump with the discharge block valve closed except for a very short time. Note any unusual vibration or operation condition.
13. Check bearings of pumps and drivers for signs of heating. Recheck all oil levels.

14. Run the pump for approximately one hour, then shut off to make any adjustment necessary and check parts for tightness. Since it is not possible to run the pump at operating temperature, a final check of alignment must be made during normal operation by switching to the spare pump.
15. Start the pump and run it for at least four hours.
16. Shut the pump down and pull the strainer. Clean the strainer and replace it in the suction line. Remove the temporary fine mesh liner from the strainer after water flushing is complete.

On a new unit, the screens are sometimes left in service for the first run on all locations where spare pumps have been provided.

When water is used for pressure testing and washing, it is sometimes better to have packing in the pumps for a seal to prevent dirt from ruining the mechanical seal.

After the lines and equipment are judged to be clean and all the pumps have been run in, the water should be drained from the various systems. Lines containing low spots should be broken at the low spot if no drain is provided. Underground lines, without drains, should be blown free of water. Before draining any vessel, a vent must be opened on that vessel so that a vacuum will not be created on draining. If the towers are to be left standing for a long period of time before steam drying or before operation, an inert gas, such as nitrogen or sweet fuel gas, must be introduced to the vessels to prevent rusting of the internals from oxygen in the air.

Of course, no water circulation should be carried out through the gas compressors. It is important that the catalyst and the compressors are not exposed to excessive moisture.

## 6. Break in Recycle Gas Compressor

In hydrotreating process unit service, most reciprocating compressors are non-lubricated type machines. The compressors will be started and operated according to the manufacturer's instructions.

**NOTE:** Before starting any reciprocating compressor, the machine should be barred or jacked over by hand to make certain it is free.

### a. Prestartup Checks

There are several points that must be checked before the compressor is ready to run.

1. The lube oil system must be cleaned and temporary 10 Angstrom filters with 20 mesh wire screen backings must be installed at the lube oil supply to each bearing. The lube oil is then circulated with the 20 Angstrom filters being frequently replaced. When the filters stay clean, they can be removed and the lube oil system is ready for service.
2. The compressor suction line and the suction snubbers should be acidized. This will remove all scale and fine dirt from the suction line that could be swept into the compressor and damage the valves.
3. All trips and alarms, high discharge temperature, low lube oil pressure, etc., must be checked and be operational. In addition, the auxiliary lube oil pump auto start must be functional.
4. The cooling water to the lube oil cooler and cylinder cooling jacket must be commissioned.
5. The oiler for the packing must be filled, and usually has to be manually cranked to supply oil pressure before the machine can be started.

6. A cold alignment check must be made. After the machine has been run, a hot check must be made. For reciprocating compressors, the method for placing the machine on line should be similar to the following:

**b. Startup Procedure for the First Compressor**

1. Purge the compressor with nitrogen, if hydrogen is to be used, through the suction purge valve to the flare or the atmospheric vent line. As hydrogen may not be available, nitrogen or air probably can be used. Be sure not to over-load the horsepower requirement of the motor.
2. Roll the machine over to ensure complete purging.
3. After nitrogen purging of the machine, introduce hydrogen to the compressor via the hydrogen pressuring line or by cracking open the suction block valve.
4. After partially pressuring the compressor with hydrogen or other gas to be used, roll the machine over and vent the hydrogen to the flare or through the atmospheric vent to displace nitrogen in the machine.
5. Gradually open the compressor suction valve to pressure up the machine to line pressure.
6. Start steam to the steam tracing. Drain the suction line and snubbers of any liquid.
7. Make sure that there are no restrictions to the gas flow from the compressor. Open any upstream flow control valves or spillback control valve prior to starting the machine.
8. If the machine is fully equipped with suction unloader valves, start up the machine as follows:

- (a) After the machine is pressured with hydrogen, close the small bypass vent line, unload all of the suction valves, and open the compressor discharge line.
- (b) Check the compressor's lubricating oil level in the crank case or reservoir.
- (c) Start the compressor and check the oil pressure.
- (d) Let the compressor idle for a few minutes while closely watching the suction temperature. Then close the suction valve loaders to put the machine on line. Follow the manufacturer's loading sequence if he has specified one.

**c. Startup Procedure for the Second and Consecutive Compressors**

1. Purge the compressor with nitrogen through the suction purge valve to the flare or the atmospheric vent line.
2. Roll the machine over to ensure complete purging.
3. After nitrogen purging of the machine, introduce hydrogen to the compressor via the hydrogen pressuring line or by cracking open the suction block valve.
4. After partially pressuring the compressor with hydrogen, roll the machine over and vent the hydrogen to the flare or through the atmospheric vent to displace the nitrogen in the machine.
5. Gradually open the compressor suction valve to pressure up the machine to line pressure.
6. Start steam to the steam tracing or the in-line jacket heater. Drain the suction line and snubbers of any liquid.
7. If the machine is fully equipped with suction unloader valves, start the second compressor as follows:

- (a) After the machine is pressured with hydrogen, close the small bypass-vent line, unload all of the compressor suction valves, and unblock the compressor discharge line. Since the compressor discharge valves will act as check valves, the gas from the operating machine will not flow back to the suction through the machine which is being started.
  - (b) Check the compressor's lubricating oil level in the crankcase or reservoir.
  - (c) Start the machine and check the oil pressure.
  - (d) Let the compressor idle for a few moments while closely watching the suction temperature, then close the suction valve loaders to put the machine online. Follow the manufacturer's loading sequence if he has specified one.
8. When placing the second or additional compressors in operation in booster service, the instrumentation must be in operation in booster service, the instrumentation must be in operation so that excess flow can be spilled back to the suction through normal channels.
9. Load the suction valve loaders as necessary to put the machine in operation fully.

**d. Maintenance Suggestions for Reciprocating Non-Lubricated Compressors**

During operation in naphtha hydrotreating service, a fine, gray, powder-like deposit may collect on the internals of the machines. This material is soluble in hot water. It is non-corrosive when dry, but when exposed to the air, it absorbs moisture readily and then becomes corrosive not only to iron and carbon steel, but also to all stainless chrome steels, especially if they have been hardened. For protection of the valves, heads, and cylinders, steps must be taken to avoid contact with air whenever possible. Several precautions will assist in this matter.

The valves should be freed of salts as soon as they are removed from the machine. This is easily done by washing in a bucket of hot water which will dissolve off the corrosive powder. The valves can be tested for leakage with water during this procedure. Prolonged soaking in the water should not be done, since the acidic compounds which will build up in the water can also damage the parts. When the valves are removed from the hot water, they will dry very quickly and are then ready for reinstallation. If the valves are to be stored for some time, it is advisable to apply a coating of light oil to the valve faces to prevent possible rusting. This oil should be removed before the valve is again installed in a machine.

In order to inspect the piston and rings, it is necessary to remove the outboard head of the cylinder, remove the rod from the crosshead, and pull the piston out far enough to view the rings. The dust should be wiped from the internal surfaces with a lint-free cloth when possible.

If the piston is entirely removed, the exposed cylinder bore and valve seating surfaces should be covered with a light coat of oil to avoid contact with air and thus prevent corrosion of the honed and polished surface of the bore. All of this oil should be removed before the piston is again installed. The bore can be plugged with a pump cup or other similar plug to assist in protection from the atmosphere. A steam hose can be used to remove the powder and scale from the cylinder gas passages, but before doing this, the valve ports must be blocked to avoid getting steam or water on the highly finished cylinder bore surface. It must be emphasized that extreme care be taken if such cleaning is attempted.

When the machines are assembled before the rest of the plant is ready for operation, they should be blanketed with gas to avoid contact with air. Close the block valves and fill the compressors to about 0.3 kg/cm<sup>2</sup>g (5 psig) with nitrogen from a cylinder after purging out all of the air in the system.

### **e. Lubricating Oil — Seasonal Changes**

Naphtha hydrotreating reciprocating compressors are normally installed in outdoor locations. Therefore, the proper weight and quality of lubricating oil in the crankcase must be used during the various seasons of the year and oil should be changed with the seasons, particularly in cold climates. Use the manufacturer's recommended type of oil for the anticipated temperature.

## **7. Service and Calibrate Instruments**

Normally, instrument lead lines will be tested hydrostatically up to block valves when the balance of the unit is tested. Hydrostatic test pressure will not be made on instruments which normally handle gas and no pressure-measuring element should be subjected to test pressures above its range. Also, never pull a vacuum on a pressure instrument or gauge unless it is specifically designed for it.

All instrument air piping should be tested at 7 kg/cm<sup>2</sup>g (100 psig) with compressed air. Soap should be used on all joints to check for leakage. Care should be taken to ensure that this high air pressure is not put on any instruments or control valve diaphragms. Likewise, when pressure testing the unit, care must be taken that the fuel gas pressure balance valves are blinded off to keep high pressure off the diaphragm. Before starting up, all instruments should be serviced and calibrated. This includes carefully measuring all orifice plate bores with a micrometer.

## **8. Dry Out Fired Heaters**

Before a heater is put into service for the first time, it will be necessary to slowly expel the excess moisture from the insulating concrete (setting) by gradually raising its temperature before any appreciable load is put on the heater. To be assured of a long heater life with minimum maintenance, this work must be done with extreme care. If the heaters are UOP heaters, UOP Heater Specification 2-18 or 2-19 (whichever applies) must be carefully adhered to for the drying operation. If they are non-UOP heaters, the manufacturer's drying procedures should be followed; however, the general procedure utilized is usually similar to the following:

**a. General Procedure**

During the initial heater refractory drying out period, it is preferable that no material be flowing through the tubes.

1. Make a temporary installation of thermocouples through the pipe sleeves in the hip section of the heater. The tips of these thermocouples should extend 150 mm (6 inches) beyond the inside of the insulating concrete, but should not contact the tubes.
2. It is preferable to use gaseous fuel (refinery gas or LPG) for drying out the setting. If no gas is available, liquid fuel may be used, but it should be free of sediment and heated as required to give the proper viscosity (about 200 SSU) for good atomization and clean combustion.
3. Light one or more burners, as required, in each section of the heater and fire slowly, so that the temperature, as indicated by the hip thermocouples, is increased at a rate of about 14°C (25°F) per hour until it reaches 482°C (900°F). Hold this temperature for 10 hours, or 2 hours per inch of refractory thickness, whichever applies.
4. While increasing the temperature, the burner operation should be rotated frequently in order to distribute the heat as evenly as possible over the entire length of the setting.
5. After the 10-hour holding period, all burners should be shut off and the heater setting allowed to cool slowly by keeping the air inlet doors and stack damper(s) fully closed.
6. After drying has been accomplished, the temporary hip thermocouples should be removed and the plugs replaced in the pipe sleeves. If the setting has been dried as outlined above, temperature may be subsequently raised or lowered at any desired rate within the design limits of the heater.

**b. For Gas-Fired Heaters**

1. When unit is shut down, always blind off the fuel gas supply line because gas may leak through the block valves at the heaters and fill a furnace.
2. Before starting to light any pilot burner, see that all individual burner block valves are closed and steam out firebox to remove any gas accumulation. Make sure the damper is opened. Steam out the box until a steady plume of steam can be seen rising out of the stack. Stop steaming and pinch in the damper.
3. When all pilot burners are lit, light each burner individually by opening the gas valve to each burner after the torch is inserted in front of the burner. After a few burners are lit, it will be necessary to open the damper to provide enough draft to light the remainder of the burners.
4. Burners should be fired to produce a blue flame with a yellow tip, obtained by regulating the primary and secondary air supply. The heaters should be checked frequently for dirty burners which might give either too long, too short, or a misdirected flame. There must be some excess of air to the burners so that an increase in fuel gas flow will have sufficient air to produce complete combustion.
5. If for any reason the fires in a heater go out:
  - (a) Shut off gas supply immediately by closing the block valves at the fuel gas control valves. Bypass and pilot lines which might be open around the control valves must also be closed.
  - (b) Put snuffing steam in the firebox.
  - (c) Close all individual burner valves.
6. As in all heaters, care should be taken that no flame impingement on the tubes is permitted.

**c. For Oil-Fired Heaters**

1. When the unit is shut down and before entering heaters, always double block the oil supply line on both the supply and return headers and pull the oil guns from the burners as oil may leak through the block valves at the heaters and fill a furnace.
2. Before starting to light any pilot burners, see that all individual oil guns are removed from the burners, and steam out the firebox and header to remove any gas accumulation. Make sure that the dampers are opened slightly.
3. Oil burners without gas pilots should be lighted from a regulation torch. When there is a gas pilot, light it first and then light the oil from the pilot. Have fuel oil circulating through the fuel oil return at normal operating temperature.
4. Burners should be fired to produce a yellow flame with a good pattern obtained by regulating the primary and secondary air supply. The furnaces should be checked frequently for dirty burners which might give either too long, too short, or a misdirected flame. There should be some excess air to the burners so that an increase in fuel flow will have sufficient air to produce complete combustion.
5. If for any reason the fires in the furnace go out, then:
  - (a) Shut off the fuel supply immediately. Do this by closing the main block valve in the fuel supply to the furnace. This will take care of any bypass lines which might be open around the control valves. Be sure the check valve on the fuel oil return does not leak allowing fuel to back into the firebox.
  - (b) Put snuffing steam in the firebox.
  - (c) Block in the pilot gas line. close individual burner valves.

6. As in all heaters, care should be taken that no flame impingement on the tubes is permitted.

**d. Safe Procedure for Lighting Oil Burners:**

1. Push the oil gun forward, and then turn on steam by fully opening the steam block valve and the steam control valve. Close off when the steam is dry.
2. Make sure the oil block valve is closed, then open the steam bypass valve to clean and warm the burner.
3. When condensate has been removed and the steam is dry (dry steam is invisible), close the bypass steam valve.
4. Adjust atomizing steam valve for a small flow of steam.
5. Open oil block valve gradually until the oil starts burning. The oil will ignite from the pilot gas flame or an oil torch. Take care to see that unburned oil is not put into the firebox. Accumulated oil will become hazardous as the firebox heats up.
6. Adjust the atomizing steam valve and oil valve to obtain correct flame pattern. Never let the flame touch the tubes.

**9. Reactor Circuit Dry Out**

It is not necessary for the reactor circuit to be bone dry, but any free water should be removed. Drain all low points in the system and air blow the lines as dry as possible. Individual charge heater passes should be blown clear separately to ensure that no liquid pockets are present. Small amounts of moisture are not harmful to the catalyst, but care must be taken so it does not become wet.

## 10. Catalyst Loading

For the catalyst loading to go smoothly, well-thought out planning and thorough preparation must be done prior to the actual loading. It must be determined how the catalyst will be loaded and what materials, equipment, and personnel will be required to do the loading. (See Catalyst Loading discussion in Section XIII.)

## 11. Purging and Gas Blanketing

It must be remembered that oil or flammable gas should never be charged into process lines or vessels indiscriminately. The unit must be purged before admitting hydrocarbons. There are many ways to purge the unit and ambient conditions may dictate the procedure to be followed: nitrogen or inert gas purging, displacement of air by liquid filling followed by gas blanketing, or steaming followed by gas blanketing.

For the remainder of the unit other than the reactor section, steam purging followed by fuel gas blanketing can be used to air free the unit. The following steps will briefly outline this method.

Potential problems or hazards could develop during the steam purge are as follows:

- a. Collapse due to vacuum: some of the vessels are not designed for vacuum. This equipment must not be allowed to stand blocked in with steam since the condensation of the steam will develop a vacuum. Thus, the vessel must be vented during steaming and immediately followed up with fuel gas purge at the conclusion of the steamout.
- b. Flange and gasket leaks: thermal expansion and stress during warm-up of equipment along with dirty flange faces can cause small leaks at flanges and gasket joints. These must be corrected at this time.
- c. Water hammering: care must be taken to prevent "water hammering" when steam purging the unit. Severe equipment damage can result from water hammering.

Block in the cooling water to all coolers and condensers.

Shut down fans on fin-fan coolers and condensers. Open high point vents and low point drains on the vessels to be steam purged.

Start introducing steam into the bottom of the columns, towers, and at low points of the various vessels. It may be necessary to make up additional steam connections to properly purge some piping which may be "dead-ended."

Thoroughly purge all equipment and associated piping of air. Be sure to open sufficient drains to drain condensate which will accumulate in low spots and receivers.

When purging is completed, close all vents and drains. Start introducing fuel gas into all vessels and cut back the steam flow until it is stopped completely when the systems are pressured. Regulate the fuel gas flow and the reduction of steam so that a vacuum due to condensing steam is not created in any vessel or that the refinery fuel gas system pressure is not appreciably reduced.

## **C. INITIAL STARTUP**

### **1. Discussion**

This procedure is designed to prepare UOP Hydrobon® catalyst for service in the fastest and safest manner without sacrificing catalyst activity or cycle length. If the procedure is not followed, catalyst activity or cycle length may be diminished, or equipment may be damaged. The procedure has been prepared for a startup with fresh or freshly regenerated catalyst. It is not intended to apply to individual units and refinery situations. THE PURPOSE OF THIS PROCEDURE IS TO PROVIDE GUIDELINES FOR THE REFINER WHEN HE IS PREPARING SPECIFIC PROCEDURES FOR AN INDIVIDUAL UNIT.

Fresh or freshly regenerated Hydrobon® Hydrotreating catalyst is a complex of metal and nonmetal oxides. During normal operation, the catalyst exists as a complex of nonmetal oxides and metal sulfides. Conversion of the metals from oxides to sulfides during startup must be done in a careful, prescribed manner in order to achieve optimum catalyst activity. An improper startup can result in depressed catalyst activity, reduced catalyst stability and possible temperature runaways.

The startup naphtha used to sulfide the Hydrobon® catalyst should be straight run material with a maximum end point of 205°C (400°F) and a bromine number of 1 or less. This minimizes the possibility of polymerization taking place in the reactor at lower temperatures, and avoids excessive heat of reaction due to olefin hydrogenation during sulfiding. In the event that the startup naphtha is quite low in sulfur, organic sulfur may be added to the feed to the unit in order to reduce the time required for sulfiding. Typically the sulfiding procedure should take 8 - 12 hours. If the time is too short it will be difficult to properly monitor the H<sub>2</sub>S in the recycle gas and insure that all the metal sites were properly sulfided. Too long a sulfiding period can start to affect the catalyst and may have some impact on the metal oxide state. The objective is to conduct the sulfiding in a controlled, orderly fashion.

Sulfur compounds added to the charge for accelerated sulfiding may be any light, liquid, organic sulfur compound (e.g., dimethyl sulfide, propyl- or butylmercaptan) which will easily decompose in the system. H<sub>2</sub>S may be used in place of a liquid sulfur compound, but the source must be examined for detrimental contaminants such as olefinic gases, sulfur oxides, carbon oxides, and ammonia, which may damage the catalyst. The total detrimental contaminants in the H<sub>2</sub>S-rich gas should be limited to a maximum of 0.1 mol-%.

Disulfides, such as carbon disulfide, are not recommended for sulfiding, since there is a safety and handling problem. Also carbon disulfide (CS<sub>2</sub>) may not hydrogenate completely at sulfiding temperatures, resulting in excessive coking of the catalyst. There is also evidence that a temperature runaway is more likely than when using other sulfides.

The following table is a list of common sulfiding agents and their associated properties.

|                                   | SA-200<br>(UOP) | DMS   | DMDS | TNPS |
|-----------------------------------|-----------------|-------|------|------|
| Sulfur, wt%                       | 40              | 51    | 68   | 37   |
| Specific Gravity @ 60°F           | 1.045           | 0.854 | 1.06 | 1.03 |
| Thermal Decomposition<br>Temp, °F | 320             | 482   | 392  | 320  |

Since feed must be started to the unit while the system is relatively cold, the reactor charge heater flow will be two phase during the period temperatures are being increased. For units with a multiple pass charge heater, a coil could be damaged if it were blocked by a liquid pocket and the heater firing continued. To ensure that the feed to the heater becomes single phase (all vapor) at relatively low temperatures, the reactor inlet pressure is initially limited to 14 kg/cm<sup>2</sup>g (200 psig).

When a Platforming Unit is the only potential source of hydrogen for startup and the Naphtha Hydrotreating Unit will be supplying charge for the Platforming Unit, a sweet, stripped, low-sulfur naphtha should be stored prior to the unit shutdown for startup purposes. It is strongly recommended that a hydrotreated naphtha be made available, but when this is not possible, straight run naphtha may be used, subject to the following limitations:

|                       |                       |
|-----------------------|-----------------------|
| Total sulfur          | 100 wt ppm maximum    |
| Total nitrogen        | 1 wt ppm maximum      |
| Arsenic               | 5 wt ppb maximum      |
| Lead                  | 25 wt ppb maximum     |
| Halides               | 1 wt ppm maximum      |
| Distillation endpoint | 205°C (400°F) maximum |
| Bromine No.           | 1 maximum             |
| Aromatics             | 15 vol-% maximum      |

The above stock may also be used for sulfiding the Hydrobon<sup>®</sup> catalyst if a sufficient amount is available, particularly if it is planned to sulfide using additional organic sulfur or H<sub>2</sub>S.

The charge stock to the Platforming Unit should be as free of water as possible during the startup. The Naphtha Hydrotreating Unit fractionation or stripping section should be in service with reflux if possible, preferably at about the design rate prior to routing naphtha to the Platforming Unit.

### **PRECAUTION: HYDROGEN SULFIDE (H<sub>2</sub>S) IS A POISONOUS GAS**

During sulfiding of the hydrotreating catalyst, hydrogen sulfide will be released to the gas and liquid streams of the unit as sulfur-bearing compounds are decomposed. Hydrogen sulfide may also be utilized as additional sulfur in the sulfiding step. The safety procedures for handling H<sub>2</sub>S should be reviewed with the appropriate operating personnel before starting the unit. Make certain that each person in the operating area is familiar with the dangers of H<sub>2</sub>S, approved methods for handling it, and first aid in case of H<sub>2</sub>S poisoning.

### **PRECAUTION**

Organic sulfur-bearing compounds which may be used for adding sulfur to the Naphtha Hydrotreater charge are dangerous materials. Make certain that each person in the operating area is familiar with the dangers of the materials being used, approved methods for handling them and appropriate first aid procedures in case of contact with the materials.

## **2. Detailed Procedure – Fresh or Freshly Regenerated Hydrobon<sup>®</sup> Catalyst Naphtha Hydrotreating Unit**

**NOTE:** This procedure is general in nature and is not intended to cover every possible mechanical and process combination. Before proceeding with a startup, each unit should be examined and a detailed procedure should be prepared to deal with that specific unit. Particular care should be taken not to exceed equipment limitations.

1. Remove oxygen from the fractionation or stripping section of the unit following the suggested procedure described in the commissioning section of the manual or normal refinery practices.
2. Establish acceptable startup naphtha charge to the fractionation or stripping section, and establish heat input (if possible) to allow a sufficient reflux/feed volume ratio (0.25 on a stripper) to remove essentially all water from the bottoms product. Slowly heat-up of the column bottoms at a rate of 20°C (35°F) per hour. When the temperature approaches 100°C (212°F) reduce the heat-up rate to 10°C (18°F) per hour to allow any water in naphtha to expand slowly. After most of the water has been sent overhead, then the temperature can be increased to the required.
3. If an associated Platforming Unit is the only source of makeup hydrogen to the naphtha hydrotreater, the Platforming Unit must be placed on stream. If hydrogen-rich makeup gas is to be supplied from an independent source, ensure that a sufficient supply is available. Hydrogen will be used to pressure the reactor circuit, after the last vacuum, up to the various operating pressures detailed below. During the sulfiding procedure some hydrogen will be dissolved in the naphtha stream and thus some hydrogen will be lost out of the Stripper column.

Hydrogen-rich makeup gas supplied from an independent source should be at least 75 mol-% hydrogen, and should be sufficient to maintain the hydrogen to hydrocarbon at a minimum of 35 nm<sup>3</sup>/m<sup>3</sup> (200 SCFB) with the reactor products separator at 28 kg/cm<sup>2</sup>g (400 psig) (or at design if the design pressure is lower). It should contain less than 0.5 mol-% sulfur and carbon oxides, less than 0.5 mol-% unsaturated hydrocarbons, and less than 50 mol ppm halides.

4. Evacuate the reactor section to 500-600 mm of mercury (20-25 in. of Hg) vacuum, and hold for at least 30 minutes to check the tightness of the unit. Vacuum loss should be less than 25-50 mm of Hg/hour (1-2 inches of Hg/hour). Break the vacuum with nitrogen to 0.3 kg/cm<sup>2</sup>g (5 psig). Evacuate

and purge with nitrogen a second time. Pull a third vacuum and break with hydrogen.

**NOTE:** Any time the unit has been opened (i.e., for maintenance or catalyst regeneration), a pressure test should be conducted to ensure the tightness of the unit.

5. Pressure the reactor section to 14 kg/cm<sup>2</sup>g (200 psig) with hydrogen, and establish once-through or recycle gas flow at the maximum possible rate.
6. If reactor temperatures are between ambient and 150°C (300°F), charge startup naphtha to the reactor section at approximately one-half of the design charge rate. Continue the bypass flow to the stripper. If any reactor temperature is above 150°C (300°F), cool the reactor with gas flow so that all catalyst temperatures are below 150°C (300°F) before bringing startup naphtha into the unit if the catalyst is fresh or freshly regenerated.
7. When a liquid level is established in the reactor products separator, discontinue routing startup naphtha directly to the stripper section. Make the transition smoothly so that downstream units are not upset. Maintain the naphtha hydrotreater feed rate at approximately one-half of the design charge rate. For a hydrotreater startup with an independent source of makeup hydrogen, it is preferable to circulate the naphtha used for sulfiding from the stripping section, through cooling and back to the feed section, making up naphtha as necessary. This minimizes the production of off-specification material during the startup.
8. Purge the reactor charge heater firebox and light fires following normal refinery practice. Increase the reactor inlet temperature to 230°C (450°F) at approximately 30°C/hr (50°F/hr). Maintain a minimum hydrogen to hydrocarbon ratio of 35 nm<sup>3</sup>/m<sup>3</sup> (200 SCFB) and maintain the reactor products separator pressure at 14 kg/cm<sup>2</sup>g (200 psig).

**NOTE:** Throughout this phase of the sulfiding, monitor the separator boot for water accumulation. When water is detected, drain it from the separator.

**NOTE:** For those units with a multiple-pass reactor charge heater, the individual charge heater pass outlet temperatures should be checked at least every 5 minutes as the heater outlet temperature is increased. If one or more pass outlet temperatures lag behind, this could indicate a liquid seal or pocket obstructing flow. This may cause localized overheating of the tube(s). If this occurs, shock the system momentarily by changing the charge flow abruptly. If the seal persists, lower temperatures and shock the system again by abruptly changing the charge rate. If the seal persists, stop heater firing, stop the naphtha charge and make certain the pass is cleared before restarting charge to the unit. Ensure that the heater is not overfired during any of these activities.

A liquid seal can be broken or prevented by adjusting the flow so that the charge heater delta P is greater than the head developed by a liquid pocket in any pass.

9. After the reactor inlet and outlet temperatures have been stabilized at 230°C (450°F), increase the reactor products separator pressure to the normal operating level or 28 kg/cm<sup>2</sup>g (400 psig), whichever is lower.
10. At 230°C (450°F), sulfiding will take place using the native sulfur in the charge. If this proves to be a time-consuming operation (assume 90% desulfurization of the native sulfur), additional sulfur in the form of an organic sulfur compound may be added to the feed, or H<sub>2</sub>S may be added to the gas to the reactor. The total amount of sulfur charged to the catalyst (native plus added) should not exceed 0.25 wt-% of the naphtha charge at this point. However, to extend the sulfiding period for better control, the total amount of sulfur injected should be controlled at 0.08 – 0.10 wt% of the naphtha charge, depending on the catalyst metal loading. Calculate the sulfur injection rate required, for the actual catalyst loaded, so that the sulfiding step takes 8-12 hours.

Hold the reactor inlet temperature at 230°C (450°F) and maintain a minimum hydrogen to hydrocarbon ratio of 35 nm<sup>3</sup>/m<sup>3</sup> (200 SCFB). Increase the feed rate to design, or the maximum available.

**NOTE:** In the event of a rapid reactor outlet or catalyst temperature rise above 250°C (480°F), stop sulfur addition (whether H<sub>2</sub>S or organic sulfur is to be added) to the unit immediately and reduce the firing in the reactor charge heater. If necessary, stop the charge to the unit to limit the temperature rise. When temperature control is regained, adjust the reactor inlet temperature to 230°C (450°F), and slowly restart sulfur addition to the unit.

11. When unspiked start-up oil is used for catalyst sulfiding and if the conditions indicate very little desulfurization is taking place at 230°C (450°F) catalyst temperatures, then the bed peak temperature can be increased slowly up to a maximum of about 250°C (480°F). It should not be necessary to exceed a 230°C (450°F) catalyst peak temperature if an organic sulfiding compound is being added.
12. During the sulfiding period, increase the stripping section reflux ratios as much as possible to remove any H<sub>2</sub>S, water, or light mercaptans which might otherwise contaminate the product. If necessary, the operating pressure of the fractionation or stripping section should be reduced to obtain sufficient material for reflux.
13. If additional sulfur is used, after the unit has stabilized at 0.08 – 0.10 wt% (maximum 0.25 wt-%) sulfur in the reactor feed, smoothly increase the amount of added sulfur until the total sulfur being charged to the catalyst is 0.15 – 0.20 wt% (maximum 0.50 wt-%) of the naphtha charge. Maintain 230°C (450°F) reactor inlet temperature and continue sulfiding. Drain water from the reactor products separator and the fractionation or stripping section water boots as it accumulates.
14. Continue sulfiding at these conditions for a period of 1-2 hours.
15. Increase the reactor inlet temperature to 290°C (550°F) at a rate of 17°C (30°F) per hour.

**NOTE:** Do not exceed 17°C (30°F) temperature rise across any catalyst bed.

16. The catalyst can be considered sulfided when the total amount of sulfur injected has reached the maximum shown in the following Table.

| <b>Hydrobon<sup>®</sup> Catalyst</b> | <b>Sulfur Level, Based on<br/>Loaded Catalyst Weight</b> |
|--------------------------------------|----------------------------------------------------------|
| S-6                                  | 6.0 wt-%                                                 |
| S-9                                  | 6.0 wt-%                                                 |
| S-12                                 | 8.5 wt-%                                                 |
| S-12H                                | 9.0 wt-%                                                 |
| S-12T                                | 8.5 wt-%                                                 |
| S-15                                 | 4.5 wt-%                                                 |
| S-16                                 | 8.5 wt-%                                                 |
| S-18                                 | 6.0 wt-%                                                 |
| S-19H                                | 9.0 wt-%                                                 |
| S-19T                                | 10.5 wt-%                                                |
| S-19M                                | 8.5 wt-%                                                 |
| S-120                                | 9.6 wt-%                                                 |
| N-204                                | 7.2 wt-%                                                 |
| N-108                                | 9.4 wt-%                                                 |
| HC-K                                 | 11.3 wt-%                                                |

17. Establish normal plant operation in the following sequence:

- Adjust naphtha charge to the desired rate.
- Increase the reactor inlet temperature to 315°C (600°F). Adjust temperature as required to produce on-specification product.
- Increase the reactor products separator pressure to normal, if this was not done in Step 9.
- Increase the hydrogen-to-hydrocarbon ratio to normal, if this was not already done.

- 
- e. For units that process charge different from the startup naphtha, normal charge can now be routed to the unit and startup naphtha stopped. The change should not be made abruptly to avoid upsets, and control of the reactor temperatures is maintained.
  - f. Establish water injection to the reactor products condenser, just after the last combined-feed exchanger bundle, at a rate equal to 3 liquid volume-% of the charge rate.

## VII. NORMAL STARTUP

### A. Discussion

This procedure is designed to prepare UOP Hydrobon® catalyst for service in the fastest and safest manner without sacrificing catalyst activity or cycle length. If the procedure is not followed, catalyst activity or cycle length may be diminished, or equipment may be damaged. The procedure has been prepared for a startup with fresh or freshly regenerated catalyst. It is not intended to apply to individual units and refinery situations. THE PURPOSE OF THIS PROCEDURE IS TO PROVIDE GUIDELINES FOR THE REFINER WHEN HE IS PREPARING SPECIFIC PROCEDURES FOR AN INDIVIDUAL UNIT.

Fresh or freshly regenerated Hydrobon® Hydrotreating catalyst is a complex of metal and nonmetal oxides. During normal operation, the catalyst exists as a complex of nonmetal oxides and metal sulfides. Conversion of the metals from oxides to sulfides during startup must be done in a careful, prescribed manner in order to achieve optimum catalyst activity. An improper startup can result in depressed catalyst activity, reduced catalyst stability and possible temperature runaways.

The startup naphtha used to sulfide the Hydrobon® catalyst should be straight run material with a maximum end point of 205°C (400°F) and a bromine number of 1 or less. This minimizes the possibility of polymerization taking place in the reactor at lower temperatures, and avoids excessive heat of reaction due to olefin hydrogenation during sulfiding. In the event that the startup naphtha is quite low in sulfur, organic sulfur may be added to the feed to the unit in order to reduce the time required for sulfiding. Typically the sulfiding procedure should take 8 - 12 hours. If the time is too short it will be difficult to properly monitor the H<sub>2</sub>S in the recycle gas and insure that all the metal sites were properly sulfided. Too long a sulfiding period can start to affect the Platforming catalyst and may have some impact on the metal oxide state. The objective is to conduct the sulfiding in a controlled, orderly fashion.

Sulfur compounds added to the charge for accelerated sulfiding may be any light, liquid, organic sulfur compound (e.g., dimethyl sulfide, propyl- or butylmercaptan) which will easily decompose in the system.  $H_2S$  may be used in place of a liquid sulfur compound, but the source must be examined for detrimental contaminants such as olefinic gases, sulfur oxides, carbon oxides, and ammonia, which may damage the catalyst. The total detrimental contaminants in the  $H_2S$ -rich gas should be limited to a maximum of 0.1 mol-%.

Disulfides, such as carbon disulfide, are not recommended for sulfiding, since there is a safety and handling problem. Also carbon disulfide ( $CS_2$ ) may not hydrogenate completely at sulfiding temperatures, resulting in excessive coking of the catalyst. There is also evidence that a temperature runaway is more likely than when using other sulfides.

The following table is a list of common sulfiding agents and their associated properties.

|                                   | SA-200<br>(UOP) | DMS   | DMDS | TNPS |
|-----------------------------------|-----------------|-------|------|------|
| Sulfur, wt%                       | 40              | 51    | 68   | 37   |
| Specific Gravity @ 60°F           | 1.045           | 0.854 | 1.06 | 1.03 |
| Thermal Decomposition<br>Temp, °F | 320             | 482   | 392  | 320  |

Since feed must be started to the unit while the system is relatively cold, the reactor charge heater flow will be two phase during the period temperatures are being increased. For units with a multiple pass charge heater, a coil could be damaged if it were blocked by a liquid pocket and the heater firing continued. To ensure that the feed to the heater becomes single phase (all vapor) at relatively low temperatures, the reactor inlet pressure is initially limited to 14 kg/cm<sup>2</sup>g (200 psig).

When a Platforming Unit is the only potential source of hydrogen for startup and the Naphtha Hydrotreating Unit will be supplying charge for the Platforming Unit, a sweet, stripped, low-sulfur naphtha should be stored prior to the unit shutdown for startup purposes. It is strongly recommended that a hydrotreated naphtha be made

available, but when this is not possible, straight run naphtha may be used, subject to the following limitations:

|                       |                       |
|-----------------------|-----------------------|
| Total sulfur          | 100 wt ppm maximum    |
| Total nitrogen        | 1 wt ppm maximum      |
| Arsenic               | 5 wt ppb maximum      |
| Lead                  | 25 wt ppb maximum     |
| Halides               | 1 wt ppm maximum      |
| Distillation endpoint | 205°C (400°F) maximum |
| Bromine No.           | 1 maximum             |
| Aromatics             | 15 vol-% maximum      |

The above stock may also be used for sulfiding the Hydrobon® catalyst if a sufficient amount is available, particularly if it is planned to sulfide using additional organic sulfur or H<sub>2</sub>S.

The charge stock to the Platforming Unit should be as free of water as possible during the startup. The Naphtha Hydrotreating Unit fractionation or stripping section should be in service with reflux if possible, preferably at about the design rate prior to routing naphtha to the Platforming Unit.

#### **PRECAUTION: HYDROGEN SULFIDE (H<sub>2</sub>S) IS A POISONOUS GAS**

During sulfiding of the hydrotreating catalyst, hydrogen sulfide will be released to the gas and liquid streams of the unit as sulfur-bearing compounds are decomposed. Hydrogen sulfide may also be utilized as additional sulfur in the sulfiding step. The safety procedures for handling H<sub>2</sub>S should be reviewed with the appropriate operating personnel before starting the unit. Make certain that each person in the operating area is familiar with the dangers of H<sub>2</sub>S, approved methods for handling it, and first aid in case of H<sub>2</sub>S poisoning.

#### **PRECAUTION**

Organic sulfur-bearing compounds which may be used for adding sulfur to the Naphtha Hydrotreating Unit charge are dangerous materials. Make certain that

each person in the operating area is familiar with the dangers of the materials being used, approved methods for handling them and appropriate first aid procedures in case of contact with the materials.

## **B. Detailed Procedure – Fresh or Freshly Regenerated Hydrobon® Catalyst Naphtha Hydrotreating Unit**

**NOTE:** This procedure is general in nature and is not intended to cover every possible mechanical and process combination. Before proceeding with a startup, each unit should be examined and a detailed procedure should be prepared to deal with that specific unit. Particular care should be taken not to exceed equipment limitations.

1. Remove oxygen from the fractionation or stripping section of the unit following the suggested procedure described in the commissioning section of the manual or normal refinery practices.
2. Establish acceptable startup naphtha charge to the fractionation or stripping section, and establish heat input (if possible) to allow a sufficient reflux/feed volume ratio (0.25 on a stripper) to remove essentially all water from the bottoms product. Slowly heat-up of the column bottoms at a rate of 20°C (35°F) per hour. When the temperature approaches 100°C (212°F) reduce the heat-up rate to 10°C (18°F) per hour to allow any water in naphtha to expand slowly. After most of the water has been sent overhead, then the temperature can be increased to the required.
3. If an associated Platforming Unit is the only source of makeup hydrogen to the Naphtha Hydrotreating Unit, the Platforming Unit must be placed on stream. If hydrogen-rich make-up gas is to be supplied from an independent source, ensure that a sufficient supply is available. Hydrogen will be used to pressure the reactor circuit, after the last vacuum, up to the various operating pressures detailed below. During the sulfiding procedure some hydrogen will be dissolved in the naphtha stream and thus some hydrogen will be lost out of the Stripper Column.

Hydrogen-rich make-up gas supplied from an independent source should be at least 75 mol-% hydrogen, and should be sufficient to maintain the hydrogen to hydrocarbon ratio at a minimum of 35 nm<sup>3</sup>/m<sup>3</sup> (200 SCFB) with the reactor products separator at 28 kg/cm<sup>2</sup>g (400 psig) (or at design if the design pressure is lower). It should contain less than 0.5 mol-% sulfur and carbon oxides, less than 0.5 mol-% unsaturated hydrocarbons, and less than 50 mol ppm halides.

4. Evacuate the reactor section to 500-600 mm of mercury (20-25 in. of Hg) vacuum, and hold for at least 30 minutes to check the tightness of the unit. Vacuum loss should be less than 25-50 mm of Hg/hour (1-2 inches of Hg/hour). Break the vacuum with nitrogen to 0.3 kg/cm<sup>2</sup>g (5 psig). Evacuate and purge with nitrogen a second time. Pull a third vacuum and break with hydrogen.

**NOTE:** Any time the unit has been opened (i.e., for maintenance or catalyst regeneration), a pressure test should be conducted to ensure the tightness of the unit.

5. Pressure the reactor section to 14 kg/cm<sup>2</sup>g (200 psig) with hydrogen, and establish once-through or recycle gas flow at the maximum possible rate.
6. If reactor temperatures are between ambient and 150°C (300°F), charge startup naphtha to the reactor section at approximately one-half of the design charge rate. Continue the bypass flow to the stripper. If any reactor temperature is above 150°C (300°F), cool the reactor with gas flow so that all catalyst temperatures are below 150°C (300°F) before bringing startup naphtha into the unit if the catalyst is fresh or freshly regenerated.
7. When a liquid level is established in the reactor products separator, discontinue routing startup naphtha directly to the stripper section. Make the transition smoothly so that downstream units are not upset. Maintain the naphtha hydrotreating feed rate at approximately one-half of the design charge rate. For a hydrotreating startup with an independent source of make-up hydrogen, it is preferable to circulate the naphtha used for sulfiding from the stripping section, through cooling and back to the feed section, making up

naphtha as necessary. This minimizes the production of off-specification material during the startup.

8. Purge the reactor charge heater firebox and light fires following normal refinery practice. Increase the reactor inlet temperature to 230°C (450°F) at approximately 30°C/hr (50°F/hr). Maintain a minimum hydrogen to hydrocarbon ratio of 35 nm<sup>3</sup>/m<sup>3</sup> (200 SCFB) and maintain the reactor products separator pressure at 14 kg/cm<sup>2</sup>g (200 psig).

**NOTE:** Throughout this phase of the sulfiding, monitor the separator boot for water accumulation. When water is detected, drain it from the separator.

**NOTE:** For those units with a multiple-pass reactor charge heater, the individual charge heater pass outlet temperatures should be checked at least every 5 minutes as the heater outlet temperature is increased. If one or more pass outlet temperatures lag behind, this could indicate a liquid seal or pocket obstructing flow. This may cause localized overheating of the tube(s). If this occurs, shock the system momentarily by changing the charge flow abruptly. If the seal persists, lower temperatures and shock the system again by abruptly changing the charge rate. If the seal persists, stop heater firing, stop the naphtha charge and make certain the pass is cleared before restarting charge to the unit. Ensure that the heater is not overfired during any of these activities.

A liquid seal can be broken or prevented by adjusting the flow so that the charge heater delta P is greater than the head developed by a liquid pocket in any pass.

9. After the reactor inlet and outlet temperatures have been stabilized at 230°C (450°F), increase the reactor products separator pressure to the normal operating level or 28 kg/cm<sup>2</sup>g (400 psig), whichever is lower.
10. At 230°C (450°F), sulfiding will take place using the native sulfur in the charge. If this proves to be a time-consuming operation (assume 90% desulfurization of the native sulfur), additional sulfur in the form of an organic sulfur

compound may be added to the feed, or  $H_2S$  may be added to the gas to the reactor. The total amount of sulfur charged to the catalyst (native plus added) should not exceed 0.25 wt-% of the naphtha charge at this point. However, to extend the sulfiding period for better control, the total amount of sulfur injected should be controlled at 0.08 – 0.10 wt% of the naphtha charge, depending on the catalyst metal loading. Calculate the sulfur injection rate required, for the actual catalyst loaded, so that the sulfiding step takes 8-12 hours.

Hold the reactor inlet temperature at 230°C (450°F) and maintain a minimum hydrogen to hydrocarbon ratio of 35  $nm^3/m^3$  (200 SCFB). Increase the feed rate to design, or the maximum available.

**NOTE:** In the event of a rapid reactor outlet or catalyst temperature rise above 250°C (480°F), stop sulfur addition (whether  $H_2S$  or organic sulfur is to be added) to the unit immediately and reduce the firing in the reactor charge heater. If necessary, stop the charge to the unit to limit the temperature rise. When temperature control is regained, adjust the reactor inlet temperature to 230°C (450°F), and slowly restart sulfur addition to the unit.

11. When unspiked start-up oil is used for catalyst sulfiding and if the conditions indicate very little desulfurization is taking place at 230°C (450°F) catalyst temperatures, then the bed peak temperature can be increased slowly up to a maximum of about 250°C (480°F). It should not be necessary to exceed a 230°C (450°F) catalyst peak temperature if an organic sulfiding compound is being added.
12. During the sulfiding period, increase the stripping section reflux ratios as much as possible to remove any  $H_2S$ , water, or light mercaptans which might otherwise contaminate the product. If necessary, the operating pressure of the fractionation or stripping section should be reduced to obtain sufficient material for reflux.
13. If additional sulfur is used, after the unit has stabilized at 0.08 – 0.10 wt% (maximum 0.25 wt-%) sulfur in the reactor feed, smoothly increase the amount of added sulfur until the total sulfur being charged to the catalyst is 0.15 – 0.20

wt% (maximum 0.50 wt-%) of the naphtha charge. Maintain 230°C (450°F) reactor inlet temperature and continue sulfiding. Drain water from the reactor products separator and the fractionation or stripping section water boots as it accumulates.

14. Continue sulfiding at these conditions for a period of 1-2 hours.
15. Increase the reactor inlet temperature to 290°C (550°F) at a rate of 17°C (30°F) per hour.

**NOTE:** Do not exceed 17°C (30°F) temperature rise across any catalyst bed.

16. The catalyst can be considered sulfided when the total amount of sulfur injected has reached the maximum shown in the following Table.

| Hydrobon <sup>®</sup> Catalyst | Sulfur Level, Based on<br>Loaded Catalyst Weight |
|--------------------------------|--------------------------------------------------|
| S-6                            | 6.0 wt-%                                         |
| S-9                            | 6.0 wt-%                                         |
| S-12                           | 8.5 wt-%                                         |
| S-12H                          | 9.0 wt-%                                         |
| S-12T                          | 8.5 wt-%                                         |
| S-15                           | 4.5 wt-%                                         |
| S-16                           | 8.5 wt-%                                         |
| S-18                           | 6.0 wt-%                                         |
| S-19H                          | 9.0 wt-%                                         |
| S-19T                          | 10.5 wt-%                                        |
| S-19M                          | 8.5 wt-%                                         |
| S-120                          | 9.6 wt-%                                         |
| N-204                          | 7.2 wt-%                                         |
| N-108                          | 9.4 wt-%                                         |
| HC-K                           | 11.3 wt-%                                        |

17. Establish normal plant operation in the following sequence:

- a. Adjust naphtha charge to the desired rate.
- b. Increase the reactor inlet temperature to 315°C (600°F). Adjust temperature as required to produce on-specification product.
- c. Increase the reactor products separator pressure to normal, if this was not done in Step 9.
- d. Increase the hydrogen-to-hydrocarbon ratio to normal, if this was not already done.
- e. For units that process charge different from the startup naphtha, normal charge can now be routed to the unit and startup naphtha stopped. The

change should not be made abruptly so that upsets are avoided, and control of the reactor temperatures is maintained.

- f. Establish water injection to the reactor products condenser, just after the last combined-feed exchanger bundle, at a rate equal to 3 liquid volume-% of the charge rate.

### C. SUBSEQUENT STARTUP

The procedure used for the initial startup should be followed except that sulfiding is not required for used catalyst so those steps should be omitted.

It is not necessary to cool the reactor beds to less than 290°C (550°F) before cutting in the feed if the catalyst is already sulfided.

The procedure to use is as follows:

1. Pressure the reactor section to 14 kg/cm<sup>2</sup>g (200 psig) with H<sub>2</sub> and establish once-through or recycle gas flow at the maximum possible rate.
2. If the reactor temperatures are between ambient and 290°C (550°F), charge startup naphtha to the reactor at about one-half of the design rate.
3. Purge the charge heater firebox with steam and light fires following normal refinery practices. Increase the reactor temperatures to 315°C (600°F) at about 40°C (75°F) per hour.

**NOTE:** The reactor inlet temperatures will decrease sharply when oil is cut into the unit. Do not overfire the charge heater in an attempt to hold the existing reactor temperature.

**NOTE:** For those units with a multiple-pass reactor charge heater, the individual charge heater pass outlet temperatures should be checked at least every 5 minutes as the heater outlet temperature is increased. If one or more

pass outlet temperatures lag behind, this could indicate a liquid seal or pocket obstructing flow. This may cause localized overheating of the tube(s). If this occurs, shock the system momentarily by changing the charge flow abruptly. If the seal persists, lower temperatures and shock the system again by abruptly changing the charge rate. If the seal persists, stop heater firing, stop the naphtha charge and make certain the pass is cleared before restarting charge to the unit. Ensure that the heater is not overfired during any of these activities.

A liquid seal can be broken or prevented by adjusting the flows so that the charge heater delta P is greater than the head developed by a liquid pocket in any pass.

4. After the reactor inlet and outlet temperatures have exceeded 260°C (500°F), increase the reactor products separator pressure to the normal operating level.
5. Establish normal plant operation by increasing the charge rate to normal, increasing the reactor inlet temperatures as required to produce on-spec product, and switching to normal feed if sweet naphtha was used for startup. Restart water injection to the products condenser, just after the last combined-feed exchanger bundle, at a rate equal to 3 liquid volume-% of the charge rate.

## VIII. NORMAL OPERATIONS

The Naphtha Hydrotreating Unit is fairly simple to monitor from a calculation and data review standpoint. The chapter describes calculations that are typically done in a Naphtha Hydrotreating Unit. This does not include the Splitter section.

### A. CALCULATIONS

Before any calculations are performed, the data should be reviewed to verify the unit was lined out during the period of time the calculations will cover. Usually, this is 24 hours. Good practice dictates that calculations be performed routinely, such as once a day, so that changes in performance can be quickly noted. Also, engineers find it very useful to have some data and calculations plotted in order to monitor trends and maintain a unit operating history.

Before the unit's performance can be properly monitored, the unit must first weight balance. Kilograms of liquid and gas in should equal kilograms out. A good balance is one where the percentage of kgs of products divided by the kgs of feeds equals 100 percent, plus or minus 2% maximum. If it is outside this range, the engineer will have to try to evaluate which indicator(s) is reading wrong and have it corrected.

On the Naphtha Hydrotreating Unit there can be one to multiple naphtha feed streams and hydrogen make-up. The products typically consist of two streams: the stripper off-gas and stripper bottoms. If there is not a flow meter on the stripper bottoms, the product flow meter(s) from downstream vessels (splitter, intermediate tanks, Platforming Unit feed, etc.) should be used. Water injection and sour water product are not considered in the weight balance. Water and oil do not mix.

The first step of the calculations is to correct the feed and product flows to their actual mass flows at standard conditions. The averages on the logsheets are only approximations of the actual value. Liquid streams have to be corrected for changes in density as measured at a standard temperature due to variations in flowing temperatures. Gases have to be corrected for variations in specific gravity,

operating pressure and operating temperatures. The corrected flows are then used to check the unit weight balance and for other calculations as is noted below.

The following calculations are typically performed on the Naphtha Hydrotreating Unit daily:

**1. Weight Balance**

$$\frac{\text{kg/hr products}}{\text{kg/hr feed}} \times 100$$

An acceptable weight balance is within 98 to 102 weight %.

**2. Liquid Hourly Space Velocity (hr<sup>-1</sup>)**

$$\text{LHSV} = \frac{\text{volume of charge per hour}}{\text{volume of catalyst}}$$

**3. Hydrogen to Hydrocarbon Ratio**

$$\frac{\text{Nm}^3/\text{hr of hydrogen recycle gas}}{\text{m}^3/\text{hr of naphtha charge}}$$

**4. Stripper Offgas**

$$\frac{\text{Nm}^3/\text{hr of stripper offgas}}{\text{m}^3/\text{hr of naphtha charge}}$$

**5. Stripper Reflux Ratio**

$$\frac{\text{m}^3/\text{hr of reflux}}{\text{m}^3/\text{hr of naphtha charge}} \times 100$$

**6. Hydrogen Consumption**

$$\frac{(\text{Nm}^3/\text{hr H}_2 \text{ makeup}) - (\text{Nm}^3/\text{hr hydrogen out of the unit})}{\text{m}^3/\text{hr of naphtha charge}}$$

**7. Cumulative Charge**

Total m<sup>3</sup> of charge to the unit. Usually, calculated from beginning of a run to a regeneration. If the unit has more than 1 feed source, the individual rates should be recorded as well.

**8. Catalyst Life**

$$\frac{\text{cumulative charge, m}^3}{\text{kg of catalyst}}$$

Catalyst life is measured from original startup to catalyst replacement.

**9. Metals Contamination**

$$\frac{\text{wt\% metals in charge} \times \text{kg charge}}{\text{kg of catalyst}} \times 10^{-2}$$

Usually, kg of charge is the total from the last time a metals analysis was performed on the feed to the latest one. The total metals contamination is then the summation of the incremental contaminations between analyses. See Section III, Part F for more information.

**10. Water Injection**

$$\frac{\text{m}^3/\text{hr of water}}{\text{m}^3/\text{hr of naphtha charge}} \times 100$$

The typical continuous water injection target is 3 liquid volume percent of the charge rate. This is for when the water is injected just after the last combined-feed exchanger bundle. If the water is injected further upstream where the

process temperature is higher, then the water rate must be increased. The goal is to maintain at least 25% of the injected water in the liquid phase.

The injection rate may require adjustments based on the separator water analysis. The separator water should be analyzed 3 times per day to insure it has the following qualities:

|          |                                       |
|----------|---------------------------------------|
| pH       | $6.0 \pm 0.5$ (avoid 6.8 – 7.3 range) |
| Iron     | 2 wt-ppm or less                      |
| Chloride | Less than 500 wt-ppm                  |

If the pH decreases below 5.5, then increase the water injection rate to bring the pH up to the desired range. Note that different water rates change the acceptable level of iron. It is the mass of iron being removed that is important to monitor. If increased water injection does not bring the pH into the proper range, then a "basic" water injection may be required. Contact UOP for further details.

## 11. Reactor pressure drop

*Reactor inlet pressure – reactor outlet pressure*

The maximum pressure drop of the reactor is typically set by the allowable pressure drop across the outlet basket. This is for guideline purposes only. For older units this is typically 60 psig (4.2 kg/cm<sup>2</sup>) and about 100 psig (7 kg/cm<sup>2</sup>) for newer designs. However, the pressure drop usually occurs at the top of the reactor bed. Thus, product quality and hydrogen flow are usually the limiting factor.

## 12. Reactor delta Temperature

*Reactor outlet temperature – reactor Inlet temperature*

For most straight run naphthas, there will be no temperature rise across the reactor, and may actually show a loss of temperature depending on heat loss. Naphthas that contain olefins, such as cracked naphthas, will exhibit a temperature rise. The magnitude will depend on the amount of olefins

present. As the olefin content increases so does the exotherm. As the outlet temperature approaches 343°C (650°F) then sulfur recombination can occur.

## **IX. ANALYTICAL**

Included in this section is the laboratory test method schedule for the Naphtha Hydrotreating Unit. This laboratory schedule is general in nature and is customized for each customer depending on the equipment included in the design. Please refer to the 934 specifications in the UOP Schedule A books for your unit.

## LABORATORY TEST METHOD SCHEDULE

### Naphtha Hydrotreating Unit

| Sample Number | Stream Name                                  | Test Method Number            | Frequency |         |
|---------------|----------------------------------------------|-------------------------------|-----------|---------|
|               | Test Name                                    |                               | Normal    | Startup |
| 1             | Charge to NHT (Reactor Feed)                 |                               |           |         |
|               | Gravity                                      | ASTM D 4052 or<br>ASTM D 1298 | 1/D       | 3/D     |
|               | API                                          | ASTM D 287                    | 1/D       | 3/D     |
|               | Distillation                                 | ASTM D 86                     | 1/D       | 3/D     |
|               | Color                                        | ASTM D 156                    | 3/D       | 3/D     |
|               | Sulfur                                       | ASTM D 4045                   | 1/D       | 3/D     |
|               | Chloride                                     | UOP 588                       | Occas.    | 1/D     |
|               | Nitrogen                                     | ASTM D 4629                   | Occas.    | 1/D     |
|               | Composition (PONA)                           | UOP 880                       | Occas.    | Occas.  |
|               | Arsenic                                      | UOP 946                       | Occas.    | 1/D     |
|               | Lead                                         | UOP 952                       | Occas.    | 1/D     |
|               | Bromine Number                               | UOP 304                       | Occas.    | 1/D     |
|               | Dissolved Oxygen                             | UOP 678                       | Occas.    | Occas.  |
|               | Trace Metals                                 | UOP 389                       | Occas.    | Occas.  |
|               | Mercury                                      | UOP 938                       | Occas.    | Occas.  |
| 2             | Recycle Gas (Separator Off-Gas)              |                               |           |         |
|               | Relative Density                             | UOP 114                       | 1/D       | 3/D     |
|               | Composition                                  | UOP 539                       | 3/W       | 1/D     |
| 3             | Stripper Off-Gas                             |                               |           |         |
|               | Relative Density                             | UOP 114                       | 1/D       | 3/D     |
|               | Composition                                  | UOP 539                       | 3/W       | 1/D     |
| 4             | Stripper Overhead Liquid                     |                               |           |         |
|               | Composition (requires high pressure sampler) | UOP 551                       | Occas.    | 1/D     |
|               | H <sub>2</sub> S                             | UOP 212                       | Occas.    | 1/D     |

| Sample Number | Stream Name<br>Test Name         | Test Method Number            | Frequency |         |
|---------------|----------------------------------|-------------------------------|-----------|---------|
|               |                                  |                               | Normal    | Startup |
| 5             | <b>Stripper Bottoms</b>          |                               |           |         |
|               | Relative Density                 | ASTM D 4052 or<br>ASTM D 1298 | 3/D       | 3/D     |
|               | API                              | ASTM D 287                    | 3/D       | 3/D     |
|               | Distillation                     | ASTM D 86                     | 3/D       | 3/D     |
|               | Color                            | ASTM D 156                    | 3/D       | 3/D     |
|               | Sulfur                           | ASTM D 4045                   | 1/D       | 1/D     |
|               | Chloride                         | UOP 395                       | 1/W       | 1/D     |
|               | Nitrogen                         | ASTM D 4629                   | 1/W       | 1/D     |
|               | Composition (PONA)               | UOP 880                       | 1/W       | 1/D     |
|               | Composition (C <sub>6</sub> -)   | UOP 551                       | Occas.    | 1/D     |
|               | Arsenic                          | UOP 296                       | 1/M       | 1/W     |
|               | Lead                             | UOP 350                       | 1/M       | 1/W     |
|               | Copper                           | UOP 144                       | 1/M       | 1/W     |
|               | Water Content                    | UOP 481                       | Occas.    | 1/W     |
|               | Bromine Number                   | UOP 304                       | 1/M       | 1/W     |
|               | Silicones                        | UOP 787                       | 1/W       | 1/W     |
|               | Fluoride                         | UOP 619                       | 1/M       | 1/W     |
| 6             | <b>Naphtha Splitter Overhead</b> |                               |           |         |
|               | Composition                      | UOP 551                       | As req.   | 1/D     |
|               | Relative Density                 | ASTM D 4052 or<br>ASTM D 1298 | 3/D       | 3/D     |
|               | API                              | ASTM D 287                    | 3/D       | 3/D     |
|               | Distillation                     | ASTM D 86                     | 3/D       | 3/D     |
| 7             | <b>Naphtha Splitter Bottoms</b>  |                               |           |         |
|               | Composition                      | UOP 880                       | As req.   | 1/D     |
|               | Relative Density                 | ASTM D 4052 or<br>ASTM D 1298 | 3/D       | 3/D     |
|               | API                              | ASTM D 287                    | 3/D       | 3/D     |
|               | Distillation                     | ASTM D 86                     | 3/D       | 3/D     |
|               | Nitrogen                         | ASTM D 4629                   | As req.   | As req. |
| 8             | <b>Make-Up Hydrogen</b>          |                               |           |         |
|               | Relative Density                 | UOP 114                       | 1/D       | 3/D     |
|               | Composition                      | UOP 539                       | 1/D       | 1/D     |
| 9             | <b>Charge Heater Flue Gas</b>    |                               |           |         |
|               | Oxygen Content                   | Orsat                         | Occas.    | Occas.  |

| Sample Number | Stream Name<br>Test Name                                                                               | Test Method<br>Number                    | Frequency                            |                                   |
|---------------|--------------------------------------------------------------------------------------------------------|------------------------------------------|--------------------------------------|-----------------------------------|
|               |                                                                                                        |                                          | Normal                               | Startup                           |
| 10            | <b>Stripper Reboiler Heater Flue Gas</b><br>Oxygen Content                                             | Orsat                                    | Occas.                               | Occas.                            |
| 11            | <b>Naphtha Splitter Reboiler Heater Flue Gas</b><br>Oxygen Content                                     | Orsat                                    | Occas.                               | Occas.                            |
| 12            | <b>Product Separator Water</b><br>pH, Iron, Copper<br>NH <sub>3</sub><br>H <sub>2</sub> S<br>Chlorides | UOP 314<br>UOP 740<br>UOP 683<br>UOP 456 | Occas.<br>Occas.<br>Occas.<br>Occas. | 1/W<br>Occas.<br>Occas.<br>Occas. |
| 13            | <b>Stripper Overhead Receiver Water</b><br>pH, Iron, Copper                                            | UOP 314                                  | Occas.                               | 1/W                               |

**REGENERATION CASE**

|    |                                                                                                         |                                                                      |                                                          |  |
|----|---------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------|----------------------------------------------------------|--|
| 14 | <b>Recycle Gas</b><br>CO <sub>2</sub> by Orsat<br>O <sub>2</sub><br>H <sub>2</sub> S<br>SO <sub>2</sub> | UOP 172<br>Portable Anal.<br>Detector Tube<br>Detector Tube          | As Required<br>As Required<br>As Required<br>As Required |  |
| 15 | <b>Reactor Effluent Gas</b><br>CO <sub>2</sub> by Orsat<br>O <sub>2</sub>                               | UOP 172<br>Portable Anal.                                            | As Required<br>As Required                               |  |
| 16 | <b>Spent Caustic</b><br>Percent NaOH<br>pH (pH Meter)<br>pH<br>Total Solids<br>Settleable Solids        | UOP 210<br>ASTM D 1293<br>Litmus Paper<br>APHA 2540-A<br>APHA 2540-F | As Required<br>As Required<br>As Required<br>1/W<br>1/W  |  |
| 17 | <b>Circulating Caustic</b><br>Percent NaOH<br>pH (pH Meter)<br>pH<br>Total Solids<br>Settleable Solids  | UOP 210<br>ASTM D 1293<br>Litmus Paper<br>APHA 2540-A<br>APHA 2540-F | As Required<br>As Required<br>As Required<br>1/W<br>1/W  |  |

## **X. TROUBLESHOOTING**

No information is provided in this section. Please refer to the Process Principles and Process Variables sections for potential resolutions. If further assistance is required, please contact UOP.

## **XI. NORMAL SHUTDOWN**

### **A. NORMAL SHUTDOWN PROCEDURE**

The following shutdown procedures cover normal planned complete shutdowns of the Naphtha Hydrotreating Unit such as would be required for a complete catalyst change and/or the periodic cleaning and inspection of vessels. Variations of this procedure may be required from time to time because of special operating conditions which may arise.

1. Notify operating foreman and other operating units concerned as to the exact time when shutdown activity will begin. Changes in fuel gas composition, steam demand, etc., may affect other units. Pumpers, tank farm, and others who may be involved should be notified.
2. Reduce the hydrotreating reactor(s) inlet temperature to 316°C (600°F) and the charge to about 50% of design. The Platforming Unit must be fed sweet naphtha at this time, or it must be shut down also.
3. Cut charge out of the unit and continue to sweep the unit with gas to remove hydrocarbons.
4. The prefractionator and rerun columns should be cooled down by stopping reboiler heat input, and should be left under positive fuel gas pressure. If entry into the columns is required, at a minimum, they must be drained, steamed out, blinded off from other equipment, and air purged for safe entry.
5. After approximately one hour of gas sweeping at a minimum reactor(s) temperature of 260°C (500°F), begin reducing reactor(s) temperatures by 30-40°C (50-75°F) per hour to 65°C (150°F) or 38°C (100°F) if the catalyst is to be dumped unregenerated.

6. If the catalyst is to be regenerated, the reactor(s) can be left at 260°C (500°F) when the gas flow is shut down. More specific procedures are given in the Regeneration Section of the Section XIII Special Procedures.
7. Block in the product separator level control valve when liquid stops accumulating. Drain the separator and all reactor section low points to remove all hydrocarbons.
8. The stripper and splitter should be cooled down and drained if any maintenance work is required. Shut down stripper bottoms and/or splitter bottoms pump.
9. Shut down the recycle gas or once-through gas flow when the reactor is cool.
10. The unit may be depressured to about 1 kg/cm<sup>2</sup>g (15 psig) pending maintenance.

## **XII. EMERGENCY PROCEDURES**

Emergencies must be recognized and acted upon immediately. The operators should carefully study, in advance, the steps to be taken in such situations. While some of the emergencies listed in this section may not result in a unit shutdown, they could cause serious trouble on the unit if not handled properly. In addition, damage to the catalyst might occur. Hard and fast rules cannot be made to cover all situations which might arise. The following outline lists those situations which might arise and suggested means of handling the situation. Because the Naphtha Hydrotreating Unit and Platforming Unit are so intimately connected, an emergency in one unit will usually cause an emergency in the other.

### **A. LOSS OF RECYCLE COMPRESSOR**

1. Stop Naphtha Hydrotreating Unit charge heater fires immediately and cut steam through the furnace boxes for its cooling effect. The charge heater should shut down automatically on low recycle gas flow.
2. Switch the Platforming Unit to sweet naphtha feed, bypassing the hydrotreater, so it can continue to run. If this is not possible, shut down the Platforming Unit in the normal manner.
3. Shut off Naphtha Hydrotreating Unit charge pumps and block in.
4. Block in Naphtha Hydrotreating Unit offgas valves so that the system pressures are maintained. Block in liquid levels.

**NOTE:** The above four steps must be performed as quickly as possible.

5. Start the compressor as quickly as possible. Remember that with no flow through the heater, the material in the tubes may become excessively hot, and if it was put through the reactors, could result in damage to the catalyst. Thus, when the compressor is started after such a shutdown, immediately check the

reactor inlet temperatures; if over 343°C (650°F), stop recycle flow and continue cooling the heater with purging steam until the reactor inlet temperatures, with recycle gas flowing, are below 343°C (650°F).

6. When the compressor is again in service, come back on stream in normal manner.
7. If a compressor cannot be started within an hour, the hydrotreating reactor pressure should be bled off gradually to the fuel gas system or flare until 7.0 kg/cm<sup>2</sup>g (100 psig) of pressure is released. This is done to purge out hydrocarbons present in the reactor as much as possible and minimize coking.

#### **B. REPAIRS WHICH REQUIRE STOPPING COMPRESSOR WITHOUT DEPRESSURING OR COOLING REACTORS**

1. Drop reactor(s) temperature to 315°C (600°F) by cutting back on heater outlet temperatures while reducing reactor charge rate to one-half. Cut out reactor charge. Block in separator and columns as in normal shutdown.
2. If unit has been operating at a low pressure, it would be proper to increase the unit pressure before the shutdown, so that leakage during the down period will not be so great as to require extra outside hydrogen to be purchased and used in the subsequent startup. However, **DO NOT EXCEED DESIGN PRESSURE FOR THE UNIT.**
3. Continue firing heaters and circulate recycle gas for one hour while reducing reactor inlets to 260-290°C (500-550°F).
4. Cut fires from all heaters and stop compressor.

**C. EXPLOSION, FIRE, LINE RUPTURE, OR SERIOUS LEAK –  
DO IF POSSIBLE**

1. Cut fire from all heaters. If heaters or control valve are beyond reach, main gas valve can be used.
2. Stop reactor charge pumps. Stop other pumps, if possible.
3. Leave compressor running if possible while other items are attended to since it will contribute little extra pressure to the system.
4. Shut down compressor.
5. Depressure plant. Use of separator safety relief valve will depressure the separator and the system.
6. Shut down balance of plant as circumstances permit or require.
7. If leak or line rupture is in a heater, add snuffing steam to cool down firebox **AFTER THE SOURCE OF COMBUSTIBLES HAS BEEN ELIMINATED. DO NOT CLOSE THE HEATER DAMPER** as it may force the fire out of the heater.
8. Purge unit of hydrocarbon as soon as possible. **DO NOT EVACUATE.**

**D. INSTRUMENT AIR FAILURE**

1. Locate reason for failure.
2. If in air drier system, bypass that section. This can then be repaired when possible.

3. If air cannot be obtained, the alternatives are a plant shutdown, or operation of the various controls on hand control. Action will depend upon the desires of the supervisor.
4. Due to the possibility of instrument air being used for breathing equipment and/or pneumatic instruments venting in the control room, do not add nitrogen to the instrument air system.
5. Be familiar with action of all control valves – memorize all actions on air failure.

## **E. POWER FAILURE**

Emergency procedures during a power failure will vary greatly, depending on: (1) the extent of the failure, (2) which utilities may be affected by a failure, and (3) the length of the failure. In general, the first consideration in case of a power failure, is to bring the plant to a safe standby condition. Of almost equal importance, however, is the protection of the catalyst.

1. If the charge pump and recycle compressor are motor driven, they will stop. Be sure the charge heater fires trip out and that the individual burner fuel valves are blocked in.

If the compressor is turbine driven, it should be run long enough to sweep hydrocarbons from the system and cool down the reactor and charge heater before it is shut down.

2. If the stripper reboiler pump is motor driven, be sure the fires trip out and the individual burners are blocked in.
3. When power is restored, follow the procedure for startup after a recycle compressor failure.

**F. LOSS OF COOLING WATER**

Watch condenser and cooler temperatures, especially on recycle gas compressor system. A shutdown may be necessary if temperatures rise too much. Do not jeopardize the recycle gas compressor by allowing lube oil temperatures to increase above 70°C (160°F). If this occurs, follow emergency procedure for shutdown of the unit.

## **XIII. SPECIAL PROCEDURES**

### **A. CATALYST LOADING**

#### **1. Catalyst Loading Preparation**

Loading of the reactor(s) is normally the last item attended to before the unit starts up. Reactor loading consists of the following:

1. Planning and preparation including a loading diagram
2. Installation and inspection of the bottom internals
3. Loading of the catalyst support material
4. Loading of the catalyst
5. Loading of the catalyst graded bed material
6. Installation of the inlet distributor and bolting up of the reactor

#### **2. Catalyst Loading Procedure**

It must first be decided how the catalyst is to be lifted to the top of the reactors. It could be lifted in its original drums by a monorail or pulley system, but the quickest and probably best way is to lift the catalyst by a crane if one is available. If a crane is used, the loading time can be greatly reduced by constructing two large transfer hoppers to move the catalyst from the ground level to the top of the reactors. In this case a transfer hopper loading platform must be constructed in a convenient place close to the reactors. The loading platform can be constructed of scaffolding and wooden planks or of any other convenient material. The platform area should be at least large enough to accommodate enough drums to load one hopper and to allow working room for the personnel who will do the loading.

Regardless of the way the catalyst will be lifted, a convenient, temporary storage place near the reactor must be found for the catalyst. The catalyst should be stored on pallets and completely covered by canvas to give a certain measure of protection against the elements. A forklift or some other means of moving the

catalyst from this site to the loading platform (or to any other place) should be available.

The catalyst loading path, both on the ground and through the air, must be checked so that it is entirely free from obstruction. To assure this, it may be necessary to remove or modify the reactor superstructure, the piping support, or the piping itself. Failure to obtain a clear loading path could result in a slow and hazardous loading procedure. A typical lay-out for the catalyst loading is shown in Figure XIII-1.

A list must be made of all the accessory equipment which will be needed to do the loading. A partial list of some of the items that will be needed follows:

1. One crane (not necessary but very helpful if available) and safety hitch for the crane hook.
2. One forklift.
3. Two large transfer hoppers (also not necessary, but desirable) and separate lifting cables for each hopper.
4. One transfer hopper loading platform if transfer hoppers are to be used.
5. One loading hopper to rest above the reactor.
6. Explosion-proof light inside the reactor (and also flashlights).
7. Ceramic rope to protect the manway ring joint and to fill crevices created between certain reactor internals.
8. Ceramic gasket or wooden cover to protect the reactor manway and elbow flange gasket surfaces.
9. Canvas for covering the catalyst drums and for covering the reactor inlets between loading intervals as protection against rain, snow, etc.

10. A safe ladder to enter and leave the reactor.
11. Loading socks of correct diameter and sufficient length to do the loading in the manner planned.
12. Wooden boards to stand on while inside the reactor and to level the catalyst and catalyst support material.
13. A vacuum eductor to remove catalyst dust.
14. Dust masks for the personnel who will be working with the catalyst. Fresh air masks may be desirable for the personnel who will be doing the actual loading inside the reactors.
15. Measuring tapes for both small and large measurements (such as catalyst loading outages).
16. Chalk, crayons, or other types of markers to mark the reactor walls.
17. Wooden covers or other means (such as plastic and tape) to the reactor baskets. Cover the reactor baskets.
18. Miscellaneous hand tools such as pliers, screwdriver, etc.
19. Air hose to supply air to the dense loading machine (if the catalyst is to be dense loaded).
20. A planned loading diagram is prepared and supplied to the loading supervisor.

The bottom internals consist of the outlet basket, the catalyst loading sleeve and the catalyst unloading support plate. The outlet basket is constructed of perforated plate and, in newer designs consists of multiple sections that are fitted together once inside the reactor. The slots in the plate are usually 10 mm by 40 mm oblong slots on 25 mm centers with 13 mm between the ends of the slots. The entire outlet basket should be inspected for deviations from the project specification and for

structural weaknesses. For older designs, the basket is centered over the outlet nozzle by four equally spaced lugs, which fit inside the outlet nozzle. For newer designs, the outlet collector is attached to the bottom head with three hold down legs. Verify that the bottom centering ring, which is cut to the bottom head radius, is flush along the bottom and there will be proper containment.

The catalyst unloading sleeve is made of 1.5 mm (16 ga.) plate. It is loosely fit into the catalyst unloading nozzle. Into the catalyst unloading nozzle is fitted the catalyst support plate. A ceramic rope must be placed on top of the plate to plug the unloading nozzle. The removable support plate rests on three equally spaced lugs. This support plate allows the removal of the nozzle blank flange and the installation of an external unloading spout prior to the actual removal of any catalyst.

Once the bottom internals are in place, the loading can begin. Be sure to record all the amounts and types of material loaded and construct an "as loaded" diagram similar to Figure XIII-2. Begin loading the catalyst support material as follows (Project Specifications take precedence for actual dimensions):

1. 19 mm (3/4") diameter ceramic balls are loaded to a level height above the outlet basket as shown in Figure XIII-2. Typically 100 mm above the outlet basket top.
2. A level layer of 6 mm (1/4") diameter ceramic balls are placed on top of the 19 mm balls as shown in Figure XIII-2.
3. A level layer of 3 mm (1/8") spherical Hydrobon® catalyst base, ceramic balls or NMRS, is usually placed on the 6 mm balls as shown in Figure XIII-2.

When loading each layer of the catalyst support material, care must be taken so that the previous layer of balls is not disturbed. Cratering of any layer may cause migration of the balls resulting in the migration of the catalyst bed.

The catalyst unloading nozzle is also filled with 6 mm (1/4") balls to within 100 mm (4 in.) of the top. The remaining 100 mm (4") space is filled with 3 mm (1/8") catalyst base or ceramic balls. An alternate is that the sleeve may be

completely filled with 3 mm spherical Hydrobon® catalyst base or ceramic balls. Catalyst should not be used due to coking potential.

4. The UOP Hydrotreating catalyst may be sock loaded or "dense" loaded by using UOP's dense loading machine. Dense loading has the primary advantage of being able to load more catalyst into the same reactor volume. In general, there are only a few differences in the sock loading and dense loading procedures. In dense loading, the dense loading machine is anchored either above or inside the reactor. A loading sock attached to the loading hopper feeds the loading machine with catalyst. The operator of the machine regulates it so that the catalyst is loaded uniformly and so that the level rises evenly. Even when dense loading, it will probably be necessary to level the bed after reaching the desired catalyst height. Afterwards, an outage measurement should be taken and recorded.

If the sock loading method is used, the sock should only extend to the reactor top tangent line when connected to the hopper in the reactor inlet manway. The catalyst must be loaded slowly at first to prevent cratering of the support material. The sock should be kept moving in a figure eight pattern to prevent the catalyst from forming hills. It is important to keep the catalyst bed as level as possible during loading so the loaded catalyst density is uniform throughout the bed.

5. During the loading of the catalyst, an accurate count of the drums loaded and the drum lot numbers must be recorded. It is advisable to retain a composite catalyst sample for future reference composed of about 1 oz. of catalyst from each drum loaded.
6. Load catalyst to the specified level.
7. Load the bottom layer of graded bed material (usually TK-550) to the depth specified, typically 300 mm (12 inches), but can be as much as 600 mm (24 inches).

8. Load the top layer of graded bed material (usually TK-10) to the depth specified, typically 150 mm (6 inches). The loading should be calculated such that the distance from the top of the TK-10 to the bottom of the inlet distributor is not less than that indicated in the following table:

| <u>Reactor ID</u> | <u>Height of Open Space</u> |
|-------------------|-----------------------------|
| 4' – 6'           | 18" minimum                 |
| 6.5' – 8'         | 15" minimum                 |
| >8.5'             | 12" minimum                 |

9. A catalyst loading diagram (as loaded) similar to Figure XIII-2 should be drawn and returned for reference.

**NOTE:** While loading the last layers of catalyst and the graded bed materials, careful measurements must be taken. This is especially true if loading different types of catalyst in a reactor. The top layer of the top graded bed material (TK-10) must not be above the reactor tangent line. The minimum distance from the top of the graded bed material to the bottom of the inlet distributor is as shown in the loading diagram. Catalyst should be leveled before the graded bed material.

## **B. UNLOADING OF UNREGENERATED CATALYST CONTAINING IRON PYRITES**

The following precautions are recommended for use during unloading of unregenerated hydrotreating catalyst. The main concern is that no oxygen be allowed to contact the catalyst inside the reactors, since this can result in spontaneous combustion of the iron pyrites. The temperature generated by this combustion can be quite high, and left unchecked can result in severe damage to the catalyst and reactor internals. Of secondary, but no less importance, is protection of personnel and proper handling of catalyst during unloading. All personnel involved with the unloading must be properly informed of the dangers involved and the proper safety measures.

1. Follow the shutdown procedure as outlined in the procedures section. Be very careful to drain all residual hydrocarbons from the system low points, the separator and the feed line down stream of the feed flow control valve.
2. After all residual hydrocarbons have been drained from the system, cut out the heater fires and cool the reactor beds to less than 66°C (150°F) preferably to 57°C (135°F). At temperatures above this level, combustion of iron pyrites is greatly accelerated and more difficult to control. If the catalyst is to be screened during unloading, the catalysts beds should be cooled to less than 54°C (130°F).
3. After cooling the beds to 66°C (150°F), the unit should be evacuated and purged with N<sub>2</sub> at least twice. The unit should then be properly isolated and a small N<sub>2</sub> purge established at the compressor discharge or preferably at the inlet to each reactor. Do not open the reactors at the top until all catalyst has been unloaded.
4. Connect the unloading nozzle and be sure that a full opening but positive shutoff valve is installed. This is best accomplished by using a ball-type or slide valve.
5. Remove all combustible materials from the area.
6. Be sure that several CO<sub>2</sub> extinguishers are available.
7. Use only metal drums for unloading, and dump directly into drums if possible. It is best to screen the catalyst after it has had time to cool to ambient temperature. Each drum should be either purged with N<sub>2</sub> during unloading or a piece of dry ice placed at the bottom of each drum. Should the catalyst be screened at the same time as it is being dumped from the reactors, nitrogen should be purged through the dumping nozzle to the top of the first screen to provide additional protection from pyrite ignition.

Do not seal the drum air tight since this could result in sudden rupture of the drum should combustion occur. Burning of catalyst in the drums is not serious and can be quickly extinguished with nitrogen or CO<sub>2</sub>.

8. It is expected that some "sparking" of the pyrites will take place in any event. Therefore, all workmen in the area must be supplied with face and eye protection. In addition, they should wear long sleeve shirts with collars and cuffs tightly buttoned.
9. Maintain a positive flow of N<sub>2</sub> out of the unloading nozzle throughout the unloading. If the catalyst becomes bridged in the unloading nozzle or is not free flowing, break the plug with a blast of N<sub>2</sub> or steam. Do not allow air to be drawing into the reactor.
10. If ignition of pyrites takes place inside a reactor, stop unloading in that reactor and increase the N<sub>2</sub> purge to maximum until burning has stopped.

### C. CATALYST SKIMMING PROCEDURE

The amount of catalyst to skim off is dependent on the amount of fines deposited in the catalyst. The extent of this is difficult to determine without seeing the condition of the catalyst, but generally has been in the 1 meter (3 feet) range. The catalyst should be inspected as it is skimmed and continue until fines are no longer observed. If the depth of catalyst skimmed is insufficient then the skimming operation may not be successful.

Follow the procedure above for "UNLOADING OF UNREGENERATED CATALYST CONTAINING IRON PYRITES" but do not unload the catalyst from the bottom. Since the top layers collect most of the iron and other inorganic material, special precautions and care must be taken when handling this material.

The catalyst can be vacuumed out from the top of the reactor. The reactor should be under a nitrogen atmosphere, especially for nickel containing catalyst. This is best performed by catalyst handling companies that specialize in this operation.

There are several catalyst handling companies who are experienced with catalyst loading and unloading in an inert atmosphere.

If "fresh" catalyst is being added, sulfiding of this new catalyst is required during the restart.

## **NAPHTHA HYDROTREATING CATALYST REGENERATION**

During operation, carbon will gradually accumulate on the catalyst. The rate of accumulation will depend upon the type of feedstock and the type of operation to which the catalyst is being subjected. The rate at which the carbon accumulates will increase if heavier feedstocks are used or if improper operating conditions are employed. The accumulated carbon, polymer, or metals deposited on the catalyst by the feed will eventually cause the catalyst to become deactivated to the point where it will not produce acceptable product quality.

When the catalyst does become deactivated, it must either be discarded or regenerated. If carbon is the prime cause of the deactivation, the activity can be substantially restored by burning off the carbon under carefully controlled conditions. It must also be noted that the process of burning off the carbon does not remove the metals that have been deposited on the catalyst. If metals are the reason for the deactivation, then that catalyst must be discarded.

Special precautions must be followed throughout the procedures if there is austenitic stainless steel in the reactor section of the unit. Water in the liquid phase plus oxygen should never be allowed to come in contact with austenitic stainless steel since there will be a light deposit of iron sulfide on the metal. If water, oxygen, and sulfur come in contact with austenitic stainless steel, the areas of stress, such as welds, could suffer from stress cracks. This hazard should at all times be considered by the supervisor and operating personnel who will be conducting the regeneration.

The temperature of austenitic stainless steel should be kept above the dew point of water when water and oxygen are both present to prevent stress cracking due to polythionic acid. Neutralization is not recommended unless absolutely necessary, such as when austenitic tube bundles are to be pulled for maintenance.

**NOTE:** (Only for austenitic stainless steel) If, for any reason, it is necessary to open the reactor system prior to regeneration, the unit must first be properly evacuated and purged with dry nitrogen. The nitrogen blanket should then be maintained with a small nitrogen purge in the section to be opened to prevent air and moisture from entering. Other sections should be blinded at this time.

**NOTE:** For the purpose of naphtha hydrotreating catalyst regeneration with austenitic stainless steel in the reactor section, nitrogen containing 100 mol-ppm or less of oxygen must be used.

There are two basic procedures for regeneration of Hydrobon® catalyst: steam-air or inert gas. The steam-air procedure is still commonly used. However, the steam-air procedure should only be used for S-6 and S-9 Hydrobon® catalysts as an activity loss may result in the other catalyst formulations. For other UOP Hydrobon® catalysts, these must be regenerated using the inert gas technique to ensure no loss of activity. All of the Hydrobon® catalysts can be regenerated by the inert gas procedure, and it is commonly used on units that are designed with recycle gas rather than once-through gas.

Throughout both procedures, the steps shown in capital letters are intended specifically for units with austenitic stainless steel in the reactor circuit. These steps may be omitted on units without austenitic stainless steel.

### **C. STEAM-AIR REGENERATION PROCEDURE (FOR S-6 AND S-9 HYDROBON® CATALYSTS)**

#### **Shutdown**

1. Cut the feed out of the unit, and increase the flow of hydrogen through the reactor section to the maximum available.
2. Raise the reactor inlet temperature to 370-400°C (700-750°F), continuing maximum hydrogen flow over the catalyst. Do not exceed the reactor design temperature.
3. Hold until the reactor outlet temperature is 400°C (750°F) or slightly higher for at least one hour, and until the accumulation of hydrocarbon in the separator and reactor section low points is zero (nil).
4. Stop the flow of hydrogen to the reactor. IMMEDIATELY REDUCE CHARGE HEATER FIRES AS NECESSARY TO MAINTAIN 290-315°C (700-750°F) FIREBOX TEMPERATURES, AS MEASURED BY THERMOCOUPLES PLACED IN THE HIP SECTIONS OF THE HEATER BELOW ANY CONVECTION COILS THAT MAY EXIST. ONLY FUEL GAS FIRING SHOULD BE USED FOR THIS OPERATION BECAUSE OF THE DIFFICULTY IN CONTROLLING AND MAINTAINING SUFFICIENTLY SMALL FLAMES WHEN BURNING FUEL OIL. BE CAREFUL AT THIS POINT TO PREVENT OVERFIRING THE CHARGE HEATER. If the heater coils and reactor are not austenitic steel, the heater firing should be stopped.
5. Depressure, evacuate and purge with nitrogen in the normal manner. BE SURE THAT PURGING NITROGEN GETS HEATED IN THE CHARGE HEATER BEFORE PASSING THROUGH THE REACTOR IF THE REACTOR CONTAINS AUSTENITIC STEEL.

6. AFTER EVACUATION AND PURGING IS COMPLETED, REDUCE THE CHARGE HEATER FIRES TO MAINTAIN 205°C (400°F) FIREBOX TEMPERATURES IF THE COILS ARE FABRICATED FROM AUSTENITIC STAINLESS STEEL.
7. AFTER EVACUATION AND PURGING OF HYDROGEN IS COMPLETE, A LOW NITROGEN PURGE SHOULD BE CONTINUED TO PREVENT AIR FROM ENTERING THE SYSTEM WHILE PREPARING FOR REGENERATION. REDUCE THE CHARGE HEATER FIRES TO MAINTAIN 205°C (400°F) FIREBOX TEMPERATURES IF THE COILS ARE FABRICATED FROM AUSTENITIC STAINLESS STEEL. IF THE REACTOR CONTAINS AUSTENITIC STAINLESS STEEL INTERNALS, IT WILL BE NECESSARY TO MAINTAIN 290-315°C (550-600°F) CHARGE HEATER FIREBOX TEMPERATURES TO ASSURE PREHEATING THE PURGING NITROGEN. Otherwise, it is most convenient to merely maintain small gas pilots in the interim while mechanical preparations are being made prior to beginning the regeneration.

### Preparations

Be prepared to hook up all required regeneration equipment rapidly to avoid any unnecessary loss of heat from the reactor.

1. Maintain low nitrogen purge during the following mechanical preparations.
2. Disconnect the effluent line from the reactor at the designated location, and hook up a temporary vent line to atmosphere at a safe location to dispose of the steam and waste gases during regeneration. A thermocouple should be maintained or installed as necessary at the reactor outlet for temperature indication during regeneration.
3. Blind off the combined feed line ahead of the reactor charge heater and connect the regeneration steam line.

4. Hook up the regeneration air manifold to the combined feed line between the heater and the reactor. The line should be sized so that it will provide enough air to give an oxygen content of up to 2.0 mol-% in the steam-air mixture during regeneration. The air line is provided with an FRC assembly to measure and control the quantity of air being used.
5. Connect the sample cooler and the measuring apparatus to the regeneration vent gas line from the reactor.

### Regeneration

BEFORE ADMITTING STEAM TO A REACTOR CONTAINING AUSTENITIC STAINLESS STEEL INTERNALS, IT MUST BE ASSURED THAT THE CATALYST BED TEMPERATURE IS ABOVE 205°C (400°F) TO PREVENT ANY CONDENSATION OF STEAM IN THE REACTOR.

1. Drain the regeneration steam lines of condensate to assure the availability of dry saturated steam for regeneration.
2. Raise the charge heater firebox temperature, as measured by thermocouple(s) placed in the hip section, to about 260°C (500°F). Slowly establish a flow of steam to the reactor 0.225 kg/hr (0.5 lb/hr) per pound of catalyst in the reactor, adjusting the heater fires simultaneously as necessary to hold the heater transfer temperature above 260°C (500°F). Be careful to avoid overfiring the heater.

NOTE: Until steam flow is established through the heater tubes, the heater transfer temperature can not be used to control the firing.

3. Increase the steam flow rate to the maximum possible and simultaneously increase the reactor inlet temperature at 28°C/hr (50°F/hr) to 288°C (550°F). Do not exceed 3.5 kg/cm<sup>2</sup> (50 psi) pressure drop across the reactor; reduce the steam flow rate if necessary. Again, be careful to avoid overfiring the heater.

4. When the reactor inlet temperature has been held at 288°C (550°F) for at least one hour, cut in enough air so that there will be approximately 0.5 mol-% oxygen in the steam-air mixture. Take two or three volume samples of the condensate and the waste gas to verify the accuracy of the steam and air meters.
5. If the meter readings are verified and the reactor outlet temperature is less than 370°C (700°F), slowly raise the air rate to give 1.0 mol-% of oxygen in the steam-air mixture. Verify this figure by taking volume samples of condensate and waste gas. Continue taking volume samples every two hours to check the accuracy of the meters.
6. An Orsat or sniffer tube should also be used every two hours to check the CO<sub>2</sub> content of the waste gas.
7. Keep a record of the reactor outlet temperatures and if it appears that this temperature is going to exceed 370°C (700°F) at any time during the regeneration, the reactor inlet temperature or oxygen content should be adjusted accordingly.
8. Continue analyzing the waste gas for CO<sub>2</sub> content and when this figure falls below 10% and the delta T is zero, raise the reactor(s) inlet temperature to 343°C (650°F) at the rate of 42°C (75°F) per hour.
9. Hold 343°C (650°F) reactor inlet temperature until the CO<sub>2</sub> content of the waste gas falls below 2 mol-%. At this time, the regeneration can be considered complete as long as the reactor(s) outlet temperature has cooled to the same as the reactor inlet temperature. If not, continue maintaining these conditions as necessary.
10. After completion of regeneration, stop the air addition. REDUCE THE HEATER FIRES TO MAINTAIN 205°C (400°F) FIREBOX TEMPERATURES IF THE COILS ARE AUSTENITIC STAINLESS STEEL. IF THE REACTORS CONTAIN AUSTENITIC STAINLESS STEEL INTERNALS, FIRE THE CHARGE HEATER TO MAINTAIN 260°C (500°F) REACTOR INLET TEMPERATURE WHILE COOLING THE CATALYST WITH STEAM.

11. COOL THE CATALYST WITH STEAM TO A REACTOR OUTLET TEMPERATURE OF 290°C (550°F). If the charge heater and reactors are not austenitic steel, stop the charge heater firing and cool the catalyst to 232°C (450°F).
12. Stop the steam and immediately follow with a large flow of nitrogen to rapidly purge the steam from the reactors. IF THE REACTORS CONTAIN AUSTENITIC STAINLESS STEEL INTERNALS, MAINTAIN 290-315°C (550-600°F) CHARGE HEATER FIREBOX TEMPERATURES UNTIL ALL THE STEAM HAS BEEN PURGED FROM THE REACTOR.
13. Maintain nitrogen purges to prevent air from entering the system while disconnecting regeneration equipment and piping the plant for normal processing.

### **Dump and Screen Catalyst**

After the first regeneration, it is advisable to dump and screen catalyst. This will give an indication of the completeness of the regeneration plus an indication of the amount of fines and scale that can be expected on future regenerations.

Even though a catalyst regeneration has been conducted, small amounts of iron sulfide scale can still remain in the system. Therefore, with austenitic stainless steel in the heater/ reactors, it is necessary to cool the catalyst with nitrogen to prevent steam from condensing in the system.

1. Cool the catalyst with nitrogen flow until the reactor has dropped to about 50°C (120°F). Cooling to about 50°C (120°F) is necessary to allow safe handling of the catalyst, and to cool the reactors sufficiently to allow personnel to enter to clean and inspect the reactor, and to reload catalyst.
2. IF THE REACTORS CONTAIN AUSTENITIC STAINLESS STEEL INTERNALS, MAINTAIN A NITROGEN BLANKET IN THE REACTOR WHILE DUMPING AND SCREENING CATALYST, TO PREVENT AIR FROM ENTERING THE REACTORS.

3. AFTER THE CATALYST HAS BEEN DUMPED FROM A REACTOR CONTAINING AUSTENITIC STAINLESS STEEL, UNHEAD THE TOP OF THE REACTOR. THOROUGHLY WASH THE INSIDE REACTOR WALLS AND INTERNALS WITH A COPIOUS AMOUNT OF SODA ASH SOLUTION, AS EXPLAINED IN THE "PROTECTION OF AUSTENITIC STAINLESS STEEL" APPENDIX F BEFORE ALLOWING AIR TO ENTER THE REACTOR. AFTER THE REACTOR HAS BEEN THOROUGHLY NEUTRALIZED, AIR CAN BE ALLOWED TO ENTER THE REACTOR TO DRY IT OUT PRIOR TO ENTRY.
4. AFTER WASHING WITH THE SODA ASH SOLUTION, ALLOW THE SURFACES TO DRY WITH A FINE DEPOSIT OF SODA ASH. DO NOT RINSE THIS RESIDUE OFF WITH WATER, BUT LET IT REMAIN AS A PROTECTIVE FILM.
5. ANY CATALYST THAT REMAINED IN THE BOTTOM OF THE REACTOR WHILE WASHING WITH SODA ASH SOLUTION SHOULD BE CONSIDERED CONTAMINATED WITH SODIUM AND SHOULD BE DISCARDED AND REPLACED WITH FRESH CATALYST.
6. After cleaning out the bottom of the reactor, install reactor internals, catalyst support material, and catalyst as recommended in Section V. Start up the unit in the normal manner, treating the regenerated catalyst in the same manner as fresh catalyst.

### **Emergency Procedures During Steam-Air Regeneration**

1. If, at any time, the catalyst temperatures become excessive, block in the air injection control valve immediately.
2. If steam flow is interrupted and cannot be immediately resumed, then do the following:
  - a. Block in air injection immediately.

- b. If heater/reactors are not austenitic stainless steel, cut out heater fires.
  - c. IF HEATER/REACTORS ARE AUSTENITIC STAINLESS STEEL AND STEAM CAN BE RESTORED WITHIN ONE-HALF HOUR, MAINTAIN FIREBOX TEMPERATURES AT 288-343°C (550-650°F) AS MEASURED BY THERMOCOUPLES PLACED IN THE HIP SECTIONS OF THE HEATERS BELOW ANY CONVECTION COILS THAT MAY EXIST.
  - d. IF HEATER/REACTORS ARE AUSTENITIC STAINLESS STEEL AND STEAM CANNOT BE RESTORED WITHIN ONE-HALF HOUR, CUT IN HIGH NITROGEN PURGE RATE THROUGH HEATER AND REACTOR. MAINTAIN FIREBOX TEMPERATURES AT 205°C (400°F) IF FIREBOX COILS ARE AUSTENITIC AND 290-315°C (550-600°F) IF REACTOR INTERNALS ARE AUSTENITIC.
3. In general, whatever the emergency, take steps to prevent condensation of water in any austenitic system while oxygen is present. Also, avoid pumping caustic into the catalyst beds.

**D. INERT GAS REGENERATION PROCEDURE**  
**(FOR S-6, S-9, S-12, S-15, S-16, S-18, S-19, S-120, N-204, N-108 AND HC-K HYDROBON® CATALYSTS)**

**Shutdown**

1. Shut down the unit in the normal manner following the procedure detailed earlier. Continue recycle gas circulation for at least one hour 260°C (500°F) and until no more liquid accumulates in the product separator and the reactor section low points. Shut down the charge heater and then the recycle compressor. IF THE CHARGE HEATER COIL IS AUSTENITIC STAINLESS STEEL, MAINTAIN 205°C (400°F) FIREBOX TEMPERATURE. Drain all liquid hydrocarbon from the system.

2. Depressure the plant to fuel gas and then to the flare system. Block in the recycle and booster compressors and purge with nitrogen independent of the reactor system. Connect the ejector and evacuate the reactor system two times to at least 500-635 mm of Hg (20-25 inches of mercury), breaking each time with nitrogen.

### Preparation

1. With the unit under a slight positive pressure of  $N_2$ , install blinds as required to isolate the unit.
2. Evacuate the unit again to at least 500-635 mm (20-25 inches) of mercury, then pressure the plant to 3.5 kg/cm<sup>2</sup>g (50 psig) with nitrogen and establish maximum circulation with the recycle compressor.
3. Light the fires and line out the reactor inlet temperatures at 290°C (550°F). Allow the reactor pressure to increase to any pre-chosen positive pressure less than 21 kg/cm<sup>2</sup>g (300 psig). Regenerating at pressures higher than normal plant air pressure will require that air be boosted. This may be done with auxiliary air booster compressors.
4. Determine the inert gas circulation rate as measured by the recycle gas meter, correcting for pressure, temperature, and density.
5. A level of water can be pumped into the high-pressure separator when the reactor inlet temperatures are stabilized at 290°C (550°F) and the reactor outlet temperatures reach their maximum level. Also the recycle gas flow rate should be steady and the heater outlet temperatures must be on automatic control and steady.

**PRECAUTION:** DO NOT CHARGE ANY WATER OR CAUSTIC INTO THE HIGH-PRESSURE CIRCUIT UNTIL HEATER AND REACTOR TEMPERATURES ARE HIGH ENOUGH SUCH THAT THE WATER WILL BE ABOVE ITS DEW POINT IN ALL LOCATIONS WHERE AUSTENITIC STAINLESS

STEEL IS PRESENT. THIS PRECAUTIONARY MEASURE IS TO MAKE CERTAIN THAT MOISTURE WILL NOT CONDENSE ON THE AUSTENITIC STAINLESS STEEL.

NOTE THAT IN THE CASE OF AUSTENITIC HEAT EXCHANGERS IN THE RECYCLE CIRCUIT THAT ARE NOT BYPASSED, TEMPERATURES BOTH IN AND OUT MUST BE ABOVE THE DEW POINT OF WATER BEFORE ADDING WATER, CAUSTIC, OR AIR.

6. Using the wash water pump, begin injecting clean condensate into the normal process injection point upstream of the condenser for the high pressure separator.

When a working level has been established in the separator, set the normal hydrocarbon level control instrument (now connected to the normal water draw control valve) to dump the excess.

7. Establish the design rate of water to the trays in the high compressor suction drum. At no time during the regeneration should this water rate be allowed to drop below the design rate for the trays. This prevents any entrainment of sodium salts in the separator gas to the recycle compressor, and keeps the total solids content in the circulating liquid stream below 10 weight percent.
8. Start the caustic circulation pump and maximize the rate. This is to ensure complete removal of the  $\text{SO}_2$  in the circulating gas.
9. Circulate the condensate from the compressor suction drum to the product condenser as long as desired to wash and flush the product condenser. Make whatever adjustments necessary to establish steady flow rates and a steady level. Note that the level will be held at about the same level that hydrocarbon is normally held.
10. Start pumping caustic into the circulating water stream prior to air injection and adjust the injection rate such that the total concentration of NaOH circulated to the product condenser will be about 3 wt-% to 6 wt-% in the beginning. The

addition of fresh caustic during the regeneration plus the water being pumped to the compressor suction drum trays should be such that the concentration of NaOH in the total caustic plus water added does not exceed 6 wt-% at any time. This will prevent the accumulation of excessive amounts of dissolved salts, which could drop out of solution in the products condenser and plug the tubes. The total solids content of the circulating caustic should be checked hourly during the regeneration to assure that it does not exceed 10 wt-% at any time.

11. The pH of the circulating caustic at this time will be about 14. Later when the rate of carbon and sulfur burning has been stabilized, the pH will drop to some lower level and also stabilize. The rate of caustic addition will then be adjusted to hold the pH of the spent caustic dumped to disposal at 7.5 to 8.0. There will then be a continuous withdrawal of spent caustic of 7.5 to 8.0 pH, and a continuous injection of fresh sodium hydroxide of any convenient strength and at the required rate necessary to control the pH of the spent caustic. The fresh NaOH may have to be diluted with fresh water in the separator to keep below 6 wt-% NaOH in the circulating water.
12. Prior to the addition of air, with the reactor inlet temperatures stable at 290°C (550°F) and the reactor outlet temperatures stable, measure and record the reactor outlet temperatures.

### Regeneration

1. Add makeup air as necessary to increase the final stage booster discharge pressure as required, so that when the air line block valves are opened, air will flow into the circulating regeneration gas stream. Adjust the air rate such that the oxygen content of the gas stream is 0.8 mol-%, or the delta T is 70°C (125°F), whichever comes first.
2. After the burning starts, adjust the air injection rate such that the oxygen content of the gas stream is 1.0 mol-% or the delta T is 70°C (125°F), whichever comes first.

3. Begin checking the circulating caustic at regular intervals and record the pH and periodically measure and record the total dissolved solids content. As the caustic becomes spent, the pH will drop from an initial of 14 to 12, down to 7, if allowed. When the pH reaches 7.5, the CO<sub>2</sub> content, as measured by Orsat analyses, should increase to about 3 mol-%. Do not let the pH drop below about 7.5. Adjust the caustic addition rate as necessary to hold the pH at 7.5 while continuously circulating caustic and continuously discarding spent caustic.
4. Orsats should be read and recorded at least every 30 minutes at the beginning. Finally when the system is stabilized, Orsats should be run about once per hour and recorded. The use of a portable oxygen analyzer for more frequent readings is highly recommended.
5. Continue as outlined until there is a breakthrough of oxygen at the reactor outlet. At that time, continue to hold conditions constant, including the oxygen level in the gas to the reactor, until the reactor outlet temperature drops back to the temperature measured in Step 12. Note that after oxygen breaks through the reactor outlet, it will be necessary to reduce the rate of air injection to maintain the same oxygen level at the reactor inlet.
6. Reduce or stop air addition, but maintain a minimum of 0.3 mol-% oxygen at all times, especially during the period when reactor temperatures are being increased. If the oxygen concentration drops to zero, it is possible that some catalyst reduction can occur due to the presence of CO<sub>2</sub>. Since this is undesirable, an Oxidizing atmosphere should be maintained at all times.
7. Raise the reactor inlet temperatures to 343°C (650°F). The caustic addition rate will also have to be reduced or stopped completely at this time, but continue caustic circulation. Maintain 343°C (650°F) reactor inlet temperatures and wait until the reactor outlet temperatures stabilize. Once these temperatures stabilize, record them.

8. Add air now for the second burn and adjust the air injection rate such that the oxygen content of the gas stream is 0.5-1.0 mol-% or the delta T is 70°C (125°F), whichever comes first. If caustic addition was cut back, then readjust the injection rate as necessary when burning is again resumed.
9. When oxygen breakthrough is observed again, continue to hold conditions constant, including the oxygen level in the gas to the reactor, until the reactor outlet drops back to the temperature measured in Step 7. A reduction in the air injection rate will be required.
10. Once again, do not block in air addition. Maintain an oxidizing atmosphere and do not allow the oxygen concentration to drop below 0.3 mol-%. Raise the reactor inlet temperature to 399°C (750°F).
11. Add air now for the third burn and adjust the air injection rate to hold about 0.5-1.0 mol-% oxygen in the gas stream to the reactor and observe to see if any delta T results. No delta T is expected; however, if one does occur, adjust the air injection rate as required to keep all catalyst bed temperatures below 426°C (800°F).
12. If a delta T is observed, reduce the air injection rate as before to maintain 0.5 mol-% oxygen in the gas stream to the reactor. Continue until the reactor outlet temperature drops back to the reactor inlet temperature or slightly below.
13. When the last burning wave is completed, maintain the 399°C (750°F) reactor inlet temperatures and increase the oxygen content of the gas to about 1.0 mol-%. Again, no delta T is expected; however, if one should occur, reduce the air injection rate as required to keep all catalyst bed temperatures below 426°C (800°F). Continue the 1.0 mol-% oxygen soak until the reactor outlet temperatures drop back to the reactor inlet temperatures or slightly below. Continue caustic circulation during this entire period.

14. If there is no austenitic steel in the reactor circuit after the last burning wave is completed, cut the fires and cool the catalyst to 150°C (300°F) or less. Shut down the caustic circulation system and drain completely when SO<sub>2</sub> is nil. Flush the system as often as necessary with fresh water to remove all traces of caustic. If the catalyst is to be removed and screened, cool down to 50°C (120°F) or less. After the first regeneration, it is advisable to dump and screen the catalyst. This will give an indication of the completeness of the regeneration plus an indication of the amount of fines and scale that can be expected on future regenerations.
15. IF AUSTENITIC STEELS ARE INVOLVED, MAINTAIN 399°C (750°F) REACTOR TEMPERATURES, MAINTAIN CAUSTIC CIRCULATION AND RECYCLE COMPRESSOR OPERATION; AND DEPRESSURE TO THE MINIMUM ALLOWABLE FOR COMPRESSOR OPERATION. THEN REPRESSURE WITH NITROGEN. CONDUCT THIS PROCEDURE AT LEAST THREE TIMES, OR AS NECESSARY TO REDUCE THE OXYGEN CONCENTRATION BY DILUTION, TO 100 PPM OR LESS.
16. SHUT DOWN THE CAUSTIC CIRCULATION SYSTEM AND DRAIN COMPLETELY WHEN SO<sub>2</sub> IS NIL. FLUSH THE SYSTEM AS OFTEN AS NECESSARY WITH FRESH WATER TO REMOVE ALL TRACES OF CAUSTIC. THEN SHUT DOWN THE WATER WASH TO THE SEPARATOR AND COMPLETELY DRAIN ALL WATER FROM THE SYSTEM. THE RECYCLE COMPRESSOR SHOULD STILL BE RUNNING AT THIS TIME AND THE CATALYST BED SHOULD STILL BE AT 399°C (750°F) AT THE INLET.
17. REDUCE THE REACTOR TEMPERATURES UNTIL THE REACTOR OUTLETS REACH 150°C (300°F). IF THE REACTORS ARE TO BE OPENED, REDUCE THE REACTOR TEMPERATURES UNTIL THE REACTOR OUTLET TEMPERATURES REACH 50°C (120°F).
18. SHUT DOWN THE RECYCLE COMPRESSOR, AND IF THE CHARGE HEATER COIL IS AUSTENITIC STEEL, MAINTAIN A 205°C (400°F) TEMPERATURE IN THE FIREBOX.

19. IF THE REACTORS ARE TO BE OPENED, DEPRESSURE THE UNIT TO 0.4 TO 0.7 KG/CM<sup>2</sup>G (5 TO 10 PSIG) AND HOLD THIS PRESSURE UNTIL THE UNIT IS OPENED.
20. DURING THIS PERIOD FOLLOWING REGENERATION OF THE CATALYST, AMMONIA SHOULD NOT BE USED IN ANY PURGING OPERATIONS CONDUCTED BEFORE THE CATALYST IS SULFIDED. EQUIPMENT CONTAINING AUSTENITIC STAINLESS STEEL SHOULD BE ISOLATED FROM THE REACTOR WITH BLINDS. WHEN ISOLATING THE EQUIPMENT, MAINTAIN JUST ENOUGH NITROGEN PURGE TO THE REACTOR TO PREVENT AIR FROM ENTERING WHILE BLINDING.

#### **Dump and Screen Catalyst**

1. THE CATALYST SHOULD BE DUMPED AND SCREENED AFTER EACH REGENERATION OR AT TWO YEAR INTERVALS. THE DUMPING SHOULD BE DONE UNDER AN ATMOSPHERE OF NITROGEN. AFTER DUMPING CATALYST, THE AUSTENITIC STAINLESS STEEL REACTOR WALLS AND INTERNALS SHOULD BE WASHED VERY THOROUGHLY WITH COPIOUS AMOUNTS OF 2-5 WT-% SODA ASH SOLUTION BEFORE ALLOWING AIR TO ENTER THE REACTORS. REFER TO "AUSTENITIC STAINLESS STEEL PROTECTION" INSTRUCTIONS IN THE APPENDIX FOR FURTHER DETAILS. NOTE THAT ANY CATALYST REMAINING IN THE BOTTOM OF THE REACTORS, OR HUNG UP ON TRAYS OR OTHER INTERNALS, SHOULD BE DISCARDED IF IT WAS CONTACTED WITH SODA ASH WASHING SOLUTION.

Note that any personnel entering a reactor must be aware of the hazards when entering a vessel containing an inert atmosphere. Rigidly follow all proper safety precautions and make sure all safety equipment is in good working order.

After the reactors have been properly washed with the soda ash solution, it can be aerated to allow workers to enter for cleaning, inspection or maintenance. Once again, strict safety rules should be adhered to. If a nickel-containing catalyst is being regenerated, proper consideration should be given to the possible presence of nickel carbonyl.

2. The regenerated catalyst should be treated in the same manner as fresh catalyst during the startup. Low temperature sulfiding with charge or charge with added sulfur compounds will be required.

**NOTE:** Although with the above procedure the regenerated catalyst might contain up to 0.5 wt-% combustibles, this small residual carbon does not in any way adversely affect recovery of catalytic activity.

### Emergency Procedures During Inert Gas Regeneration

1. If, at any time, the catalyst temperatures become excessive, block in the air injection control valve immediately.
2. If the recycle compressor fails or must be shut down, and if it is known that it cannot be immediately restarted, then do the following:
  - a. Block air injection immediately.
  - b. Shut down booster compressor, double block air inlet.
  - c. Cut out heater fires.
  - d. Shut down the caustic circulating pump immediately.

3. In the event of caustic circulation failure, block in the air injection immediately, but keep the system hot and continue gas circulation.
4. In general:

Whatever the emergency, take steps to prevent condensation of water in any austenitic system while oxygen is present. Also, avoid pumping caustic into the catalyst beds.

#### **E. DESCALING OF HYDROTREATING PROCESS HEATER TUBES (NOT FOR AUSTENITIC STEEL)**

The procedure employed for descaling hydrotreating heater tubes is a combination of two separate procedures; namely, (1) burning or conversion, and (2) acidizing. Burning is employed to convert iron disulfide ( $\text{FeS}_2$ ) to iron sulfide ( $\text{FeS}$ ) and sulfur dioxide ( $\text{SO}_2$ ), and acidizing to dissolve and remove the iron sulfide.

##### **1. Scale Conversion by Burning**

The burning portion of the procedure is a modification of the method used to remove coke from thermal cracking tubes. Whereas, coke is readily combustible at elevated temperatures in the presence of air (oxygen) and therefore requires the use of steam as a diluent to control the burning rate, the dense hard scale in hydrotreating heater tubes is essentially iron disulfide and requires air alone, and even higher temperatures, to accomplish its conversion to iron sulfide and sulfur dioxide. Steam for this operation is employed primarily as a purging and flushing medium.

Piping and manifold connections for the introduction of steam and air are shown on Figure XIII-3.

Heat for converting the scale is produced by firing the heater, thus raising the temperature of the tube walls and of the scale.

A general description of the procedure in sequence of operation is as follows.

After shutting down and purging the unit of hydrocarbons, the inlet and outlet flanges of the coils are broken in order to connect up the air steam supply line and discharge manifolds.

A good flow of steam (valve wide open with 7-11 kg/cm<sup>2</sup>g (100-150 psig) instrument board gauge pressure) is then introduced and the burners lit. Furnace temperatures, as determined by the thermocouples in the hip sections are brought up to 730°C (1350°F) at a rate of 220°C (400°F) per hour.

During this period of purging with steam, the outlet pipe quench water will probably appear cloudy. As soon as it clears up, the steam can be cut off while simultaneously and gradually introducing air for conversion. With 2.8-3.5 kg/cm<sup>2</sup>g (40-50 psig) air pressure on the instrument board gauge, the valve in the air supply line should be wide open.

It is desired that conversion of the scale in the bottom wall tubes (both sides simultaneously) be started first and then to progress upward through the hip and convection tubes (although periodically reversing the flow of air, to further promote conversion in the convection coil). To accomplish this, the burners should be initially adjusted to give a short flame for relatively high heat input rates to the lower wall tubes. At this time, the air injection rate will probably have to be reduced to attain sufficiently high scale temperature for ignition. As conversion takes place, a close observation should be maintained to see that the tube wall temperatures do not exceed 760°C (1400°F), a dull cherry red.

Evidence of scale conversion can be determined by noting the exit gases, which will have a brownish color, or the quench water, which will be cloudy. The exit gases will contain SO<sub>2</sub>, an extremely toxic gas, the inhalation of which should be carefully avoided.

As burning proceeds, or rather, in order that it may proceed, and progress through the upper wall tubes and hip tubes, burner adjustments will probably have to be made in order to produce a longer luminous flame by increasing the amount of

secondary air and backing off the primary air. With respect to the convection coil, it may be necessary to increase the amount of excess air to obtain satisfactory tube wall and scale temperatures. Inasmuch as these tubes cannot be observed easily, much will have to be left to the judgment of the supervisor of the descaling operation.

It is best not to attempt complete conversion of the scale in the entire coil in one continuous flow of air through the tubes. Cycles of air and steam purging should be employed. After the initial conversion has proceeded for fifteen minutes, the air should be shut off and steam for purging introduced. As soon as outlet water (with reddish-brown tinge) clears up, the purging steam flow should be reversed, and flow continued until the outlet water again is clear. During this particular period of purging when steam is introduced to the convection section, the flow through the two hip and wall sections should be alternately pinched down so as to make certain that a good purging effect has been obtained through each of the individual coils.

Air should then again be introduced at the charge inlet. Burner adjustments as referred to in the foregoing may have to be made and the air flow closely controlled to initiate conversion. Too high a rate of flow of air may have too much cooling effect and thus prevent conversion of the scale. The outlets should be alternately pinched down to ensure adequate flow through each coil and to make certain that there is no blockage. Flow should be continued in this direction for fifteen minutes followed by steam purging until exit water clears up.

Air flow is then started again to the outlet connections. From here on, proceed as previously described but with an increase of the airflow periods by increments of fifteen minutes until they reach one hour. Continue converting and purging with one hour burning periods until the outlet streams give evidence that the reaction has been completed. Evidence of this regard, as mentioned before, will be the color of the outlet water flush and absence of  $\text{SO}_2$  in the gas stream.

After the heater has cooled down to normal atmospheric temperatures, the tubes should be given a downward flow final blast of steam to remove any remaining loose material in them. The coils on each side of the heater should be given a purging separately in order to obtain maximum benefit.

A word of caution when the temperature is near 650°C (1200°F), keep a close watch on the color of the tubes. Observe to determine when the tubes just turn red, then read the temperature. Hold this temperature and regulate air flow up and down as needed to stay in a safe color range of the tube metal.

If it appears that the tubes are getting too red and are exceeding a safe temperature, lower the air pressure some. This will slow down the burning rate. Using firebox temperatures is not always a true temperature indication, so watch the tubes and use some judgment. When in doubt, reduce or stop air and put in steam to lower the temperature a little. Then in a few minutes, start air in again and bring up temperatures as needed. Length of time required to burn out a heater depends upon thickness of scale. The average time should be from 8 to 12 hours. After the heater has cooled down, you will be ready to acidize the coils.

Example of temperatures while burning:

|         |                  |
|---------|------------------|
| Firebox | 750°C (1380°F)   |
| Stack   | 525°C ( 977°F)   |
| Outlet  | 650°C (1200°F)   |
| Tubes   | Light cherry red |

## 2. Scale Removal By Acidizing

It is necessary to acidize naphtha hydrotreating heater tubes after descaling by the burning method. Acidizing will remove the iron sulfide from the tubes. The frequency of this descaling operation depends upon the type of charge stock.

Heaters for all units should be piped with a manifold so flow can be reversed during the acidizing and flushing operation.

## Acid

Concentrated hydrochloric acid should contain an inhibitor, a detergent and an emulsifier. There are several brand names of satisfactory inhibitors. The inhibitor must be good for use with HCl in dissolving iron sulfide scale and hydrogen sulfide scale. Hydrogen sulfide is very hard on inhibitors. There are other prepared solutions for acidizing prepared by chemical companies for use by the refiners.

When acidizing, the maximum temperature allowed is set by the inhibitor. The temperature should never be allowed to go above the recommended temperature given by the manufacturer of the inhibitor. This temperature will normally be from 71-82°C (160-180°F).

A 68 m<sup>3</sup>/hr (300 gallon per minute) pump or larger should be used to handle the acid. This pump should be built for handling acid. The pump needs to be large enough so that it will produce sufficient velocity to carry away the suspended sludge. Refer to Figure XIII-4 for a typical acidization system piping layout.

If a commercial acidizer has the contract, he will most likely furnish the fresh acid storage and acid mix tanks. The customer may have to furnish an acid circulating tank to vent off the H<sub>2</sub>S and let the acid sludge and scale settle out. The circulating tank should be about 1500 U.S. gallon size and should contain a vapor disengaging space behind a perforated baffle so that the H<sub>2</sub>S can readily release itself from the circulating acid solution and escape through the vent system.

Poisonous H<sub>2</sub>S gas generated during the acidizing must be vented to the refinery flare or to some sort of sour gas disposal system.

Acid concentration should start at about 18 - 20 wt%. When it drops to 8 - 10 wt%, it should either be dumped and new acid brought into the system, or it should be built up in strength. It is recommended to dump the acid if it is very dirty. Normally, maintaining acid strength needs only one addition. The second drop will be slow and when it holds steady for one hour, the acidizing is normally complete (refer to Figure XIII-5). Neutralizing and flushing can then be started.

Violent reactions may take place when acidizing is first started. It will be necessary to shut down the pump until reaction settles down. Also, shut down the heating coil during this time. Any time circulation stops, the heat must be stopped.

The iron content of the acid should be monitored. If iron content gets too high, dump out acid and put new acid and inhibitor into circulation. When the iron content is high, 0.4% by weight, this is an indication the inhibitor is suspected in the solution. Water wash the tubes before and after neutralizing with soda ash and detergent to remove all traces of acid and neutralizers.

## **F. PROTECTION OF AUSTENITIC STAINLESS STEEL**

### **1. Introduction**

Since corrosion cracking of austenitic stainless steel can lead to failure of the equipment involved, it is of the utmost importance that this equipment be properly protected to prevent corrosive environments from occurring. Therefore, all operating personnel, and especially the supervisory personnel, must be familiar with the locations of piping and equipment fabricated from austenitic stainless steel. They should also recognize the need for special handling of these sections of the unit during startup, shutdown, flushing, cleaning, maintenance and inspection, and should be thoroughly familiar with the procedures to be used for the proper protection of the equipment.

It is recognized that corrosion cracking of austenitic stainless steel is much less likely to occur in hydrodealkylation units than in hydrocracking and hydrodesulfurization units, because of the relatively low sulfur concentrations involved, combined with the wide use of the stabilized grades of austenitic stainless steel. Nevertheless sufficient danger of corrosion cracking exists in hydrodealkylation units to warrant due consideration for adequate protection procedures.

A number of both general and detailed instructions are contained in this discussion; however, all possibilities cannot be covered. Therefore, if situations arise which are not covered in this discussion, the refiner is advised to discuss the matter with UOP before proceeding.

## **2. General**

### **a. Austenitic Stainless Steel**

Austenitic stainless steels are those of the "300 series," the compositions of which are nominally 18% chromium and 8% nickel. The most common types used in the petroleum industry are Types 304, 316, 321 and 347. Because of their inherent high temperature strength properties and high corrosion resistance, they are particularly suitable for use in hydrocracking, hydrodesulfurization, and hydrodealkylation units in areas of moderate and high temperature, and where substantial resistance to hydrogen sulfide corrosion is required, such as in heater tubes, reactors, reactor effluent exchangers and piping. Types 321 and 347 are stabilized to minimize intergranular carbide precipitation and are preferred because they are more resistant to the intergranular corrosion cracking caused by polythionic acid attack, which can occur particularly during downtime periods when exposed to air and moisture. Since these stabilized grades are not completely immune to intergranular corrosion cracking, special handling procedures are recommended for the protection of these materials as well as the unstabilized grades.

### **b. Chloride Attack**

The presence of halides (chlorides are usually the most serious offenders) along with an aqueous phase and tensile stresses can result in stress corrosion cracking of austenitic stainless steels.

This type of cracking is predominantly transgranular and is somewhat dependent on time, temperature and chloride concentration. Therefore, precautions should be taken to minimize the amount of chloride in the process material which will come in contact with austenitic stainless steel equipment. Under normal shutdown period conditions, chloride cracking is not likely to be a problem as long as chlorides are not allowed to accumulate and concentrate in hot equipment, and as long as precautions are taken to limit the chloride content to low levels in any flushing, purging or neutralizing agents used in the system.

#### **c. Polythionic Acid Attack**

Once a unit has been placed on stream, even if the sulfur content of the feed stock is low, all items made of austenitic stainless steel should be considered to contain a layer of iron sulfide scale. Even though these layers of scale in many cases may be very thin, they represent a potential hazard to the underlying steel. The action of water and oxygen on this sulfide scale forms weak sulfurous type acids, commonly referred to as polythionic acids, which can attack austenitic stainless steels and cause intergranular corrosion and cracking. These stainless steels are vulnerable to this type of corrosion, particularly in areas of residual tensile stresses and in areas where intergranular carbides may exist, such as the heat-affected zones adjacent to welds. Therefore, special precautions should be taken to protect austenitic stainless steel from this corrosive environment.

#### **d. Protection Against Polythionic Acid Attack**

Protection against polythionic acid attack can be accomplished by preventing the corrosive environment from forming or by providing an agent which will neutralize any corrosive acids as they are formed:

### **(1) Preventing the Formation of Polythionic Acids**

Since these acids are formed by the action of water and oxygen with hydrogen sulfide or sulfide scale, elimination of either liquid phase water or oxygen will prevent these acids from being formed. Since there will usually be an equilibrium amount of water vapor present during the normal operation of a unit, during shutdown periods this water vapor can be prevented from condensing by maintaining the temperature of the austenitic stainless steel equipment above the dew point of water.

Under normal operations (other than a startup immediately following a catalyst regeneration, where there may be significant amounts of oxygen present before purging), there should be essentially no oxygen present in the system. The only other time any significant amount of oxygen might enter the system would be during a shutdown period when the system is depressured and the equipment is opened and exposed to air. Under these conditions a suitable purge of nitrogen should be established through the equipment involved to prevent any air from entering the system, and maintained until the system is again closed. If possible, the equipment should be blinded or blanked-off during this period and kept under a slight positive pressure of nitrogen.

### **(2) Neutralization**

Whenever austenitic stainless steel cannot be adequately protected by maintaining temperatures above the dew point of water or by an adequate nitrogen purge, a protective neutralizing environment should be established in this equipment prior to exposure to air. An effective neutralizing environment can be provided by purging with and maintaining an ammoniated nitrogen blanket, or by washing with a dilute soda ash solution.

### 3. Purging and Neutralizing

#### a. Purging Nitrogen

Nitrogen used for the purging and protection of austenitic stainless steels should be dry and the oxygen content should be limited to a maximum of 100 mol-ppm. The oxygen content of the nitrogen used should be specified by the supplier, since the analysis for oxygen in this low concentration range requires elaborate analytical equipment which may not normally be available in the refinery laboratory. If the only nitrogen available has an oxygen content in excess of 100 mol ppm, or if the oxygen content is unknown, then as a safeguard, ammoniated nitrogen should be used where possible. However, for this case, catalyst safety considerations might be necessary.

#### b. Ammoniated Nitrogen

To prepare ammoniated nitrogen for use in purging or blanketing an austenitic stainless steel system, sufficient ammonia is added to the nitrogen to provide a minimum concentration of 5000 mol ppm of ammonia. Whenever ammonia is added to the reactor system, the ammonia content of the recycle gas should be checked frequently. One convenient method of adding ammonia to the system, especially when the system is at high pressure, is to use a high pressure "blow case." With this type of arrangement, liquid ammonia is pressured into the blow case at low pressure from the ammonia cylinder, and then the blow case is isolated. High pressure gas from the discharge of the recycle gas compressor is then used to pressure up the blow case and to force the ammonia into the system at a location of lower pressure.

#### CAUTION

1. All personnel working in the unit should be familiar with the toxic nature of ammonia, and must follow proper safety precautions in working with the system when it contains ammonia. For example, workers opening flanges or manways in a system containing ammonia should be equipped with fresh air masks or other oxygen breathing equipment.

2. In order to preserve the activity of the catalyst in the reactors, ammonia is not to be passed over the catalyst when it is in its oxidized form, that is, whenever the catalyst is either fresh or freshly regenerated. When dealing with platinum type catalysts or HC-2 catalyst, ammonia should be excluded regardless of the state of the catalyst.
3. Brass and most other copper alloys are subject to corrosion attack from ammonia. Therefore, arrangements should be made to isolate this equipment from the system before admitting any ammonia.

**c. Soda Ash Solutions**

**(1) Composition**

Aqueous neutralizing solutions of soda ash ( $\text{Na}_2\text{CO}_3$ ) should be prepared in the range of 2% to 5% by weight. Preheating the water to about 38°C (100°F) will facilitate dissolving all the soda ash. In this range a sufficiently high level of alkalinity will be provided to affect neutralization of any reasonable amount of polythionic acids which may be formed. To avoid exposing the austenitic stainless steel equipment to a concentration of chlorides, the chloride content of the soda ash used to prepare the solution should be limited to a maximum of 500 wt-ppm, while the chloride content of the water should not exceed 50 wt-ppm. As added protection against chloride attack from the small amount of chloride present in the neutralizing solution, 0.5% by weight of sodium nitrate should be added to the soda ash solution. Sodium nitrate concentrations much above 0.5% should not be used, however, in order to avoid the possibility of stress corrosion cracking of carbon steel piping and equipment in the system.

## **(2) Neutralization Techniques**

Whenever a soda ash solution is used for neutralizing and protecting austenitic stainless steel, the piping or piece of equipment involved should be filled completely full with the solution.

The equipment should then be allowed to soak for a minimum of two hours before the soda ash solution is drained and the equipment is exposed to the air. If there are any pockets of unvented high areas in the equipment which cannot be reached by filling with the soda ash solution, then the solution should be vigorously circulated through the equipment to assure thorough contact of all austenitic stainless steel surfaces. This circulation should be continued for a minimum period of two hours before draining and exposing the equipment to air. For extremely large surfaces, such as reactor or large vessel walls and internals, where filling with soda ash solution is not only impractical but in some cases impossible because of foundation load limitations, it is recommended to wash the areas very thoroughly by means of a high pressure hose equipped with a spray nozzle. This type of washing will have to be done after the vessel has been opened to allow entry. Until the soda ash washing has been completed, the vessel should be maintained under a nitrogen blanket to prevent the entry of air.

## **(3) Protective Film**

In all cases of flushing or washing with soda ash solution, after the solution is drained from the equipment, the surfaces should be allowed to dry so that a film or fine deposit of soda ash remains on all surfaces for added protection against polythionic acid formation. Therefore, after draining the soda ash solution, do not rinse the system with steam or water.

For large accessible surfaces, such as vessels or reactor walls and internals, the excess dried soda ash can be removed just prior to

startup with a brush or dry cloths; do not use wet cloths and do not flush with steam or water. The small amount of soda ash remaining on the reactor surfaces, even if it were all deposited on the catalyst, would not have any significant effect on the activity of all but the platinum-type catalysts under consideration in this paper.

#### (4) **Precautions with Platinum Catalysts**

When platinum-type catalyst is used in the system, any soda ash film which may have been applied and is present should be removed just prior to reloading catalyst or just prior to connecting the austenitic stainless steel equipment back into the reactor circuit, as applicable, to prevent sodium contamination of the catalyst and loss of activity. To remove the soda ash film, first check to assure that the equipment involved is properly isolated from the reactors to prevent any contact of the catalyst by soda ash. Then thoroughly purge the system with nitrogen to remove all oxygen. While maintaining a nitrogen blanket on the system to prevent the entry of air, flush the system thoroughly with clean deaerated condensate to remove the soda ash deposits. Continue to maintain a nitrogen blanket on the system and drain the condensate. Then purge with nitrogen at maximum rate to remove any remaining pockets of water.

Where austenitic stainless steel heater tubes are involved, small fires should be lit as soon as the nitrogen purging is started and adjusted for the nominal 205°C (400°F) FIREBOX temperatures, to thoroughly dry the tubes. Make all necessary connections to prepare the unit for startup and normal operation while maintaining sufficient nitrogen purges from both sides of flanges, etc., to be closed so as to prevent any air from entering the system.

#### **4. Hydrotesting**

##### **a. New Austenitic Stainless Steel**

When conducting hydrostatic tests on new austenitic stainless steel equipment, the water used should have a chloride content not exceeding 50 ppm by weight, in order to reduce the possibility of concentrating chlorides in pockets or dead areas of the system. If chlorides were allowed to accumulate and concentrate (such as during subsequent heating operations) in such areas, stress corrosion cracking could result. If the only water available has a chloride content in excess of 50 ppm, then 0.5 wt-% of sodium nitrate should be used.

##### **b. Used Austenitic Stainless Steel**

Whenever a piece of equipment has been used for the processing of hydrocarbons in hydrocracking, hydrodesulfurization or hydrodealkylation service, it must be assumed that some degree of sulfide scale can be present. Therefore, even if this sulfide scale is so slight that it is difficult to detect, the possibility of polythionic acid formation with resulting intergranular corrosion cracking exists. Even if the equipment has been cleaned by mechanical means, burning or acidizing, it is difficult to assure that no traces of sulfide scale remain. Therefore, any hydrostatic testing (and any cleaning by hydroblasting) operations on used equipment should be conducted using the dilute soda ash solution specified for neutralizing this equipment. Here again, a protective film of dried soda ash should be allowed to remain on the surfaces of the equipment while it is exposed to the air.

## 5. Special Procedures

### a. Reactor Charge Heater Tubes

#### (1) Maintaining Small Fires

The austenitic stainless steel tubes in a reactor charge heater can best be protected by maintaining a balanced set of small fires (or pilots, as applicable) in the heater box at all times, even when there is no circulation of process material through the tubes. These small fires should be adjusted to keep the tubes warm and dry, to maintain the environment inside the tubes above the dew point of water. As a general rule about 205°C (400°F), as measured by thermocouples placed in the hip sections of the heater and directly below convection coils that may exist, will usually be sufficient for this purpose. The dew point, however, should be determined for each specific condition involved and the temperature should be adjusted as necessary. Only fuel gas firing should be used for this operation because of the difficulty in controlling and maintaining sufficiently small flames when burning fuel oil. It is important during these periods of heater operation that the heater firing be under strict control and that the firing pattern be properly established to provide good heat distribution. Sufficient thermocouples should be installed throughout the hip sections of the heater to provide a good measurement of the firebox temperatures and to monitor the distribution of heat in the firebox. These thermocouples should be located below any convection bank in the heater, and should be connected to a continuous recorder provided with high and low alarm points. The low alarm point should be set at about 150°C (300°F) and the high alarm point at about 232°C (450°F). CAUTION: Stack temperatures should never be used to control firebox temperatures.

## (2) To Shut Down Fires

If it should be necessary for any reason to shut down the fires in charge heater containing austenitic stainless steel tubes, then this should be done only when it is absolutely certain that the environment within the tubes does not contain both oxygen and water (or water vapor). As a result of the operation of the reactor effluent water wash facilities in units so equipped, there will normally be an equilibrium amount of water present in the entire reactor circuit both during normal operation and during or after a period of in situ catalyst regeneration. If the heater fires must be shut down during a period of normal operation, it is required only that no oxygen is present, which is usually the case during normal processing periods. As the heater tubes cool there will be small amounts of water condensing inside the tubes; however, this water should not be harmful in the absence of oxygen.

If the heater must be cooled down and it is suspected that trace quantities of oxygen might be present, then before cooling the heater the system should be depressured completely, but do not evacuate. Evacuation at this point is perhaps possible, but not recommended because it introduces the possibility of allowing air to enter the system. Continue to maintain the 205°C (400°F) FIREBOX temperatures while depressuring and purging. After the system has been depressured, pressure with nitrogen to any convenient pressure level. Repeat this depressuring/pressuring procedure as many times as required to reduce the oxygen concentration, by dilution, to as much below 100 mol ppm as is possible and reasonable. Then the fires can be shut down and the heater allowed to cool.

### **(3) Neutralization**

If neutralization is necessary, such as when a tube or tubes are cut out of the coil, or any time when exposure to air at temperatures below the dew point of water cannot be avoided, the tubes should be filled with soda ash solution and allowed to soak for a minimum of two hours. With vertical coils, where it is not possible to completely fill the unvented upper return bends, it is necessary instead to vigorously circulate the soda ash solution through the tubes for a minimum of two hours to assure contact of all surfaces. After draining the soda ash solution, do not flush with steam or water but instead allow a film of protective soda ash to remain in the tubes.

### **(4) Exterior Surfaces**

Whenever heater fires must be shut down and the tubes are allowed to cool, it is recommended that the exterior tube surfaces be protected, especially in heaters where fuel oil or high sulfur content fuel gas is employed. As a result of the sulfur in the fuel, a sulfide or sulfate scale can build up on the exterior tube surfaces. If moisture is allowed to condense on the tubes as the heater box is cooled, the action of oxygen and moisture on the scale can form polythionic acids which can attack the austenitic stainless steel tube surfaces and lead to intergranular stress corrosion cracking. There are two recommended procedures that can be followed to prevent this from occurring: First, it is possible to prevent any moisture from condensing on the tubes, and thus prevent the formation of polythionic acids, by purging the firebox with copious amounts of dry air. Normal instrument air is prepared by processing through a set of driers where the dew point is reduced to a sufficiently low level to prevent condensation from occurring at ambient conditions. This air can be effectively used to maintain a dry air blanket in the heater box both during cooling and throughout the entire period the fires are out. In order to minimize the consumption of instrument air, and to prevent moist air from entering the heater box, the stack damper, all burner air registers, and all doors and ports in the heater box should be kept closed.

Second, an alternate method of protecting the tubes from polythionic acid attack is to cover the exterior tube surfaces with a protective film of soda ash, which will act to neutralize any polythionic acids as they are formed. The neutralizing soda ash should be the same dilute solution recommended for general neutralization, and should be applied to the tube surfaces as soon as the heater box has cooled sufficiently to prevent vaporizing the soda ash solution, and preferably before any moisture has begun to condense out on the tube surfaces. A fairly efficient and effective method of applying the soda ash solution is to utilize a vat or tank with a small portable pump which can pump the solution through a hose fitted with a spray nozzle which will produce a fairly fine mist. NOTE: A low pressure spray is advisable as high pressure may erode the refractory. Small diameter pipe extensions can be fitted to the hose to allow reaching up to the tube areas at the top of the heater box. This type of spray equipment will minimize the soda ash consumption and provide a reasonable means to reach all tube surfaces which are exposed to the heater flames. Once the soda ash solution has been applied, it should be allowed to dry to form a protective film on the surfaces of the tubes; do not wash off this protective film.

If the exterior tube surfaces are heavily coated with an oxide or carbonaceous material, it should be removed by wire brushing or sandblasting. This cleaning, however, will also remove any protective soda ash film which may have been applied. In this case, the tube surfaces should be further protected by maintaining a dry air blanket in the heater box both during cooling and throughout the entire period the fires are out. In order to minimize the consumption of instrument air, and to prevent moist air from entering the heater box, the stack damper, all burner air registers, and all doors and ports in the heater box should be kept closed.

**b. Fractionator Heater Tubes**

Where the fractionator heater tubes are made of austenitic stainless steel and it is necessary to work on the fractionator section, and in particular to prepare for opening the fractionator column, the following steps should be taken.

- (1) Pump or pressure all the oil from the flash drum or fractionator feed drum into the fractionator and out to storage or slop. Fire the heater as necessary to maintain a FIREBOX temperature, as measured by thermocouples placed in the hip section of the heater, of about 205-315°C (400-600°F) to keep the tubes warm and dry, above the dew point of water, and to assure that the steam that will be used to purge the system will not condense in the tubes. Some judgement by operators and supervisors is necessary at this time.
- (2) Drain all condensate from the steam lines, then open the steam valves at the inlet of the fractionator heater wide open on all heater passes. Superheated steam is preferred for this purging operation. At the same time increase the heater firing as necessary to heat the steam above its saturation temperature to assure it will not condense in the tubes. A heater transfer temperature of about 315°C (600°F) is generally preferred, as long as the temperature limitations of the heater or the fractionator system are not exceeded.
- (3) After the system has been thoroughly purged with steam, stop the steam and immediately cut in a nitrogen purge through the coils and into the column to sweep out any remaining steam. The nitrogen flow will usually be less than the steam flow, and as a result a corresponding reduction in heater firing will be necessary.
- (4) If the heater is not to be entered, then the firebox should be reduced only to the point where a 205°C (400°F) FIREBOX temperature, as measured by the previously discussed hip section thermocouples, can be maintained. If the heater must be entered, cut the fires and

maintain a continuous nitrogen purge through the tubes. The exterior tube surfaces should be protected from polythionic acid attack whenever the heater fires are shut down, in accordance with the recommendations in the "Exterior Surfaces" section.

- (5) If the fractionator column is to be opened, install blinds at the column to isolate the heater coils, and maintain a positive pressure of nitrogen on the coils. If heater tubes are to be cut, or for some reason the insides of the tubes will be exposed to air, then the tubes should first be thoroughly soaked or flushed with soda ash solution, as discussed in the section on "Reactor Charge Heater Tubes."

#### **c. Heat Exchangers**

If lines leading to or from heat exchangers containing austenitic stainless steel are to be opened, blinds can be rapidly inserted to isolate the exchanger, while maintaining a nitrogen purge through the exchanger involved to prevent air from entering. A nitrogen blanket or continuous nitrogen purge should then be maintained in the exchanger during this maintenance period.

If shell and tube exchangers containing austenitic stainless steel are to be opened and inspected, or if the tube bundles are to be pulled, then before exposing this equipment to air, both shell and tube sides should be flooded with soda ash solution and allowed to soak for a minimum of two hours. If there are any pockets or high areas which cannot be reached with the soda ash solution, then the soda ash solution should be vigorously circulated through the exchanger for a minimum of two hours. Do not rinse with water, but instead allow a film of soda to remain on the surfaces.

If tube bundles of austenitic stainless steel are to be cleaned by hydroblasting, then soda ash solution should be used for this purpose instead of just water.

#### **d. Reactor Internals**

Any time a reactor is to be opened, maintain sufficient nitrogen purges to prevent the entry of air into any part of the system and isolate the charge heater coils and the reactor effluent system with blinds. A blanket of nitrogen should also be maintained in the reactor, especially if it contains unregenerated catalyst. A slight amount of air coming in contact with the reactor internals for relatively short periods of time is normally not considered to be harmful to the metal; however, precautions should be taken to prevent contact with water or moisture, especially in the presence of air. If any exposure to air has occurred, the air should be purged out with nitrogen as soon as possible.

When the reactor internals are to be exposed to air for a prolonged period of time, such as during a catalyst change, the reactor walls and internals should be washed very thoroughly as soon as possible with a high pressure hose, using copious amounts of soda ash solution. A portable pump and a vat of soda ash solution on skids is advisable for this operation. In order to do this washing properly, a workman equipped with a fresh air mask and following all other proper safety precautions, might have to enter the vessel to make sure all surfaces, including the underside of the top head, are thoroughly wetted. Be especially careful to thoroughly soak welded areas with particular emphasis on welds normally required to support heavy loads, such as those on support beams, grids and trays. When the reactors contain trays, which would make wetting all surfaces with soda ash solution difficult, a sufficient amount of soda ash solution should be sprayed around the top of the reactors, and allowed to rain down through the reactors to wet as much of the surfaces as possible in the areas below the top tray. Be sure to thoroughly soak and keep wetted any used catalyst remaining in the reactors. Then air can be drawn through the reactor so that personnel can enter. During this time, a small flow of soda ash solution to the reactor should be maintained, and as each tray manway is removed, the vessel area beneath that tray and the underside of the tray should be thoroughly washed with soda ash solution.

Whenever spent, unregenerated catalyst is unloaded from a reactor, some amounts of catalyst will inadvertently remain on the trays and in the bottom of the reactor. This catalyst must be kept wet to prevent ignition of sulfide scale when air is admitted, which is another reason for conducting a thorough washing operation with the soda ash solution.

After washing with soda ash solution, allow the surfaces to dry with a fine deposit of soda ash. Do not rinse this residue off with water. Later, just prior to reloading catalyst, wipe as much excess soda ash residue from the surfaces as possible with brushes or dry cloths; do not use water or wet cloths.

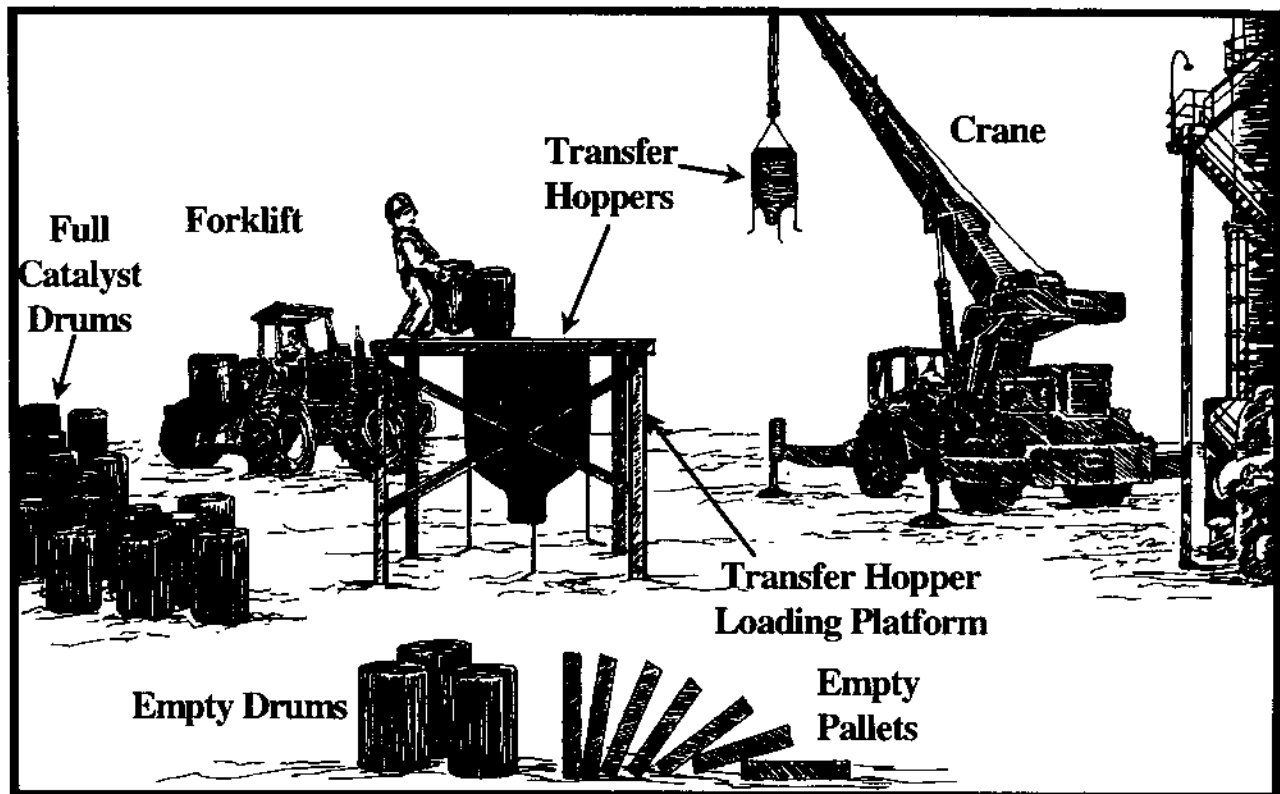
**e. Cooling Catalyst After Regeneration**

When it is necessary to reduce the temperature in an austenitic stainless steel charge heater coil below the dew point of water when oxygen is present, such as during the procedure of cooling the catalyst bed to a temperature which would allow entering the reactor following a catalyst regeneration, the oxygen must first be reduced to an acceptable level. Maintain the final reactor temperature used in the regeneration and continue gas circulation; and depressure to the minimum allowable for recycle compressor operation. Then pressure with nitrogen. Conduct compressor operation. Then pressure with nitrogen. Conduct this procedure at least three times, or as necessary to reduce oxygen concentration in the circulating gas by dilution to as much below 100 mol ppm as is possible and reasonable. Maintain reactor temperatures and gas circulation, after shutting down and draining the caustic and water systems, until the reactor system is dry and no more water collects in the separator. Then the system can be cooled to about 65°C (150°F) at the reactor outlet. At this point, shut down the recycle gas compressor but maintain heater fires sufficient to maintain about 205°C (400°F) FIREBOX temperatures throughout the shutdown period.

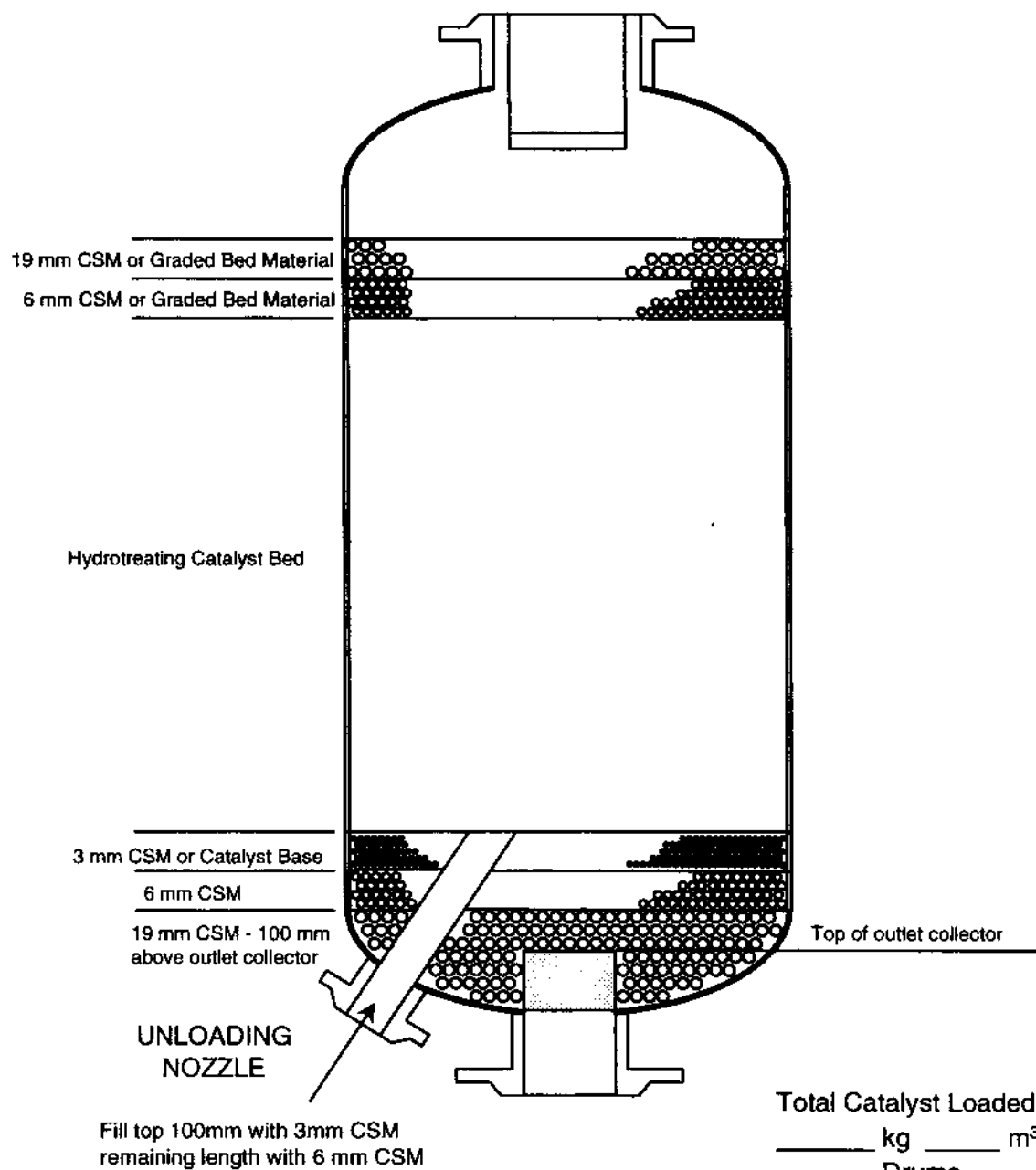
## 6. References

Additional information on the subject of protection of austenitic stainless steel can be found in the NACE Standard RP-01-70, entitled "Recommended Practice, Protection of Austenitic Stainless Steel in Refineries against Stress Corrosion Cracking by use of Neutralizing Solutions during Shut Down" and approved October 1970. Copies may be obtained from NACE Headquarters, 2400 West Loop South, Houston, Texas 77027.

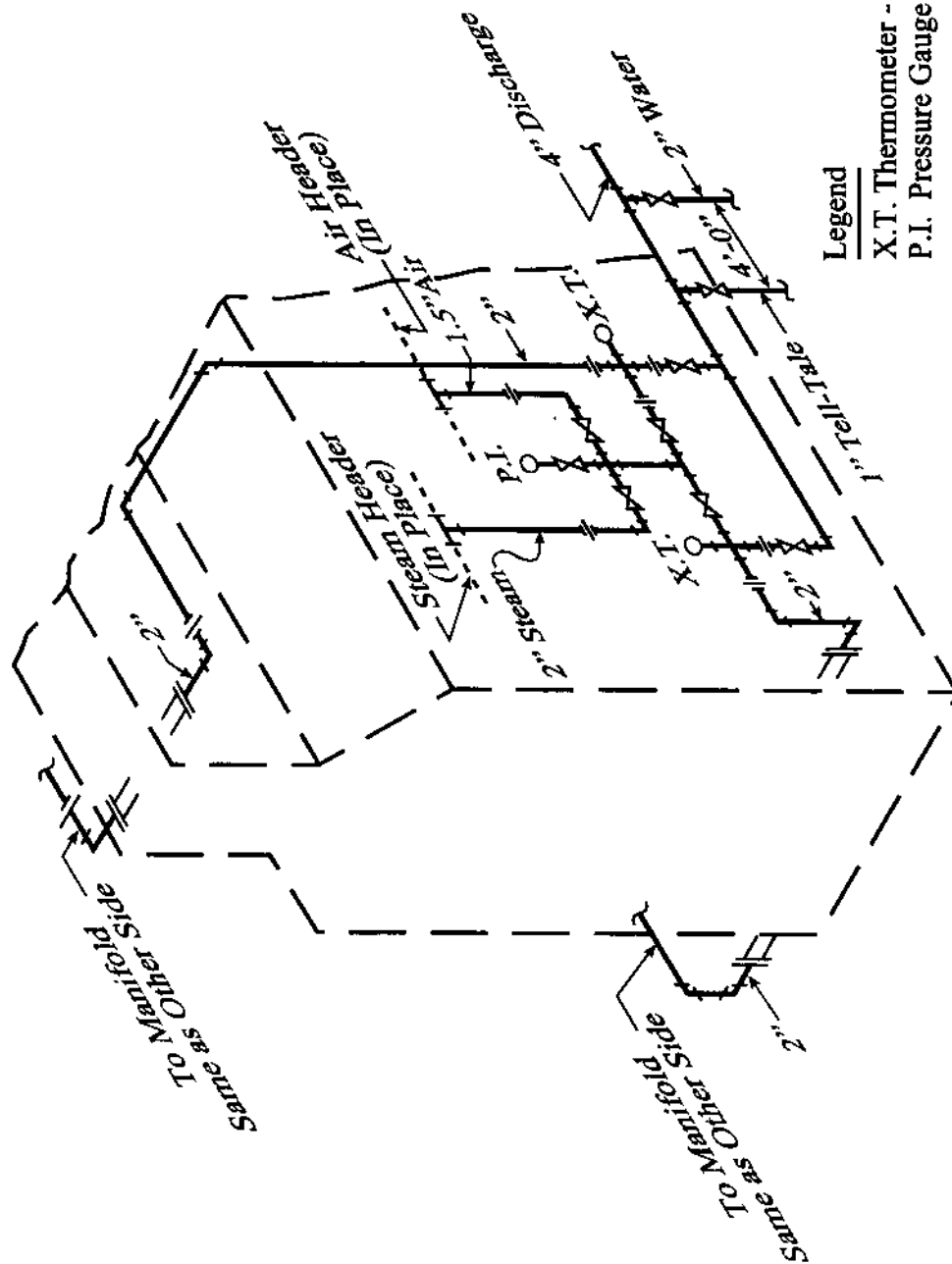
**Figure XIII-1  
TYPICAL LAYOUT FOR CATALYST LOADING**



**Figure XIII-2**  
**Typical Reactor Loading Diagram**



**Figure XIII-3**  
**Descaling Piping Arrangement**



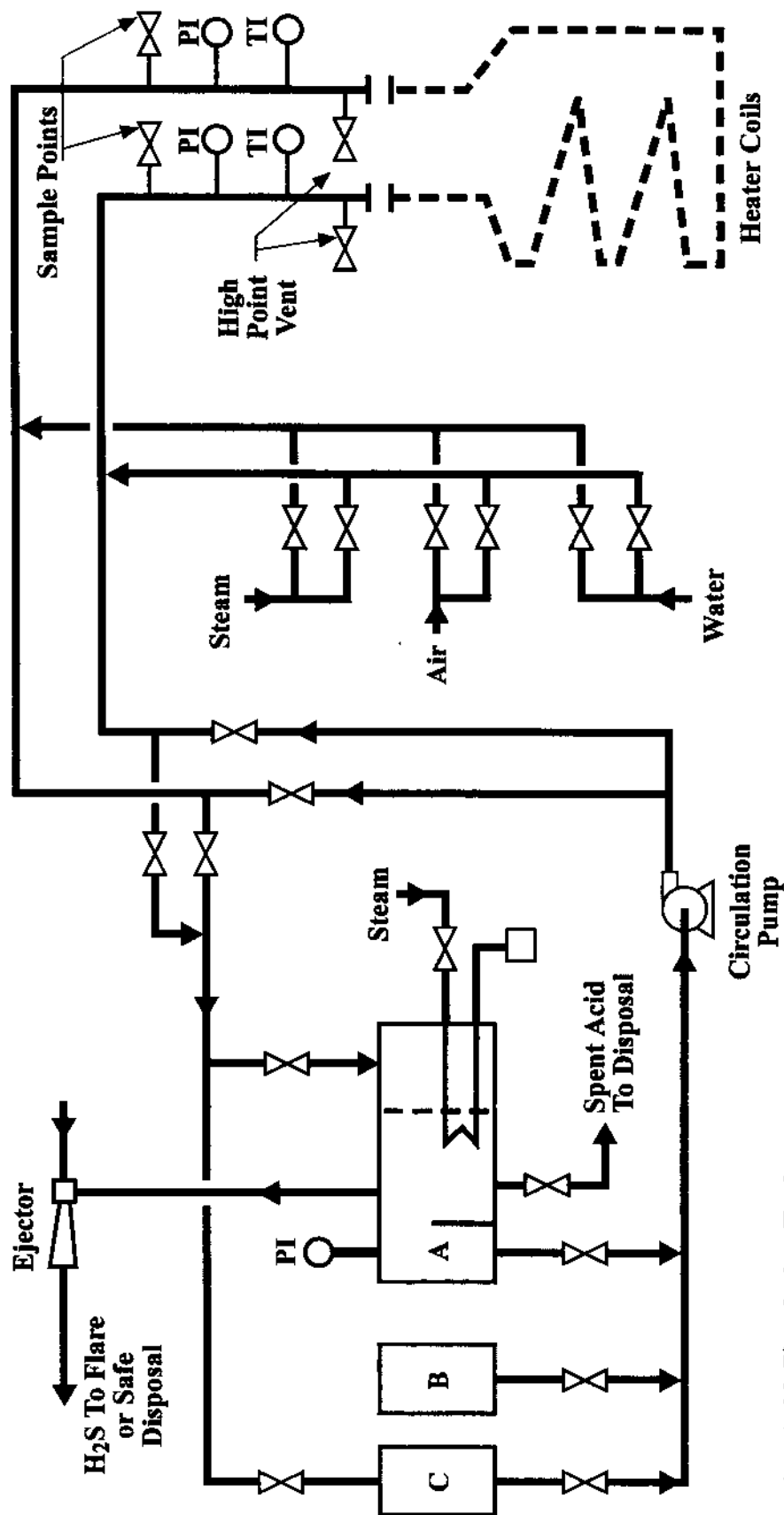
**Legend**

X.T. Thermometer - Max. 1500°F

P.I. Pressure Gauge

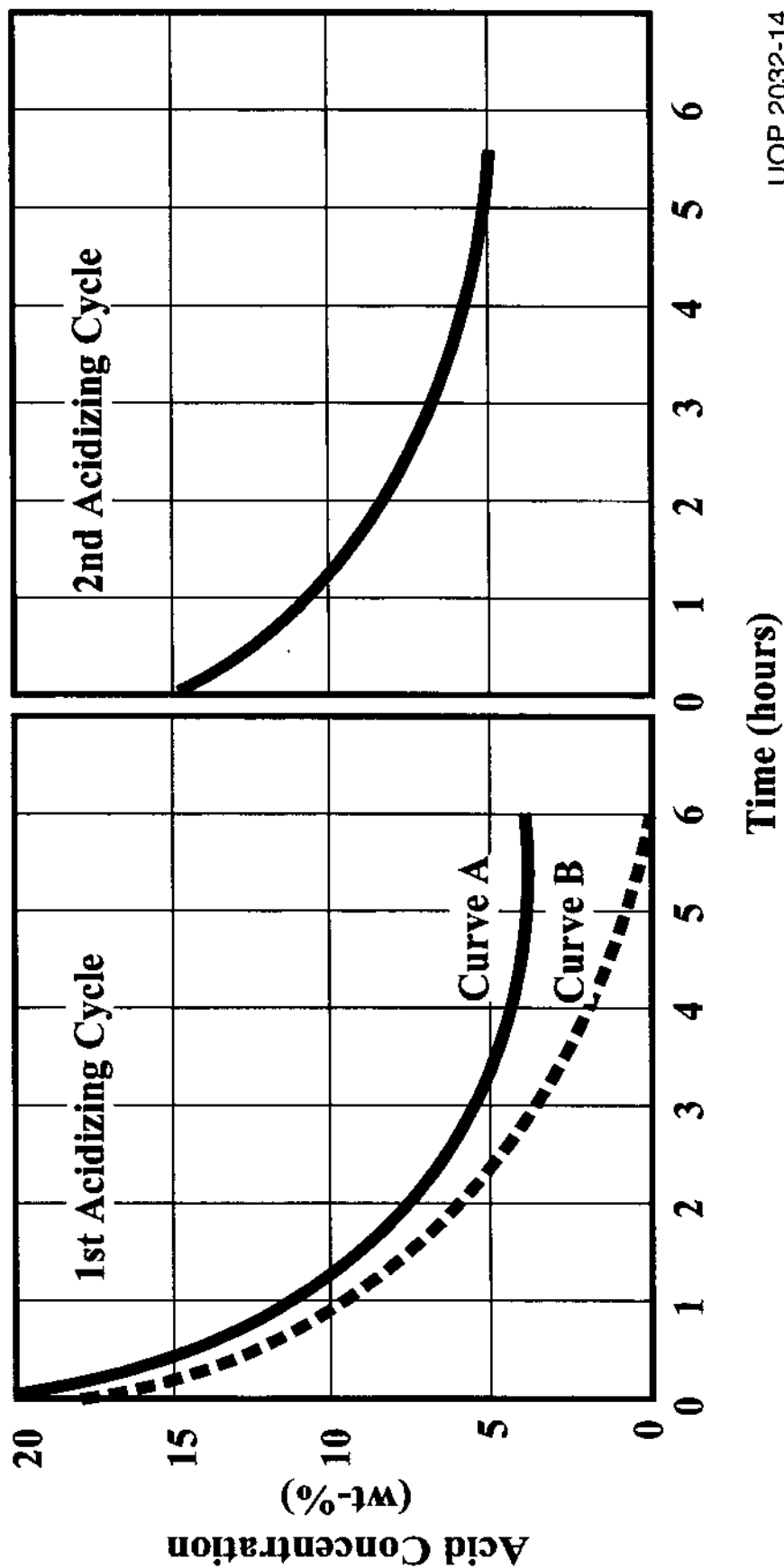
UOP 3055-18

# Figure XIII-4 Acidizing System Piping



UOP 2032-13  
UOP-3054-29

**Figure XIII-5**  
**Typical Acid Concentration Curves**



UOP 2032-14  
UOP-3054-30

## **XIV. SAFETY**

This chapter on safety includes the following sections:

- A. OSHA Hazard Communication Standard
- B. Hydrogen Sulfide Poisoning
- C. Nickel Carbonyl Formation
- D. Precautions for Entering Any Contaminated or Inert Atmosphere
- E. Preparations for Vessel Entry
- F. MSDS Sheets

The information and recommendations contained in this manual have been compiled from sources believed to be reliable and to represent the best opinion on the subject as of 1989. However, no warranty, guarantee or representation, expressed or implied, is made by UOP as to the correctness or sufficiency of this information or to the results to be obtained from the use thereof. Each refiner should determine the suitability of the following material for his purposes before adopting them.

Since the use of UOP products by others is beyond UOP control, no guarantee, expressed or implied, is made and no responsibility assumed for the use of this material or the results obtained therefrom. Moreover, the recommendations contained in this manual are not to be construed as a license to operate under, or a requirement to infringe, any existing patents, nor should they be confused with state, municipal or insurance requirements, or with national safety codes.

### **A. OSHA HAZARD COMMUNICATION STANDARD**

All references to environmental, occupational safety and material transport laws are based on U.S.A. federal, state and local laws which are applicable only within the U.S.A. and its territories. It cannot be assumed that all necessary warnings and precautionary measures are contained in this manual, or that any additional warning and or measures may not be required or desirable because of particular exceptional conditions or circumstances, or because of applicable federal, state or local law.

As of May 25, 1986, all U.S. employers covered under the Specific Industrial Classification (SIC) Codes 20-39 must be in compliance with the Occupational Safety and Health Standard, Subpart Z - Toxic and Hazardous Substances Hazard Communications, Section 1910.1200 of the Federal Regulations. This standard is commonly referred to as the "Right-to-Know Law."

The OSHA standard is a U.S. Federal regulation requiring chemical manufacturers, importers and distributors to evaluate the hazards of their chemical products and convey hazard information through labels and material safety data sheets to its employees and customers which fall within SIC Codes 20-39. The customers in turn must pass the hazard information on to its employees and contractors which come on the premises. In this context, UOP employees who are working in or visiting a refinery are considered contractors to the refiner.

It is the responsibility of all refiners in the United States to inform all contractors of the hazardous chemicals the contractor's employees may be exposed to while performing their work, and to provide any suggestions for appropriate protection measures. It is then the responsibility of UOP to provide the information to its employees about the hazardous chemicals to which they could be exposed by means of 1) a written hazard communications program, 2) training and information, 3) labels and other forms of warning, and 4) material safety data sheets.

## **1. Written Hazard Communications Program**

The OSHA standard requires that U.S. employers make available to their employees the company's written Hazard Communication Program. This document is intended to describe how the company will implement the OSHA standard. The program explains the company's labeling system, material safety data sheets (MSDS), and employee information and training. The latter includes a listing of hazardous chemicals known to be present in the work place, and methods the company will use to inform its employees and contractors of the hazardous chemicals.

## **2. Training and Information**

All UOP employees receive classroom training in compliance with the OSHA standard. This includes an overview of the standard, an explanation on how to interpret and use the information on a MSDS, the location and availability of UOP's file of MSDS's, labeling requirements and their meaning, and an introduction to toxicology.

UOP employees working in or visiting U.S. refiners are considered contractor employees of that refinery. The OSHA standard states that contractors performing work in these facilities are required to train their people before they enter the refinery. However, it is the responsibility of U.S. refiners to inform UOP of the specific hazardous chemicals to which UOP's employees may be exposed. UOP complies with the OSHA standard by making available to its employees this list of hazardous chemicals and by appraising them of the hazards they will be exposed, relevant symptoms and appropriate emergency treatment and proper conditions and precautions of safe use or exposure.

## **3. Labels and Other Forms of Warning**

The OSHA standard states that all portable containers of hazardous chemicals must have a large, readable label or tag which has on it:

- a. The name and address of the manufacturer
- b. The name of the chemical
- c. A numerical hazard warning or other appropriate warnings supplied by the manufacturer

For the latter, the National Fire Protection Association (NFPA) Diamond is commonly used. An explanation of the NFPA Diamond may be found in Figure XIV-1 and Table XIV-1.

Labels can also be color coded according to the following:

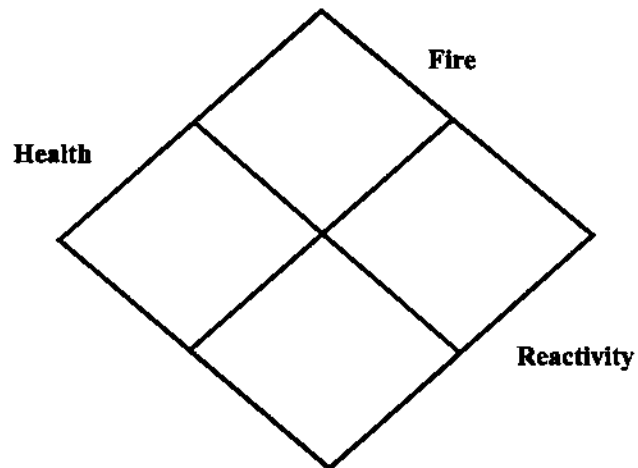
|        |                                             |
|--------|---------------------------------------------|
| Orange | Carcinogen Hazard (i.e.: Benzene)           |
| Red    | Chemical Burn Hazard (i.e.: Acids, Bases)   |
| Yellow | Toxic Vapor Hazard (i.e.: H <sub>2</sub> S) |
| White  | All Others                                  |

Contractor employees must label all containers of hazardous materials which they bring into the refinery. This applies to UOP employees who are visiting or working in refineries.

#### **4. Material Safety Data Sheet (MSDS)**

The MSDS requirement falls primarily on chemical manufacturer, importers and distributors. The OSHA standard requires them to develop and provide a MSDS for each hazardous chemical they produce or handle. These manufacturers, importers and distributors are required to provide the MSDS to the purchasers of the hazardous chemical. MSDS sheets for the following UOP hydrotreating catalysts are included at the end of this section: S-120, N-108, N-204 and HC-K. For MSDS sheets on other UOP hydrotreating catalysts, please contact UOP.

Figure XIV-1

***NFPA 704 Diamond***

**CAUTION: CONTAINS HAZARDOUS CHEMICAL**  
**Transportation Emergencies .... Call**  
**Health Emergencies .....**

**HAZARD RANKING**

**0 = Least**  
**1 = Slight**  
**2 = Moderate**  
**3 = High**  
**4 = Extreme**

PLT-R00-99

TABLE XIV-1

**National Fire Protection Association  
Identification of Color Coding**

| <b><u>Color Blue:</u></b>    | <b><u>Type of Possible Injury</u></b>                                                                                                                                          |
|------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Signal 4:                    | Materials which on very short exposure could cause death or major residual injury even though prompt medical treatment was given.                                              |
| Signal 3:                    | Materials which on short exposure could cause serious temporary or residual injury even though prompt medical treatment was given.                                             |
| Signal 2:                    | Materials which on intense or continued exposure could cause temporary incapacitation or possible residual injury unless prompt medical treatment is given.                    |
| Signal 1:                    | Materials which on exposure would cause irritation but only minor residual injury even if no treatment is given.                                                               |
| Signal 0:                    | Materials which on exposure under fire conditions would offer no hazard beyond those of ordinary combustible materials.                                                        |
| <br><b><u>Color Red:</u></b> | <br><b><u>Susceptibility of Materials Burning</u></b>                                                                                                                          |
| Signal 4:                    | Materials which will rapidly or completely vaporize at atmospheric pressure and normal ambient temperature, or which are readily dispersed in air and which will burn readily. |
| Signal 3:                    | Liquid and solids that can be ignited under almost all ambient temperature conditions.                                                                                         |
| Signal 2:                    | Materials that must be moderately heated or exposed to relatively high ambient temperatures before ignition can occur.                                                         |
| Signal 1:                    | Materials that must be preheated before ignition can occur.                                                                                                                    |
| Signal 0:                    | Materials that will not burn.                                                                                                                                                  |

**TABLE XIV-1**  
**(cont'd)**

**National Fire Protection Association**  
**Identification of Color Coding**

**Color Yellow:**      **Susceptibility of Release of Energy**

- Signal 4:      Materials which in themselves are readily capable of detonation or of explosive decomposition or reaction at normal temperature and pressure.
- Signal 3:      Materials which in themselves are capable of detonation or explosive reaction but require a strong initiating source or which must be heated under confinement before initiation or which react explosively with water.
- Signal 2:      Materials which in themselves are normally unstable and readily undergo violent chemical change but do not detonate. Also materials which may react violently with water or which may form potentially explosive mixtures with water.
- Signal 1:      Materials which in themselves are normally stable, but which can become unstable at elevated temperatures and pressures or which may react with water with some release of energy but not violently.
- Signal 0:      Materials which in themselves are normally stable, even under fire exposure conditions and which are not reactive with water.

Although the format of the MSDS can vary, they should all include the following information:

- Chemical and common name
- Ingredient information
- Physical and chemical characteristics
- Physical hazards - Potential for reactivity, fire and/or explosion
- Health hazards
- Symptoms of exposure
- Primary route of likely entry into the body upon exposure
- OSHA permissible exposure levels
- Precaution for use
- Waste disposal
- Protective measures and equipment, including during spills and maintenance
- Emergency and first-aid procedures
- Date of MSDS preparation and last revision
- Emergency contact of manufacturer or distributor

The OSHA standard requires that the manufacturer or distributor provide quick and easy access to all MSDS's applicable to their work place.

## **5. MSDS Sheets for UOP Naphtha Hydrotreating Process**

Included in Section F are typical MSDS for some chemicals normally used in the NHT processes. These are not meant to replace those which must be supplied by the U.S. refiners nor is this meant to suggest that this is a complete list of the hazardous chemicals which can be found in and around the unit. Some of the information on these typical MSDS may be out of date.

## B. HYDROGEN SULFIDE POISONING

Hydrogen sulfide is both an irritant and an extremely poisonous gas. Breathing even low concentrations of hydrogen sulfide ( $H_2S$ ) gas can cause poisoning. Many natural and refinery gases contain more than 0.10 mol-%  $H_2S$ . The current OSHA permissible exposure limits are 20 mol-ppm ceiling concentration and 50 mol-ppm peak concentration for a maximum 10 minute exposure.

The naphtha hydrotreating recycle gas and high pressure stripper gas can contain from 0.5 to 5 mol-%  $H_2S$ , while the low pressure stripper gases can contain from 10 to 50 mol-%  $H_2S$ . These gases must NEVER be inhaled. One full breath of high concentration hydrogen sulfide gas will cause unconsciousness and could cause death, particularly if the victim falls and remains in the presence of the  $H_2S$ .

The operation of any unit processing gases containing  $H_2S$  remains safe, provided ordinary precautions are taken and the poisonous nature of  $H_2S$  is recognized and understood. No work should be undertaken on the unit where there is danger of breathing  $H_2S$ , and one should never enter or remain in an area containing it without wearing a suitable fresh air mask.

There are two general forms of  $H_2S$  poisoning - acute and subacute.

### 1. Acute Hydrogen Sulfide Poisoning

Breathing air or gas containing more than 500 mol-ppm  $H_2S$  can cause acute poisoning and possibly be fatal.

### 2. Symptoms of Acute Hydrogen Sulfide Poisoning

The symptoms of acute  $H_2S$  poisoning are muscular spasms, irregular breathing, lowered pulse, odor to the breath and nausea. Loss of consciousness and suspension of respiration quickly follow.

Even after the victim recovers, there is still the risk of edema (excess accumulation of fluid) of the lungs which may cause severe illness or death in 8 to 48 hours.

### **3. First Aid Treatment of Acute Hydrogen Sulfide Poisoning**

Move the victim at once to fresh air. If breathing has not stopped, keep the victim in fresh air and keep him quiet. If possible, put him to bed. Secure a physician and keep the patient quiet and under close observation for about 48 hours for possible edema of the lungs.

In cases where the victim has become unconscious and breathing has stopped, artificial respiration must be started at once. If a Pulmotor or other mechanical equipment is available, it may be used by a trained person; if not, artificial respiration by mouth-to-mouth resuscitation must be started as soon as possible. Speed in beginning the artificial respiration is essential. Do not give up. Men have been revived after more than four hours of artificial respiration.

If other persons are present, send one of them for a physician. Others should rub the patient's arms and legs and apply hot water bottles, blankets or other sources of warmth to keep him warm.

After the patient is revived, he should be kept quiet and warm, and remain under observation for 48 hours for the appearance of edema of the lungs.

### **4. Subacute Hydrogen Sulfide Poisoning**

Breathing air or gas containing  $H_2S$  anywhere between 10 to 500 mol-ppm for an hour or more may cause subacute or chronic hydrogen sulfide poisoning.

### **5. Symptoms of Subacute Poisoning**

The symptoms of subacute  $H_2S$  poisoning are headache, inflammation of the eyes and throat, dizziness, indigestion, excessive saliva, and weariness. These can be the result of continued exposure to  $H_2S$  in low concentrations. Edema of the lungs may also occur.

## 6. Treatment of Subacute Poisoning

Keep the patient in the dark to reduce eyestrain and have a physician treat the inflamed eyes and throat. Watch for possible edema.

Where subacute poisoning has been suspected, the atmosphere should be checked repeatedly for the presence of  $H_2S$  by such methods as testing by odor, with moist lead acetate paper, and by Tutweiler  $H_2S$  determination to make sure that the condition does not continue.

## 7. Prevention of Hydrogen Sulfide Poisoning

The best method for prevention of  $H_2S$  poisoning is to stay out of areas known or suspected to contain it. **The sense of smell is not an infallible guide as to the presence of  $H_2S$ ,** for although the compound has a distinct and unpleasant odor (rotten eggs), it will frequently paralyze the olfactory nerves to the extent that the victim does not realize that he is breathing it. This is particularly true of higher concentrations of the gas.

Fresh air masks or gas masks suitable for use with hydrogen sulfide must be used in all work where exposure is likely to occur. Such masks must be checked frequently to make sure that they are not exhausted. People who must work on or in equipment containing appreciable concentrations of  $H_2S$ , must wear fresh air masks and should work in pairs so that one may effect a rescue or call for help should the other be overcome. Table XIV-2 shows the respiratory requirements for hydrogen sulfide atmospheres.

As mentioned above, the atmosphere in which people work should be checked from time to time for appreciable concentrations of  $H_2S$ .

**REMEMBER - JUST BECAUSE YOUR NOSE SAYS IT'S NOT THERE, DOESN'T MEAN THAT IT IS NOT.**

TABLE XIV-2

**RESPIRATORY PROTECTION FOR HYDROGEN SULFIDE**

| <b>Condition</b>                                                     | <b>Minimum Required Protection*<br/>Required Above 10 ppm</b>                                                                                                                                                                                                                                                                                                                                                               |
|----------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Gas Concentration                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 300 ppm or less                                                      | Any supplied-air respirator with a full facepiece, helmet, or hood.<br><br>Any self-contained breathing apparatus with a full facepiece.                                                                                                                                                                                                                                                                                    |
| Greater than 300 ppm or entry and escape from unknown concentrations | Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.<br><br>A combination respirator which includes a Type C supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure or continuous-flow mode and an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive pressure mode. |
| Fire Fighting                                                        | Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.                                                                                                                                                                                                                                                                                                       |
| Escape                                                               | Any gas mask providing protection against acid gases or hydrogen sulfide.<br><br>Any escape self-contained breathing apparatus.                                                                                                                                                                                                                                                                                             |

\*Only NIOSH-approved or MSHA-approved equipment should be used.

## 8. Further Information

A more detailed information booklet, The Chemical Safety Data Sheet SD36, may be obtained by writing to:

Manufacturing Chemists Association  
1825 Connecticut Avenue, NW  
Washington, DC 20009

### C. NICKEL CARBONYL FORMATION

Nickel carbonyl [ $\text{Ni}(\text{CO})_4$ ] is known to be an extremely toxic gas. Its primary effect is to cause lung damage with a lesser effect on the liver. The maximum average exposure to nickel carbonyl recommended by NIOSH is 0.001 ppm (1 ppb), and a maximum spot exposure of 0.04 ppm (40 ppb).

In naphtha hydrotreating units, the potential for forming nickel carbonyl exists only with catalysts containing nickel (S-6, S-7, S-15, S-16, S-19, N-204, HC-K), and only during regeneration or during the handling of unregenerated catalyst. Care must be used to ensure that the procedures used will prevent the formation of nickel carbonyl. Data has been published showing the equilibrium concentration of  $\text{Ni}(\text{CO})_4$  versus temperature, pressure and CO concentration in a gas. The nickel carbonyl concentration drops rapidly with increasing temperature and decreasing CO concentration. At 7 kg/cm<sup>2</sup>g (100 psig) with 0.5 mol-% CO in the gas, the nickel carbonyl concentration is at the maximum recommended spot level of 0.04 ppm at 149°C (300°F), and 0.001 ppm at 182°C (360°F).

The following practices should be followed to prevent the formation of nickel carbonyl:

1. Once a reactor containing a nickel catalyst has been exposed to oxidizing conditions (regeneration), a measurable concentration of oxygen must be maintained until the combustion of all carbon ceases and all  $\text{CO}_2$  has been purged from the system.

2. Once a reactor containing a nickel catalyst is in a reducing atmosphere and regeneration is not desirable, maintain the system in a reducing or inert atmosphere until all the catalyst has been cooled to at least 66°C (150°F). Unregenerated catalyst should be unloaded with N<sub>2</sub> purge before receiving used catalyst. Oxidation (burning) must be avoided.

There are many published techniques for determining the concentration of nickel carbonyl in air (such as a vessel to be entered for maintenance), and several direct reading instruments are available commercially. For further information see:

American Industrial Hygiene Assoc. Journal  
May - June, 1968  
Jan. - Feb., 1965

#### **D. PRECAUTIONS FOR ENTERING ANY CONTAMINATED OR INERT ATMOSPHERE**

Nitrogen is non-toxic. 79 mol-% of the air we breathe is nitrogen; 21 mol-% is oxygen. However, in vessels or areas where there is a high concentration of nitrogen, there is also a deficiency of oxygen for breathing. Breathing an atmosphere deficient in oxygen (i.e. an inert atmosphere) will rapidly result in dizziness, unconsciousness, or death depending on the length of exposure. Do not enter or even place your head into a vessel which has a high concentration of nitrogen. Do not stand close to a valve where nitrogen is being vented from equipment at a high rate which might temporarily cause a deficiency in oxygen close to the valve.

**UOP policy is not to allow any UOP technical advisors to perform work in a vessel which is known to be contaminated or under an inert atmosphere. UOP does not permit its technical advisors to perform "inert entry" work inside any vessel.**

Refinery personnel who do have to enter a contaminated or inert atmosphere should follow all prescribed standard safety precautions and regulations which

apply for the refinery. OSHA regulations concerning the use of respirators (29 CFR Subpart 1, Section 1910.134) should be read and thoroughly understood.

It is also important to emphasize that if a person has entered a vessel and become unconscious, no individual should go in to help him without first putting on a fresh air mask, confirming that the air supply is safe, donning a safety harness, and enlisting the aid of a minimum of two other people to remain immediately outside of the vessel to assist him. This may seem to be an obvious warning, but people do forget this in the trauma of an emergency situation. Often the first thought is to save the person in distress and people enter the vessel without proper protection only to succumb to the same hazard without anyone else being present to save them.

## **E. PREPARATIONS FOR VESSEL ENTRY**

Whenever a UOP technical advisor must enter a vessel, a meeting should be arranged between UOP and the refinery personnel who will be involved. The meeting should include review of the UOP vessel entry procedures, the refiner's safety requirements and facilities, preparation of a vessel entry schedule, assignment of responsibility for the preparation of a blind list, and assignment of responsibility for the vessel entry permits.

The most common tasks of a UOP technical advisor which could involve a potentially hazardous vessel entry are:

- Unit Checkout Prior to Startup
- Tumaround Inspections
- Reactor Loading
- Reactor Unloading

There are many precautions common to each situation which will be discussed in more detail in the remaining part of this section. The precautions apply equally to entry into all forms of vessels, including those enclosed areas which might not normally be considered vessels. Examples include:

---

|                     |                 |
|---------------------|-----------------|
| Reactors            | Regenerators    |
| Fractionators       | Separators      |
| Receivers           | Drums           |
| Fired Heaters       | Sumps           |
| Neutralizing Basins | Storage Tanks   |
| API Separators      | Water Treatment |
| Basins              |                 |

The API publication "Guide for Inspection of Refinery Equipment" or the NIOSH publication No. 87-113; "A Guide to Safety in Confined Spaces" can be referred to for additional information on safety procedures for vessel entry and accident prevention measures.

## **1. Positive Vessel Isolation**

Every line connecting to a nozzle on the vessel to be entered must be blinded at the vessel. This includes drains connecting to a closed sewer, utility connections and all process lines. The location of each blind should be marked on a master piping and instrumentation diagram (P&ID), each blind should be tagged with a number and a list of all blinds and their locations should be maintained. One person should be given responsibility for the all blinds in the unit to avoid errors.

The area around the vessel manways should also be surveyed for possible sources of dangerous gases which might enter the vessel while the person is inside. Examples include acetylene cylinders for welding and process vent or drain connections in the same or adjoining units. Any hazards found in the survey should be isolated or removed.

## **2. Vessel Access**

Safe access must be provided both to the exterior and interior of the vessel to be entered. The exterior access should be a solid, permanent ladder and platform or scaffolding strong enough to support the people and equipment who will be involved in the work to be performed.

Access to the interior should also be strong and solid. Scaffolding is preferred when the vessel is large enough to permit it to be used. The scaffolding base should rest firmly on the bottom of the vessel and be solidly anchored. If the scaffolding is tall, the scaffolding should be supported in several places to prevent sway. The platform boards should be sturdy and capable of supporting several people and equipment at the same time and also be firmly fastened down. Rungs should be provided on the scaffolding spaced at a comfortable distance for climbing on the structure.

If scaffolding will not fit in the vessel a ladder can be used. A rigid ladder is always preferred over a rope ladder and is essential to avoid fatigue during lengthy periods of work inside a vessel. The bottom and top of the ladder should be solidly anchored. If additional support is available, then the ladder should also be anchored at intermediate locations. When possible, a solid support should pass through the ladder under a rung, thereby providing support for the entire weight should the bottom support fail. Only one person at a time should be allowed on the ladder.

When a rope ladder is used, the ropes should be thoroughly inspected prior to each new job. All rungs should be tested for strength, whether they be made of metal or wood. Each rope must be individually secured to an immovable support. If possible, a solid support should pass through the ladder so that a rung can help support the weight and the bottom of the ladder should be fastened to a support to prevent the ladder from swinging. As with the rigid ladder, only one person should climb the ladder at a time.

### **3. Wearing of a Safety Harness**

Any person entering a vessel should wear a safety harness with an attached safety line. The harness is not complete without the safety line. The harness should be strong and fastened in such a manner that it can prevent a fall in the event the man slips and so that it can be used to extricate the man from the vessel in the event he encounters difficulty. A parachute type harness is preferred over a belt because it allows an unconscious person to be lifted from the shoulders, making it easier to remove him from a tight place such as an internal manway.

A minimum of one harness for each person entering the vessel and at least one spare harness for the people watching the manway should be provided at the vessel entry.

#### **4. Providing a Manway Watch**

Before a person enters a vessel, there should be a minimum of two people available outside of the vessel, one of whom should be specifically assigned responsibility to observe the activity of the people inside of the vessel. The other person must remain available in close proximity to the person watching the manway so that he can assist the or go for help, if necessary. He must also be alert for events outside of the vessel which might require the people inside to come out of the vessel, for example, a nearby leak or fire. These people should not leave their post until the people inside have safely evacuated the vessel.

A communication system should be provided for the manway watch so that they can quickly call for help in the event that the personnel inside of the vessel encounter difficulty. A radio, telephone, or public address system is necessary for that purpose.

#### **5. Providing Fresh Air**

The vessel must be purged completely free of any noxious or poisonous gases and inventoried with fresh air before permitting anyone to enter. The responsible department, usually the safety department, must test the atmosphere within the vessel for toxic gases, oxygen and explosive gases before entry. This must be repeated every 4 hours while there are people inside the vessel. When possible the UOP technical advisor should personally witness the test procedure. Each point of entry and any dead areas inside of the vessel, such as receiver boots or areas behind internal baffles, where there is little air circulation should be checked.

Fresh air can be circulated through the vessel using an air mover, a fan, or, for the cases where moisture is a concern, the vessel can be purged using dry certified instrument air from a hose or hard piped connection. When an air mover is used, make certain that the gas driver uses plant air, not nitrogen, and direct the exhaust of the driver out of the vessel to guarantee that this gas does not enter the vessel.

When instrument air is used, the UOP technical adviser must confirm that a check of the supply header is made to ensure that it is properly lined up and that there are no connections where nitrogen can enter the system (nitrogen improperly used as a backup for instrument air by some refiners). The fresh air purge should be continued throughout the time that people are inside of the vessel. The responsible control room should be informed that instrument air is being used for breathing so that if a change to nitrogen is required the people are removed from the affected vessel.

A minimum of one fresh air mask for each person entering the vessel and at least one spare mask for the hole watcher should be provided at the vessel entry. These masks should completely cover the face, including the eyes, and have a second seal around the mouth and nose. When use of the mask is required, it must first be donned outside of the vessel where it is easy to render assistance in order to confirm that the air supply is safe. Each mask must have a backup air supply that is completely independent of the main supply. It must also be independent of electrical power. This supply is typically a small, certified cylinder fastened to the safety harness and connected to the main supply line via a special regulator that activates when the air pressure to the mask drops below normal. The auxiliary supply should have an alarm which alerts the user that he is on backup supply and it should be sufficiently large to give the user 5 minutes to escape from danger.

## **6. Preparation of Vessel Entry Permit**

Before entering the vessel a vessel entry permit must be obtained. A vessel entry permit ensures that all responsible parties know that work is being conducted inside of a vessel and establishes a safe preparation procedure to follow in order to prevent mistakes which could result in an accident. The permit is typically issued by the safety engineer or by the shift supervisor. The permit should be based on a safety checklist to be completed before it is issued. The permit should also require the signatures of the safety engineer, the shift supervisor, and the person that performed the oxygen toxic and explosive gas check on the vessel atmosphere. Four copies of the permit should be provided. One copy goes to the safety engineer, one to the shift supervisor, one to the control room, and one copy should be posted prominently on the manway through which the personnel will enter the

vessel. The permit should be renewed before each shift and all copies of the permit should be returned to the safety engineer when the work is complete. Additional requirements or procedures may be imposed by the refiner, but the foregoing is considered the minimum acceptable for good safety practice.

## **7. Checkout Prior to New Unit Startup**

The risk of exposure to hydrocarbon, toxic or poisonous gases, and catalyst dust is low during a new unit checkout; the primary danger is nitrogen. There will be pressure testing, line flushing, hydrotesting, and possibly chemical cleaning being conducted in the unit and nitrogen may be used during any of these activities. Some of the equipment may have been inventoried with nitrogen to protect the internals from corrosion. An additional hazard is posed by operations in other parts of the plant which may be beyond the control of the people entering the vessel so that action taken at a remote location could admit nitrogen, fuel gas, steam, or other dangerous material through a connecting process line into the vessel which is being entered. For these reasons vessel entry procedures must still be rigorously followed during the checkout of a new unit.

The oxygen content of the atmosphere inside of the vessel should be checked before every entry and the vessel should be blinded. Independent blinds at each vessel nozzle are preferred. However, in the event that many vessels are to be entered in a new unit which is separate from the rest of the plant, the entire unit can be isolated by installing blinds at the battery limits rather than by individually isolating every vessel nozzle.

## **8. Inspections During Turnarounds**

In turnaround inspections, the possibility that vessels will contain dangerous gases is much higher. Equipment which has been in service must be thoroughly purged before entry. The vessel should have been steamed out unless steam presents a hazard to the internals and then fresh air circulated through it until all traces of hydrocarbons are gone. If liquid hydrocarbon remains or if odors persist afterwards, repeat the purging procedure until the vessel is clean. The service history of the vessel must also be investigated before entry so that appropriate precautions may

be taken. The service may require a neutralization step or a special cleaning step to make the vessel safe. Internal scale can trap poisonous gases such as hydrogen sulfide or hydrogen fluoride which may be released when the scale is disturbed. If this sort of danger is present, fresh air masks and protective clothing may be required to be worn while working inside of the equipment.

In a turnaround inspection, every vessel nozzle must be blinded at the vessel with absolutely no exceptions. There will always be process material at the low and high points in the lines connecting to the vessel because it is not possible to purge them completely clean. The blinds must all be in place before the vessel is purged.

Another factor to be cautious of, especially if entering a vessel immediately after the unit has been shut down, is heat stress. The internals of the vessels can still be very hot from the steam-out procedure or from operations prior to the shutdown. If that is the case, the period of time spent working inside of the vessel should be limited and frequent breaks should be taken outside of the vessel.

## **9. Reactor Loading**

Catalyst loading has perhaps the highest risk for asphyxiation or injury because some of the safety practices could be overlooked in the rush to complete the loading and get the unit on stream. If the reactor being loaded is new, the main concerns are catalyst dust and nitrogen. If the loading is a reload of an existing unit, any of the dangerous conditions described for turnaround inspections may also be present.

During reactor loading, dust will always be present. The effect of dust on the lungs is cumulative and even small concentrations with short exposure times should not be tolerated.

People who are exposed to the catalyst either outside or inside the reactor should wear MSHA/NIOSH approved dust masks or fresh air respirators. Goggles are also recommended. Exposure to catalyst dust can be minimized greatly by staying outside of the vessel during catalyst loading and by allowing the dust to settle before entering the vessel for inspection after loading.

Catalyst handling should be done in a well-ventilated area, using local mechanical exhaust ventilation if natural ventilation is insufficient.

Section XIII in this manual contains the detailed reactor loading procedure.

## 10. Reactor Unloading

Reactor unloading can present extraordinary health risks, especially to personnel working in the reactor. During the unloading, large quantities of catalyst dust may be generated. Additional hazards may include a contaminated atmosphere in the reactor, residual hydrocarbons or toxic forms of catalyst chemicals (e.g. nickel carbonyl). Unloading of unregenerated catalyst is covered in Section XIII. Here there are added precautions as the catalyst could contain iron pyrites which will spontaneously combust on contact with air.

UOP believes that the OSHA exposure limits to catalyst chemicals will not be exceeded if proper handling procedures are followed, and the proper protective clothing and safety devices are used. UOP recommends that the following minimum safety procedures be established and adhered to:

- Personnel working in reactors being unloaded should wear a fresh air respirator with a hood or helmet, operated in a pressure demand or other positive pressure mode, or in a continuous flow mode (NIOSH Respirator Code SAFE: PD, PP, CF). This respirator should have a primary, secondary, and emergency supply of air.
- Personnel in the reactor should be equipped with safety harnesses and safety lines for rescue and a means for visual, voice or signal line communication with standby personnel, who should be strategically located with suitable rescue equipment.
- The OSHA regulations concerning use of respirators (29 CFR, Subpart 1, Section 1910.134) should be read and thoroughly understood before

undertaking to place personnel in reactors during catalyst loading and unloading operations.

- Protective clothing and all safety devices should be thoroughly decontaminated after each use. Worn-out, broken or defective safety equipment and clothing should be removed from service and repaired or replaced. Good personal hygiene after handling a catalyst or being exposed to catalyst dust is an essential part of a responsible catalyst safety program. Do not eat, drink, or smoke in areas where the catalyst is being handled or where exposure to catalyst dust is likely.

#### **F. MSDS SHEETS FOR UOP HYDROBON® CATALYSTS**

The following MSDS Sheets are available from UOP: S-6, S-9, S-12, S-15, S-16, S-18, S-19M, S-19T, S-120, N-108, N-204 and HC-K.

## XV. EQUIPMENT EVALUATION

While the majority of UOP unit performance tests are conducted in order to satisfy contractual agreements between UOP and the customer, the potential significance of a mechanical evaluation is much greater. From the information generated and collected during an evaluation test, the refiner has the means to assess the potential of the unit, to plan for future debottlenecking and to optimize refinery operations.

The following description includes data necessary for contractual tests plus information required for evaluating hydraulic systems, heater, exchangers, pumps, compressors, etc. A large amount of the information would be gathered in any case (flows, temperatures, pressures, samples, etc.), and much of the rest can be obtained on a one-time basis.

However, the test information may not be of much value unless the following criteria are met:

1. The unit **must** weight balance. The weight balance must be consistently between 98 and 102 wt.%, and preferably between 99 and 101 wt.%.
2. All operations must be steady, including quality of charge stock, product specifications, exchanger outlet temperatures, etc.
3. Sufficient sample containers and laboratory analytical time must be available, including containers for sample shipment to UOP (optional).
4. Sufficient technical manpower must be available to gather data and take samples, in addition to those normally available for operating the unit.
5. The instrument technicians will be required before and during the performance test in order to calibrate the instrumentation daily during the test.

The following list indicates the amount and type of information required:

1. Flows: All process flows into and out of the unit, and also intermediate streams such as recycle gas and reflux, utility flows such as steam, BFW, instrument and plant air, cooling water, fuel gas, power consumption.
2. Temperatures: All process temperatures, including those not usually measured, but provided for by thermowells, heater and exchanger temperatures, storage tank temperatures.
3. Pressures: All process pressures, including single gauge hydraulic surveys on reactor systems and columns, pump and compressor suction and discharge pressures.
4. Levels: Particularly storage tank levels for feed and products, chemical consumption (inhibitor, etc.), process levels in columns, drums, receivers, compressor seal oil and/or cylinder oil losses, etc.
5. Samples: Samples of feed and products, intermediate streams such as reflux, recycle gas, fuel gas, flue gas, sour water.

Why is all this data required? There are many reasons, but those used most frequently are to establish a unit baseline performance, to predict the unit's maximum capacity, and to identify where the unit bottlenecks are. Another reason is for UOP's Engineering Department to evaluate actual unit and equipment performance compared to design. It is suggested that the data be accumulated at one time (during the performance run for contract demonstration), and that evaluation of the equipment be made later. It is important, however, to have **all** the necessary information available. To this end, the following lists and data sheets are given to use as guides in collecting data.

**GENERAL**

Ambient air conditions:    temperature  
                                         relative humidity  
                                         barometric pressure  
                                         wind velocity and direction  
                                         (shown on rough plot plan)

General description of unit – includes process flow diagram.

Unit system used (e.g. USA, Imperial, Metric) and definition of any uncommon units (e.g. kPa) and Standard Conditions (0°C, 760 mm; 60°F, 14.7 psia, etc.)

**Guarantee**

Data as required for Guarantee Agreement.

Complete weight balance, including meter correction factors.

**Exchangers**

Flow through exchangers on both sides (gas and liquid), composition and mass flow.

Temperature in and out on both sides, also between shells, bundles.

Pressures in and out on both sides, if possible.

Air coolers; air temperature out, air velocity out, motor amps, note any belt slippage, variable pitch positions, louver positions, etc.

In preparing data, submit overall heat transfer coefficient, specifics on exchangers.

## Heaters

Process flows (volume and mass, avg. mol wt., composition, etc.)

Process pressures in and out

Process temperatures in and out

Flue gas composition

Fuel gas composition

Fuel gas rate, pressure, temperature

Temperatures at bridgewall, any intermediate convection points, stack, tube skins, firebox skins

Temperature of BFW coils in and out, superheated steam pressure and temperature

Steam generation rate, pressure, temperature

BFW pressure, rate, temperature

Draft

Basic data on process coils (size, number, material, layout sketch, etc.)

Basic data on convection coils (size, number, material, layout sketch, etc.)

Burner data (rating, design release, etc.)

Need sufficient data to calculate heat flux from process side, heat flux from fire side, calculate total heat release, calculate heater efficiency.

**Chemical Consumption**

Feed Inhibitor

Sulfide Agents

Water

Stripper Overhead Inhibitor

**Hydraulic Survey and Process Separations**

Single gauge pressure survey of every point available on reactor circuit

All control valve positions (including fuel, BFW, etc.)

Pump and compressor motor amps

Pump suction, discharge pressure, flow rates, composition, temperatures, with manufacturer's curve data for comparison

Compressor suction, discharge pressures, flow rates, composition, temperatures, with manufacturer's curve data for comparison

Single gauge pressure survey of fractionation systems, with sufficient data to calculate internal reflux, number of theoretical trays, etc.

Samples of separator liquid and vapor and recontactor liquids and vapors for phase separation data

**Utility consumption/production data:**

Steam (all pressures)  
Air (Plant and Inst.)  
N<sub>2</sub>  
Cooling water  
BFW  
Utility water  
Steam condensate  
Process condensate

**Samples**

Unit charge from Feed Surge Drum  
Unit charge from each feed stream  
Separator (recycle) liquid  
Separator gas  
Makeup gas  
Stripper ovhd gas  
Stripper ovhd liquid (reflux)  
Stripper bottoms  
Flue gas  
Fuel gas  
Naphtha splitter ovhd  
Naphtha splitter bottoms  
Prefractionator Feed  
Stripper Ovhd (prefrac. section)  
Rerun column bottoms

**Comments:**

It is not necessary to obtain all the data at one time. It is acceptable to run various segments of the survey at different times, and one possible period would be during the line-out period prior to the guarantee test period. Data collections for heater and air-fin exchangers, in particular, are lengthy processes, and may be done at any time when the unit is stable, provided all the required process data are available.

If the data are collected, it obviously is necessary to have a good weight balance ( $100 \pm 2\%$ ) for the information to be meaningful. For most pieces of information, if the unit is lined out, spot data will be sufficient, rather than long-term averaged data. It might be possible, taking into consideration, to obtain the spot data in sections spread out during the guarantee test (one exception is column performance).

In presenting the data, some order should be kept. UOP suggests keeping sections by type of information, i.e., one section on the guarantee test results, one on heaters, one on exchangers, one on hydraulics, etc. Attached are some typical summary sheets for this purpose.

**COLUMN SUMMARY**

page \_\_\_\_\_

date \_\_\_\_\_

by \_\_\_\_\_

Item No.: \_\_\_\_\_

Service: \_\_\_\_\_

Type of Operation: \_\_\_\_\_

No. of Trays: \_\_\_\_\_ Reflux Ratio: \_\_\_\_\_

Type of Trays: \_\_\_\_\_

|                       | Feed  | Reflux | Off Gas | Btms. | Net Ovhd. Liquid | Other |
|-----------------------|-------|--------|---------|-------|------------------|-------|
| Mass Flow, _____      | _____ | _____  | _____   | _____ | _____            | _____ |
| Temperature, ° _____  | _____ | _____  | _____   | _____ | _____            | _____ |
| Pressure, _____       | _____ | _____  | _____   | _____ | _____            | _____ |
| Composition, _____ %  | _____ | _____  | _____   | _____ | _____            | _____ |
| H <sub>2</sub>        | _____ | _____  | _____   | _____ | _____            | _____ |
| N <sub>2</sub>        | _____ | _____  | _____   | _____ | _____            | _____ |
| H <sub>2</sub> S      | _____ | _____  | _____   | _____ | _____            | _____ |
| H <sub>2</sub> O      | _____ | _____  | _____   | _____ | _____            | _____ |
| C <sub>1</sub>        | _____ | _____  | _____   | _____ | _____            | _____ |
| C <sub>2</sub>        | _____ | _____  | _____   | _____ | _____            | _____ |
| C <sub>3</sub>        | _____ | _____  | _____   | _____ | _____            | _____ |
| iC <sub>4</sub>       | _____ | _____  | _____   | _____ | _____            | _____ |
| nC <sub>4</sub>       | _____ | _____  | _____   | _____ | _____            | _____ |
| iC <sub>5</sub>       | _____ | _____  | _____   | _____ | _____            | _____ |
| nC <sub>5</sub>       | _____ | _____  | _____   | _____ | _____            | _____ |
| C <sub>6</sub> +      | _____ | _____  | _____   | _____ | _____            | _____ |
| Avg. Mol. Wt.         | _____ | _____  | _____   | _____ | _____            | _____ |
| Gravity               | _____ | _____  | _____   | _____ | _____            | _____ |
| Distillation, ° _____ | _____ | _____  | _____   | _____ | _____            | _____ |
| IBP                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 5%                    | _____ | _____  | _____   | _____ | _____            | _____ |
| 10%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 20%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 30%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 40%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 50%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 60%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 70%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 80%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 90%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| 95%                   | _____ | _____  | _____   | _____ | _____            | _____ |
| EP                    | _____ | _____  | _____   | _____ | _____            | _____ |

(Sketch system showing flows, P, T, Q on separate page) \_\_\_\_\_

Weight balance \_\_\_\_\_ Heat balance \_\_\_\_\_

Deviations from UOP Specifications: \_\_\_\_\_

\_\_\_\_\_

**RECIPROCATING COMPRESSOR DATA**

(can substitute metric values)

page \_\_\_\_\_

date \_\_\_\_\_

by \_\_\_\_\_

Item No.: \_\_\_\_\_

Service: \_\_\_\_\_

Manufacturer: \_\_\_\_\_

Type, Model: \_\_\_\_\_

No. of Stages, No. of Cylinders: \_\_\_\_\_

Cylinder Lubrication: \_\_\_\_\_

Clearance Pockets: (yes/no)

Sparing Description: \_\_\_\_\_

**OPERATING CONDITIONS/PERFORMANCE**

Flow Rate: \_\_\_\_\_

Suction Temperature: \_\_\_\_\_ °F

Suction Pressure: \_\_\_\_\_ psig

Discharge Temperature: \_\_\_\_\_ °F

Discharge Pressure: \_\_\_\_\_ psig

HP/stage: \_\_\_\_\_ hp

MW: \_\_\_\_\_

Operating Speed: \_\_\_\_\_ rpm

Cylinder Diameters: \_\_\_\_\_

Piston Speed: \_\_\_\_\_ ft/s

# of Suction/Discharge Valves: \_\_\_\_\_

Actual Rod Loadings, T/C: \_\_\_\_\_ lbf

Max Allowable Rod Loadings, T/C: \_\_\_\_\_ lbf

**DRIVER**

Motor Manufacturer: \_\_\_\_\_

Rating: \_\_\_\_\_

Service Factor: \_\_\_\_\_

Insulation Class: \_\_\_\_\_

Voltage/phase/cycle: \_\_\_\_\_

Turbine Manufacturer: \_\_\_\_\_

Speed: \_\_\_\_\_

Steam Supply: \_\_\_\_\_ psig \_\_\_\_\_ °F

Steam Rate: \_\_\_\_\_

Steam Exhaust: \_\_\_\_\_ psig \_\_\_\_\_ °F

Gear Manufacturer: \_\_\_\_\_

Rating: \_\_\_\_\_

Service Factor: \_\_\_\_\_

Type: \_\_\_\_\_

Power Loss: \_\_\_\_\_

Deviations from UOP Specification: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**CONTROL VALVE SUMMARY**

page \_\_\_\_\_  
date \_\_\_\_\_  
by \_\_\_\_\_  
Item No.: \_\_\_\_\_  
Service: \_\_\_\_\_  
Description of Valve: \_\_\_\_\_ Design CV: \_\_\_\_\_  
Mfr. and Catalog No.: \_\_\_\_\_  
Positioner? \_\_\_\_\_

|                               | <b>Actual</b> | <b>Design</b> |
|-------------------------------|---------------|---------------|
| Percent open (valve position) | _____         | _____         |
| Flow rate: _____              | _____         | _____         |
| Upstream pressure: _____      | _____         | _____         |
| Downstream pressure: _____    | _____         | _____         |
| Flowing temperature: _____    | _____         | _____         |

Deviations from UOP Specification: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**AIR FIN COOLER SURVEY**

Item No.: \_\_\_\_\_ page \_\_\_\_\_  
 Service: \_\_\_\_\_ date \_\_\_\_\_  
 Manufacturer: \_\_\_\_\_ by \_\_\_\_\_  
 Type, Model: \_\_\_\_\_  
 No. of Bundles: \_\_\_\_\_ No. of Passes: \_\_\_\_\_  
 No. of Tubes per Pass: \_\_\_\_\_ Fans/bundle: \_\_\_\_\_  
 Tube Size \_\_\_\_\_ ID x \_\_\_\_\_ Gauge x \_\_\_\_\_ Length  
 Piping Geometry: \_\_\_\_\_ Type\*: \_\_\_\_\_  
 Overall Heat Transfer Coefficient: \_\_\_\_\_

|                 |       | Pressure      | Temperature |
|-----------------|-------|---------------|-------------|
| Inlet           |       | _____         | _____       |
| Outlet          |       | _____         | _____       |
| Air             | In    | _____         | _____       |
|                 | Out   | _____         | _____       |
| No. fans on     | _____ | Pitch control | _____       |
| Louver position | _____ |               |             |

|                      | Air   | Process |
|----------------------|-------|---------|
| Mass flow            | _____ | _____   |
| Q (calc.)            | _____ | _____   |
| Composition, _____ % |       |         |
| H <sub>2</sub>       |       | _____   |
| N <sub>2</sub>       |       | _____   |
| H <sub>2</sub> S     |       | _____   |
| H <sub>2</sub> O     |       | _____   |
| C <sub>1</sub>       |       | _____   |
| C <sub>2</sub>       |       | _____   |
| C <sub>3</sub>       |       | _____   |
| iC <sub>4</sub>      |       | _____   |
| nC <sub>4</sub>      |       | _____   |
| iC <sub>5</sub>      |       | _____   |
| nC <sub>5</sub>      |       | _____   |
| C <sub>6</sub> +     |       | _____   |
| Avg. Mol. Wt.        |       | _____   |
| Relative Humidity    | _____ |         |

Gravity  
 Distillation, ° \_\_\_\_\_  
 IBP  
 10%  
 30%  
 50%  
 70%

90%

EP

---

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Deviations from UOP Specification: 

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\*Include sketch of piping geometry if different from UOP standard practice types.

**FLOW METER SUMMARY**

page \_\_\_\_\_

date \_\_\_\_\_

Item No.: \_\_\_\_\_

by \_\_\_\_\_

Service: \_\_\_\_\_

Type of Fluid: \_\_\_\_\_ Normal Units of Flow: \_\_\_\_\_

Type of Meter: \_\_\_\_\_

Meter Reading: \_\_\_\_\_

Pressure \_\_\_\_\_

Temperature \_\_\_\_\_

Sp. Gr.\*\* \_\_\_\_\_

Meter Factor \_\_\_\_\_

Corrected Flow Rate \_\_\_\_\_

Mass Flow Rate \_\_\_\_\_

Avg. mol. wt. \_\_\_\_\_

Molar Flow Rate \_\_\_\_\_

\*\*Sketch piping layout, showing distances in nominal pipe IDs.

**HEAT EXCHANGER SURVEY**

page \_\_\_\_\_  
 date \_\_\_\_\_  
 by \_\_\_\_\_

Item No.: \_\_\_\_\_  
 Service: \_\_\_\_\_  
 Manufacturer: \_\_\_\_\_  
 Type, Model: \_\_\_\_\_  
 No. of Bundles: \_\_\_\_\_  
 No. of Passes/Bundle: \_\_\_\_\_ Tubes per Pass: \_\_\_\_\_  
 Tube Size \_\_\_\_\_ ID x \_\_\_\_\_ Gauge x \_\_\_\_\_ Length \_\_\_\_\_  
 Heat Exchange Surface Area/Bundle: \_\_\_\_\_  
 Piping Geometry (sketch if necessary): \_\_\_\_\_  
 Length of Service: \_\_\_\_\_  
 Design Heat Transfer Coefficient: \_\_\_\_\_

|               |        | Stream | Pressure | Temperature |
|---------------|--------|--------|----------|-------------|
| Effluent Side | Inlet  | A      | _____    | _____       |
|               | Outlet |        | _____    | _____       |
| Feed Side     | Inlet  | B      | _____    | _____       |
|               | Outlet |        | _____    | _____       |

Q (calc.) Effluent side \_\_\_\_\_  
 Q (calc.) Feed side \_\_\_\_\_

Composition, \_\_\_\_\_%

|                  | A     | B     |
|------------------|-------|-------|
| H <sub>2</sub>   | _____ | _____ |
| N <sub>2</sub>   | _____ | _____ |
| H <sub>2</sub> S | _____ | _____ |
| H <sub>2</sub> O | _____ | _____ |
| C <sub>1</sub>   | _____ | _____ |
| C <sub>2</sub>   | _____ | _____ |
| C <sub>3</sub>   | _____ | _____ |
| iC <sub>4</sub>  | _____ | _____ |
| nC <sub>4</sub>  | _____ | _____ |
| iC <sub>5</sub>  | _____ | _____ |
| nC <sub>5</sub>  | _____ | _____ |
| C <sub>6+</sub>  | _____ | _____ |
| Mass Flow        | _____ | _____ |
| Avg. Mol. Wt.    | _____ | _____ |

Gravity  
 Distillation, ° \_\_\_\_\_

|     |       |       |
|-----|-------|-------|
| IBP | _____ | _____ |
| 10% | _____ | _____ |
| 30% | _____ | _____ |
| 50% | _____ | _____ |
| 70% | _____ | _____ |
| 90% | _____ | _____ |
| EP  | _____ | _____ |

Deviations from UOP Specification: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**CHARGE HEATER AND COMBINED FEED EXCHANGER DATA**

(can substitute metric values)

**Flow Rates**

Fresh Feed (BPD) \_\_\_\_\_

Recycle Gas (MMSCFD) \_\_\_\_\_

**Compositions**

Fresh Feed

SpGr or API \_\_\_\_\_

D-86 Dist, (°F)

IBP \_\_\_\_\_

10% \_\_\_\_\_

30% \_\_\_\_\_

50% \_\_\_\_\_

70% \_\_\_\_\_

90% \_\_\_\_\_

EP \_\_\_\_\_

Recycle Gas

Molecular Wt \_\_\_\_\_

Chromatograph, (Mol%)

H<sub>2</sub> \_\_\_\_\_C<sub>1</sub> \_\_\_\_\_C<sub>2</sub> \_\_\_\_\_C<sub>3</sub> \_\_\_\_\_C<sub>4</sub> \_\_\_\_\_C<sub>5</sub> \_\_\_\_\_C<sub>6+</sub> \_\_\_\_\_**Pressures (psig)**

Separator/Compressor Suction \_\_\_\_\_

Compressor Discharge \_\_\_\_\_

Reactor 1 Outlet \_\_\_\_\_

Reactor 2 Outlet \_\_\_\_\_

**Temperatures (°F)**

Inlet

Outlet

Charge Heater \_\_\_\_\_

Reactor No. 1 \_\_\_\_\_

Reactor No. 2 \_\_\_\_\_

**Combined Feed  
Exchangers****Exch  
No. 1****Exch  
No. 2****Exch  
No. 3****Exch  
No. 4****Hot Side**

Rx Effluent In \_\_\_\_\_

Rx Effluent Out \_\_\_\_\_

**Cold Side**

Liquid In \_\_\_\_\_

Recycle Gas In \_\_\_\_\_

Comb Feed In \_\_\_\_\_

Comb Feed Out \_\_\_\_\_

Exchanger Type (S&amp;T or Packinox) \_\_\_\_\_

Number of Exchangers \_\_\_\_\_

Tube Length, ft \_\_\_\_\_

Shell Diameter, in. \_\_\_\_\_

Manufacturer \_\_\_\_\_

Number of Tubes \_\_\_\_\_

Tube OD, in. \_\_\_\_\_

| Heaters       | Oxygen<br>Readings<br>Mole % | Fuel Gas<br>Flow<br>SCFH |
|---------------|------------------------------|--------------------------|
| Charge Heater | _____                        | _____                    |

## Fuel Composition, Mole %

|                  |       |
|------------------|-------|
| H <sub>2</sub>   | _____ |
| N <sub>2</sub>   | _____ |
| H <sub>2</sub> S | _____ |
| H <sub>2</sub> O | _____ |
| C <sub>1</sub>   | _____ |
| C <sub>2</sub>   | _____ |
| C <sub>3</sub>   | _____ |
| iC <sub>4</sub>  | _____ |
| nC <sub>4</sub>  | _____ |
| iC <sub>5</sub>  | _____ |
| nC <sub>5</sub>  | _____ |
| C <sub>6</sub> + | _____ |

**HEATER SURVEY**

page \_\_\_\_\_

date \_\_\_\_\_

by \_\_\_\_\_

Item No.: \_\_\_\_\_

Service: \_\_\_\_\_

Manufacturer: \_\_\_\_\_

Type, Model: \_\_\_\_\_

No. of Passes: \_\_\_\_\_ Tubes per Pass: \_\_\_\_\_

Tube Size \_\_\_\_\_ ID x \_\_\_\_\_ Wall x \_\_\_\_\_ Length \_\_\_\_\_

Geometry (Process): \_\_\_\_\_

Geometry (Flue Gas): \_\_\_\_\_

|                             |        | Stream | Pressure | Temperature |
|-----------------------------|--------|--------|----------|-------------|
| Radiant                     | Inlet  | A      | _____    | _____       |
|                             | Outlet |        | _____    | _____       |
| Convection                  | Inlet  | B      | _____    | _____       |
|                             | Outlet |        | _____    | _____       |
| Fuel Gas                    |        | C      | _____    | _____       |
| Flue Gas Under Convection   |        | D      | _____    | _____       |
| Flue Gas Under Stack Damper |        | E      | _____    | _____       |
| Flue Gas Above Floor        |        | F      | _____    | _____       |

**HEATER SURVEY**

page \_\_\_\_\_  
 date \_\_\_\_\_  
 by \_\_\_\_\_

| Stream _____                  | A _____ | B _____ | C _____ | D _____ | E _____ | F _____ |
|-------------------------------|---------|---------|---------|---------|---------|---------|
| Mass Flow, _____              | _____   | _____   | _____   | _____   | _____   | _____   |
| Composition, _____ %          | _____   | _____   | _____   | _____   | _____   | _____   |
| H <sub>2</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| N <sub>2</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| O <sub>2</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| CO                            | _____   | _____   | _____   | _____   | _____   | _____   |
| CO <sub>2</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| H <sub>2</sub> S              | _____   | _____   | _____   | _____   | _____   | _____   |
| SO <sub>2</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| C <sub>1</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| C <sub>2</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| C <sub>3</sub>                | _____   | _____   | _____   | _____   | _____   | _____   |
| iC <sub>4</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| nC <sub>4</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| iC <sub>5</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| nC <sub>5</sub>               | _____   | _____   | _____   | _____   | _____   | _____   |
| C <sub>6</sub> -205°C (400°F) | _____   | _____   | _____   | _____   | _____   | _____   |
| 205°C (400°F)+                | _____   | _____   | _____   | _____   | _____   | _____   |
| Avg. Mol. Wt.                 | _____   | _____   | _____   | _____   | _____   | _____   |
| Gravity                       | _____   | _____   | _____   | _____   | _____   | _____   |
| Viscosity                     | _____   | _____   | _____   | _____   | _____   | _____   |
| Total Sulfur, _____           | _____   | _____   | _____   | _____   | _____   | _____   |
| Metals, _____                 | _____   | _____   | _____   | _____   | _____   | _____   |
| Q (calc.) Absorbed            | _____   | _____   | _____   | _____   | _____   | _____   |
| Q (calc.) Released            | _____   | _____   | _____   | _____   | _____   | _____   |
| Heater Gross Efficiency       | _____   | _____   | _____   | _____   | _____   | _____   |
| Excess Air, %                 | _____   | _____   | _____   | _____   | _____   | _____   |
| Tube Skin Temps., ° _____     | _____   | _____   | _____   | _____   | _____   | _____   |

Burner Pressure \_\_\_\_\_ % of Rating \_\_\_\_\_

Provide sketch showing piping and controls for process piping.

Deviations from UOP Specification: \_\_\_\_\_

**CENTRIFUGAL PUMP SURVEY**

Item No.: \_\_\_\_\_ page \_\_\_\_\_  
Service: \_\_\_\_\_ date \_\_\_\_\_  
Manufacturer: \_\_\_\_\_ by \_\_\_\_\_  
Type, Model: \_\_\_\_\_  
No., Size and Style (Mfgs. Designation) \_\_\_\_\_

|           | Pressure | Temperature |
|-----------|----------|-------------|
| Suction   | _____    | _____       |
| Discharge | _____    |             |

**Other Information**

|                                             |                                           |
|---------------------------------------------|-------------------------------------------|
| Rated Flow (STP) _____                      | Seal Type? Single, Tandem, Double, Bellow |
| Sp. Gr. _____                               | Spillback? Yes/No                         |
| Viscosity _____                             | NPSHR? _____                              |
| Static Suction Head _____                   | Suction Specific Speed: _____             |
| Speed _____                                 |                                           |
| Differential Head (flowing condition) _____ |                                           |

Driver Type: \_\_\_\_\_  
Manufacturer: \_\_\_\_\_  
No., Size, Rating and Style (Mfgs. designation): \_\_\_\_\_  
Rating: \_\_\_\_\_ Insulation Class: \_\_\_\_\_  
Service Factor: \_\_\_\_\_ Voltage/Phase/Cycle: \_\_\_\_\_

**Motor:**

Power consumption \_\_\_\_\_  
Speed \_\_\_\_\_

**Turbine:**

|                         |          |             |
|-------------------------|----------|-------------|
| Steam consumption _____ | Pressure | Temperature |
| Steam supply            | _____    | _____       |
| Steam exhaust           | _____    | _____       |
| Speed _____             |          |             |

Supply copy of Mfgs. pump curve and plot operating point.

Deviations from UOP Specification: \_\_\_\_\_

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**REACTOR SECTION PRESSURE SURVEY**

page \_\_\_\_\_  
date \_\_\_\_\_  
by \_\_\_\_\_

|                                                 |        | Pressure | Temperature |
|-------------------------------------------------|--------|----------|-------------|
| Feed CV                                         | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| Combined Feed Exchanger                         | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| Charge Heater                                   | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| No. 1 RX                                        | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| No. 2 RX                                        | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| Combined Feed Exchanger                         | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |
| Fractionator Feed/Reactor<br>Effluent Exchanger | Inlet  | _____    | _____       |
|                                                 | Outlet | _____    | _____       |

**REACTOR SECTION PRESSURE SURVEY**

page \_\_\_\_\_  
 date \_\_\_\_\_  
 by \_\_\_\_\_

|                                     |                  |   | Pressure | Temperature |
|-------------------------------------|------------------|---|----------|-------------|
| Reactor Effluent Fin Fan            | Inlet            |   | _____    | _____       |
|                                     | Outlet           |   | _____    | _____       |
| Reactor Effluent Condenser          | Inlet Intershell | A | _____    | _____       |
|                                     |                  | B | _____    | _____       |
|                                     |                  | C | _____    | _____       |
|                                     |                  | D | _____    | _____       |
|                                     |                  | E | _____    | _____       |
|                                     |                  | F | _____    | _____       |
|                                     | Outlet           |   | _____    | _____       |
| Separator                           |                  |   | _____    | _____       |
| Separator Pump                      | Suction          |   | _____    | _____       |
|                                     | Discharge        |   | _____    | _____       |
| Separator Liquid CV                 | Inlet            |   | _____    | _____       |
|                                     | Discharge        |   | _____    | _____       |
| Separator Offgas CV                 | Inlet            |   | _____    | _____       |
|                                     | Discharge        |   | _____    | _____       |
| Separator Gas to Net Gas Compressor |                  |   | _____    | _____       |
| Recycle Compressor A                | Suction          |   | _____    | _____       |
|                                     | Discharge        |   | _____    | _____       |
| Recycle Compressor B                | Suction          |   | _____    | _____       |
|                                     | Discharge        |   | _____    | _____       |
| Recycle Gas to CFE                  |                  |   | _____    | _____       |

**OPERATING DATABASE**

(can substitute metric values)

Customer \_\_\_\_\_  
Catalyst Batch Number \_\_\_\_\_  
Regeneration Cycle Number \_\_\_\_\_

Refiner Name \_\_\_\_\_  
Location \_\_\_\_\_

Catalyst Type: \_\_\_\_\_

Total Reactor Catalyst Loading, lb \_\_\_\_\_

Total Reactor Catalyst Volume, ft<sup>3</sup> \_\_\_\_\_

Catalyst Distribution, wt%:

Reactor No. 1 \_\_\_\_\_

Reactor No. 2 \_\_\_\_\_

Design Inlet Temperature, °F:

Reactor No. 1 \_\_\_\_\_

Reactor No. 2 \_\_\_\_\_

## OPERATING DATABASE

**LINE 1**

|              |       |       |       |       |
|--------------|-------|-------|-------|-------|
| Date of Test | _____ | _____ | _____ | _____ |
| Start Time   | _____ | _____ | _____ | _____ |
| End Time     | _____ | _____ | _____ | _____ |

**LINE 2**

|                         |       |       |       |       |
|-------------------------|-------|-------|-------|-------|
| Days on Stream          | _____ | _____ | _____ | _____ |
| Cumulative Charge, bbl  | _____ | _____ | _____ | _____ |
| Liquid Flow Rates, bpsd | _____ | _____ | _____ | _____ |
| Reactor Charge          | _____ | _____ | _____ | _____ |
| Total Stripper Bottoms  | _____ | _____ | _____ | _____ |
| Stabilizer Ovhd Liquid  | _____ | _____ | _____ | _____ |
| Excess Liquid           | _____ | _____ | _____ | _____ |

**LINE 3**

|                                 |       |       |       |       |
|---------------------------------|-------|-------|-------|-------|
| Gas Flow Rates, mscfd           | _____ | _____ | _____ | _____ |
| Makeup Gas                      | _____ | _____ | _____ | _____ |
| Separator Gas                   | _____ | _____ | _____ | _____ |
| Stabilizer Ovhd Gas             | _____ | _____ | _____ | _____ |
| H <sub>2</sub> /HCBN Mole Ratio | _____ | _____ | _____ | _____ |
| RX #1, Inlet Temp., °F          | _____ | _____ | _____ | _____ |
| RX #2, Inlet Temp., °F          | _____ | _____ | _____ | _____ |

**LINE 4**

|                        |       |       |       |       |
|------------------------|-------|-------|-------|-------|
| RX #1, Delta Temp., °F | _____ | _____ | _____ | _____ |
| RX #2, Delta Temp., °F | _____ | _____ | _____ | _____ |

|                |       |       |       |       |
|----------------|-------|-------|-------|-------|
| Pressure, psig | _____ | _____ | _____ | _____ |
| Last RX Inlet  | _____ | _____ | _____ | _____ |
| Separator      | _____ | _____ | _____ | _____ |

|                     |       |       |       |       |
|---------------------|-------|-------|-------|-------|
| Temperatures, °F    | _____ | _____ | _____ | _____ |
| Separator           | _____ | _____ | _____ | _____ |
| Charge Heater Inlet | _____ | _____ | _____ | _____ |

**LINE 5**

Combined RX Feed Characteristics

API Gravity

Distillation Method

|       |       |       |       |
|-------|-------|-------|-------|
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |

**LINE 6**

Feed Distillation, °F

IBP

10%

30%

50%

70%

90%

EP

|       |       |       |       |
|-------|-------|-------|-------|
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |

## OPERATING DATABASE

|                              |       |       |       |       |
|------------------------------|-------|-------|-------|-------|
| PONA Method                  | _____ | _____ | _____ | _____ |
| Paraffins, lv%               | _____ | _____ | _____ | _____ |
| Olefins, lv%                 | _____ | _____ | _____ | _____ |
| Naphthenes, lv%              | _____ | _____ | _____ | _____ |
| Aromatics, lv%               | _____ | _____ | _____ | _____ |
| Water Addition, lv%          | _____ | _____ | _____ | _____ |
| Feed Sources, lv%            | _____ | _____ | _____ | _____ |
| Gas Conc. Unit               | _____ | _____ | _____ | _____ |
| Gas Conc. Unit               | _____ | _____ | _____ | _____ |
| Coker                        | _____ | _____ | _____ | _____ |
| Blending System              | _____ | _____ | _____ | _____ |
| Tankage                      | _____ | _____ | _____ | _____ |
| Other _____                  | _____ | _____ | _____ | _____ |
| Feed Qualities, wt ppm       |       |       |       |       |
| Sulfur                       | _____ | _____ | _____ | _____ |
| Nitrogen                     | _____ | _____ | _____ | _____ |
| Chloride                     | _____ | _____ | _____ | _____ |
| Silicon                      | _____ | _____ | _____ | _____ |
| RX Feed GC, lv%              |       |       |       |       |
| nC <sub>4</sub>              | _____ | _____ | _____ | _____ |
| iC <sub>5</sub>              | _____ | _____ | _____ | _____ |
| nC <sub>5</sub>              | _____ | _____ | _____ | _____ |
| C <sub>6</sub> +             | _____ | _____ | _____ | _____ |
| Stripper Bottoms Distill. °F |       |       |       |       |
| IBP                          | _____ | _____ | _____ | _____ |
| 10%                          | _____ | _____ | _____ | _____ |
| 30%                          | _____ | _____ | _____ | _____ |
| 50%                          | _____ | _____ | _____ | _____ |
| 70%                          | _____ | _____ | _____ | _____ |
| 90%                          | _____ | _____ | _____ | _____ |
| EP                           | _____ | _____ | _____ | _____ |

## OPERATING DATABASE

## Recycle Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Rec. Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## Makeup Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Makeup Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> O |  |  |  |  |
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## OPERATING DATABASE

## Recycle Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Rec. Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## Makeup Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Makeup Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> O |  |  |  |  |
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## OPERATING DATABASE

## Recycle Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Rec. Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## Makeup Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

## Makeup Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> O |  |  |  |  |
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

## OPERATING DATABASE

Stripper Ovhd Gas GC, mole%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub>   |  |  |  |  |
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

Stab. Ovhd. Gas Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |

Stab. Ovhd. Liquid GC, lv%

|                  |  |  |  |  |
|------------------|--|--|--|--|
| C <sub>1</sub>   |  |  |  |  |
| C <sub>2</sub>   |  |  |  |  |
| C <sub>2</sub> = |  |  |  |  |
| C <sub>3</sub>   |  |  |  |  |
| C <sub>3</sub> = |  |  |  |  |
| iC <sub>4</sub>  |  |  |  |  |
| nC <sub>4</sub>  |  |  |  |  |
| C <sub>4</sub> = |  |  |  |  |
| iC <sub>5</sub>  |  |  |  |  |
| nC <sub>5</sub>  |  |  |  |  |
| C <sub>6</sub> + |  |  |  |  |

Stab. Ovhd. Liquid Impurities, mol ppm

|                  |  |  |  |  |
|------------------|--|--|--|--|
| H <sub>2</sub> S |  |  |  |  |
| HCl              |  |  |  |  |