
UOP/Cepsa Detal Unit

General Operating Manual

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UOP/CEPSA DETAL UNIT

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I. INTRODUCTION

A. HISTORICAL BACKGROUND

The Detal Process is a catalytic process to alkylate benzene with linear olefins to make linear alkylbenzene (LAB). The linear alkylbenzenes produced from the C₁₀ to C₁₃ or C₁₁ to C₁₄ range linear olefins are useful detergent intermediates and can be readily sulfonated to yield linear alkylbenzene sulfonates (LAS). These compounds constitute the "active" ingredients of many household detergents. They are surface active compounds (surfactants) which are combined with various builders (often inorganic salts) to make up a detergent formula.

During the 1940s and 1950s, the detergent market was primarily captured by dodecylbenzene (DDB), a product formed by alkylation of benzene with propylene tetramer in a "hard" detergent alkylation unit. It was found, however, that the branched structure of the alkyl group was responsible for the poor biodegradability of this detergent. The linear alkylbenzenes (LAB) which are produced in the "soft" detergent alkylation units that were introduced in the early 1960s have substantially replaced their branched counterparts.

B. PROCESS DESCRIPTION

Since the late 1960s, HF acid has been the principal catalyst for the alkylation reaction that produces LAB. Due to the high capital and maintenance costs involved with a very corrosive acid like HF, and due to the growing concerns about the safety and waste disposal in an HF acid unit, there are several advantages in using a solid, non-corrosive catalyst in place of HF acid. Such a catalyst was developed in the early 1990s and is the basis for the Detal process.

In a typical LAB complex (Figure I-1) there is an n-paraffin extraction unit (such as the UOP Molex unit) which extracts n-paraffins from a hydrotreated kerosene feed. The pure n-paraffins are then fed to a UOP Pacol unit where about 13% of the

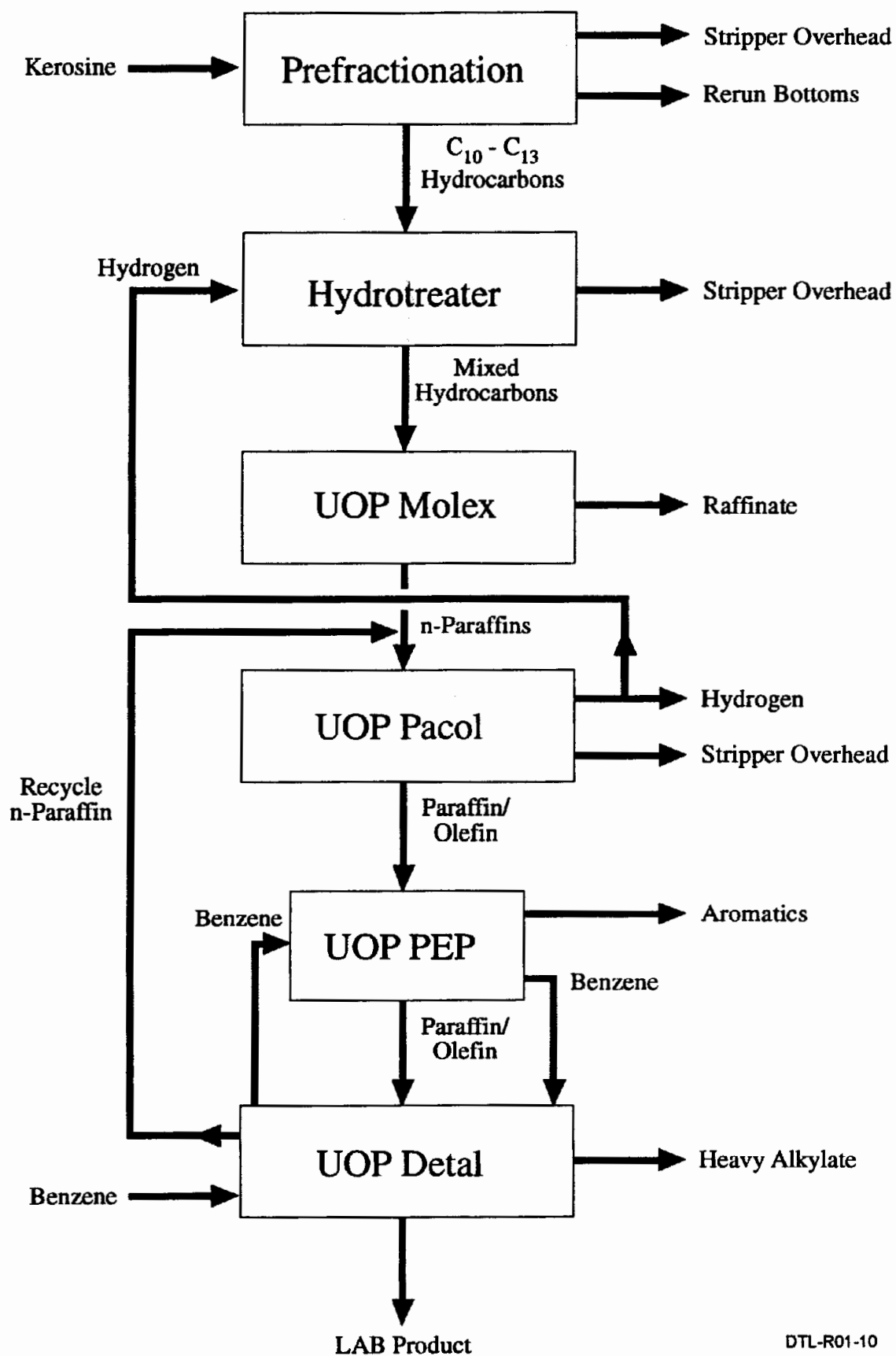
n-paraffins are dehydrogenated to n-olefins. This mixture of n-olefins and n-paraffins is then fed to the Detal unit where the n-olefins react with benzene to form LAB. The unreacted normal paraffins are continuously recycled to the Pacol feed after passing through the Detal unit. It has been found that adjusting the Pacol reactor to about 13 wt-% olefin conversion is the most economical way to operate in terms of product quality and operating costs. All Pacol units which are associated with Detal units must have a DeFine reactor to treat the Pacol reactor product. DeFine selectively hydrogenates the diolefins which are formed in the Pacol reactors, reducing the formation of by-products which would foul the Detal catalyst. The PEP unit removes aromatics from the Pacol product that is fed to the Detal unit. These aromatics are a byproduct in the Pacol reaction and they inhibit the Detal catalyst activity and can cause Detal catalyst deactivation.

The n-olefins produced in the Pacol unit are alkylated with benzene in the Detal reactor section to yield linear alkylbenzene (LAB). Due to the buildup of polymer-like by-products on the catalyst, it is necessary to regenerate the catalyst with hot benzene after about 24 hours of operation. For this reason, a two-reactor system is used so that one reactor can always be on-line while the other is being regenerated.

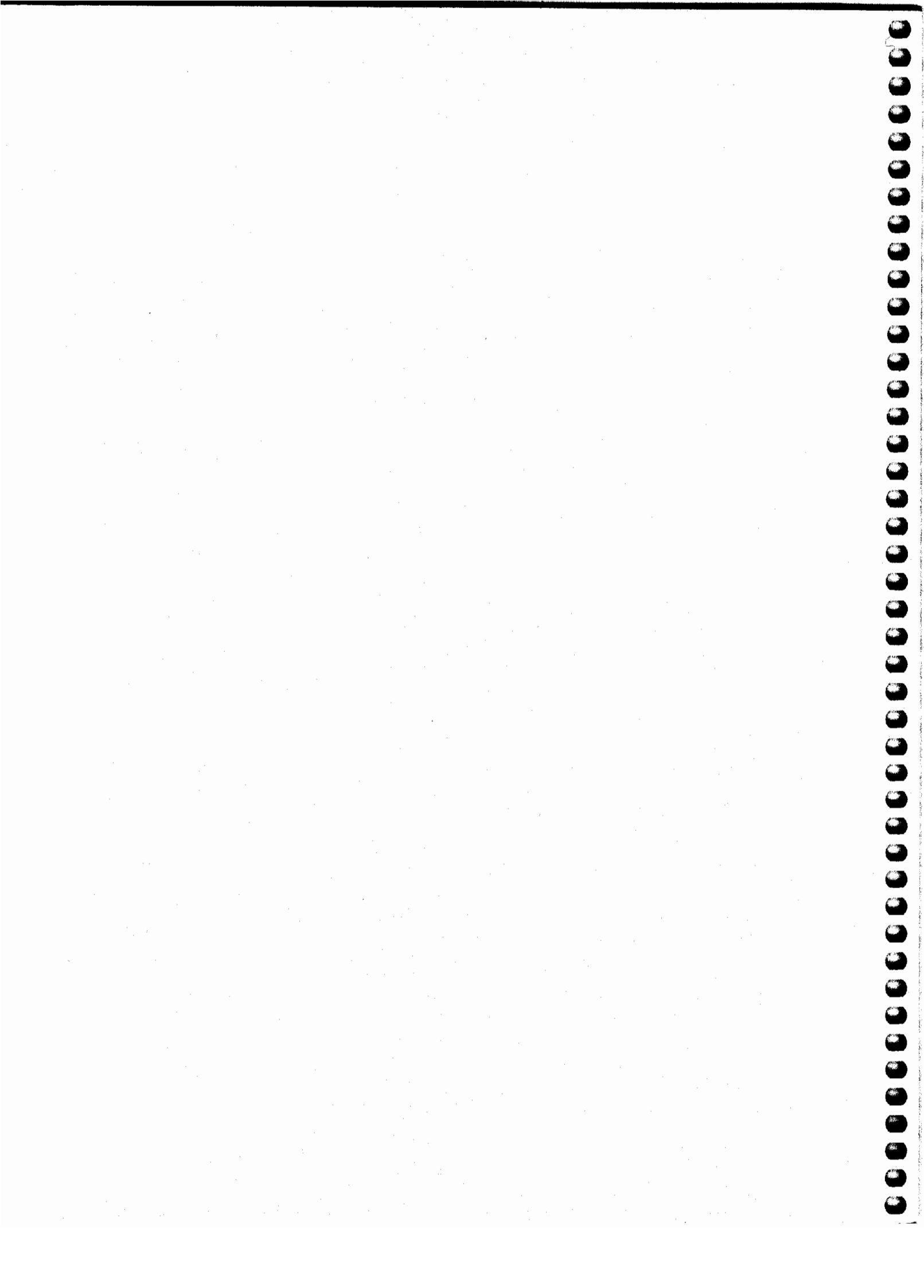
The Detal reactor section effluent proceeds to a series of fractionation columns where the various remaining reactants and the products are separated. The unreacted benzene is recovered in the benzene column and recycled back to the Detal reactor. The benzene column also separates the heavy by-products from the regenerant benzene. The unreacted n-paraffins are recovered in the recycle paraffin stripper and in the paraffin column and recycled back to the Pacol unit. The LAB product is then separated from the heavy alkylate (HAB) by-product and these two streams are sent to the respective storage tanks. Clay treatment is generally used to improve the quality of the LAB. Typical examples of the Detal unit process flow are shown in Figures IV-1 and IV-2.

The product composition and quality is a function of various operating parameters which we will discuss in more detail in the following pages.

Figure I - 1
LAB Complex





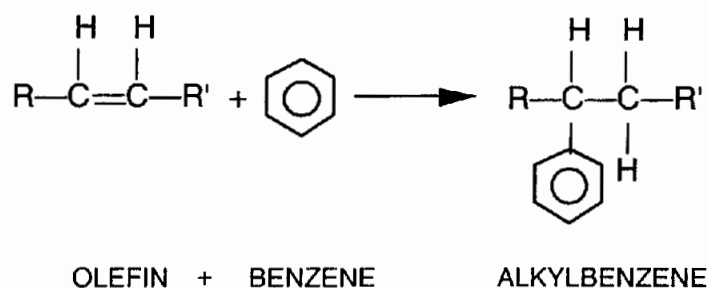


II. PROCESS PRINCIPLES

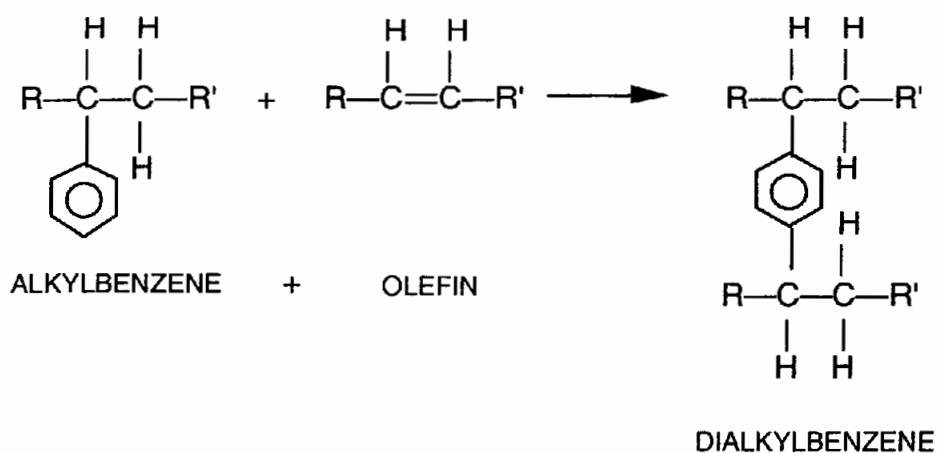
A. CHEMISTRY

The following describes various alkylation reactions based on linear olefins:

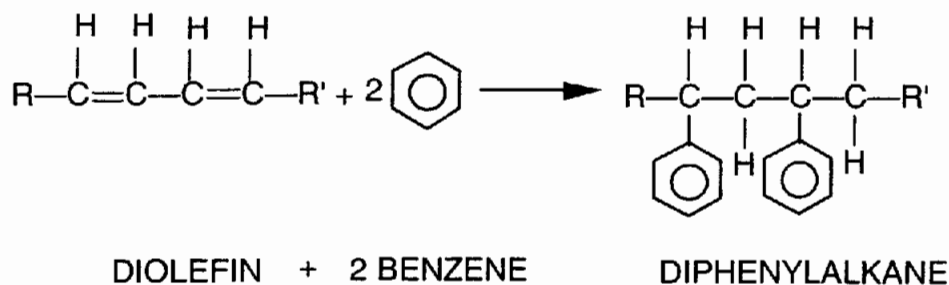
1. The main reaction is the alkylation of benzene with the linear chain olefins to make linear alkylbenzene (LAB). Of course, this is the desired reaction. Process conditions are adjusted to maximize this reaction and to minimize the side-reactions.



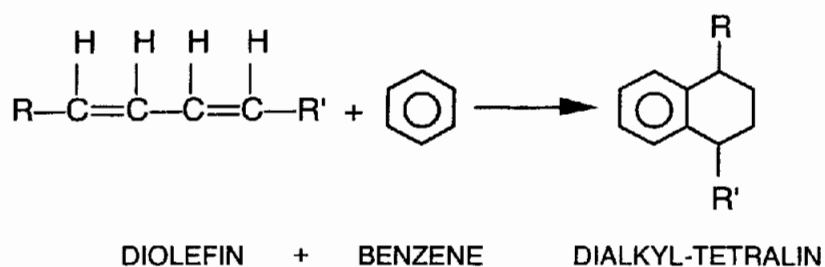
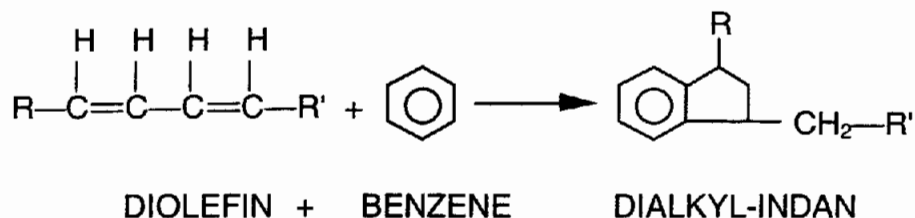
2. At the existing conditions, some undesirable side-reactions take place, like the reaction of LAB with olefin to make dialkylbenzene. Dialkylbenzene is heavy alkylate (HAB) which has a significantly lower value than LAB.



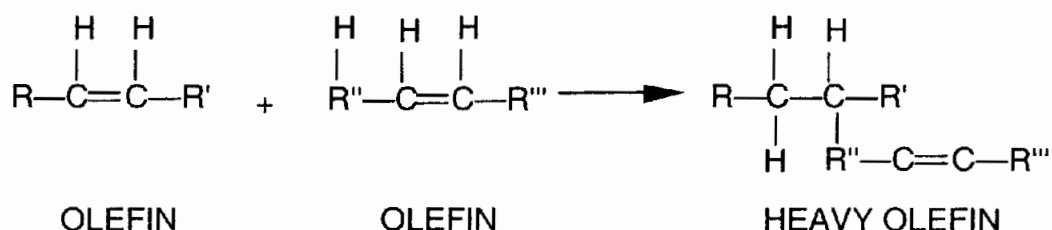
3. Another undesirable side-reaction is that of a diolefin molecule reacting with two benzene molecules to form a diphenylalkane. Diphenylalkanes are also heavy alkylate (HAB) and have a significantly lower value than LAB.



4. A diolefin molecule can also react with just one benzene molecule to form an indan or tetralin. Indans and tetralins wind up in the LAB product but they are undesirable because they are generally thought to have poorer biodegradability than linear alkylbenzenes.



5. Another undesirable side-reaction is when olefins react with other olefins to form heavy olefins.



These heavy olefins can then further react with olefins, benzene, or alkylbenzene to form heavy polymers. Some of these heavy polymers will pass through the reactor and wind up in the HAB product stream. This is undesirable because HAB has a lower value than LAB. An even more undesirable result is that some of these heavy polymers tend to stick on the catalyst, blocking the active sites on the catalyst. This reduces the activity of the catalyst. At normal operating conditions, most of these polymers can be removed by regenerating the catalyst with a hot benzene wash every 24 hours. However, if the amount of polymer formation is excessive, the polymer may become so heavy that it cannot be washed off the catalyst and the catalyst becomes permanently deactivated.

B. CATALYST

HF acid has been the principal catalyst for the production of LAB from linear olefins. However, the handling of corrosive catalysts such as HF acid have always had some disadvantages such as; higher equipment capital cost, and disposal of small quantities of neutralization products generated by the process. The advantage of a heterogeneous non-corrosive catalyst for this process has long been recognized.

Aromatic alkylation has been demonstrated over many solid acids such as; clay minerals, zeolites, metal oxides, and sulfides. Many of these catalysts are highly active, but they are usually lacking in selectivity or stability. Research at CEPSA and UOP has resulted in the development of a catalyst that is active, selective, and

stable over prolonged periods of operation. The Detal process (developed jointly by CEPSA and UOP) utilizes this catalyst in a fixed bed reactor. The reaction takes place at relatively mild conditions in the liquid phase.

The Detal catalyst is very active and selective for the production of LAB from n-olefins and benzene. It has been specifically developed so that its high activity can be maintained by a simple periodic solvent regeneration. Expected life of the catalyst is 2 years or more.

During normal operation, heavy tar-like polymers build up on the surface of the catalyst. These polymers tend to reduce the activity of the catalyst. A periodic solvent regeneration (using hot benzene) is used to remove these polymers. If the polymers are allowed to build up for too long or too quickly, it can be very difficult to remove them. This could lead to a permanent reduction in the activity of the catalyst.





III. PROCESS VARIABLES

A. PROCESS VARIABLES

1. Benzene/Olefin Ratio

The molar ratio of benzene to olefin in the feed to the Detal reactor is an important process variable. The benzene/olefin ratio affects both yield and product quality. The higher the ratio of benzene to olefin, the more likely it is for the olefin molecules to react with benzene (forming LAB) instead of reacting with something else (forming unwanted by-products).

A decrease in the benzene to olefin ratio increases the formation of heavy alkylate, reducing the yield of LAB product. A decrease in the benzene to olefin ratio also tends to increase the bromine index and decrease the linearity of the LAB product. A drastic decrease in benzene to olefin ratio could cause catalyst deactivation due to excessive tar formation.

Increasing the benzene to olefin ratio improves both yield and product quality, but it also increases operating cost. The recirculated benzene is all taken as fractionator overhead and this requires a large amount of heat. The economic trade-off between yield and utility cost must be considered in order to optimize the benzene to olefin ratio.

There are two different basic Detal reactor designs. The benzene/olefin ratio is significantly different in the two designs.

a) Split Feed Detal Reactor

In the Split Feed design, the reactor is divided into three beds with all of the benzene flowing through each bed in series, and one-third of the olefin feed going to the inlet of each bed. All of the olefin in the feed to each bed is reacted before leaving that bed so no olefin is carried over into the feed to the next bed. Splitting

the olefin feed in this manner increases the effective benzene to olefin ratio by a factor of about 3 with no increase in utility cost for the circulating benzene. In a typical Split-Feed design, the overall benzene to olefin ratio is 15, but the effective benzene to olefin ratio at the inlet to each bed is actually about 45. The drawback to this design is that in the lower two beds, the ratio of LAB to benzene increases. The olefin can react with the benzene ring on the LAB molecule in the same way as it can react with the benzene molecules themselves, and this reaction of an olefin with LAB produces dialkyl benzene which ends up in the HAB byproduct – thus reducing the LAB yield.

b) Single Feed Detal Reactor

In the Single Feed design, all of the olefin feed is mixed with the recycle benzene and this mixture is the single feed to the top bed of the reactor. A second reactor bed may be used in this design to “finish off” the reaction at a higher temperature, but no additional olefin feed is injected to the second bed. A higher flow rate of recycle benzene is required for this design compared to the Split Flow design. In a typical Single Feed design, the overall benzene to olefin ratio is about 30. However, most of the heat required for the additional recycle benzene can be recovered from waste heat in the fractionation system so the utilities cost is about the same as for the Split Feed design. The main advantage of the Single Feed design is lower HAB production due to the lower ratio of LAB to benzene in the part of the reactor where olefins are present. A second advantage of the Single Feed design is that the total amount of catalyst in the reactor is a bit smaller compared to the Split Feed design.

2. Temperature

Reactor temperature is another important process variable. Reactor temperature affects conversion and product quality. The Detal unit will typically run at a reactor inlet temperature between 120°C and 160°C.

Higher reactor temperature increases the reaction rate. As a result, an increase in reactor temperature will decrease the bromine index of the reactor effluent because more of the bromine-reactive compounds will have a chance to react before leaving

the reactor. An increase in the reactor temperature will also increase the reaction rate of the isomerization of n-olefin to iso-olefin. This isomerization reaction decrease the linearity of the LAB product. As a rough estimate, increasing the reactor inlet temperature by 10°C will decrease the LAB linearity by about 1%.

If the reactor temperature is decreased beyond a certain point, the conversion will drop from 100% and olefin breakthrough from the outlet of the reactor will occur. When the reactor temperature decreases enough to cause olefin breakthrough, there is a possibility of tar formation in the reactor which could damage the catalyst. If the reactor temperature is adjusted such that the LAB bromine index is less than 20, this will be sufficient to prevent olefin breakthrough.

The alkylation reaction is exothermic – it produces heat. As the olefin reacts inside the catalyst bed the temperature increases, so the temperature at the outlet of the catalyst bed is higher than the inlet temperature.

In Split Feed reactor designs, the temperature of the olefin feed to the middle and bottom beds in the reactor is controlled so that the combined feed to the middle and bottom beds is cooled to the same temperature as the combined feed to the top bed. This gives a more uniform reaction temperature in the three catalyst beds.

In Single Feed reactor designs that have a small “finishing bed” at the bottom of the reactor, a heater is used to increase the temperature of this finishing bed. Higher temperature in this “finishing bed” gives a reduction in the bromine index without decreasing the linearity of the LAB product. The reason that the higher temperature does not decrease the linearity of the LAB is because there is no olefin present in this “finishing reactor” and it is the isomerization of the olefin that causes the decrease in linearity at higher temperature. Once a molecule of linear alkylbenzene is formed, it does not isomerize to iso-alkylbenzene inside the Detal reactor at any temperature.

As the catalyst gets older, the activity will slowly decrease. It will be necessary to gradually increase the reactor temperature as the catalyst ages to compensate for this gradual loss of activity.

3. Liquid Hourly Space Velocity

Liquid hourly space velocity (LHSV) is defined as the liquid feed rate to the reactors divided by the catalyst volume. The design overall LHSV is about 2.0/hr for Split Feed designs, and about 3.75/hr for Single Feed designs with a finishing bed. An increase in LHSV will require a slightly higher reactor temperature to maintain the same conversion and bromine index.

4. Pressure

The reactor pressure has essentially no effect on the reaction as long as it is high enough to keep the hydrocarbon in the liquid phase. It is very important to make sure there is no vapor inside the reactor for several reasons.

- a) The reactors are not designed for mixed phase flow. Severe flow maldistribution will be caused by vapor in the feed to the reactor.
- b) If some of the hydrocarbon vaporizes inside the reactor, the catalyst bed could be disturbed. This will cause permanent flow maldistribution (even after returning to 100% liquid phase conditions). If the support balls at the bottom of the reactor are disturbed, catalyst can leak out of the bottom of the reactor.
- c) If vapor accumulates in the top of the reactor, serious pressure upsets can occur during the reactor switching sequence.

5. Cycle Time

During operation, heavy tar-like polymers build up on the catalyst. In order to prevent deactivation of the catalyst by this material, it is necessary to periodically clean the catalyst with a hot benzene regeneration. The cycle time is the amount of time that a reactor is on-line between benzene regenerations. The Detal unit is designed for a cycle time of 24 hours. Exceeding 24 hours cycle time could result in diminished activity and poor product quality near the end of the cycle. Permanent loss in activity could result from radically excessive cycle times or repeatedly exceeding the design cycle time.

6. Water

The presence of water will reduce the activity of the catalyst. Small concentrations of water in the feed (in the 1 to 10 ppm range) normally only act as temporary poisons to the catalyst. Once the water level in the feed is returned to the normal low level, the small amount of water desorbs off of the catalyst and the activity normally is recovered. However, larger amounts of water, especially if present as a separate phase, will permanently deactivate the catalyst.

Water analyzers are provided on the reactor feed and effluent streams. The reactor feed streams should be kept below 0.1 to 0.2 ppm water. Under normal circumstances, there should be very little water going to the reactor because both the paraffin and benzene feed are stripped before entering the unit.

B. CATALYST POISONS

A catalyst poison is something that deactivates the catalyst. A loss of catalyst activity will cause a significant increase in the bromine index of the reactor product due to breakthrough of olefins. Depending on the severity, it may be possible to compensate for the activity loss by increasing the reactor temperature. However, this will tend to reduce the linearity of the LAB product.

1. Water

Water can act as either a temporary or a permanent catalyst poison. The water content of the reactor feed streams (including the regenerant) must be kept below 1 ppm (by weight), and normally it should be possible to keep the water below 0.1 to 0.2 ppm.

At relatively low concentrations (less than 10 ppm in the feed), water tends to act as a temporary poison. In this case, the water adsorbs on the catalyst and decreases the activity because it affects the acid function of the catalyst. Once the water has

been eliminated from the feed, water will slowly desorb off of the catalyst and the activity will return to normal.

Contact with higher concentrations of water, especially water in the liquid aqueous phase, will permanently deactivate the catalyst. In this case, fluoride is chemically displaced from the catalyst by the water. This type of deactivation is permanent.

2. Polymer

Typically there will not be any heavy polymers in the feed to the Detal unit. However, due to the presence of olefins, and the action of the acid function catalyst, there is a potential to form polymers in the Detal unit. Heavy polymers can build up on the catalyst, blocking the active sites. This will reduce the activity of the catalyst. Such a loss in activity can be either temporary or permanent, depending on the amount of polymer and the manner in which it was formed.

The high benzene/olefin ratio helps prevent excessive polymer formation in the Detal unit. The Detal catalyst promotes the reaction of olefins. The presence of a large excess of benzene makes it more likely that the olefins will react with benzene rather than other olefins. If there is not enough benzene present in the reactor, the olefins will react with other olefins, forming long polymer molecules. These polymers will adsorb on the catalyst, reducing the activity. In mild cases, the polymer can be removed with hot benzene during a normal regeneration cycle. However, in severe cases, it may not be possible to remove all of the polymer, and some permanent catalyst deactivation will result.

Low Detal reactor temperature, combined with the presence of olefins (even with normal benzene/olefin ratio), can also cause excessive polymerization. Low reactor temperatures with olefin in the reactor must be avoided.

3. Heavy Hydrocarbons

Heavy hydrocarbons are usually not present in the Detal unit feed. If they do appear in the feed, they would affect the catalyst in the same way as heavy polymers.

Heavy hydrocarbons (such as paraffins, LAB, and HAB) can be present in the regenerant if the benzene column is upset. These heavy hydrocarbons can permanently damage the Detal catalyst at regeneration conditions. The regeneration is performed at a much higher temperature than the alkylation reaction, and the heavy hydrocarbons will tend to polymerize and permanently attach to the catalyst at the high temperature (similar to coke).

4. Diolefins

Diolefins tend to form heavy alkylate and polymer. This is due to the presence of two reactive olefinic bonds instead of just one. Increased polymer formation will cause catalyst deactivation as described above. Proper operation of the DeFine unit will reduce the diolefin concentration to an acceptable level.

5. Oxygenates

Oxygenates (such as alcohols, glycols, ketones, and ethers) are typically not present in the feed to the Detal unit but would be poisons to the catalyst. Oxygenates can break down to form water in the reactor. The water will then act as a catalyst poison as described above. Some oxygenates are strongly polar compounds, which can be strongly adsorbed on the catalyst, causing deactivation as described below.

6. Polar Compounds

Polar compounds tend to adsorb on the catalyst. If the polar attraction is strong enough, the compound will not desorb from the catalyst during regeneration. In this case there will be some permanent deactivation of the catalyst due to physical

blocking of the active catalyst sites by the adsorbed polar compound. Such strongly polar compounds are not usually present in the Detal unit. Glycols and most corrosion inhibitor chemicals are strongly polar, and should be avoided.

7. Nitrogen Compounds

Nitrogen compounds are typically not present in the Detal unit. Basic nitrogen compounds (such as amines) modify the acid function of the catalyst causing permanent deactivation. One potential source of nitrogen compounds is the injection of corrosion inhibitors.

8. Sulfur

Sulfur (in the form of H_2S) tends to act as a temporary poison - similar to low levels of water. The low levels of H_2S that might come from the DeFine unit (less than 1 ppm) are not a problem. If there is an upset of H_2S in the feed to the unit, the H_2S should slowly desorb from the catalyst once the H_2S is removed from the feed.

Heavier organic sulfur compounds are not normally present, but may cause deactivation due to an increase in polymerization of the olefins in the reactor.

9. Chloride

Chloride is not expected in the Detal feed, but small amounts should not be a problem. Larger amounts of chloride could affect the acid function of the catalyst.

10. Metals

There should not be any metals in the feed to the Detal unit. However, most metals would be expected to cause permanent catalyst damage. The alkaline metals (Na, K, Ca, Mg) are expected to be the worst due to their potential neutralizing effect on the acid function of the catalyst.





IV. PROCESS FLOW AND CONTROL

A. GENERAL

The basic process flow for a Split-Feed Reactor type of Detal unit is shown in Figure IV-1 and the basic process flow for a Single Feed type of Detal unit is shown in Figure IV-10. In either type of unit, a C_{10} - C_{13} or C_{11} - C_{14} n-paraffin stream containing about 13% n-olefins is produced in the Pacol unit. This paraffin/olefin feed is mixed with an excess of benzene and fed to one of the two Detal reactors where the olefins react with the benzene to produce linear alkyl benzene (LAB). The reactors are switched daily and the off-line reactor is regenerated with hot benzene to remove the heavy hydrocarbons that build up on the catalyst. Both the reaction effluent and the regeneration effluent go to the benzene column where the excess benzene is removed and recycled to the reactors. The bottoms of the benzene column go to the paraffin column where the paraffin is taken overhead and recycled to the Pacol unit. In many units (Figure IV-10), a recycle paraffin stripper is used upstream of the paraffin column to recover a large portion of the recycle paraffin. The recycle paraffin stripper operates at a higher pressure than the paraffin column which allows recovery of heat from the recycle paraffin stripper overhead for use in the middle reboiler in the benzene column.

In general, two different flow schemes have been used to separate and treat the LAB and HAB that is contained in the paraffin column bottoms.

1. In one scheme (Figure IV-1), the paraffin column bottoms stream is fed to the rerun column where the linear alkyl-benzene (LAB) product is taken overhead. A slipstream of LAB is taken from the rerun column, fed to a clay treater to reduce the bromine index, and then sent back to the benzene column to remove the benzene generated in the clay treater. The rerun column bottoms is sent to the recycle column to maximize the recovery of LAB from the rerun column bottoms. The heavy alkyl-benzene (HAB) byproduct is taken from the bottom of the recycle column, and the overhead is sent to a clay treater and then recycled

back to the paraffin column. The advantage of this first scheme is that the final separation of LAB and HAB is done in the recycle column where the operating conditions are very stable thus minimizing the loss of LAB into the HAB product stream.

2. In the second scheme (Figure IV-10), the paraffin column bottoms is fed to the LAB column. The heavy alkyl-benzene (HAB) byproduct is taken from the bottom of the LAB column and sent to storage. The LAB column overhead is sent to a clay treater to reduce the bromine index, and then is fed to the finishing column. The small amount of benzene produced in the clay treater is stripped overhead in the finishing column and is recycled back to the benzene column along with some excess LAB that is also sent overhead in the finishing column. The LAB product is taken from the bottom of the finishing column. The advantage to this second scheme is that all of the LAB product is clay treated thus minimizing the bromine index of the LAB product.

B. REACTOR SYSTEM

There are two different basic reactor designs.

1. Split Feed Reactor

In the Split Feed reactor design (Figure IV-2), there are two identical Detal reactors, each with 3 catalyst beds. The paraffin/olefin feed from the Pacol unit is divided into 3 equal streams. One-third of the paraffin/olefin feed is mixed with the entire flow of recycle benzene and is fed to the top bed in the reactor. All of the olefin in the paraffin/olefin feed to the top bed reacts in the top bed. The effluent from the top bed then exits the reactor, is mixed with the second one-third of the paraffin/olefin feed and is fed to the middle bed of the reactor. Again, the olefin is completely reacted in the middle bed. The effluent from the middle bed exits the reactor, is mixed with the final one-third of the paraffin/olefin feed, and is fed to the bottom bed of the reactor where the last of the olefin is reacted. This split-feed olefin,

series-flow benzene scheme gives roughly three times the effective benzene to olefin ratio (compared to a one-bed scheme) for a given flow of recycle benzene. So the advantages of higher benzene/olefin ratio are obtained without an increase in utility cost due to higher recycle benzene flow rate.

The reactor effluent meets with the regenerant effluent and is fed to the benzene column. The reactor effluent back-pressure valve (on the benzene column feed line) keeps the hydrocarbon in the reactor in the liquid phase.

The paraffin/olefin feed from the Pacol unit goes through a heat exchange system before mixing with the benzene and going to the reactors (see Figure IV-2). The relatively hot paraffin/olefin feed from the Pacol stripper bottoms must be cooled to give the proper reactor feed temperature. Most units will also have a heat exchange system on the recycle benzene stream to the reactor. The temperature of the feed to the top bed must be adjusted to the desired reactor inlet temperature. In order to compensate for the exothermic heat of reaction, the temperature of the paraffin/olefin feed to the middle and bottom beds must be adjusted (cooled) so that the mixture going to the middle and bottom beds is at the desired reactor inlet temperature. Normally the inlet streams to all three reactor beds are adjusted to be at the same temperature.

The alkylation reaction is exothermic, so the temperature of the paraffin/olefin feed to the middle and bottom beds will have to be lower than the desired reaction temperature in order to remove the heat of reaction. The complexity of the heat exchange system is necessary in order to maximize the recovery of heat from the paraffin/olefin feed and to allow operation over a wide range of reactor inlet temperatures (120°C-160°C). There will be a trade-off between operability and heat recovery. The exchanger bypasses should be adjusted to maximize heat recovery, while still providing sufficient operability to allow good control of the reactor temperatures. Depending on the reactor temperature, it is possible that one or more of the heat exchangers will be bypassed or shut down.

2. Single Feed Reactor

In the Single Feed reactor design (Figure IV-11), there are two identical Detal reactors. Typically each reactor will have two beds, and the top bed will be much larger than the bottom bed. All of the paraffin/olefin feed from the Pacol unit will be mixed with all of the recycle benzene stream and then fed to the top bed. All of the olefin in the paraffin/olefin feed to the top bed reacts in the top bed. The effluent from the top bed then exits the reactor, is heated up with hot oil in an exchanger, and is fed to the bottom bed of the reactor. The bottom bed is used to "finish off" the reaction at a higher temperature to minimize the bromine index at the reactor outlet. No additional olefin feed is injected to the bottom bed. Since there is no olefin present in the bottom bed, the higher temperature does not decrease the linearity of the LAB product. This single feed design requires a higher flow rate of recycle benzene than the split-feed design, but it gives a lower HAB yield and allows a small reduction in the total amount of catalyst in the reactors. The additional heat required for the additional recycle benzene required for this scheme can be recovered from waste heat in the recycle paraffin stripper overhead.

In this scheme, the reactor effluent is not mixed with the regenerant effluent. Instead, it is fed to the benzene column separately from the regenerant effluent at a different tray in the column. The reactor effluent back-pressure valve keeps the hydrocarbon in the reactor in the liquid phase.

The heat exchange scheme for the Single Feed reactor is much simpler than for the Split Feed reactor. A single feed heater is used to adjust the entire combined feed stream to the top bed of the reactor. Typically, the heat for this reactor charge heater is taken from the overhead of the recycle paraffin stripper.

The reactor effluent is heated in an exchanger by the regenerant effluent stream then the reactor effluent is fed to the benzene column. (Note that the regenerant effluent is also fed to the benzene column, so it may appear that this heat exchanger is not necessary. However, the reactor effluent and regenerant effluent are fed in different parts of the column, and the overall heat consumption in the

column is significantly less when the reactor effluent is heated with the regenerant effluent.)

There is a regenerant effluent heater that is used to maintain a constant temperature on this stream which supplies heat to the reactor effluent heater. This makes the benzene column operation more stable and prevents the need for adjustment of the benzene reboiler heat input during the regeneration cycle. The regenerant effluent temperature controller is set so that hot oil only flows through it during the colder portion of the regeneration cycle when the regenerant effluent is at low temperature. Note that there will be a lower flow of hot oil to the regenerant heater during this time, so the overall load on the hot oil system does not change very much. (It may also seem that the regenerant effluent heater is not necessary. However, during the part of the regeneration cycle when the regenerant effluent is at low temperature, it is necessary to replace the heat to the benzene column. Using the regenerant effluent heater to supply this heat allows for very stable control of the benzene column without the use of switching valves or a larger size exchanger in another location.)

3. Reactor Operation (applies to both types of reactors)

During normal operation, when there is olefin in the feed to the reactors, it is much better for the catalyst if the reactor temperature is a little too high rather than a little too low. Higher than required reactor feed temperatures (within reasonable limits) will only cause a small decrease in linearity of the LAB product and will not damage the catalyst. Low reactor feed temperatures will cause increased polymerization of the olefins and the additional polymer will accelerate the deactivation of the catalyst. Depending on the amount of polymer deposited on the catalyst, the hot benzene regeneration may not be able to restore the full activity of the catalyst. This increased polymerization only takes place when there are olefins in the feed to the reactor, so low reactor temperature is not a problem when the unit is on startup circulation or other times when only paraffin and/or benzene is in the reactor feed.

Although there are two Detal reactors, only one is on-line at a time. During operation, small amounts of heavy, tar-like, polymer material builds up on the surface of the catalyst. It is necessary to switch the reactors daily to remove this heavy material from the catalyst using a hot benzene regeneration procedure. An automatic valve sequencer controls the switching of the reactors to minimize upset to the unit.

C. REGENERATION SYSTEM

After about 24 hours of operation, the Detal catalyst must be regenerated with a hot benzene wash (Figure IV-3 or Figure IV-11). Benzene from an upper accumulator tray of the benzene column is used as regenerant. The benzene regenerant is pumped through the regenerant heater where it is heated to 250°C before entering the reactor. The hot regenerant passes through the reactor, where it removes heavy hydrocarbons from the surface of the catalyst, and then goes to the regenerant effluent exchangers and eventually into the benzene column.

A control valve on the regenerant effluent line is used to control the flow of regenerant. This control valve is also reset (via a low signal selector) by a pressure transmitter on the regenerant effluent line. The pressure controller will be set at a pressure sufficient to keep the regenerant in the liquid phase at the maximum regeneration temperature. The system is designed so that the flow controller will normally send the low signal to the low signal selector, so the control valve will work primarily as a flow controller. However, if the pressure decreases below the set point of the pressure controller, the control valve will be closed by a lower signal from the pressure controller.

The general procedure for regeneration is to switch reactors, flush the reactor with unheated benzene, slowly heat-up the reactor to 250°C, regenerate the catalyst with benzene at 250°C for several hours, cool-down the catalyst to approximately 5°C above the desired reactor inlet temperature, and finally switch the reactors and start the whole procedure over again on the other reactor. It is important not to heat

up too soon or too quickly. Removing as much heavy material as possible at lower temperature will help prevent permanent catalyst deactivation.

Two different heat exchange schemes have been used for the regenerant.

1. In the first scheme (Figure IV-3), the benzene regenerant is pumped through the regenerant exchanger, where it is heated by the regenerant effluent, then goes to the regenerant heater where it is heated to 250°C before entering the reactor. After the hot regenerant passes through the reactor, it then goes to the regenerant exchanger where it is cooled by the fresh regenerant. The regenerant then passes through the regenerant effluent heater (which is only used during cool-down), the regenerant flow control valve, and finally goes to the alkylation mix drum where it mixes with the effluent from the on-line reactor before going to the benzene column. The alkylation mix drum helps smooth out the composition changes in the feed to the benzene column during reactor switching. In this scheme, the regenerant heater is only used during the heat-up and 250°C hold steps, and the regenerant cooler is only used during the cool-down step. The regenerant heater and regenerant cooler are never used at the same time. The regenerant effluent heater is only used during the cool-down step to replace the heat that is removed by the regenerant cooler. The reboiler in the benzene column could not handle the additional duty required if the regenerant effluent temperature were allowed to decrease significantly. The regenerant benzene flow is run continuously (bypassing the reactors when necessary) to keep the operation of the benzene column constant.
2. In the second scheme (Figure IV-11), the benzene regenerant is pumped directly to the regenerant heater where it is heated to 250°C before entering the reactor. After the hot regenerant passes through the reactor, it then goes to the regenerant effluent heater (which is used during cool-down and low temperature periods), the reactor effluent heater, the regenerant flow control valve, and finally goes to the

benzene column. In this scheme, the regenerant heater is only used during the heat-up and 250°C hold steps. The regenerant effluent heater is only used during the cool-down step to replace the heat that is no longer being supplied by the regenerant heater. The reboiler in the benzene column is not designed to handle the additional duty required if the regenerant effluent temperature were allowed to decrease significantly. The regenerant benzene flow is run continuously (bypassing the reactors when necessary) to keep the operation of the benzene column constant.

D. BENZENE COLUMN

The benzene column (Figure IV-4 or Figure IV-12) is actually 2 columns in one. The top section of the column is a stripper for the make-up benzene; water and dissolved non-condensibles are removed in this section of the column. The bottom section of the column separates the benzene from the heavier hydrocarbons in the reactor and regenerant effluent streams.

The overhead vapor from the benzene column is condensed in an air-cooled condenser before going to the overhead receiver. In cold-weather locations, an air recirculation system is used on the condenser to prevent solidification of benzene at low exchanger surface temperatures. Any non-condensibles in the benzene column overhead will remain in the vapor space in the overhead receiver.

The benzene column pressure is typically controlled in one of two ways:

1. A butterfly type control valve on the overhead vapor line which is reset by the pressure above the benzene accumulator tray. In this scheme, the overhead receiver floats on the relief header pressure. A nitrogen purge into the receiver off-gas line prevents vapors from the relief header from backing into the receiver. (See Figure IV-4).

2. A split range control system with a control valve on the nitrogen purge to the receiver and a second control valve on the off gas line to the relief header. These control valves are reset by the pressure in the overhead vapor line. The split range control system is set up so that only one of the two control valves will be open at any given time. (See Figure IV-12).

Water is stripped overhead in the benzene column. If the water concentration in the overhead liquid in the receiver reaches saturation, free water will separate out and collect in the water boot on the receiver. A proportioning pump is used to pump the water to the waste water treating facilities. The proportioning pump stroke is reset by the water interface level in the receiver boot.

The overhead receiver acts as the surge vessel for the benzene inventory in the unit and the make-up benzene to the unit is fed to the receiver on the level control. Reflux from the receiver is pumped to the top tray of the benzene column and this top reflux flow is reset by the benzene level on the recycle benzene accumulator tray.

The size of the benzene column overhead receiver is relatively small compared to the hydrocarbon inventory of the reactor section. Any change in the proportion of benzene to paraffin/olefin in the reactor section (such as due to a change in benzene/olefin ratio or a loss of paraffin/olefin feed) will have a drastic effect on the level in the receiver. If the receiver becomes completely full or empty, the column will be badly upset, possibly causing catalyst damage due to water breakthrough or heavies in the regenerant. In addition, if the receiver goes completely empty, the accumulator tray will also go empty and recycle benzene will be lost.

A benzene drag stream can be taken from the overhead receiver and sent to the PEP unit (depentanizer or PEP splitter) to recover the pentane that slips into the feed to the Detal unit from the PEP unit. Any pentane that gets into the Detal unit will accumulate in the benzene column overhead receiver. If the concentration of pentane in the overhead receiver builds up to more than about 5%, the vapor pressure of the liquid in the overhead receiver will become high enough to generate a continuous off-gas flow that will cause the loss of both pentane and benzene.

This drag stream will contain dissolved water, so it must be dried before it is used in the PEP unit. The PEP unit will have either a molecular sieve dryer system, or a drying section on the depentanizer or PEP splitter column to remove the water contained in this drag stream. During normal operation, the flow rate of the drag stream to the PEP unit (and the amount of dissolved water in that stream) is sufficient to prevent free water from accumulating in the benzene column overhead receiver.

The benzene drag stream from the overhead receiver can also be sent to slops in order to remove impurities from the recycle benzene stream. Certain impurities (such as cyclohexane, which cannot be fractionated from benzene and is not consumed in the process) may come into the unit in the make-up benzene stream and can build up in the recycle benzene stream. Normally it is not necessary to send the benzene drag stream to slops as long as the make-up benzene purity is reasonably good (e.g. benzene from a Sulfolane unit).

The feed to the benzene column is comprised of both the reactor effluent and the regenerant effluent. In some units, these two streams are combined and pass through an alkylation mix drum which helps smooth out any sudden composition changes which might occur during the daily reactor switching. The benzene column feed then passes through a back-pressure control valve where a significant portion of the feed flashes before entering the column. The back-pressure control valve must be set at a high enough pressure to prevent vaporization of the liquid in the reactor and the mix drum. In other units, the reactor effluent and regenerant effluent are fed separately to the benzene column on different trays. The reactor effluent passes through a back-pressure control valve which is set to prevent vaporization of the liquid in the reactor. The reactor effluent flashes as it passes through the back-pressure valve and is then further vaporized in the reactor effluent exchanger. In all unit designs, the flashing of the feed to the benzene column is necessary because the reboiler is not capable of supplying sufficient heat for proper column operation unless a significant portion of the feed is vaporized.

The benzene column can have either a stab-in type reboiler (Figure IV-4) or an external type reboiler (Figure IV-12). In either case, the reboiler is heated by a controlled flow of circulating hot oil. In the stab-in and kettle-type reboilers, all of the liquid from the bottom tray in the column flows into a reboiler tub which keeps the reboiler tubes covered with liquid. During startup (and other times when column operation is not steady), it is necessary to open the spillback line from the discharge of the benzene column bottoms pump to the reboiler tub to insure that the reboiler tubes are always covered with liquid. During normal operation the spillback line should be closed because there is enough liquid flow through the tub that the spillback is not necessary. External reboilers with a horizontal thermosiphon type design do not have a tub and do not need the spillback line to the tub because the tubes will always be covered with liquid as long as there is a liquid level in the bottom of the column.

In some benzene columns (Figure IV-12), a middle reboiler is used to recover heat from the recycle paraffin stripper overhead liquid circulation. This is a stab-in type reboiler and there is a tub inside the column to keep the tubes covered with liquid. It is located about 20 trays above the bottom of the column where the temperature is low enough to recover heat from the circulating recycle paraffin stripper overhead liquid. Typically, the middle reboiler is operated to maximize heat recovery from the recycle paraffin overhead liquid, and minimize the heat input to the lower reboiler, while still keeping enough total heat input to the column to make a good split between the benzene and the paraffin.

The paraffins and heavier hydrocarbons (LAB and HAB) are taken from the bottom of the benzene column and this stream is the feed to the recycle paraffin stripper (or directly to the paraffin column in units that do not have a recycle paraffin stripper). The net flow of benzene column bottoms is reset by the liquid level in the bottom of the column.

The benzene contained in the feed to the column travels up the column to be drawn off from one of the accumulator trays near the top of the column. Some units just have a single benzene accumulator tray which supplies regenerant, PEP desorbent, and recycle benzene (Figure IV-4). Other units have two separate

accumulator trays; an upper one for the regenerant and PEP desorbent, and a lower one for the recycle benzene (Figure IV-12). Using a lower tray for the recycle benzene saves some energy (by using a lower reflux ratio in the lower part of the column) while still making sure that there are no heavy hydrocarbons in the regenerant and desorbent streams. If an upper accumulator tray is used, it is an overflow type tray where any liquid that is not drawn off as regenerant or PEP desorbent simply flows down to the tray below. No matter whether there is one or two accumulators, some of the liquid from the recycle benzene accumulator tray is pumped back to the column just below the accumulator tray as reflux to the lower section of the column. The reflux flow is reset by the temperature on an intermediate tray lower down in the column. This intermediate tray temperature is an indication of the composition of the net overhead and net bottoms streams.

Recycle benzene is taken from the lower accumulator tray and is pumped on flow control to the reactors. The recycle benzene flow rate is set to give the desired benzene/olefin ratio for the reactors.

Regenerant benzene is taken from the upper accumulator tray and pumped to the regeneration system. A separate pump is used for the regenerant because of the higher pressure required. PEP desorbent is also taken from this tray and is pumped to the PEP unit either with the same pump as the regenerant, or with a separate pump.

Finally, benzene from the accumulator tray can be sent to the off-spec tank. This is useful during startup and shut down operations when it is necessary to remove some or all of the benzene from the unit. Material from the off-spec tank is recovered by feeding it to the benzene column. A flow indicator is provided to meter the flow rate of off-spec material to the benzene column. This flow indicator can also be used to meter the flow rate of hydrocarbon being purged from the reactors during shut down. A bypass for the off-spec material around the flow meter is provided for occasions when a high flow rate of off-spec material is required (such as during startup inventory operations).

Note that any water or non-condensibles in the feed to the benzene column (including feed from the off-spec tank) will tend to pass from the lower section of the benzene column to the upper section and will be removed in the overhead receiver. Any water, dissolved oxygen or other non-condensibles in the make-up benzene will also be stripped overhead in the upper section of the benzene column because the make-up benzene is fed to the overhead receiver and must travel down to the accumulator tray before going to the reactors.

It is important to make a good split between the benzene (on the lower accumulator tray) and the paraffin (in the net bottoms) in the benzene column. Any benzene that leaves the column in the bottoms stream will be either lost to the overhead vacuum system in the paraffin column, or sent to the Pacol unit with the recycle paraffin where it will be lost in the Pacol product stripper overhead. A small amount of paraffin in the benzene on the lower accumulator tray does not cause any significant problems and will not be lost. Too much paraffin (more than about 0.5%) in the benzene on the upper accumulator tray can cause problems in the regenerant stream because the heavier paraffin material will tend to cause some deactivation of the catalyst at the high temperature used for regeneration. Of course, if there is just one accumulator tray, the paraffin content must be minimized because the regenerant comes from the same tray. Proper operation of the benzene column is also important in order to insure good water removal from the make-up benzene.

E. RECYCLE PARAFFIN STRIPPER

The benzene column bottoms is the feed to the recycle paraffin stripper (Figure IV-13). A large portion of the n-paraffins is taken overhead in the recycle paraffin stripper and sent to the Pacol as recycle paraffin. Some of the n-paraffins and all of the LAB and HAB are taken as net bottoms from the column and sent to the paraffin column for recovery of the rest of the n-paraffin.

The recycle paraffin stripper operates at a pressure slightly above atmospheric - about 0.5 kg/cm^2 (7 psig). This keeps the overhead liquid temperature high enough to use in the middle reboiler in the benzene column as a means of heat recovery. Without the recycle paraffin stripper column, all of the heat removed in the paraffin fractionation towers would be lost to the air. In order to run the recycle paraffin stripper at this pressure, a significant amount of paraffin must be present in the bottom of the column to keep the reboiler temperature below the temperature of the hot oil and to prevent thermal degradation of the bottoms liquid. (Note that the paraffin column runs under vacuum to allow removal of all the paraffin from the net bottoms liquid at a reasonable temperature.) The recycle paraffin stripper bottoms must contain about 50% paraffin to maintain an acceptably low temperature in the reboiler. However, there is a very large amount of paraffin in the feed to the recycle paraffin stripper, and about 70% of the total recycle paraffin stream is taken from the recycle paraffin stripper overhead. About 30% of the total recycle paraffin passes out the bottom of the recycle paraffin stripper along with the LAB and HAB, and this is enough to keep the paraffin concentration in the recycle paraffin stripper bottoms at about 50%.

Another advantage of the higher pressure in the recycle paraffin stripper is that the total column cross-sectional area required for removing the paraffin from the LAB and HAB is much smaller compared to a design where all of the paraffin is removed at full vacuum conditions. So both the recycle paraffin stripper and the paraffin column become relatively small diameter columns compared to a very large diameter column required if the entire recycle paraffin stream is recovered in just a single paraffin column.

There is a contact condenser in the top part of the recycle paraffin stripper which consists of a packed section above the accumulator tray. Paraffin from the accumulator tray is cooled and continuously circulated on flow control to the top of the contact condenser packing. The vapors that rise through the chimney past the overhead accumulator tray are condensed in the contact condenser and are collected on the accumulator tray. The contact condenser inside the column takes the place of a conventional overhead condenser heat exchanger (which would be outside of the column). The contact condenser has a much lower pressure drop

than an external overhead condenser would have, and this is a significant advantage in low pressure service. The cooled, circulating paraffin stream is often called "cold reflux" even though it is not the actual reflux stream used to control the column split. The function of the "cold reflux" in the contact condenser is similar to the cooling water used in a conventional shell & tube overhead vapor condenser. The flow rate and temperature of the "cold reflux" will affect the temperature of the liquid on the accumulator tray.

Most of the circulating liquid that goes to the top of the contact condenser is cooled in the benzene column middle reboiler where the heat is used to vaporize liquid in the benzene column. Circulating overhead liquid is also used to heat the Detal reactor feed stream. In order to maintain a stable and continuous rate of cooling, a portion of the circulating overhead liquid to the contact condenser is cooled in the paraffin column overhead air-cooler. The amount of circulating liquid flow through the air cooler is reset by the temperature of the combined liquid flow going to the top of the contact condenser. This helps to stabilize the column operation and to provide a means of cooling the overhead of the recycle paraffin stripper in case no heat is being used in the benzene column middle reboiler.

The column pressure is controlled by a split range control system with a control valve on the nitrogen purge to the top of the column and a second control valve on the off gas line to the relief header. These control valves are reset by the pressure inside the column just below the contact condenser. The split range control system is set up so that only one of the two control valves will be open at any given time.

The accumulator tray is just below the contact condenser in the top of the paraffin column and it takes the place of an overhead receiver by collecting the liquid that is condensed in the contact condenser.

Due to the unusual nature of the split that is being made in this column (some of the n-paraffin is intentionally taken out the bottom of the column), the control scheme is a little unusual. First, the net bottoms flow rate is set on straight flow control. The bottoms flow rate is set at approximately double the design flow rate of the LAB + HAB flow rate. This will give about 50% n-paraffin in the recycle paraffin

stripper bottoms. Moderate changes in the concentration of n-paraffin in the recycle paraffin stripper bottoms will not cause any problems, so normally it is not necessary to adjust the bottoms flow rate at all.

The heat input to the recycle paraffin stripper is achieved with a controlled flow of hot oil to the tubes of the reboiler. Typically, the reboiler is a shell and tube exchanger outside the column. Inside the column, there is a typically a reboiler tub that collects the liquid from the bottom tray and sends it to the reboiler. The reboiler outlet line goes back into the bottom of the column in a location where the liquid can not get back into the reboiler tub. The spillback from the discharge of the net bottoms pump to the tub should be used when column operations are unstable to keep the tub full and to make sure there is always a liquid flow to the reboiler.

Some of the paraffins from the accumulator tray are refluxed back to the column to the top fractionation tray (or packing) just below the accumulator tray. This stream is sometimes called the "hot reflux". The flow rate of "hot reflux" is reset by the liquid level in the bottom of the column.

The net overhead stream from the paraffin column is also taken from the accumulator tray, but it is taken from a point after it has been cooled in the benzene column middle reboiler. The net overhead flow rate is reset by the level controller on the accumulator tray. This stream eventually joins the paraffin column net overhead stream and the combined n-paraffin flow is recycled back to the Pacol feed surge drum.

F. PARAFFIN COLUMN

The paraffin column (Figure IV-5 or Figure IV-14) separates the n-paraffins (overhead) from the LAB and heavier hydrocarbons (net bottoms). Due to the high boiling temperature of the hydrocarbons being fractionated, the paraffin column is operated under vacuum in order to prevent excessive thermal cracking.

The feed to the paraffin column will be the net bottoms stream from the benzene column in units that do not have a recycle paraffin stripper. In units that do have a recycle paraffin stripper, the feed to the paraffin column will be recycle paraffin stripper bottoms. In either case, the paraffin column feed is pumped to the control valve which is located very close to the feed point on the paraffin column because a significant amount of flashing takes place across the control valve.

There is a contact condenser in the top part of the paraffin column which consists of a packed section above the accumulator tray. Paraffin from the accumulator tray is cooled in the paraffin column overhead air-cooler and is continuously circulated on flow control to the top of the contact condenser packing. The vapors that rise through the chimney past the overhead accumulator tray are condensed in the contact condenser and are collected on the accumulator tray. The contact condenser inside the column takes the place of a conventional overhead condenser heat exchanger (which would be outside of the column). The contact condenser has a much lower pressure drop than an external overhead condenser would have, and this is a significant advantage in vacuum service. The cooled, circulating paraffin stream is often called "cold reflux" even though it is not the actual reflux stream used to control the column split. The function of the "cold reflux" in the contact condenser is similar to the cooling water used in a conventional shell & tube overhead vapor condenser. The flow rate and temperature of the "cold reflux" will affect the temperature of the liquid on the accumulator tray.

The column vacuum is pulled by either a vacuum pump or an ejector system. A high temperature alarm is provided in the overhead vacuum line from the paraffin column to indicate a breakthrough of hydrocarbons from the contact condenser which could be the result of a problem in the contact condenser. The control scheme used to control the pressure in the column is not the same in all units.

1. In some units, a spillback line on the vacuum pump or ejector system is used to control the column pressure. In this control scheme, the spillback control valve is reset by the pressure inside the column just below the contact condenser.

2. In other units, a butterfly valve in the overhead vacuum line is used to control the column pressure. The control valve is reset by the pressure inside the column just below the contact condenser. In this scheme, a small nitrogen purge is injected to the overhead line just upstream of the control valve to allow for proper pressure control in case there are no non-condensibles leaking into the column. This nitrogen eventually passes through the vacuum pump or ejector and helps to purge the net off-gas line. Typically this scheme will also have a spillback line on the vacuum pump or ejector to control the pressure in the suction line to the pump or ejector. This scheme allows two different columns to use the same vacuum pump or ejector, and still maintain independent pressure control for each column.

The accumulator tray is just below the contact condenser in the top of the paraffin column and it takes the place of an overhead receiver by collecting the liquid that is condensed in the contact condenser.

Some of the paraffins from the accumulator tray are refluxed back to the column to the top fractionation tray (or packing) just below the accumulator tray. This stream is sometimes called the "hot reflux" and is actually the reflux stream used to help control the split in the column. The net overhead stream from the paraffin column is also taken from the accumulator tray. This n-paraffin stream sometimes flows through an exchanger system to recover heat before being recycled back to the Pacol feed surge drum.

The paraffin column split control is typically designed to operate in one of two ways:

1. Some units are designed with a "flywheel" control scheme (Figure IV-5). In this design, the total overhead flow rate (the combined "hot reflux" and net overhead) is reset by the paraffin level on the accumulator tray. The control valve for the total overhead flow is actually on the "hot reflux" line after the net overhead splits off. The net overhead flow rate is reset by the column temperature at a point

about halfway between the feed point and the top tray. In this design, an increase in column temperature indicates heavy material moving up the column and causes a decrease in the flow of net overhead. When the net overhead flow is decreased, the total overhead flow controller increases the "hot reflux" flow in order to maintain the same total overhead flow, which will tend to maintain a steady level on the accumulator tray. The additional reflux then moves the heavy material back down the column where it belongs. With this type of control scheme, it is essential to tune the accumulator level controller to hold a very constant level. If the level controller is tuned "loose" (which is typical for level controllers in other services), there will be too much lag in the response of the hot reflux flow rate and the system will not operate in a stable manner.

2. Other units are designed with a "conventional" control scheme (Figure IV-14). In this design, the "hot reflux" flow rate is reset by the column temperature at a point about halfway between the feed point and the top tray. The net overhead flow rate is reset by the paraffin level on the accumulator tray. In this design, an increase in column temperature indicates heavy material moving up the column and causes an increase in the "hot reflux" which tends to push the heavy material back down the column where it belongs. The net overhead flow rate simply increases or decreases as necessary to prevent over-filling or emptying the accumulator tray. In this design, it is not important to maintain a very constant level in the accumulator – the controller can be tuned "loose" as would be typical for most level controllers.

The heat input to the paraffin column is achieved with a controlled flow of hot oil to the tubes of the stab-in reboiler. There is a reboiler tub to keep a liquid level covering the reboiler tubes. The spillback from the discharge of the net bottoms pump to the tub should be used when column operations are unstable to keep the tub full and the reboiler tubes covered.

The net bottoms stream from the paraffin column is pumped to the rerun column or the LAB column depending the unit design. The net bottoms flow rate is reset by the level in the bottom of the paraffin column.

In units with a recycle column, the recycle column net overhead stream (from the clay treater) is normally directed to the suction of the benzene column bottoms pump, but can also be directed to either the boot on the bottom of the paraffin column or to the suction line of the paraffin column bottoms pump. The recycle column net overhead from the clay treater will be much cooler than the paraffin column bottoms and if it is directed to the boot, it will decrease the temperature of the bottoms material in the boot. This is good to a certain extent because a slightly reduced temperature in the boot will help decrease the possibility of thermal cracking. However, if the temperature in the boot is reduced too much, vapors from the reboiler can be condensed which can cause paraffin in the net bottoms stream and can seriously affect the performance of the column.

It is important to make a clean split between the paraffins and the LAB in the paraffin column. Any paraffin that is allowed to get into the net bottoms stream will contaminate the LAB product and could exceed the tight specifications on the amount of paraffin that is allowed in the LAB product. Any LAB that goes overhead in the paraffin column with the recycle paraffin will cause increased coking on the Pacol catalyst which causes faster deactivation of the Pacol catalyst. LAB in the Pacol product would tend to be removed in the PEP unit as aromatics. However, LAB in the paraffin recycle stream can be dehydrogenated in the Pacol reactor and would lead to increased indan and tetralin production in the Detal unit if it is not removed in the PEP unit. It is normally possible to operate the paraffin column so that there is less than 0.1% paraffin in the LAB product and less than 0.1% LAB in the recycle paraffin.

The carbon number range of the paraffin feed (e.g. C₁₁-C₁₄) is very important to the operation of the paraffin column. If a range of more than four carbon numbers is present, it becomes difficult or impossible to separate the paraffin from the LAB in the paraffin column because the boiling point of the highest carbon number paraffin is close to, or greater than the boiling point of the lowest carbon number LAB. The

usual paraffin feed specification is for a carbon range of 4 numbers with less than 0.5 wt% of the carbon number above and below the 4 carbon number range. (For example: if the 4 carbon number range is C₁₁-C₁₄, then there should be less than 0.5 wt% of C₁₀ paraffin and less than 0.5 wt% of C₁₅ paraffin). The four carbon number range is also important for proper operation of the Pacol reactors, and this is discussed in the Pacol operating manual.

G. RERUN COLUMN – or – LAB COLUMN

The paraffin column bottoms is sent as feed to either a rerun column or a LAB column. Rerun column and LAB column are just two different names for the same basic column. In older Detal units, the column is typically called a rerun column and in newer units it is typically called a LAB column. Every unit will have either a rerun column or a LAB column, but not both.

The objective of the rerun column (Figure IV-6) or the LAB column (Figure IV-15) is to separate the detergent range linear alkyl benzene (LAB) product from the heavy alkylate (HAB). The rerun/LAB column is typically a packed column and operates under a vacuum.

There is a contact condenser in the top part of the rerun/LAB column which consists of a packed section above the accumulator tray. The operation of the contact condenser and the accumulator tray in the rerun/LAB column are the same as in the paraffin column. The pressure control scheme for the rerun/LAB column is also the same as for the paraffin column with a vacuum pump or ejector system and a spillback or butterfly control valve controlling the pressure just below the contact condenser.

There are typically three packed sections in the rerun/LAB column (although there can be more). The top packed section is the contact condenser and is located above the accumulator tray. The packed section between the feed point and the accumulator tray is the rectification section and this packing works the same as trays in a conventional distillation column. The packed section between the feed

point and the reboiler is the stripping section and again this packing takes the place of trays in a conventional column. The reason packing is typically used instead of trays in this column is because packing has a much lower pressure drop than trays and low pressure drop is desirable in all of the vacuum columns in the Detal unit due to the very low operating pressure of these columns.

In general, a different column control scheme was used on the columns named "rerun column" in older Detal unit designs compared to the typical control scheme used on the columns named "LAB column" in newer Detal unit designs. For the sake of clarity in describing these two different control schemes in this manual, one scheme will be associated with the name "rerun column" and the other control scheme will be associated with the name "LAB column". In general, the scheme for the rerun column works best when there is a recycle column to recover LAB from the net bottoms stream, and the scheme for the LAB column works best when the net bottoms stream is the HAB product (no further fractionation).

1. Rerun Column Control Scheme (Figure IV-6)

In the typical rerun column control scheme, the flow of "hot reflux" to the top of the rectification section in the rerun column is reset by the level on the accumulator tray. The net overhead flow rate is reset by the column bottoms level. An alternate configuration used in some units has the flow of "hot reflux" reset by the bottoms level, and the net overhead flow reset by the level on the accumulator tray. Both of these schemes are somewhat unconventional but the column is controlled in this manner because: a) the net bottoms is designed to contain a significant amount of overhead product (which will be recovered in the recycle column), b) a temperature indicator stabbed into one of the packed sections in this column is not a good indicator of the desired split in this case, c) the HAB production rate is very stable and predictable, and d) only 10% to 20% of the column feed is taken as net bottoms (a difficult control situation using any scheme). So, the net bottoms flow rate is adjusted manually according to the expected heavy alkylate production rate and/or the laboratory analysis of the rerun bottoms stream. The relatively constant flow of bottoms to the recycle column significantly improves the operation of the recycle column.

In the rerun column, an increase in bottoms level is usually due to an accumulation of LAB rather than an accumulation of HAB (since less than 10% of the column feed is HAB). So, in this control scheme, an increase in rerun column bottoms level causes an increase in the flow of net overhead LAB product. This will tend to decrease the level on the accumulator tray and will cause a decrease in the flow of "hot reflux". As the "hot reflux" decreases, more material will be allowed to rise to the accumulator tray in the column, reducing the level in the bottom of the column. With this type of control scheme, it is essential to tune the accumulator level controller to hold a very constant level. If the level controller is tuned "loose" (which is typical for level controllers in other services), there will be too much lag in the response of the hot reflux flow rate and the system will not operate in a stable manner.

In the alternate control scheme, the response is somewhat more conventional. An increase in rerun column bottoms level causes a decrease in the flow of "hot reflux." This will allow more material to rise up the column, reducing the level in the bottom of the column. Also, an increase in accumulator tray level causes an increase in net overhead flow rate. The level controller in this more conventional arrangement can (and should be) be tuned "loose" because it is not necessary to control with a very constant level on the accumulator tray.

In either of these cases, the bottoms level controller should be tuned relatively "loose" because of the relatively long lag time between the change in "hot reflux" flow and the change in bottoms level. If the bottoms level controller is tuned too "hot" it will cause excessive changes in the hot reflux and create an unstable control situation. Due to the fact that the bottoms level controller must be tuned "loose" in order to get good control during normal operation, the level controller will not be able to handle any quick or sudden upsets. If the column is upset, the level controller should be taken out of cascade mode and the hot reflux controlled manually to stabilize the column.

2. LAB Column Control Scheme (Figure IV-15)

In the typical LAB column control scheme, the flow of "hot reflux" to the top of the rectification section in the LAB column is reset by a temperature indicator stabbed into the packed section below the feed point. The net overhead flow rate is reset by the level on the accumulator tray near the top of the column. The net bottoms flow rate is reset by the liquid level in the bottom of the column. This is a relatively conventional method of column control.

In this control scheme, an increase in the temperature in the bottom packed section (where the temperature indicator is stabbed in) indicates heavy alkylate moving up the column. This will increase the flow of "hot reflux" which will push the heavy alkylate back down toward the bottom of the column where it belongs. Net overhead and bottoms product are taken off as necessary to maintain the respective liquid levels. The top accumulator level controller in this conventional arrangement can (and should be) be tuned "loose" because it is not necessary to control with a very constant level on the accumulator tray.

As for the rerun column control scheme, the bottoms level controller should be tuned relatively "loose" because of the relatively long lag time between the change in "hot reflux" flow and the change in bottoms level. If the bottoms level controller is tuned too "hot" it will cause excessive changes in the hot reflux and create an unstable control situation. Due to the fact that the bottoms level controller must be tuned "loose" in order to get good control during normal operation, the level controller will not be able to handle any quick or sudden upsets. If the column is upset, the level controller should be taken out of cascade mode and the hot reflux controlled manually to stabilize the column.

The heat input to the rerun/LAB column is the same as for the paraffin column. Hot oil goes to the tubes of the stab-in reboiler on flow control. There is a reboiler tub to keep a liquid level covering the reboiler tubes. The spillback from the discharge of the net bottoms pump to the tub should be used when column operations are unstable to keep the tub full and the reboiler tubes covered. Due to the relatively

low flow rate, a pump spillback is sometimes necessary for protection of the rerun/LAB net bottoms pump.

In units that have a recycle column, it is not important to make a precise split in the rerun column. The rerun column net bottoms goes to the recycle column where the LAB in the rerun net bottoms is recovered. It is important not to allow any HAB to go out with the net overhead LAB product because the HAB has a very high bromine index and would cause the LAB to go off-spec. In units with a recycle column, typically the rerun net bottoms flow rate is adjusted to give about 50% LAB and 50% HAB in this stream.

In units that do not have a recycle column, it is important to make a good split in the LAB column. In this case, any LAB that goes out the bottom of the LAB column will end up in the net HAB product causing a reduction of LAB yield. Excessive LAB in the HAB can also have an undesirable effect on the HAB properties (e.g. reduced viscosity). In units with a finishing column, 100% of the LAB product is clay treated, so any heavies that slip up into the LAB product will definitely be sent through the clay treater. This helps reduce the increase in LAB bromine index that can occur as a result of small upsets in the LAB column.

H. RECYCLE COLUMN

Not all Detal units have a recycle column. In units that do have one, the purpose of the recycle column (Figure IV-7) is to recover the LAB from the rerun column bottoms. The LAB that is recovered is clay treated and recycled back to upstream fractionation (usually the paraffin column feed). The recycle column is a packed column and operates under a vacuum.

The recycle column is very similar to the rerun column. There is a contact condenser above the accumulator tray, and packed rectification and stripping sections. Typically, there is no separate pressure controller for the recycle column. The overhead vacuum line from the recycle column is normally connected directly to the overhead vacuum line of the rerun column so the pressure of the recycle

column rides on the pressure in the rerun column vacuum system. The recycle column has a stab-in reboiler with a tub just like the rerun column. The recycle column bottoms pump spillback to the reboiler tub should be opened during unstable operation to keep the reboiler tubes covered with liquid. There is a cooled spillback of bottoms liquid to the boot on the bottom of the column (below the reboiler tub) which is used to control the temperature of the liquid in the boot. The liquid in the boot is slightly cooled to help prevent thermal degradation. However, if the temperature in the boot is reduced too much, vapors from the reboiler can be condensed which can seriously affect the performance of the column. There may also be an uncooled bottoms pump spillback to the boot on the bottom of the recycle column for low flow protection on the pump.

The recycle column is typically designed to operate with a "flywheel" control scheme. In this design, the total overhead flow rate (the combined "hot reflux" and net overhead, which is computed by a signal summing device) is reset by the liquid level on the accumulator tray. The control valve for the total overhead flow is actually on the "hot reflux" line. The net overhead flow rate is reset by the column temperature at a point just below the accumulator tray. In this design, an increase in column temperature indicates heavy material moving up the column and causes a decrease in the flow of net overhead. When the net overhead flow is decreased, the total overhead flow controller increases the "hot reflux" flow in order to maintain the total overhead flow at a rate which will maintain a steady level on the accumulator tray. The additional reflux then moves the heavy material back down the column where it belongs.

The net bottoms from the recycle column is heavy alkylate (HAB) and is pumped to storage. The flow rate is reset by the level in the bottom of the column.

The net overhead from the column is LAB and this stream is sent to the clay treater before being recycled upstream.

It is best to minimize the amount of HAB in the net overhead stream of the recycle column because, due to the higher bromine index of the HAB, more olefinic material will be adsorbed on the clay and this will tend to shorten the life of the clay

in the treater. Also, too much HAB in the recycle column overhead will ultimately increase the amount of HAB in the feed to the rerun column. This could overload the rerun column with HAB and affect the LAB product quality. On the other hand, it is important to minimize the amount of LAB in the recycle column bottoms because any LAB that goes out in the net bottoms stream is lost in the HAB product. Typically, the recycle column is controlled so that the net bottoms stream contains about 5% LAB. Since the HAB yield is only about 10% of the LAB yield, this amounts to a loss of less than 0.5% of the total LAB produced.

I. FINISHING COLUMN

Not all Detal units have a finishing column. In units that do have one, the purpose of the finishing column (Figure IV-16)) is to strip out the small amount of benzene that is formed in the clay treaters. Along with the benzene, some of the LAB will also be stripped overhead in the finishing column. The finishing column overhead is recycled back to upstream fractionation (usually to one of the lower trays in the benzene column). The finishing column is a packed column and operates under a vacuum.

The finishing column has a contact condenser above the accumulator tray, and packed rectification and stripping sections. The operation of the contact condenser and the accumulator tray in the finishing column are the same as in the paraffin column. Typically a butterfly valve in the overhead vacuum line is used to control the finishing column pressure. The control valve is reset by the pressure inside the column just below the contact condenser. In this scheme, a small nitrogen purge is injected to the overhead line just upstream of the control valve to allow for proper pressure control in case there are no non-condensibles leaking into the column. This nitrogen eventually passes through the vacuum pump or ejector and helps to purge the net off-gas line. Typically, the finishing column will use the same vacuum pump or eductor as the paraffin column since the pressure in the finishing column is normally designed to be about the same as in the paraffin column. In this case, there will be a spillback line on the vacuum pump or ejector with a control valve on the spillback to control the pressure in the suction line to the pump or ejector. This

allows the two different columns to use the same vacuum pump or ejector, and still maintain independent pressure control for each column.

The finishing column has a stab-in reboiler with a tub just like the LAB column. The finishing column bottoms pump spillback to the reboiler tub should be opened during unstable operation to keep the reboiler tubes covered with liquid.

The finishing column is typically designed to remove an excess of net overhead instead of trying to make a split between the benzene and LAB. This helps insure that there is no benzene left in the net bottoms from the column. In this design, the net overhead flow rate is set on straight flow control. The overhead flow rate is set at approximately the design flow rate and is typically not changed. This will normally be sufficient to insure there is no benzene left in the bottoms. The flow rate of "hot reflux" to the packing just below the accumulator tray is reset by the level controller on the accumulator tray. The heat input to the feed and to the reboiler should be adjusted to keep the "hot reflux" flow rate at about the design value to insure the benzene is properly stripped out of the LAB product. In this control scheme, an increase in the level on the top accumulator tray means that too much liquid is going up the column. Since the net overhead liquid stream flow rate is constant, the increase in accumulator level causes an increase in "hot reflux" which sends the liquid back down to the bottom of the column where it belongs.

The net bottoms from the recycle column is LAB product and is pumped to storage. The bottoms flow rate is reset by the level in the bottom of the column.

The net overhead from the column is mostly LAB, but most importantly the net overhead stream also contains the trace amount of benzene that is produced in the clay treater. If there is any paraffin present in the feed to the LAB column, it will tend to end up in the finishing column overhead as well. The finishing column overhead stream is recycled to the benzene column where the benzene, paraffin, and LAB can be recovered.

J. CLAY TREATERS

The purpose of the clay treaters is to reduce the bromine index of the LAB product. In the clay treaters, trace amounts of olefins and other impurities which contribute to bromine index can either be adsorbed on the clay or they can react to form heavier hydrocarbons. Adsorption is the primary function at lower temperature, and reaction is the primary function at higher temperature. The life of the clay is much shorter for adsorption than for reaction.

An important side reaction that can take place in the clay treater is trans-alkylation. In this reaction, the straight-chain alkyl group from one molecule of LAB is transferred to another LAB molecule, creating one di-alkyl benzene and one benzene molecule. The amount of trans-alkylation increases as the clay treater temperature increases. Any benzene produced in the clay treaters must not be allowed to contaminate the LAB product. If benzene is formed in the clay treater, it must be removed, typically by fractionation of the clay treater product.

The clay treater temperature must be carefully controlled because if the temperature is too high, cracking reactions take place which could degrade the quality of the LAB product as well as cause operational problems in the vacuum columns (partial loss of vacuum). Also, if the clay treater temperature is too high, trans-alkylation reactions will take place which will result in excessive benzene and heavy hydrocarbons in the clay treater product. If the clay treater temperature is too low, the olefinic material and other impurities will not be completely removed or reacted, causing an increase in bromine index of the LAB product. The clay treater temperature will have to be increased as the clay deactivates in order to maintain the clay performance. The typical operating range for the clay treater temperature is about 80°C-120°C depending on the clay activity and the mode of operation. The clay treater pressure must be maintained high enough to keep the material in the clay treater in the liquid phase.

Many different configurations and locations are possible for clay treaters in a Detal unit. In general, only two configurations have been commonly used in Detal units:

1. Rerun & Recycle Clay Treaters

In Detal units that have a rerun and a recycle column, two clay treaters are typically used. The first (called the rerun clay treater) is the larger of the two and treats the overhead or sidecut stream from the rerun column. The second (called the recycle clay treater) is much smaller and treats the overhead of the recycle column.

a) Rerun Clay Treater

The feed to the rerun clay treater is taken from either the top accumulator tray or a sidecut accumulator tray in the rerun column. Typically the Rerun clay treater effluent is recycled back to the paraffin column feed (via the suction to the benzene column bottoms pump). In this configuration, the bromine index reduction occurs on a recycle stream of LAB which is somewhat less effective than treating the actual LAB net product stream. However, it does provide a way to remove both benzene and heavy hydrocarbons from the clay treater product using an existing column (paraffin column). This allows operation of the clay treater at a higher temperature, maximizing the bromine index reduction across the clay treater but also producing some benzene and heavy hydrocarbons by trans-alkylation.

CAUTION: Directing the clay treater effluent directly back to the rerun column (either to the "cold reflux" line, the "hot reflux" line, or the feed line via suction of paraffin column bottoms pumps) will not allow for removal of benzene. If there is benzene in the rerun column feed or reflux, at least some of the benzene will end up in the net overhead stream (LAB product). Of course, benzene in the LAB product is not acceptable and must be avoided. If the clay treaters are run at a high enough temperature to produce even 1 ppm of benzene, the clay treater effluent must be directed to the paraffin column feed (suction of the benzene column bottoms pumps), or the benzene must be somehow otherwise stripped out of the LAB product.

b) Recycle Clay Treater

The net overhead from the recycle column is LAB, but this LAB is really the heavy fraction of LAB that is produced in the unit. This heavy fraction of LAB tends to have a much higher bromine index than the lighter fraction that makes up the bulk of the LAB product. The olefinic components of the heavy fraction of LAB (which contribute to the bromine index) tend to either be adsorbed on the clay or react to form heavier hydrocarbons in the clay treater. These heavy hydrocarbons could cause product quality problems in the LAB if this stream were blended directly into the LAB product. However, when the clay treated product is fed back to the paraffin column, the benzene (resulting from trans-alkylation) goes out the top of the paraffin column with the recycle paraffin (the amount of benzene is so small that it does not affect the Pacol unit), and the heavy hydrocarbons go out the bottom of the rerun column and end up in the HAB product.

Most of the net recycle column overhead passes through the recycle column overhead cooler before going to the clay treater. A small flow of uncooled recycle column overhead bypasses the overhead cooler and this flow is reset by the inlet temperature to the clay treater. There is a back-pressure control valve on the outlet of the clay treater to keep the material in the clay treater in the liquid phase.

CAUTION: Directing the clay treater effluent directly to the rerun column will not allow for removal of benzene. If there is benzene in the rerun column feed or reflux, at least some of the benzene will end up in the net overhead stream (LAB product). Of course, benzene in the LAB product is not acceptable and must be avoided. If the clay treaters are run at a high enough temperature to produce even 1 ppm of benzene, the clay treater effluent must be directed to the paraffin column feed (suction of the benzene column bottoms pumps), or the benzene must be somehow otherwise stripped out of the LAB product.

2. LAB Product Clay Treater

In Detal units that have a LAB column and a finishing column, only one clay treater is typically used. This single clay treater (typically called the "LAB clay treater") treats the net overhead stream from the LAB column. In this configuration, the bromine index reduction occurs directly on the LAB product which is more effective than treating a recycle or reflux stream because all of the LAB product must pass through this clay treater before going to the product storage tank. However, this configuration requires an additional fractionation column to strip the benzene out of the LAB product. Therefore, a finishing column is required in this clay treater configuration.

CAUTION: Proper operation of the finishing column is very important in this configuration to prevent benzene in the LAB product. Of course, benzene in the LAB product is not acceptable and must be avoided. If the finishing column is not operating properly, the clay treater effluent must be directed to off-specification storage and re-run through the unit to insure the benzene is properly stripped out of the LAB product.

In any of the configurations, it is possible to have a spare clay treater installed to make it easier to change out the clay during normal operation. Typically, a spare treater is not required because the clay can be changed and dried out in a relatively short time and the unit operation can be adjusted slightly to minimize the LAB bromine index without the full use of the clay treater during this time. If a spare treater is installed, the piping will typically be arranged so that the freshly loaded clay treater can be brought on-stream in the lag position to dry out the clay before taking the lead treater out of service.

K. OFF-SPECIFICATION LINES AND TANKAGE

Off-spec lines are provided to export material from the benzene recycle pumps, benzene column bottoms, recycle paraffin stripper bottoms, paraffin column bottoms, rerun/LAB column overhead, rerun/LAB column bottoms, recycle column

bottoms, finishing column overhead, and finishing column bottoms to the off-spec tank in order to provide maximum operating flexibility. Off-spec material is sent to a nitrogen-blanketed, pressure controlled off-spec storage tank through the off-spec cooler. The off-spec material may be returned to the plant via a globe valve and flow indicator using the off-spec pump, and is routed to the benzene column feed line. The flow indicator can be bypassed when the amount of off-spec material coming back to the unit exceeds the meter range (such as during startup inventory operations). It is important to keep water out of the off-spec tank to avoid the moisture problems that could occur if slugs of water should enter with the reintroduced off-spec material.

When material from the off-spec tank is returned to the unit, it is usually best to keep the off-spec tank well-mixed. Benzene and paraffin can stratify if the off-spec tank is not well-mixed (with the paraffin layer on top of the benzene). This can cause an upset to the benzene column if the material being recovered from the off-spec tank suddenly changes from benzene to paraffin.

During normal operation, it is recommended to minimize the amount of material in the off-spec tank so that there is sufficient capacity available to pump the hydrocarbon out of the unit in case of an emergency.

L. VACUUM PRODUCING EQUIPMENT

The vacuum under which the paraffin, rerun/LAB, recycle, and finishing columns operate is provided by either vacuum pumps or a series of ejectors operated with steam.

1. Ejector System

In an ejector system, steam is injected at high velocities through a specially designed nozzle and transfers sufficient energy to the gases from the suction header to entrain them through the diffusers into the first-stage discharge header. The pressure in the first-stage discharge header is of course higher than the

pressure in the suction header, but if the velocity of the steam through the diffuser throat is high enough, gas cannot back into the suction header. If a single ejector is incapable of raising the gases to atmospheric pressure (so the gases can be vented), the steam is condensed and a second ejector taking suction on the non-condensable gases raises them to a higher pressure. The condensate is evacuated through long pipes discharging into a condensate separator. The long barometric legs create enough liquid head for the condensate to be drained at atmospheric pressure.

A three stage ejector system is required for the paraffin, rerun/LAB, recovery, and finishing columns, with Stage 1A from the rerun/LAB column going to the first after-condenser which also receives the outlet from ejector 1B which is connected to the paraffin column. From the top of the first after-condenser the common non-condensibles go to the Stage 2 ejectors, a second after-condenser, and then to the Stage 3 ejectors and the final after-condenser. The non-condensable gases are recirculated with a small purge of nitrogen. These non-condensibles are recirculated back to the inlet of the Stage 1A ejectors for rerun column pressure control, and also to the inlet of the Stage 1B ejector for paraffin column pressure control. Additional stage 1 ejectors can be used to provide independent pressure control for the recovery, finishing, or other vacuum columns as necessary.

The net condensate, water, and hydrocarbon goes to the condensate separator from the various after-condenser bottoms. The effluent from the first after-condenser contains most of the light hydrocarbon that breaks through the contact condenser in the top of the columns.

One end of the condensate separator is set up to collect hydrocarbon from the separator via overflow weirs. Condensate water is sent towards the other end of the separator via underflow and a second overflow weir. The water goes to the condensate pumps and is sent to refinery waste water disposal. On the other end of the separator, hydrocarbon is drained to the closed aromatic drain header. There is a level indicator on the hydrocarbon end of the separator and a level controller on the water end. The condensate water level instrument resets the stroke on the proportioning condensate pumps to control the water level.

2. Vacuum Pumps

Vacuum pumps can also be used to produce the vacuum required in the paraffin, rerun/LAB, recovery, and finishing columns. There are many types of vacuum pumps available and the manufacturer should provide information and operation instructions for these pumps.

In general, separate vacuum pumps will be necessary for the paraffin column and the rerun/LAB column because the rerun/LAB column typically operates at a significantly lower pressure. The recycle column will typically share the same vacuum pump with the rerun column, and the finishing column will typically share the same vacuum pump with the paraffin column. The paraffin column will typically have a liquid ring vacuum pump (LRVP) with a flow-through seal oil system because this type of pump can better handle the higher likelihood of light hydrocarbon breakthrough from the top of the paraffin column and finishing column. The rerun/LAB column may have either a LRVP or a rotary piston vacuum pump (RPVP).

In some units, simple spillback of non-condensable gases to the inlet of the vacuum pump is used to control the pressure in the columns. However, this only allows independent pressure control of one column per pump. In other units, the spillback is used to control the pressure in the suction line to the pump. Then each column will have an independent pressure control valve in the overhead vacuum line. This allows using one pump for more than one column while retaining the ability to control each column pressure independently.

M. REACTOR VALVE SWITCHING SEQUENCE

The reactors must be switched every 24 hours for regeneration. A valve sequence control system (sequence controller) is used to automatically switch the reactors and control the regeneration sequence.

During the reactor switching part of the sequence, the reactants and products (paraffin/olefin, recycle benzene, LAB, and HAB) are displaced from the on-line reactor with regenerant from the reactor that has just completed a regeneration. During the regeneration part of the sequence, the reactor that was taken off-line is regenerated.

A general description of the sequence follows. This description is for the case where the A reactor has been on-line (with paraffin/olefin feed and recycle benzene) for about 24 hours and the B reactor has just completed a regeneration. The sequence is the same for the case where the B reactor was on-line and the A reactor has completed a regeneration except that the reactors are reversed.

1. The regenerant is bypassed around the B reactor.
2. The paraffin/olefin feed and recycle benzene are switched from the top bed of the A reactor (which has been on-line for about 24 hours) to the top bed of the B reactor (which has just finished being regenerated). (In Split-Feed reactors, the paraffin/olefin feed remains flowing to the middle and bottom bed of the A reactor at this point.) Also, the regenerant being displaced out of the B reactor is switched to the top of the A reactor. This will displace the reactants and products out of the A reactor. The outlet of the A reactor remains lined up to the benzene column.
3. (In Split-Feed reactors, when the total volume of paraffin/olefin feed and recycle benzene to the top bed of the B reactor equals the displacement volume of the top bed, the paraffin/olefin feed to the middle bed is switched from the A reactor to the B reactor.)
4. (In Split-Feed reactors, when the total volume of paraffin/olefin feed and recycle benzene to the top and middle beds of the B reactor equals the displacement volume of the top and middle beds, the paraffin/olefin feed to the bottom bed is switched from the A reactor to the B reactor.)

5. When the total volume of paraffin/olefin feed and recycle benzene to the B reactor equals the displacement volume of the entire reactor, the outlet of the B reactor is switched from the top of the A reactor to the benzene column. Also, the outlet of the A reactor is closed. The B reactor is now in the normal on-line mode.
6. The regenerant flow is started through the A reactor and the heat-up begins.
7. When the regenerant coming out of the A reactor is hot enough, the "hot soak" begins.
8. When the "hot soak" has been held for the desired amount of time on the A reactor, the cool down begins.
9. When the cool down is completed on reactor A, the sequence pauses for a specified amount of time. Then the sequence starts again with the B reactor being regenerated this time.

A schematic of a typical Split-Feed reactor/regeneration section and the remote operated valves is given in Figure IV-8. A typical valve sequence table for the Split-Feed reactor system is shown in Figure IV-9. A schematic of a typical Single Feed reactor/regeneration section and the remote operated valves is given in Figure IV-17. A typical valve sequence table for the Split-Feed reactor system is shown in Figure IV-18.

The details of the sequence controller will differ slightly from one unit to another. A general description of a typical sequence controller follows:

1. The controller will have a switch to allow operation in the Automatic, Semi-automatic, or Manual mode.
 - a) In the Automatic mode, the controller will automatically advance through the switching and regeneration sequence based on the timer

settings and the temperature setpoints that are input into the controller. This is the normal operating mode.

- b) In the Semi-automatic mode, the controller will advance through the normal valve sequence, one step at a time, each time the "manual step advance button" is depressed. The timers will not cause the sequence to advance in this mode. All of the valve position verifications and process conditional checks are disabled in this mode.
 - c) In the Manual mode, each individual valve can be opened or closed using the sequence controller. The main purpose of this mode is for special operations (such as shutdown) and maintenance. In order to switch from the Manual mode to Semi-automatic or Automatic, all of the valve positions must correspond to the current sequence step on the controller. Manual operation of the valves should not be possible when the controller is in the Automatic or Semi-automatic modes.
2. Each remote operated valve will have limit switches to indicate when the valve is fully open or fully closed. The sequence controller will display the status of each of these limit switches as well as the status of the command signal being sent to each valve (open/closed). The sequence controller will have a valve verification system that monitors the valve limit switches and stops the sequence if a valve does not open or close properly. A valve verification timer is used to allow a given time (usually about 10 seconds) for the valves to open or close before a verification alarm occurs.
3. The sequence controller will typically have the following timers:
- a) Pressurizing and Depressurizing Timers: These timers set the time allowed for pressurizing and depressurizing the reactors via the small restriction orifices.

- b) Verification Timer: Sets the time allowed for the remote operated valves to open or close after a signal has been sent for the valve to move. Typically set at about 10 seconds.
- c) Window Timers: These timers set the minimum and maximum time for displacement of each bed in the reactor during the reactor switching part of the sequence. The purpose of these timers is to give an alarm to prevent under or over displacement in case the flow meters are not operating properly. These timers should normally be set at about +/- 10% of the expected displacement time.
- d) Ramp Timer: Sets the time for ramping up the regenerant temperature (going to the reactor) at the beginning of the regeneration part of the sequence. This timer sets the ramp-up rate.
- e) Long Heat-Up Timer: Sets the time allowed for the regenerant temperature coming out of the reactor to come up to the high temperature regeneration (soak) setpoint. If the regenerant coming out of the reactor does not come up to the desired setpoint before the Long Heat-Up Timer times out, the Long Heat-Up alarm will come on. The timer should be set about 10% longer than the expected heat-up time to prevent nuisance alarms, but still indicate a problem as quickly as possible.
- f) Soak Timer: Sets the time that the high temperature regeneration conditions are held constant. This is the "hot soak" time. This should normally be set at about 15 hours (900 minutes). A shorter soak time may not allow for complete regeneration. A longer soak time is not a problem as long as the entire sequence can still be performed within a 24 hour period.

- g) Pause Timer: Sets the time that the regeneration reactor is held at the cool down setpoint. This allows the operator to adjust the total sequence time so that the regenerations will occur at the same time every day.
4. The sequence controller will typically have the following adjustable setpoints:
- a) Reactor Inlet High Temperature Regeneration Setpoint: Sets the temperature of the regenerant going to the reactor during the "hot soak". This should typically be set at 250°C (482°F).
- b) Reactor Inlet Low Temperature Hold Setpoint: Sets the temperature of the regenerant going to the reactor during the cool-down after the "hot soak".
- c) Reactor Inlet Low Temperature Regeneration Setpoint: Sets the minimum temperature allowed for the regenerant going to the reactor during the "hot soak". If the regenerant temperature drops below this setpoint during the "hot soak", the soak timer stops counting until the regenerant temperature increases back above this setpoint. This should typically be set at about 25°C (45°F) below the Reactor Inlet High Temperature Regeneration Setpoint.
- d) Reactor Inlet Low Temperature Cool Down Setpoint: Sets the temperature at which the regenerant is switched from the regenerant heater to the regenerant cooler during the cool down step. This should normally be set about 5 to 10°C (9 to 18°F) above the on-line reactor inlet temperature.
- e) Reactor Outlet High Temperature Regeneration Setpoint: Sets the temperature at the outlet of the reactor which must be attained before the "hot soak" begins. This should normally be set at about 230°C (446°F). This will allow for the heat loss from the reactors. If the reactor heat loss is more than 10°C (18°F), the reactor inlet high

temperature regeneration setpoint should be increased (instead of decreasing the reactor outlet high temperature regeneration setpoint).

- f) Reactor Outlet Low Temperature Cool Down Setpoint: Sets the temperature which the regenerant coming out of the reactor must cool down to during the cool down step. When this setpoint is met, the "pause" timer starts. This should normally be set about 5 to 10°C (9 to 18°F) above the on-line reactor inlet temperature.
5. The timers should stop timing when the controller is put in the manual or semi-automatic mode. The timers should also stop when there is a power failure, and the Run-Stop switch should switch to the stop position. The sequence can be re-started by toggling the Run-Stop switch to the stop position and then back to the run position. The controller should not allow the sequence to automatically restart when power is restored following a loss of power.



Figure IV - 1
Detal Unit Process Flow

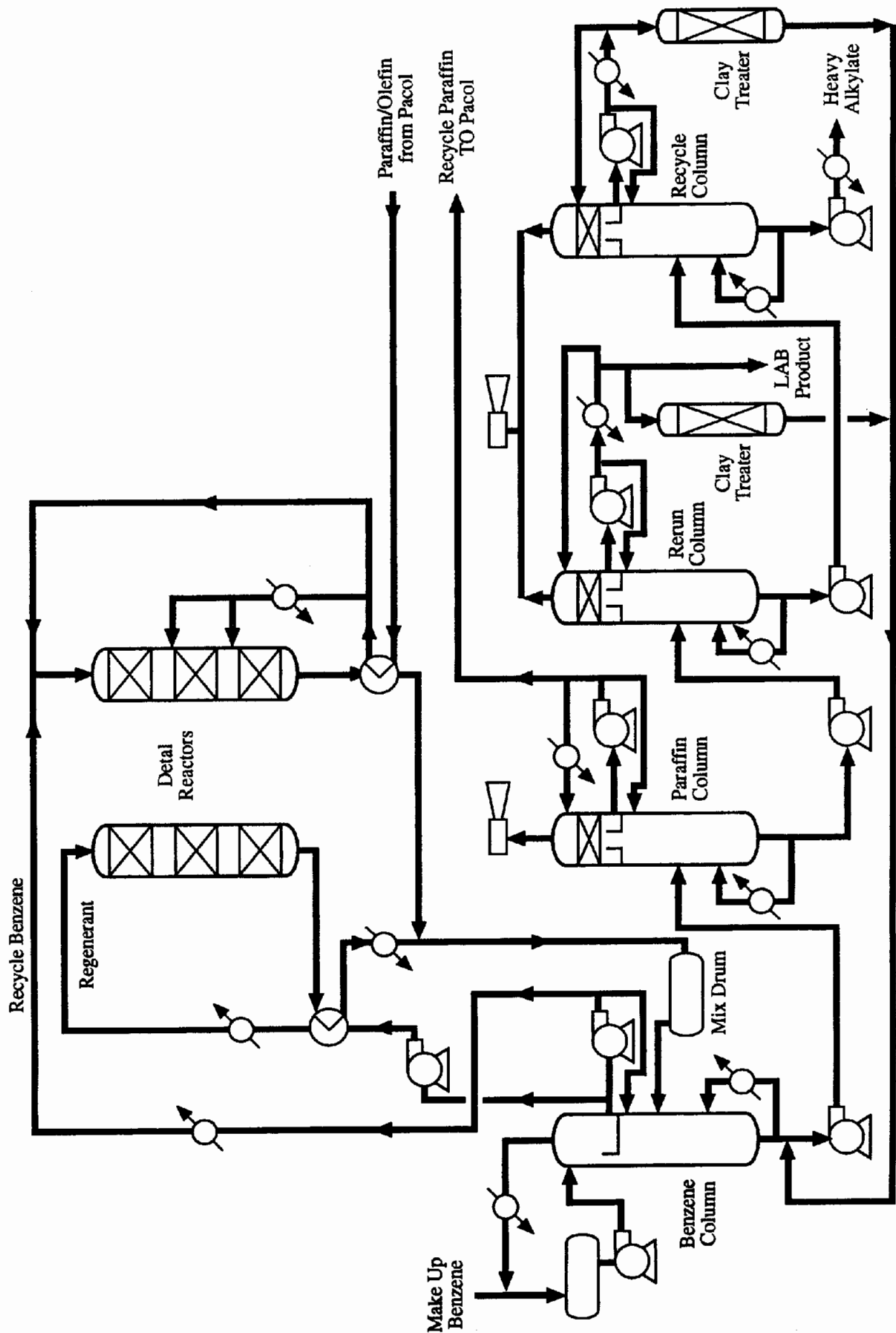


Figure IV - 2
Reactor Section

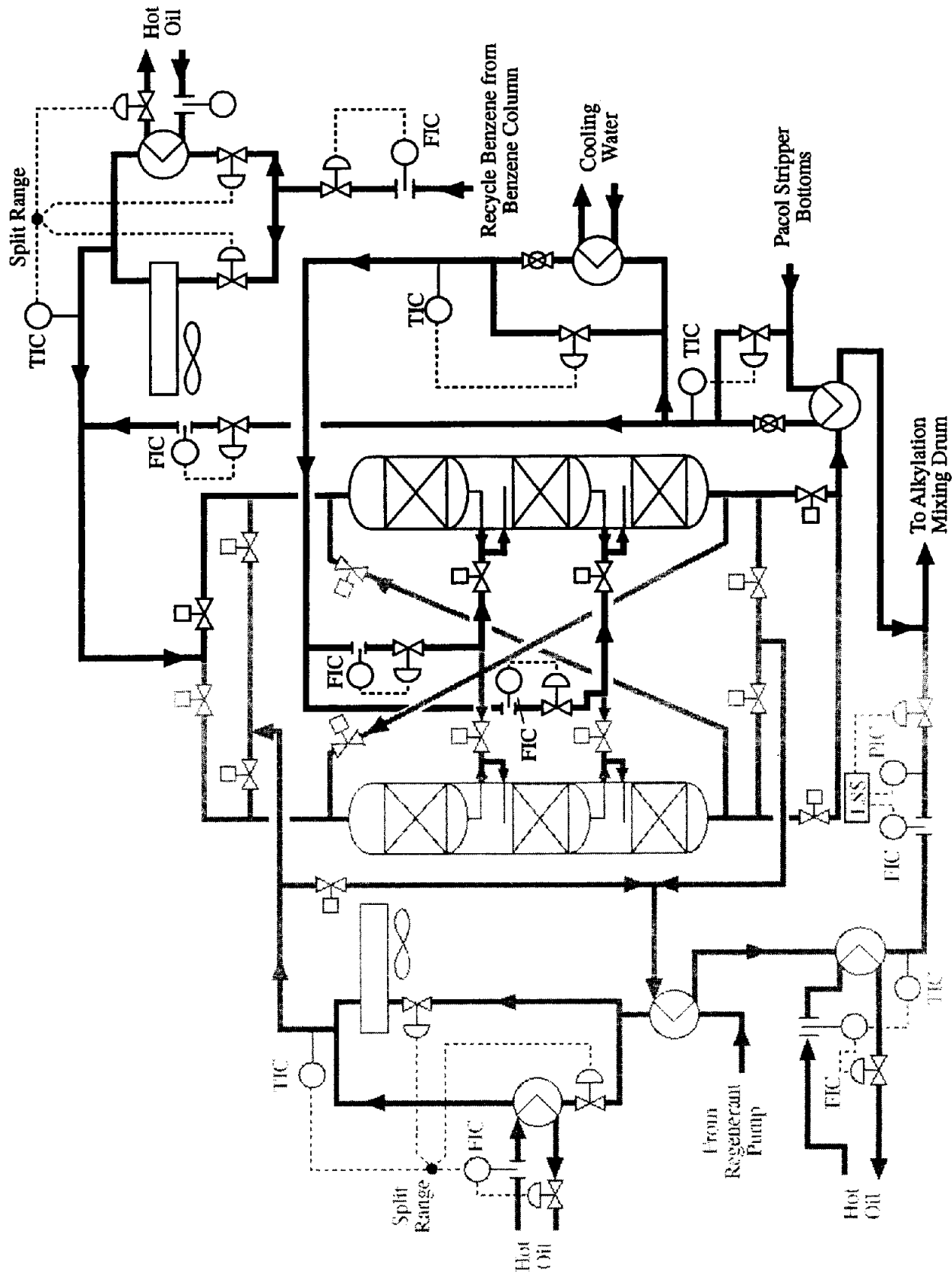


Figure IV - 3
Regeneration Section

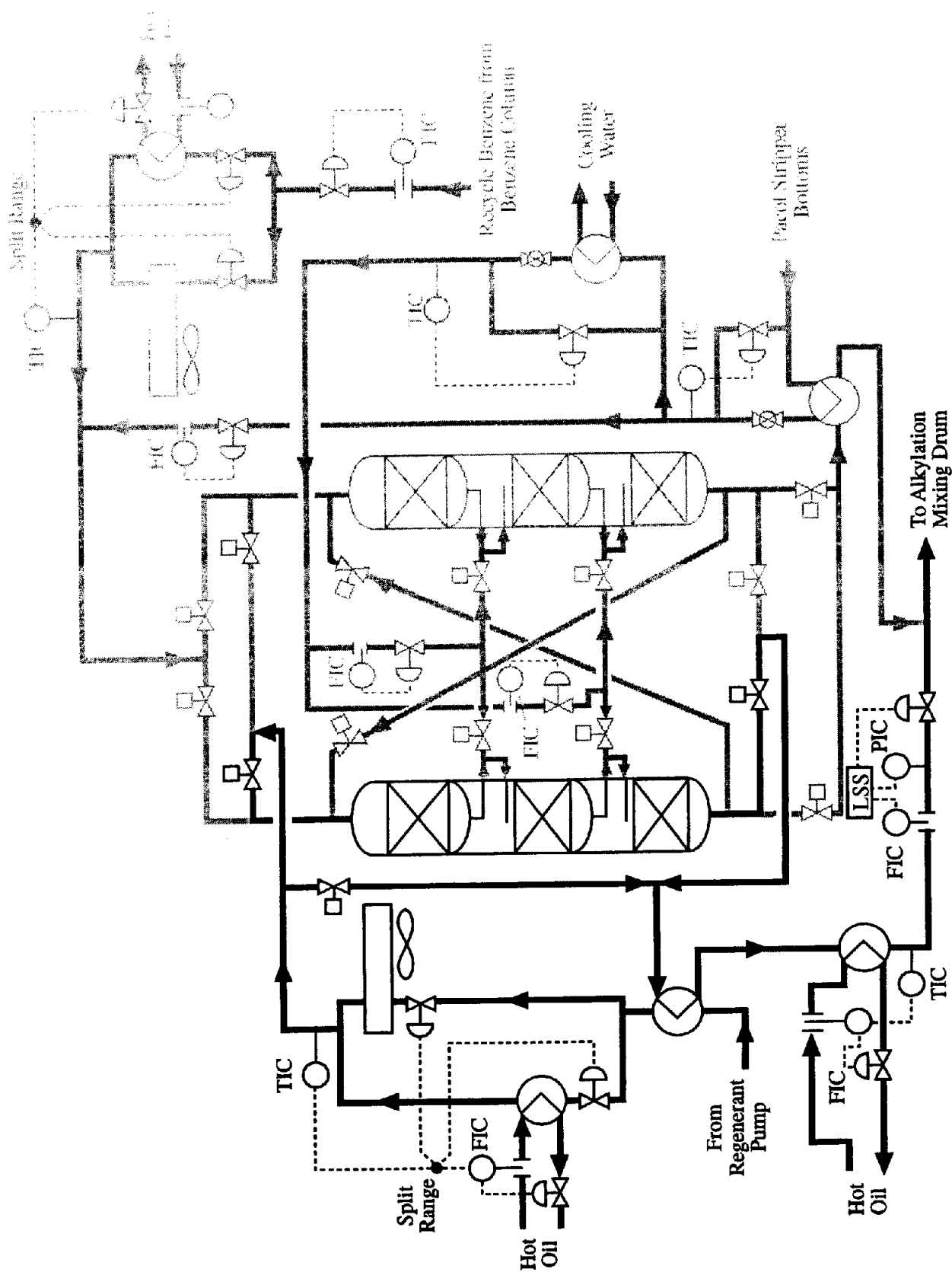


Figure IV - 4
Benzene Column

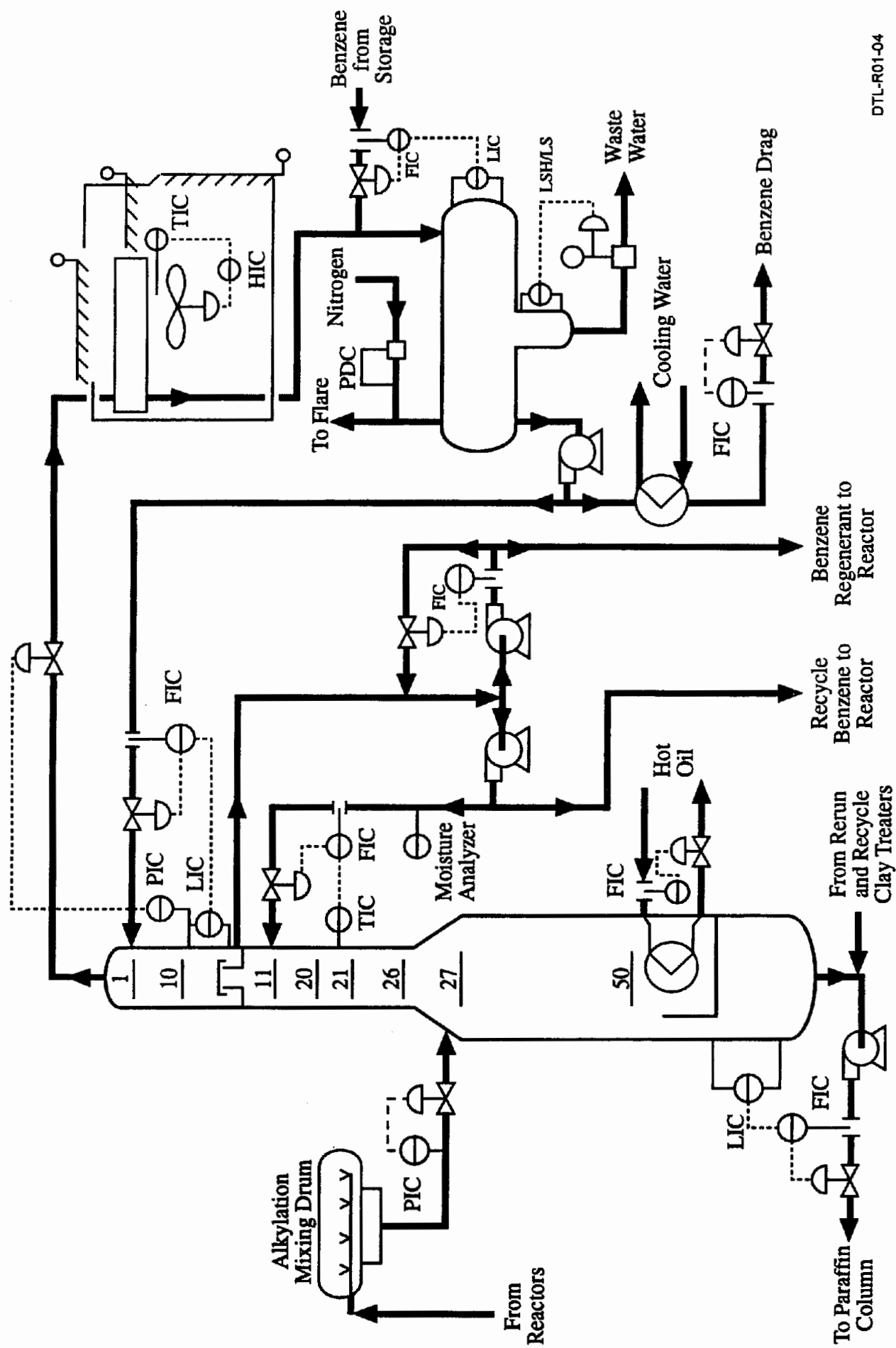


Figure IV - 5
Paraffin Column

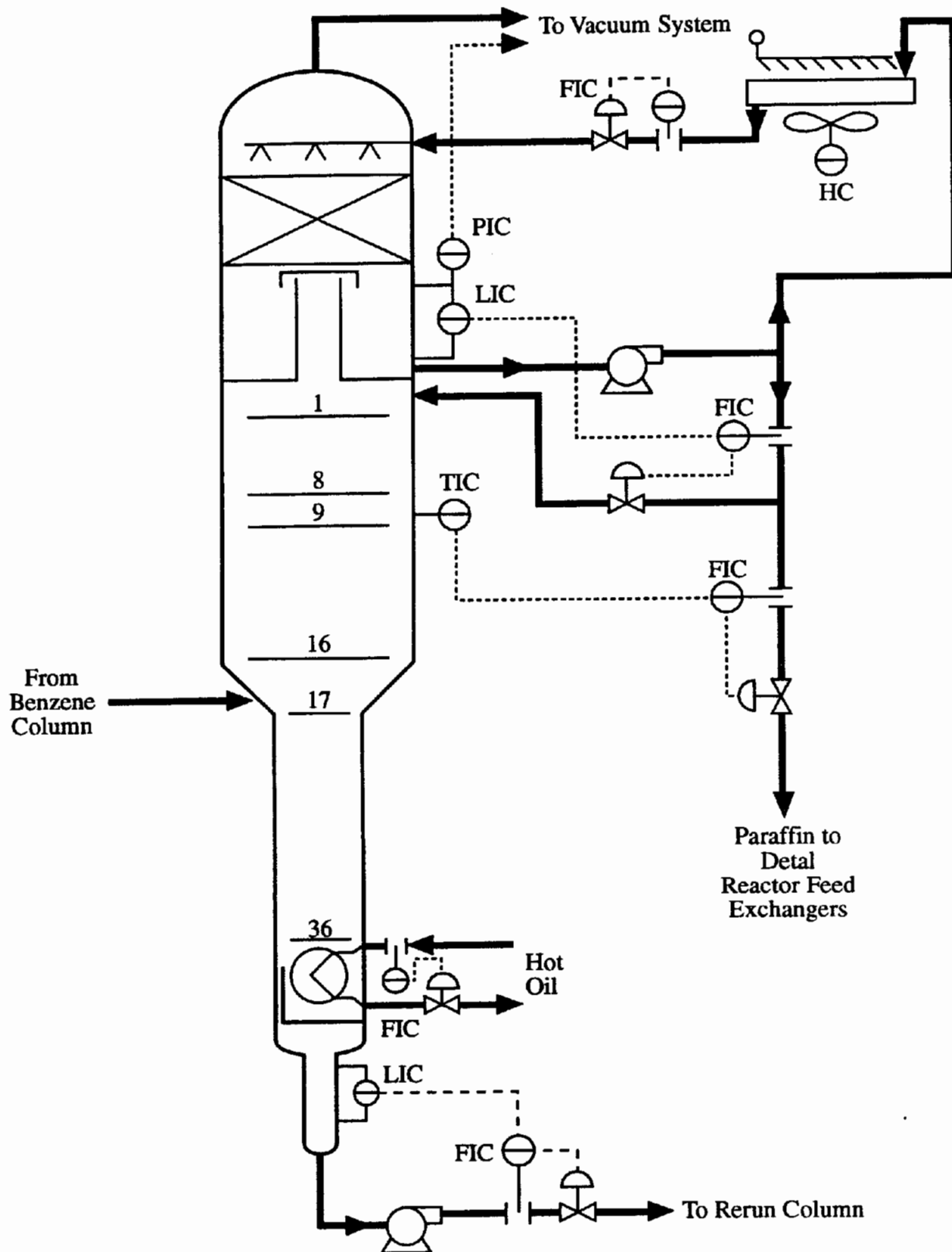
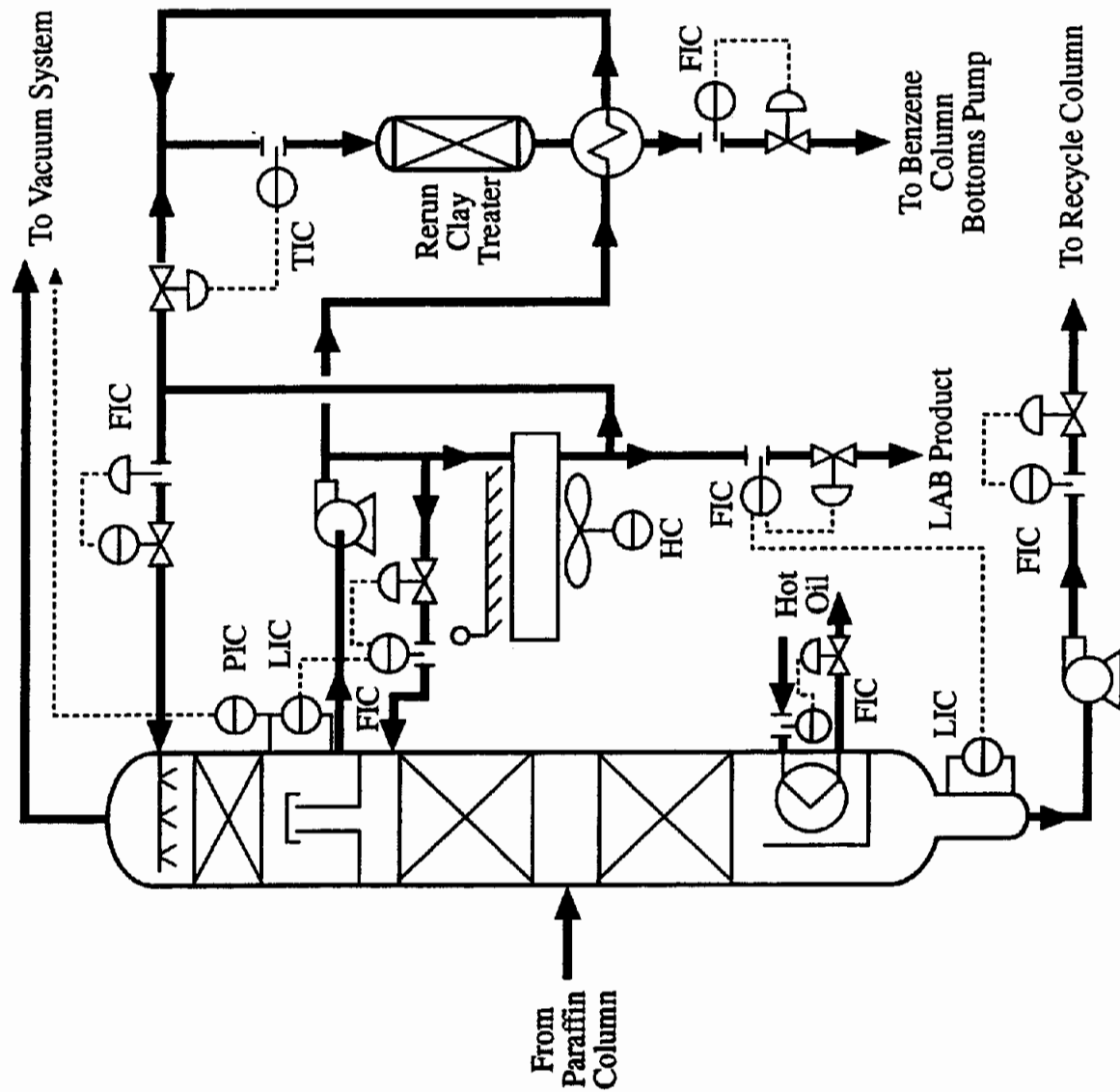


Figure IV - 6
Rerun Column



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Figure IV - 8
Reactor/Regeneration Switching Valves

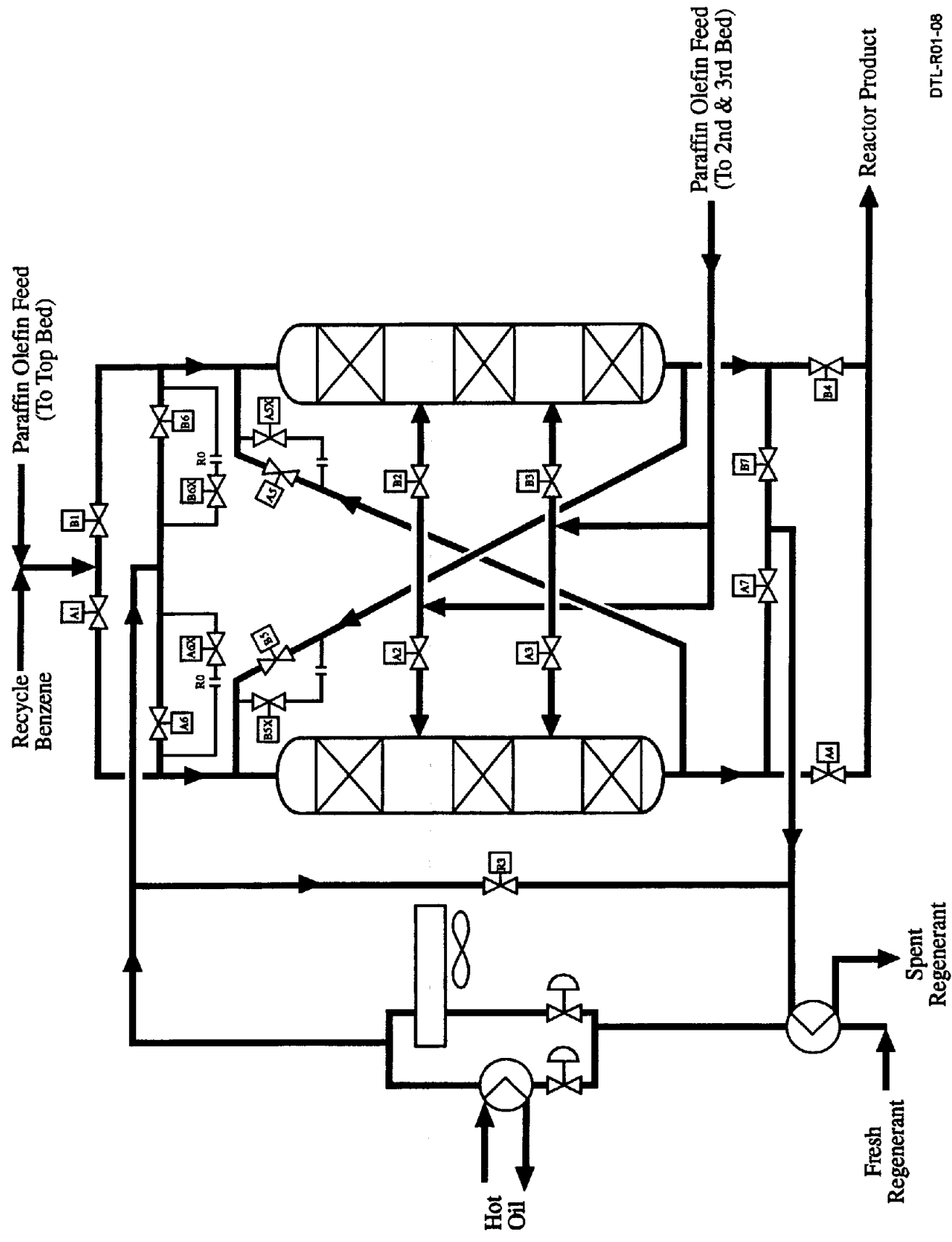


Figure IV-9

Reactor/Regeneration Valve Sequence (Split-Feed Reactor)

Step ---->	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48				
Valve																																																				
A1	o	o	o	o	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
A2	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
A3	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
A4	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
A5	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
A5X	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
A6	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
A6X	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
A7	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
B1	x	x	x	x	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
B2	x	x	x	x	x	x	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
B3	x	x	x	x	x	x	x	x	x	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o
B4	x	x	x	x	x	x	x	x	x	x	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o
B5	x	x	x	x	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
B5X	x	o	o	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
B6	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
B6X	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
B7	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x			
R3	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
Regen Step																																																				

Valve Symbols: X = Closed o = Open

Regen Step A: "Ramp-Up" indicator illuminated. Ramp timer starts and regenerant temperature is ramped up. Long heat-up timer starts.
 Regen Step B: When ramp timer times out, "Heat-Up" indicator is illuminated and the controller waits for the bed to heat up.
 Regen Step C: When the bed temperature reaches the set point, the soak timer starts and the "Soak" indicator is illuminated.
 Regen Step D: When soak timer times out, the "Cool-Down" indicator illuminated and regenerant temperature set point is reduced.
 Regen Step E: When outlet temperature reaches the cool-down setpoint, the "Ready" indicator is illuminated and the Pause timer starts.
 From the "Ready" mode, the operator must give the "Sequence Start" command to initiate the next switching sequence.

Totalizer: 1 : Totalizer counts recycle benzene and paraffin/olefin feed to top bed.
 12 : Totalizer counts recycle benzene and paraffin/olefin feed to top and middle bed.
 123 : Totalizer counts recycle benzene and paraffin/olefin feed to top, middle, and bottom bed.



Figure IV-10
 Detal Unit
 Process Flow
 (Single Feed)

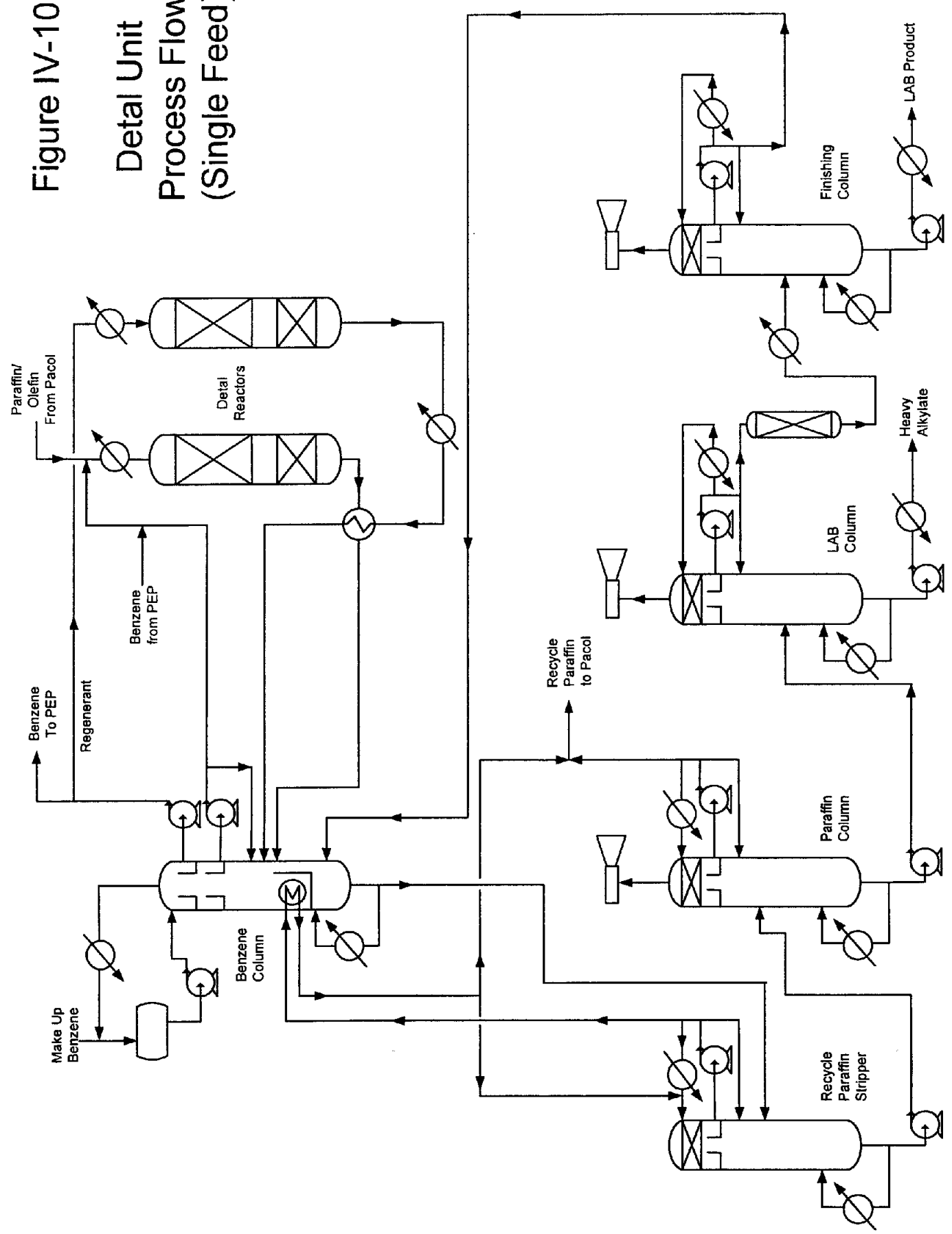


Figure IV-11
Reactor Section

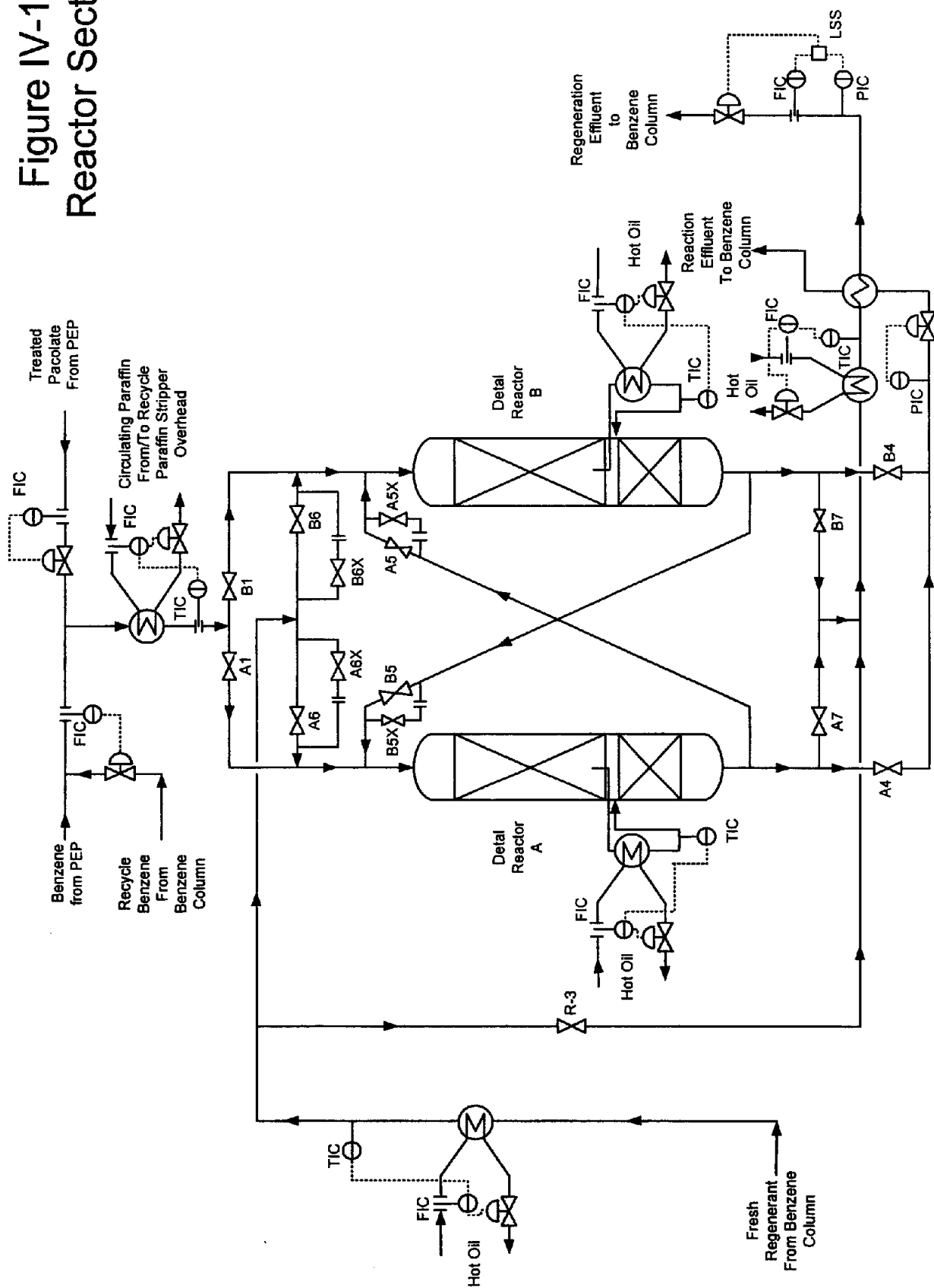


Figure IV-12
Benzene Column (With Middle Reboiler)

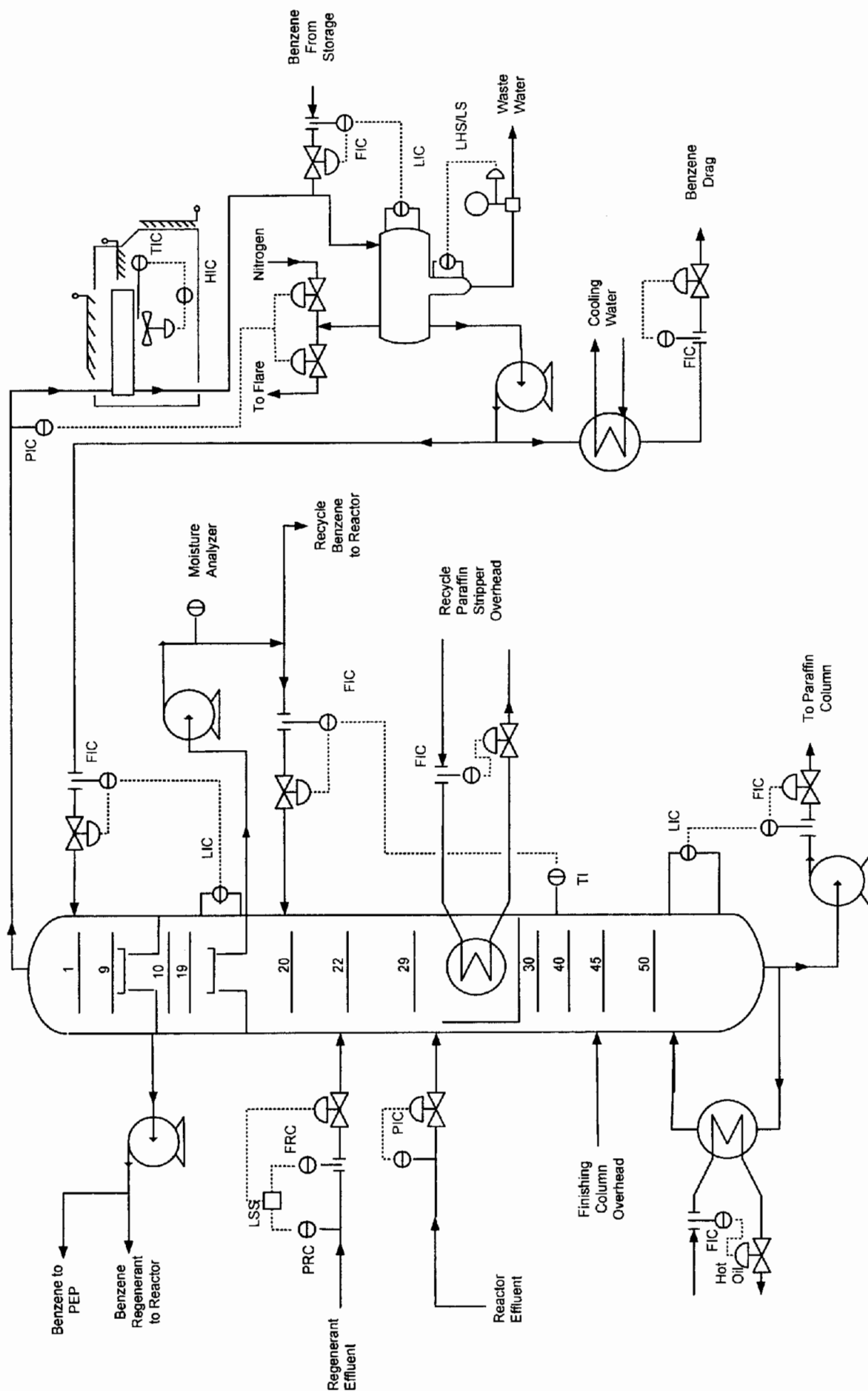


Figure IV-13
Recycle Paraffin Stripper

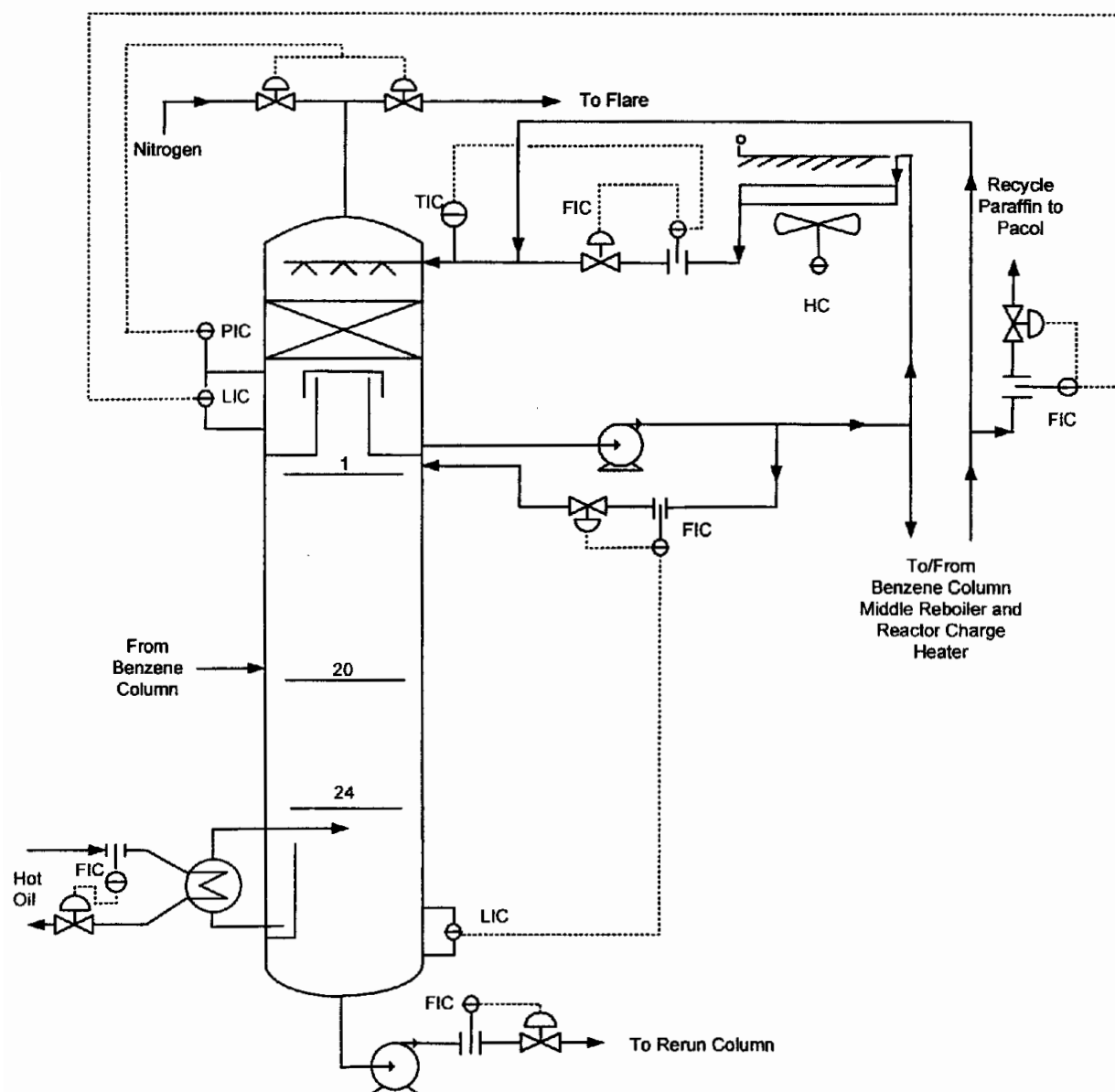


Figure IV-14
Paraffin Column

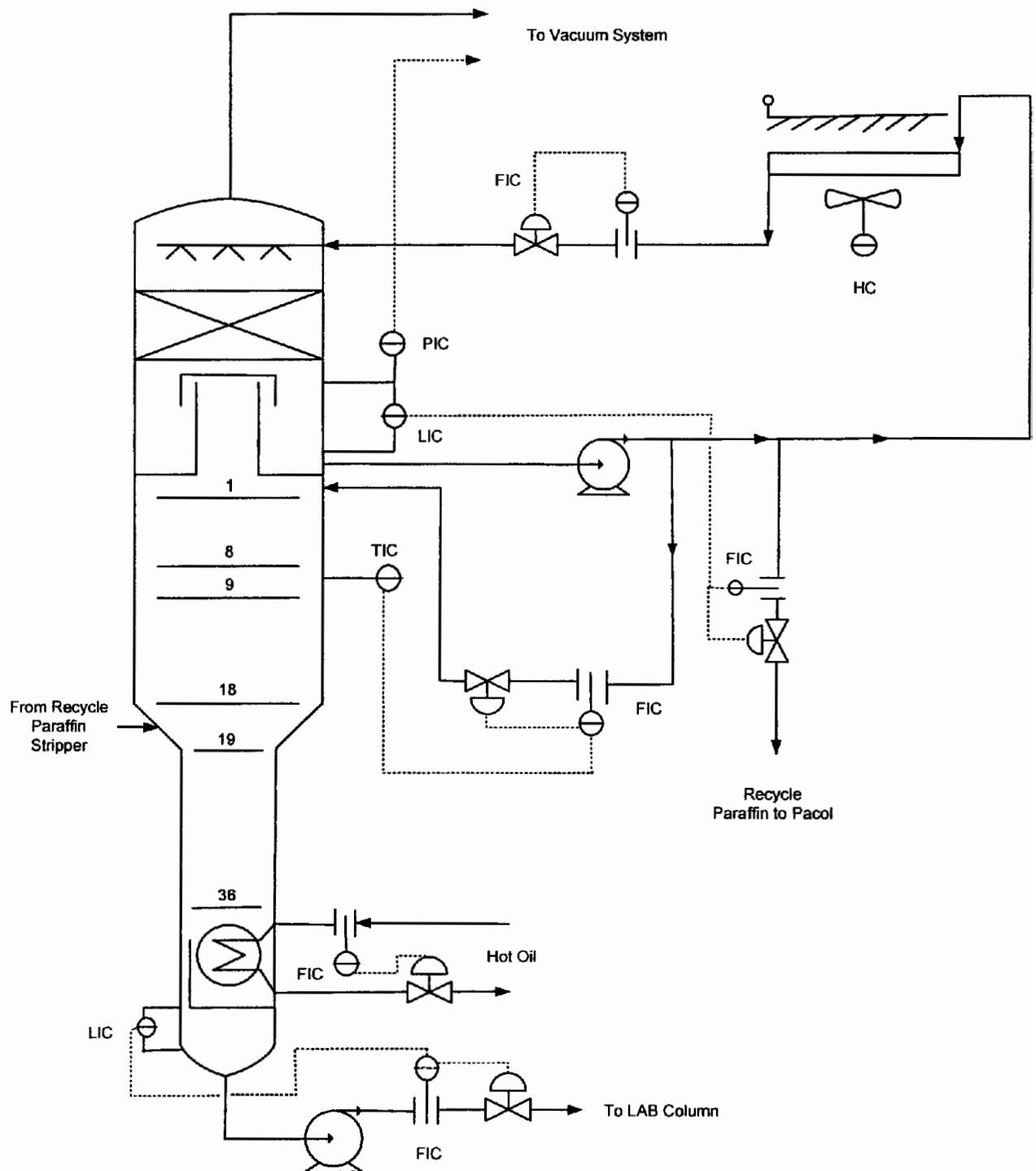


Figure IV-15
LAB Column

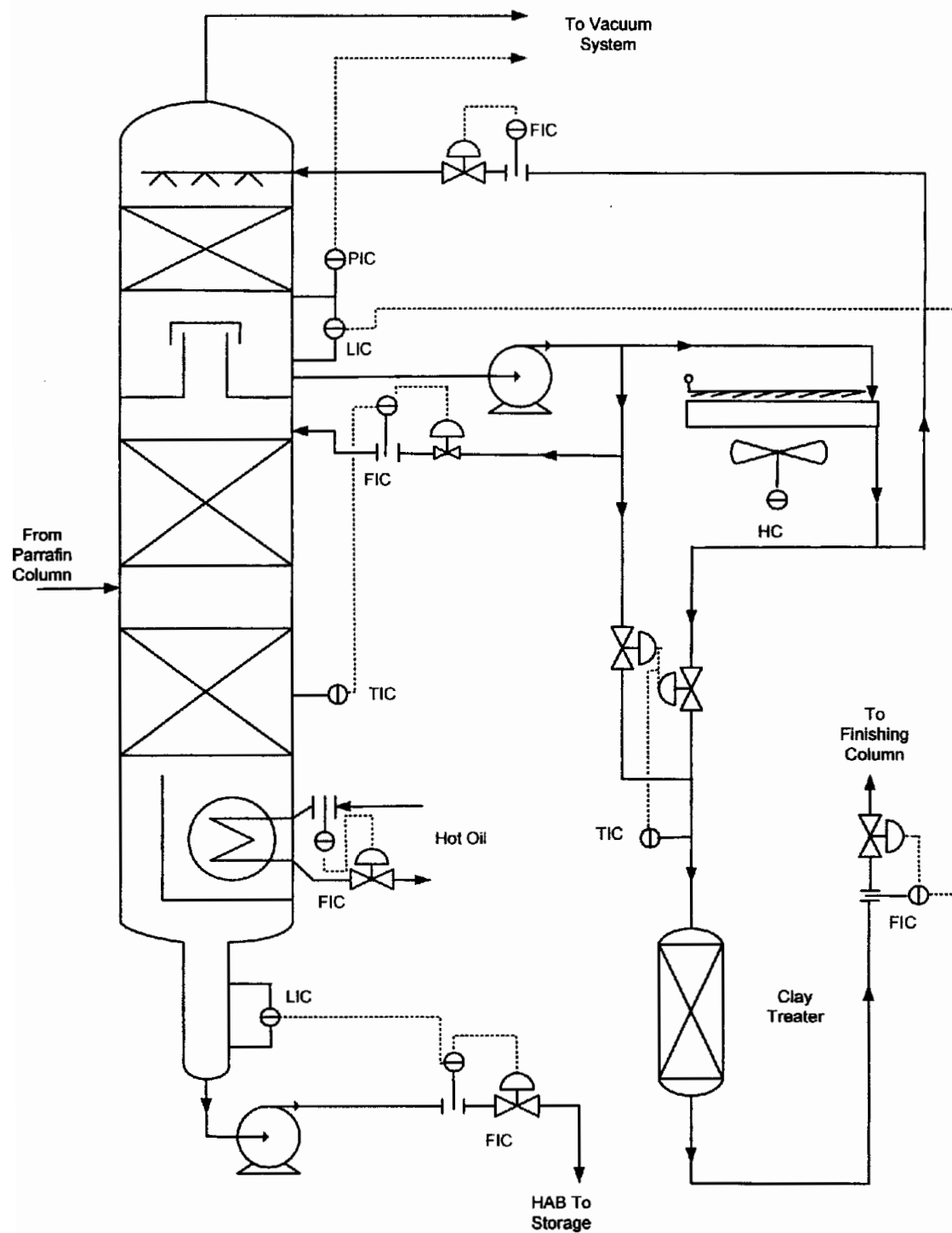


Figure IV-16
Finishing Column

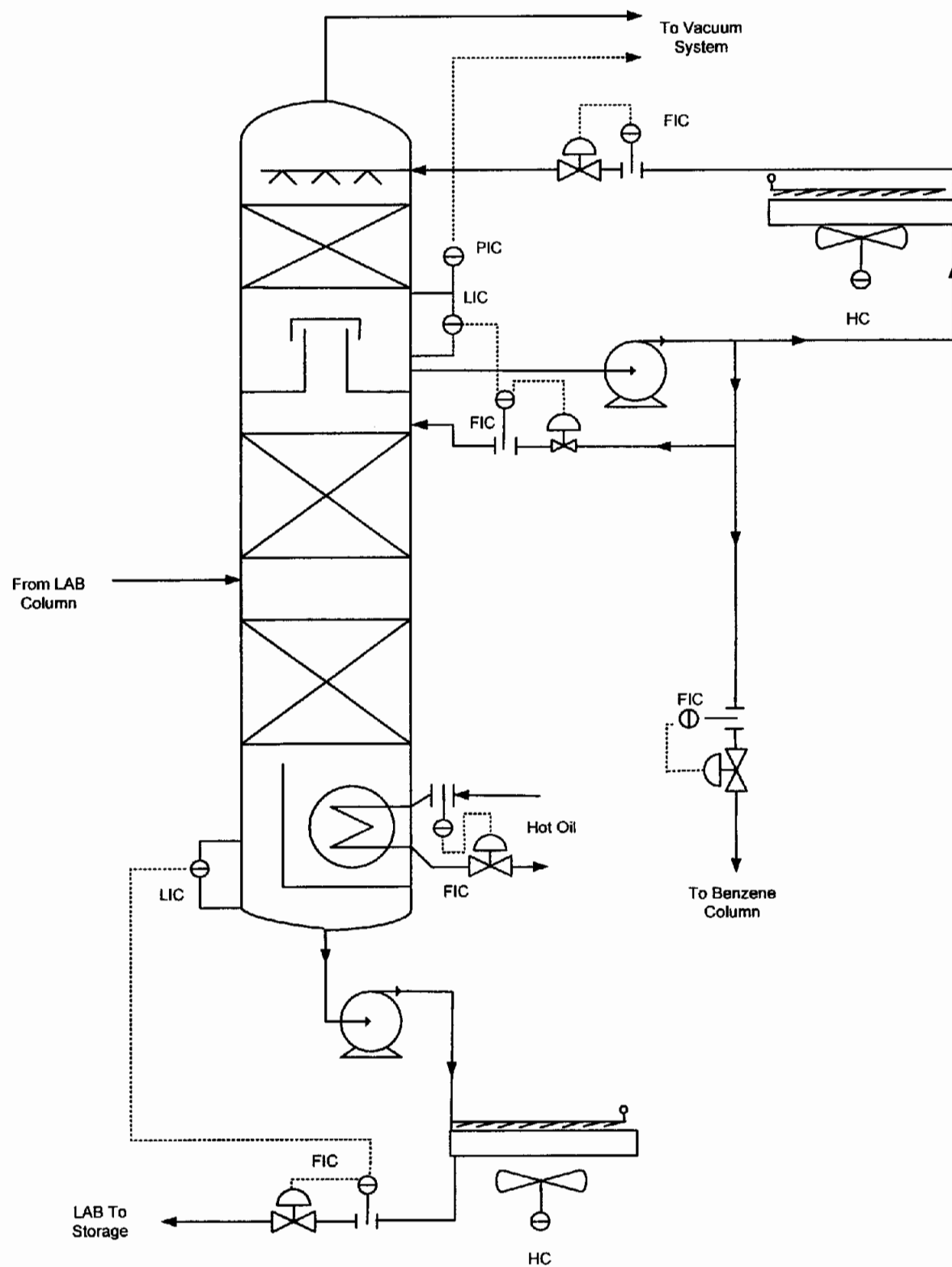


Figure IV-17 Regeneration Switching Valves

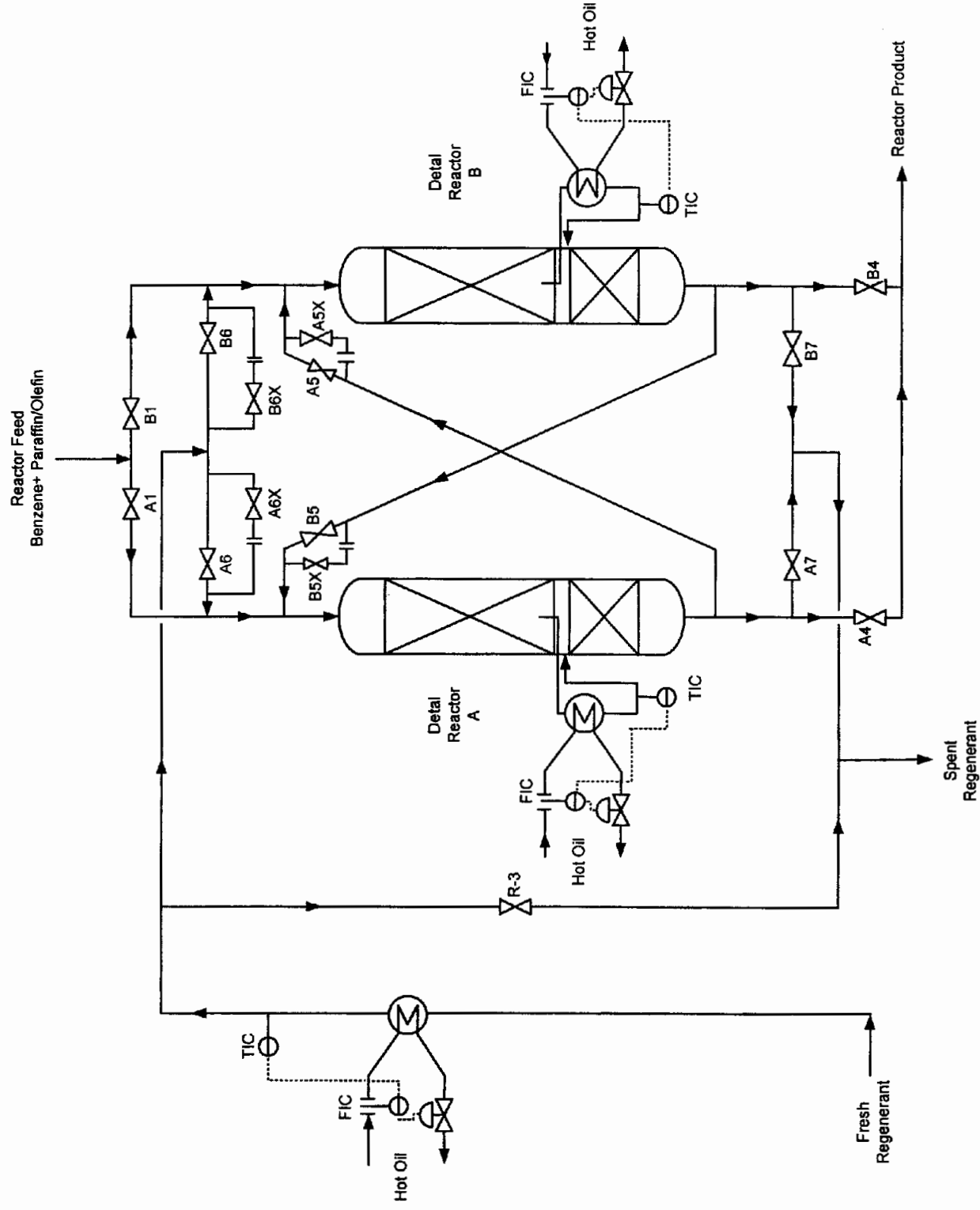


Figure IV-18

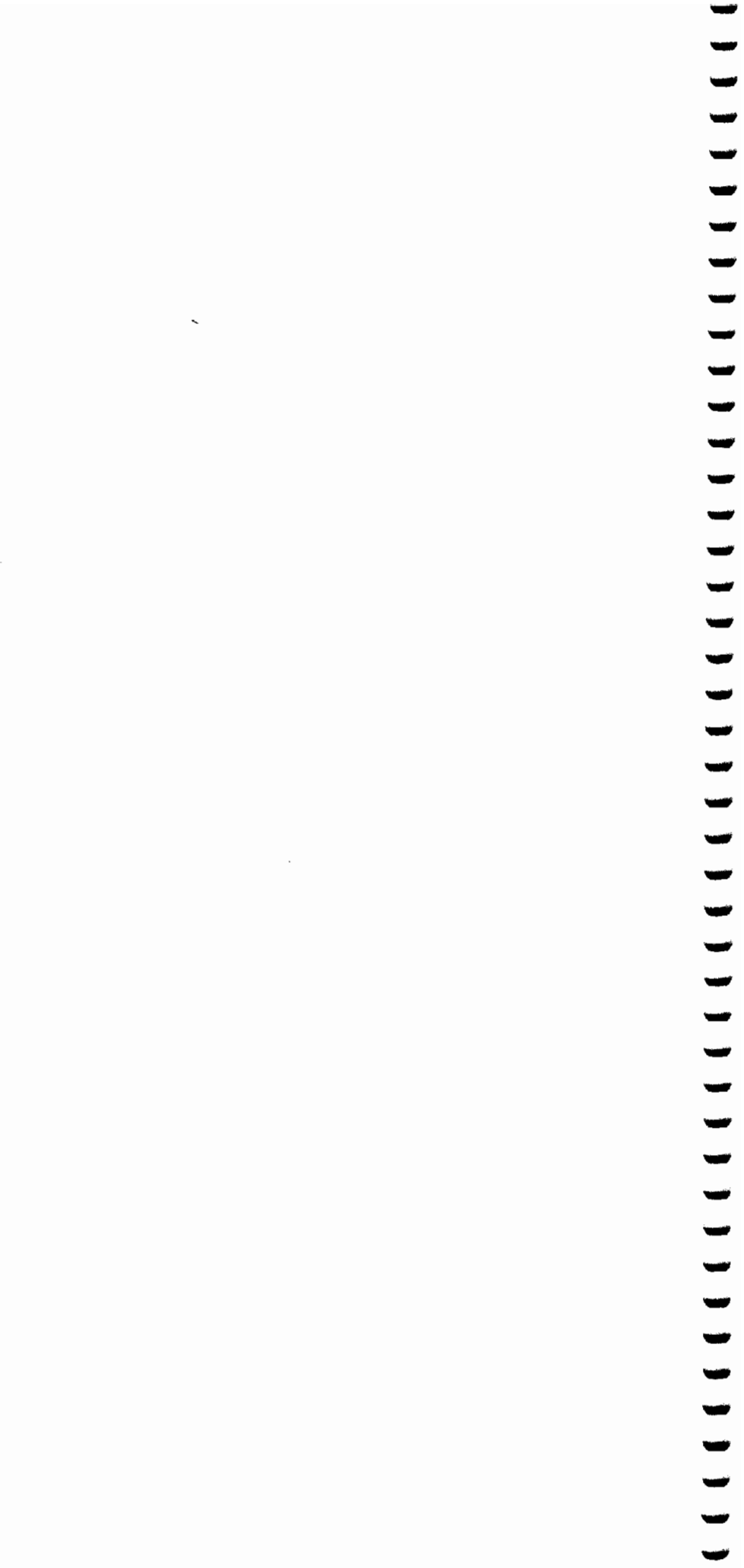
Reactor/Regeneration Valve Sequence (Single Feed Reactor)

Step ---->	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	
Valve																																					
A1	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
A4	o	o	o	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	o	o	o	o	o	o	o	o	o	o	o	o	o
A5	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	
A5X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
A6	X	X	X	X	X	X	X	X	X	o	o	o	o	o	o	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
A6X	X	X	X	X	X	X	X	X	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
A7	X	X	X	X	X	X	X	X	X	X	o	o	o	o	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B1	X	X	X	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B4	X	X	X	X	X	X	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
B5	X	X	o	o	o	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B5X	X	o	o	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B6	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B6X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
B7	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
R3	o	o	o	o	o	o	o	o	o	o	o	X	X	X	X	X	X	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	o	
Regen Step													A	B	C	D	E																				
Totalizer																																					

Valve Symbols: X = Closed o = Open

Regen Step A: "Ramp-Up" indicator illuminated. Ramp timer starts and regenerant temperature is ramped up. Long heat-up timer starts.
 Regen Step B: When ramp timer times out, "Heat-Up" indicator is illuminated and the controller waits for the bed to heat up.
 Regen Step C: When the bed temperature reaches the set point, the soak timer starts and the "Soak" indicator is illuminated.
 Regen Step D: When soak timer times out, the "Cool-Down" indicator illuminated and regenerant temperature set point is reduced.
 Regen Step E: When outlet temperature reaches the cool-down setpoint, the "Ready" indicator is illuminated and the Pause timer starts.
 From the "Ready" mode, the operator must give the "Sequence Start" command to initiate the next switching sequence.

Totalizer: 1 : Totalizer counts recycle benzene and paraffin/olefin feed to top bed.







V. PROCESS EQUIPMENT

A. VESSELS

1. Detal Reactors (see Figure V-1)
2. Benzene Column
3. Benzene Column Receiver
4. Alkylation Mixing Drum
5. Paraffin Column
6. Rerun Column
7. Rerun or LAB Clay Treater
8. Recycle Column
9. Recycle Clay Treater
10. Off-Spec Tank
11. Recycle Paraffin Stripper
12. Finishing Column

B. EXCHANGERS

1. Olefin Feed Exchanger and/or Cooler
2. Interbed Olefin Cooler
3. Benzene Feed Heater and Cooler
4. Feed/Product Exchanger
5. Paraffin Recycle Exchanger
6. Regenerant Exchanger
7. Regenerant Heater
8. Regenerant Cooler
9. Regenerant Effluent Heater
10. Benzene Column Condenser
11. Benzene Drag Cooler
12. Benzene Column Reboiler
13. Paraffin Column Overhead Cooler
14. Paraffin Column Reboiler
15. Rerun or LAB Column Overhead Cooler
16. Rerun or LAB Column Reboiler
17. Recycle Column Overhead Cooler
18. Rerun or LAB Clay Treater Exchanger
19. Recycle Column Reboiler
20. Heavy Alkylate Cooler
21. Paraffin Column Vacuum Pump Spillback Cooler

22. Rerun or LAB Column Vacuum Pump Spillback Cooler
23. Off-Spec Cooler
24. Reactor Charge Heater
25. Reactor A&B Interheaters
26. Regenerant-Reactor Effluent Exchanger
27. Benzene Column Middle Reboiler
28. Recycle Paraffin Stripper Overhead Cooler
29. Recycle Paraffin Stripper Reboiler
30. Finishing Column Overhead Cooler
31. Finishing Column Reboiler
32. Net LAB Cooler

C. PUMPS

1. Benzene Feed
2. Benzene Column Overhead
3. Benzene Recycle
4. Regenerant
5. Benzene Column Bottoms
6. Paraffin Column Overhead
7. Paraffin Column Bottoms
8. Rerun or LAB Column Overhead
9. Rerun or LAB Column Bottoms
10. Recycle Column Overhead
11. Recycle Column Bottoms
12. Off-Spec
13. Benzene Column Boot Water
14. Paraffin Vacuum Hydrocarbon Recycle
15. Paraffin Column Vacuum
16. Rerun or LAB Column Vacuum
17. Desorbent
18. Recycle Paraffin Stripper Overhead
19. Recycle Paraffin Stripper Bottoms
20. Finishing Column Overhead
21. Finishing Column Bottoms

Figure V-1 Split Feed Detal Reactor

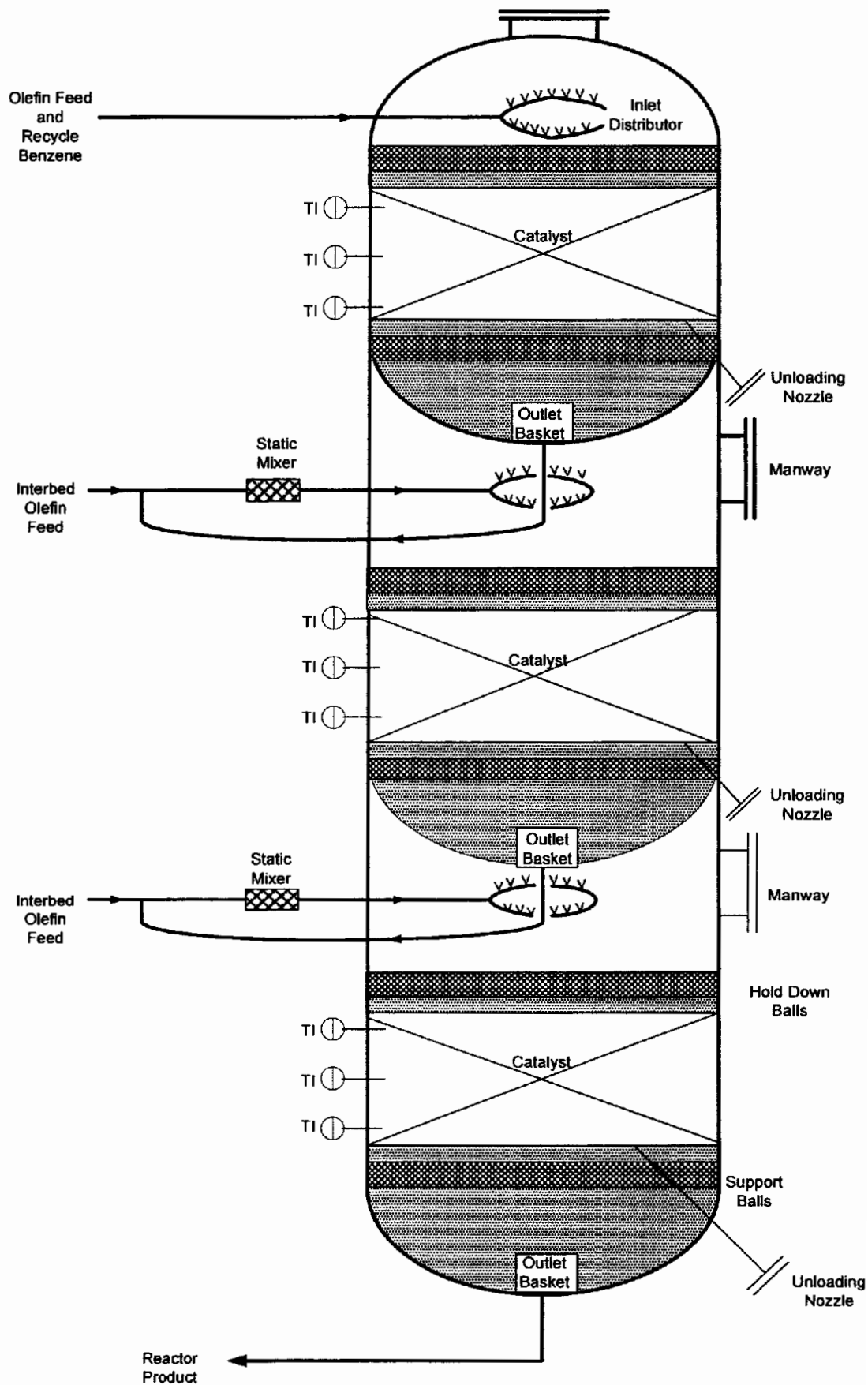
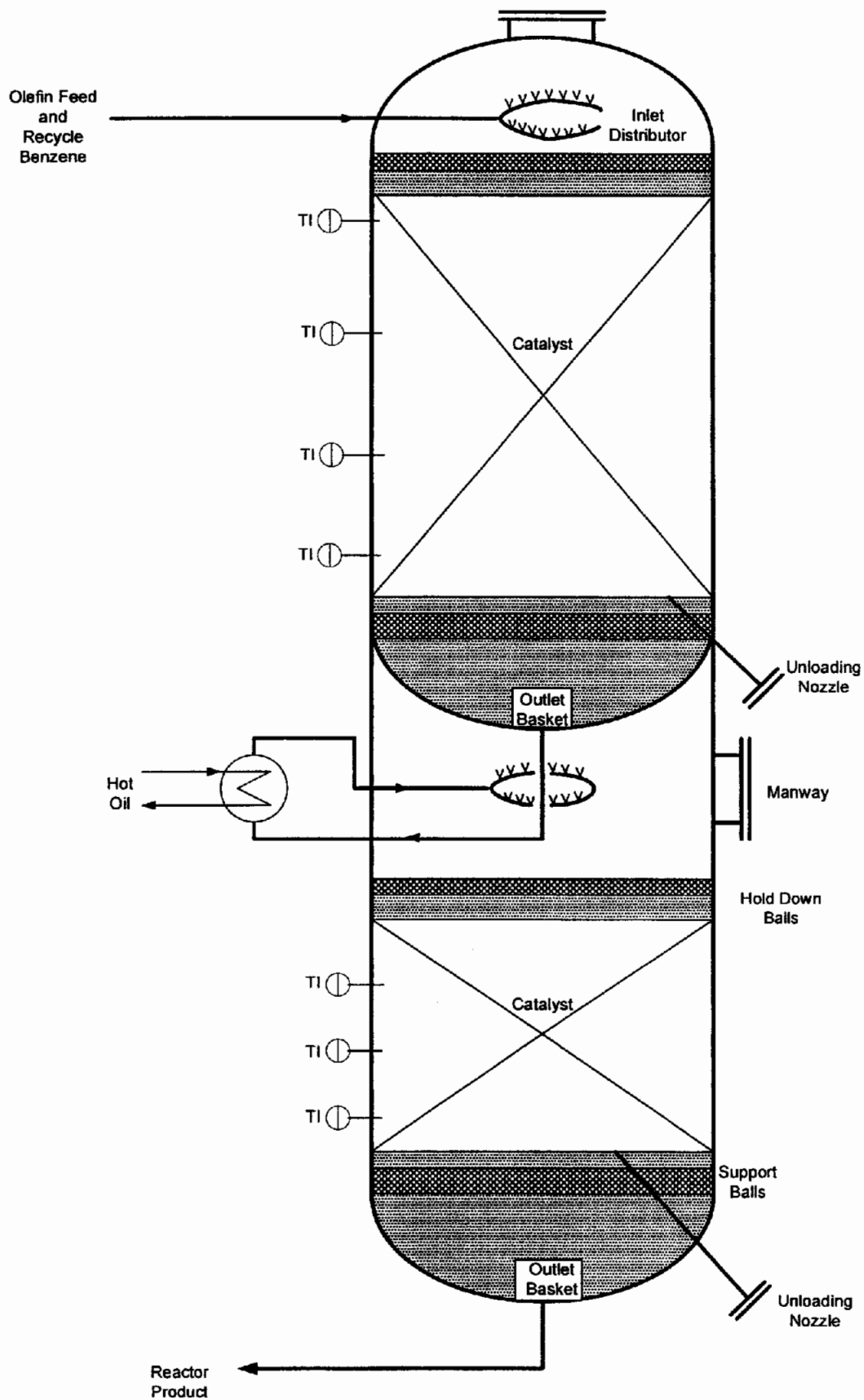
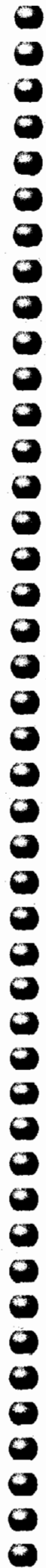


Figure V-2
Single Feed Detal Reactor







VI. COMMISSIONING

During the final stages of construction and upon completion of construction, there is a large amount of detailed preparatory work which must be done by the operating personnel in cooperation with others at the job site. The primary reasons behind this work are:

- To acquaint the operators with the equipment.
- To inspect the unit for the adherence of the construction to the piping and instrument diagram.
- To ensure the lines and equipment are free of debris and construction trash.
- To "run in" pumps and drivers.
- To commission all utilities and support facilities.
- To acidize the unit for removal of mill scale.
- To dry out the unit (initial).
- To load catalyst and clay.

This section covers the work required in preparing the unit for operation. Such work as testing and cleaning is generally carried out during the final stages of construction and, on the whole, is performed only once. However, after turnarounds, it may be necessary to perform some of these items either in part or in entirety. Careful and thorough unit preparation is a prerequisite for satisfactory performance and elimination of trouble and delays during startup and normal operation. An outline is presented for a general method of cleaning and flushing lines and equipment. The outline for checking pumps, after installation and prior to operation, is applicable on all startups. Much of the work noted in this section is

performed by construction personnel. No attempt is made herein to designate the personnel performing this preparatory work. There are some phases of the preparation, such as the run-in of pumps during water circulation for line cleaning, which are best completed by the refiner's operations personnel. Much valuable experience can be obtained in this manner.

A. EQUIPMENT AND INSTRUMENTATION INSPECTION

Piping Inspection

The piping and other construction should be checked to make sure it conforms to the specifications set out in the Schedule A design. It is necessary to confirm that the piping is arranged according to the piping and instrumentation diagram. The inspection should include the following:

1. Line sizes and materials
2. Flanges and gaskets
3. Bolting
4. Globe, gate, and check valves
5. Drains and vents
6. Relief valve settings
7. Lines and equipment supports
8. Equipment accessibility
9. Expansion provisions
10. Utility lines
11. Tie-ins with other units/storage
12. Pump ratings
13. General safety requirements

Vessel Inspection

Prior to any vessel closure, a thorough inspection should be performed. The actual installation should be compared to the vessel drawings specified on the project specification sheets. A suggested general check list includes:

1. Vessel temperature and pressure rating.
2. Metallurgy as specified.
3. Vessel elevation.
4. Nozzles.
5. Installation of trays: number, type, spacing, levelness, liquid seal, weir height, valve number and operation, openings in perforated or sieve trays, metallurgy, bolting, supports.
6. Internal distributors and mesh blankets: location, alignment, size and number of openings.
7. Internal drawoff piping.
8. Vortex breakers, splash baffles, and risers.
9. Location and length of thermowells.
10. Location and range of level instrument internal floats, external type displacement tube cages or differential type nozzles.
11. Material damage from corrosion or wear.
12. Cleanliness.
13. Completeness.

CAUTION: Before entering the vessel, the refinery's safety precautions should be observed. These usually include the following: sampling the vessel for toxic vapors and oxygen concentration, wearing a safety harness, and having an attendant outside the vessel. An unattended vessel should never be entered.

Instrument Inspection

Preparation of the plant instrumentation for startup and operation should include the following inspections and tests:

1. Visual inspection of instruments to verify compliance with specification.
2. Visual inspection of instrument installation to verify compliance with specifications and installation details.
3. Pressure testing of instrument piping.
4. Testing and calibration of instruments.
5. Testing of instrument air signal lines.
6. Testing of thermocouple wiring.
7. Loop checking.
8. Final preparation.

In inspecting instruments for compliance with specification, attention should be given to instrument range, pressure rating, connections, materials of construction, size, electrical characteristics, and special features. Special attention should be paid to notes in the specification. Orifice plates should be inspected as soon as possible so they will be ready for installation when line flushing is complete; inspection should include size, material of construction, location of weep hole (if any), counterbore, overall condition, measurement of orifice bore, and verification of information stamped on tab.

Inspection of installations should include attention to location of transmitters with respect to the connection point; to piping for proper drainage, and for proper valves and gaskets; to transmitters and control valves to ensure that they are not installed backwards; and to special treatment such as winterizing, heat tracing, and insulation. Orifice installations should be checked as soon as possible to ensure that straight run requirements are met, so that any piping changes required can be made.

Instrument piping should be inspected visually for completeness and condition of welds and connections, and tested by pressuring with instrument air and brushing joints with soap solution. During hydrostatic testing of the plant, the first block valve at the process line should be closed and a drain valve opened to relieve any pressure. Control valve assemblies may be included in the hydrostatic test, providing that it is understood by all personnel that any leaks at control valve stem packing will be corrected by a qualified instrument technician only. It is generally safer to exclude the control valve assemblies from the hydrostatic test.

Testing and calibration of instruments should include calibration of transmitters (in place, if possible), control board equipment, and local controllers. Control valves and positioners should be adjusted. Special attention should be given to instrumentation used in critical services (such as heater firing, etc.) and accounting services (such as charge and product meters). Temperature transmitters should be checked for proper thermocouple burnout protection. Meter factor tags and valve position indication on controller output indicators should be installed at this time, and control actions should be set.

Instrument air signal lines should be checked for leakage, kinked tubing, and for connections to the proper port of shutdown, solenoid valves and controllers. Thermocouple wiring should be inspected for proper polarity at the thermocouple head. Point by point verification of the switch identification at the temperature indicator on the operator's desk can be accomplished by selecting a point to be checked, opening the circuit at the thermocouple head and checking the temperature indicator to see if it drives upscale or otherwise indicates an open circuit. The same applies for TRC instruments on the control board. Loop checking

involves checking inputs and outputs to ensure that piping and wiring has been properly done. A signal should be simulated at the transmitter, and the control board instrument should be watched for response. The controller output should be adjusted manually, and the control valve watched for response. Special attention should be given to split range loops and complicated systems.

Final preparation includes checking instrument piping to verify that valves are in the proper position, making preliminary control mode adjustments, and making sure that instrument air and electrical power are supplied to all instruments. It may also be necessary to have the steam tracing operable at this time.

Commissioning of instruments will be carried out as the plant comes on stream. Most attention in commissioning of instruments will be directed toward liquid flow measurements; differential pressure instrument piping must be drained to eliminate accumulated dirt and water, and the instrument zero must be adjusted to obtain a correct zero reading on the scale on the control board instrument. Controllers must be tuned, interface level transmitters must be zero checked, scale, weld slag, and trash must be removed from sticking control valves, and other malfunctions must be corrected. Inspection and commissioning of analyzers should include attention to sample line lengths, analyzer enclosures, auxiliary equipment such as carrier gas for chromatographs, and other items as indicated in the instruction manual for the particular analyzer.

B. HYDROSTATIC TESTING

Hydrostatic tests are made on new or repaired equipment to prove strength of materials and welds. This test is made by completely water filling the equipment to be tested and increasing the pressure to the specified test pressure. Normally, a positive displacement pump will be used to raise the pressure. This test should not be compared with other less severe tests generally carried out before a startup to check the tightness of connections.

The hydrostatic test is ordinarily performed by construction personnel. If for any reason it should become necessary for the operators to carry out the test, the following suggestions should be considered:

1. Care should be taken that the temperature is not too low causing metal to become brittle; vessel and water temperature should always be above 15°C (60°F) to eliminate the possibility of cold fracturing the metal.
2. Test pressure is normally 1.5 times the design operating pressure.
3. All items under test must be liquid full with all air vented.
4. Relief valves or rupture discs must be removed, blinded, or gagged.
5. Internals, such as level ball floats, which are not designed for the test pressure must be removed.
6. Items such as connecting piping to a vessel under test, which are capable of withstanding the test, must be subjected to the test.
7. Items such as vessels connected to piping under test which are not capable of withstanding the test, must be isolated by blinding.
8. Divide the unit into sections as governed by test pressure and location of items to be tested, and isolate the sections by installing suitable blinds.
9. All relief valves (PSV's) are required to be bench tested and set before final installation. This testing and setting may be done while hydrostatic testing is in progress.
10. It should be remembered that in pressure testing all vessels and towers, the test gauge should be placed at the bottom of the vessel so that the liquid head will be taken into account.

NOTE: A water-filled system requires venting when being emptied in order to avoid collapsing equipment due to a vacuum being pulled.

C. UTILITY COMMISSIONING

Following the flush of the various utility lines, they must be put in service. Where possible, steam lines should be opened and blown out to the atmosphere, then flanged up and pressured to test for leaks. Steam lines should be warmed up slowly to prevent damage by water hammer. All steam traps and control valves in the system are to be placed in service. Cooling water lines must be flushed. Remove any control valves, flush through the line, then install and flush through the next section. Remove an ell or by some means disconnect the inlet lines to all condensers and coolers before flushing. Never flush through a cooler, as the tubes are likely to become plugged by the dirt and debris left from construction. Air lines should be blown out with steam, followed with an air purge and finally tested with air pressure. Instrument air lines should be tested with soap to detect small leaks. Electric power lines to the electric motors can be energized at this time and motor rotation checked. Nitrogen should be available where required.

D. LINE FLUSHING

All piping must be thoroughly cleaned of debris and scale. This may be done after hydrostatic testing, before the test water is drained. Generally, fluid handling lines are flushed with water and thoroughly drained, while gas handling piping may be either water flushed or air blown. Water should be blown from gas lines on completion of flushing.

Steam and utility air piping may be cleaned by blowing with their normal media. Instrument air lines should be thoroughly blown with clean, dry air. Upon completion of these and other utilities line flushing, the utilities should be commissioned and made ready for service. See "Utility Commissioning" above.

The following items are suggested as a guide for line flushing:

1. Where practical, clean water should be supplied to the vessels and the connecting lines should be flushed away from the vessel; never flush into equipment.
2. No matter what the flushing medium (steam, air, or water), maximum volume and velocity should be used for thorough cleaning.
3. Orifice plates must not be installed.
4. Control valves should be removed.
5. Instrument lines should be closed off or disconnected.
6. At pumps:
 - Disconnect suction and discharge piping and flush lines.
 - Reconnect lines for circulating water.
 - See further discussion under "Run-in of Pumps and Drivers."
7. Regulate rate of flushing medium from source. For example, when flushing with water from a tower, regulate the water flow at the tower; when flushing with steam, regulate the steam flow at the valve in the supply to the line being blown.
8. Flush through all vents and drains.
9. Where possible, flush downward or horizontally.

10. Flush or blow:

- The main header, from source to end, then
- Each lateral header, from the main to end, and finally,
- Each branch line, from the lateral header to end.

11. Always flush through bypass to an open end before flushing through equipment.

12. Disconnect lines at exchangers and flush to open end lines.

13. For steam systems, flush well through dirt leg drains and steam trap bypasses before placing the traps in service.

NOTE: Care must be taken to avoid a vacuum condition when draining any pieces of equipment. When flushing from a vessel or other equipment, adequate venting must be provided.

Upon completion of line flushing of any system, carefully check that all temporary breaks are reconnected, control valves replaced, and pump alignments are normal. Drain as much water as possible from the piping and equipment after flushing.

E. RUN-IN OF PUMPS AND DRIVERS

Proper installation and operation of pumps and drivers is essential for trouble-free performance. The pumps and drivers should receive careful handling during initial run-in. The initial run-in of pumps is generally done by circulating water through the new equipment. Temporary strainers are installed in the suction line of the pumps, conveniently located for removal and cleaning. The screens also should be positioned such that dirt particles will not gravitate to inaccessible places when flow is stopped.

During run-in of pumps, the strainers may cause some restriction of flow. As debris collects in the strainers; flow to the pump will fall off. In this event, it will be necessary to limit the flow of centrifugal pumps by throttling the discharge valve. Avoid restricting the discharge to the extent that it will cause internal slippage and excessive heat generation. The pump must not be permitted to cavitate (i.e., "lose suction"), which may cause damage to the pump. In starting a centrifugal pump, the rotation should be brought up to speed as rapidly as possible. Proper operating speed is normally attained rapidly and automatically with motor driven pumps and the usual motor starting.

The development of discharge pressure is essential to flush and lubricate the wearing rings. Subsequently, it is advisable, where practical, to close the pump discharge immediately prior to shutting down a centrifugal pump, to maintain maximum flush through wearing rings. The discharge valve on an operating positive displacement type pump, rotating or reciprocating, should not be closed as this will cause overpressuring. Pumps in hot service must be gradually warmed to near operating temperature before placing in service. Otherwise uneven expansion of the internals and casing may cause seizure and damage when starting. The following items are suggested for checking prior to run-in:

1. The manufacturer's operating instructions should be reviewed for any specific precautions that should be observed.
2. Completion of overall installation.
3. Verify that pump and driver have been aligned for cold operation, no undue strain by the piping on the pump or driver is allowable.
4. Cooling fluid piping and seal or gland oil piping:
 - a. Conventionally packed pumps in hot service are generally furnished with gland oil; verify this installation is correct and complete.

- b. For pumps with mechanical seals; verify that all of the components of the flushing system, such as strainers, separator, restriction orifices, and coolers (when required), have been correctly installed and are clean. Loss of flush, or dirty flush may cause the loss of seals. Process flushing to mechanical seals and to stuffing box jacket for hot pumps has been specified for this unit. In addition, cooled product flushing material is specified for stuffing box jackets with return to pump suction or to mechanical seals.
- 5. Verify that packing or seals are installed.
- 6. Verify bearings and shafts have been cleaned prior to final lubrication.
- 7. Check that pump and driver are lubricated according to lubrication instructions.
- 8. Check rotation of electric motor drivers uncoupled from the pump. Run-in the motor, uncoupled, verifying operability.
- 9. Water is usually heavier than the normal process fluid. The pump driver (motor) is sized for the normal pumping fluid; consequently, running-in the pump with water could cause an overload of the electric motor. To avoid overloading the motor of a centrifugal pump when running-in with water, the flow must be limited by throttling the pump discharge valve. When doing so, if possible, check the amperage usage against design.

NOTE: Due to the possibility of overloading the pump motors and damaging the mechanical seals, many refiners elect not to run-in new pumps with water.

- 10. The following items are suggested for checking during pump run-in:
 - a. Rotate pump and driver by hand, verifying they will roll freely.
 - b. Check that run-in water circulation is lined up.

- c. Open suction valves fully, venting air from piping and pumps, and completely filling with liquid.
- d. Establish flow of cooling fluid, where required.
- e. Check that lubrication is satisfactory.
- f. Make sure electric power is available from the switch gear to the starter of the electric motor driver.
- g. Barely open the discharge valve on the centrifugal pump.
- h. Start the pump; if the pressure does not build immediately, stop and resolve problem.
- i. When the discharge pressure has increased satisfactorily after starting, gradually open the discharge valve to obtain the desired flow rate.
- j. In the event of unusual noise, vibration, overheating or other abnormal conditions, shut the pump down immediately; correct the cause before resuming use of the pump. Continue to check for abnormal conditions as these may occur after prolonged operation.
- k. Check shaft sealing; mechanical seals should show no leakage; conventionally packed stuffing boxes must always be permitted to leak slightly, to provide some lubrication and prevent overheating; stuffing box gland nuts are generally only finger tight. A new mechanical seal will sometimes have a small amount of leakage on startup, however, after the pump is started and stopped a few times, the leakage may stop. Prior to pump run-in on water, check with the vendor to confirm that the pump and mechanical seal can be run on water. In some cases, mechanical seals are not installed until after water run-in with temporary packing being used during the water run-in period.

- l. Operate the pump, directing flows through all suction and discharge piping circuits.
- m. Inspect and clean screens as required.
- n. After any disturbance of the piping (such as is required for suction screen inspection if pipe flanges have to be parted for screen removal), check the pump alignment and realign if necessary.
- o. When shutting down a centrifugal pump, close the discharge valve first, thereby maintaining discharge pressure while the pump rolls to a stop; this will also help protect against the pump rolling backwards should the discharge check valve leak. Positive displacement pump discharge valves must not be closed until the pump is shut down and rolling stops.
- p. After all lines available to a pump have had suitable flushing, the temporary suction strainers are left in for the hydrocarbon dryout. Only after showing free of debris on two successive examinations during the hydrocarbon circulation are the temporary strainers taken out. The permanent strainers (with larger holes) may then be installed where required.

F. HOT OIL SYSTEM

If a hot oil system is used, the procedure in the Hot Oil System Manual is to be used in commissioning it. Before commissioning the hot oil circulation system, it will be necessary to slowly expel the excess moisture from the refractory of the system's heater. This procedure is covered in the Hot Oil System Manual.

G. VESSEL LOADING

1. Clay Treater

The clay treater should be loaded after the initial line flushing (with water) of the unit is finished and before the dryout procedure begins. The vessel should be clean and dry before the clay treater is loaded. The procedure for loading the clay treater is given in Section XIII (Special Procedures).

2. Detal Reactors

The bottom support balls in the reactors should be loaded before the dryout procedure is started. However, the Detal catalyst cannot be loaded until after the dryout procedure has been completed (see Section VII, Normal Startup). The loading procedure for both the bottom support balls and the catalyst is given in Section XIII (Special Procedures).

H. LEAK TESTING

Just before the air-freeing of the unit is performed, the unit should be pressure tested to check for leaks. This test is not the same as the hydrotest. The purpose of the leak test is to make sure all flanges, instruments, and equipment have been properly connected before bringing hydrocarbon into the unit. The leak test can be performed with either air or nitrogen. If nitrogen is used, the leak test may be used to help air-free the unit. The following items should be used as a guideline for leak testing:

1. Divide the unit into sections and test each section separately. This will make it easier to find leaks.
2. Perform a thorough visual inspection of each section to identify obvious problems such as loose flanges and open vents or drains before pressuring up.

3. Include as many instruments as possible in the leak test. All orifice plates should be installed before the leak test is performed.
4. Pumps should not be included in the leak test. Most pump seals will not hold pressure if the pump is not running. Visually check the pump connections to make sure they are properly installed and tightened.
5. Pressure relief valves should be installed and included in the leak test.
6. Pressure up with air or nitrogen to about 4 kg/cm² (60 psig), or the design operating pressure, whichever is less. Do not over-pressure the equipment.
7. During the test, every flange, joint, valve packing, and closure should be checked for leakage using soap solution.
8. The leak test should be held for at least 2 hours with no loss of pressure.

I. VACUUM EQUIPMENT

The vacuum under which some of the fractionation columns operate is provided by either vacuum pumps or a series of ejectors operated with steam.

Vacuum pumps mechanically pump the non-condensable gases from a vacuum up to atmospheric pressure so the gases can be vented. Low vapor pressure seal oil is used in most vacuum pumps and it is important to prevent higher vapor pressure hydrocarbons from building up in the seal oil or the pump will lose vacuum. A flow-through seal oil system can be used in services where higher vapor pressure hydrocarbons may be present (such as in the paraffin column).

In an ejector system, steam is injected at high velocities through a specially designed nozzle and transfers sufficient energy to the gases from the suction header to entrain them through the diffusers into the first stage discharge header. The pressure in the first stage discharge header is, of course, higher than the

pressure in the suction header, but if the velocity of the steam through the diffuser throat is high enough, gas cannot back into the suction header. If a single ejector is incapable of raising the gases to atmospheric pressure so the gases can be vented, the steam is condensed and a second ejector taking suction on the non-condensable gases raises them to a higher pressure. The condensate is evacuated through long pipes which discharge into the condensate separator. The long barometric legs create enough liquid head for the condensate to be drained at atmospheric pressure.

After the vacuum columns and associated piping have been hydrostatic pressure tested, water flushed, and leak tested, they should be vacuum tested. This serves as a check of the vacuum system and a further test on the tightness of those pieces of equipment which will operate under a vacuum. It is particularly critical that the rerun/LAB and recycle column bottoms pumps and suction lines have no air leaks, because it will be difficult to maintain suction to these pumps (relatively low flow rate) with any air leakage in the suction piping.

1. Vacuum Test

After the leak test has been performed on the vacuum columns, a vacuum test must be done. The vacuum test serves several purposes. First, the vacuum test will identify significant leaks that may not have existed or were not found during the leak test. (Many times a vacuum leak will not leak during a leak test where the pressure is on the inside of the vessels and piping.) Second, the vacuum pumps or ejector system can be commissioned and checked out during the vacuum test. Finally, the air will be removed from the piping and equipment being vacuum tested; eliminating the need for further air-freeing procedures.

IMPORTANT NOTE: Never pull a vacuum on piping or equipment that has not been designed for full vacuum. The forces due to external pressure on a vessel are much different than the forces due to internal pressure, and even vessels rated for high internal pressure may not be able to withstand an internal vacuum.

NOTE: Careful consideration must be given to the destination of the vacuum equipment vent gas during startup. Air (or oxygen) must not be sent to the flare header. Hydrocarbon vapors must not be sent to the hot oil heater box before the heater is started up.

- a. Remove any blinds in the vacuum columns which would interfere with a true vacuum test on this equipment.
- b. Close the block valves and bypass valves on the charge, reflux, and bottoms to and from these columns. This valve closure should also include the isolation of all pumps in this service. Also check to see that the pump vent lines on all of these pumps are closed.
- c. Start up the vacuum producing equipment (pumps or ejectors). Commission the column pressure controllers and line out the column pressure at design.
- d. Check each column and valve flange for leakage. Leaks may be detected by sound, or by taping the flanges and using a foam such as shaving cream.
- e. Check for valve packing gland leakage.
- f. When the vacuum test is completed the vacuum should be broken by introducing nitrogen into the columns just before the vacuum producing equipment is shut down. The columns should then be pressured up with nitrogen to about 0.7 kg/cm² (10 psig) to prevent air leakage until the unit is started up.

2. Vacuum Pump Startup

The manufacturer's instructions should be followed for starting up the vacuum pumps. This procedure will vary depending on the type of pump, so detailed vacuum pump startup instructions will not be given in this manual. The following are some general notes on starting up the vacuum pumps:

- a. Make sure the vacuum pump discharge line is open to the proper destination (usually a heater firebox). The nitrogen purge on the discharge line should be commissioned before starting the pump.
- b. The seal oil system (on pumps with a flow-through seal oil arrangement) should be commissioned before starting the pump.

3. Ejector System Startup

- a. Pressure test the vacuum ejector condensers:
 - i. Install blinds at inlet to condensers, vent nozzle after condensers, and in barometric legs.
 - ii. Fill the condensers with water and pressure test at 2 kg/cm² (30 psig).
 - iii. Drain system and remove blinds.
- b. Start cooling water to all of the ejector condensers.
- c. Open the steam to the first ejector. Operate this ejector alone to ascertain the performance of a single ejector. Once this has been attained, open the steam to the second ejector and determine the ejector system capability. Do the same for any subsequent ejector.
- d. Check that there is a liquid level in the condenser shells. If the condensers are empty, the steam piping and strainer should be checked for blockage.
- e. Check the available steam pressure. It must be maintained quite close to that for which the equipment was designed. If the steam pressure greatly exceeds that for which the nozzle was designed, the quantity of steam discharging into the diffuser will be greater than can pass through the diffuser, and steam will back into the suction header. It should be noted that wet steam will cause

random fluctuation in ejector performance and in addition will erode the nozzle and diffuser.

- f. Check the discharge pressure of the ejector. If the discharge pressure rises above design, there is an increasing probability of reverse flow. An increase in discharge pressure on an ejector discharging to atmosphere is only possible if the discharge is obstructed. But on multi-stage units, an increase in interstage pressure due to high condensate temperatures or failure of a second or third stage ejector will immediately affect the performance of the first-stage unit.
- g. Check the load on the system. A decrease in load (kg/hour of vapor to ejector) will result in a somewhat higher vacuum being obtained, but if a load is increased above design, the vacuum obtained will fall off quite suddenly and dramatically. The column pressures are controlled by adjusting the load on the ejectors using the non-condensable spillback control valve.
- h. Check the condenser cooling water temperature. The temperature at which the steam is condensed in the condensers will have a relatively minor effect on the vacuum obtained, but will substantially reduce the load at which the ejector system breaks down, since an increase in condensate temperature increases the interstage pressure. The minimum combination of equipment which will achieve a satisfactory vacuum is normally used. The vapors drawn from the top of a typical unit to the ejectors consists of air from leaks and light hydrocarbons. The steam and light hydrocarbons will condense in the first condenser so that the first and second stage ejectors can be heavily loaded under conditions which only lightly load the third stage ejector.
- i. When shutting down the ejectors, the vacuum should be broken by introducing nitrogen into the systems before the steam to the ejectors is blocked in and then the cooling water blocked in.
- j. After the vacuum test, and during turnarounds, the strainer in the steam line should be cleaned and also the ejector jets should be disassembled for cleaning.

J. AIR FREEING

Prior to the addition of hydrocarbons to the unit, a check must be made to ensure that all oxygen has been removed from the process vessels, lines, and equipment. An explosive mixture can result with the hydrocarbon if this oxygen is not removed. All equipment that has not yet been freed of oxygen should be purged with steam or nitrogen. Nitrogen should be used to purge the relief and flare system. Steaming followed by nitrogen purge and blanketing is generally the technique recommended by UOP for air freeing the unit prior to startup. Use of nitrogen purging without steam drying will tend to lengthen the dryout time in most cases. Do not use the ejector system to evacuate and purge parts of the unit that are not designed for vacuum.

Steaming Out

Following is an outline of the general refinery practice to steam out vessels and lines before startup. Block in all cooling water to coolers and condensers and drain the water. Open high point vents and low point drains in vessels to be steam purged.

1. Begin steaming process vessels by introducing steam to the bottom of the vessel. Make maximum use of the steamout connections to introduce dry steam. Drains and vents can also be used to bring steam into the unit.
2. Each vessel should have a net flow of steam from bottom to top. Each line should have a net steam flow to purge the line.
3. Systematically purge all lines and vessels with steam while draining condensate from low points.

NOTE: DO NOT steam purge the loaded clay treater during this period.

4. Continue steaming until a strong plume of dry steam comes from each of the high point vents and drains.

5. Throttle the steam vents and drain condensate while building the pressure to 2-3 kg/cm²g (25-50 psig). Check the system for leakage and maintain minimum condensate inventory. The higher the pressure, the hotter the metal, which will flash off water when the pressure is reduced. Hold the pressure long enough to heat up the metal to approximate steam temperature and then discontinue the steam purge.
6. To aid in drying the unit and expelling the steam rapidly, open vents and drains fully to expel the steam.
7. As the steam pressure starts to decrease after steam is discontinued and vents are opened fully, it is important to keep air out of vessels. This can be done by using nitrogen to maintain at least 0.7 kg/cm²g (10 psig) as steam is expelled. Drain low points to remove any residual water.
8. Check to be sure the oxygen content is below the acceptable limit (usually about 0.3%). If not, follow the nitrogen purging procedure below.
9. Start cooling water to all exchangers, condensers, and turn on all fans.

Nitrogen Purging

It is also acceptable to use nitrogen purging without steamout for air freeing the unit prior to startup; however, this will normally extend the required dryout period. Nitrogen purging may be preferred in some cases because of unusual circumstances such as: the unit has never been wet, or steam is not available in the quantity and quality needed, or in cold weather where nitrogen purging should be used rather than steamout to reduce water freezing problems. If nitrogen is used, purge according to the standard refinery practice until the oxygen content is acceptable, then pressure up to around 0.7 kg/cm²g (10 psig) and maintain this blanketing pressure until startup.





VII. NORMAL STARTUP

This procedure is for the original plant startup and covers all procedures that are likely to be required at that time. For subsequent restart operations, all the steps covered here may not be necessary and the procedure can be altered to fit the situation. The dryout procedure involves the circulation of dry benzene and paraffins through all lines and vessels. This operation will mark the first time that hydrocarbons are circulated in the unit. This is an ideal occasion for a first check of the unit and training of the operators. All equipment should be put in service at this time. All pumps should be run in and all instrument loops should be operated.

Prior to introducing hydrocarbon to the unit, all of the Commissioning activities (described in Section VI) must be completed.

The Detal catalyst should not be loaded into the reactor before dryout.

Before the unit is inventoried with hydrocarbon, the following checklist of items should have been completed:

- The bottom support balls should be loaded in the reactor. The catalyst should not be loaded yet.
- The clay treaters should be loaded.
- All vessel internals must be installed so that they will be included in the dryout.
- A leak test should have been performed to verify tightness of flanges, equipment and instruments.
- The Pacol and PEP units should be ready for startup.

- All safety equipment which will be required for hydrocarbon addition to the unit must be on-site and operable.
- Use a blind list to verify that all blinds are removed or in place as required.
- All utility systems should be in service, including the flare system.
- All block valves for pressure relief valves must be locked in the full open position with the entire relief system in service.
- All instruments should be ready for service.
- Commission all steam tracing.
- Start cooling water to all coolers and condensers and turn on fans for all air coolers.
- Commission the hot oil circuit.
- Check that all pumps are equipped with temporary strainers for protection. These strainers are to be removed when the dryout is completed, except in the case where the strainers give protection against fouling small holes, such as in the packed column feed and reflux streams.
- Check that all vessels and lines are free of oxygen.
- Check that the benzene and paraffins to be used during dryout meet specifications.

During the dryout period, constant surveillance will be required by all associated with the startup as it will be harder to hold column levels and temperatures than it will be with design materials.

During this dryout circulation it is possible that the pump suction screens will plug up. All pumps should be checked frequently to be sure they are not cavitating. They should also be checked for overheating of the bearings and packing glands, any unusual noises, or vibrations. Flush and cooling water are to be in service to the pumps that require it.

The dryout and startup operations must be coordinated with the Pacol and PEP unit startup procedures.

The sequence of operations is as follows:

A. HYDROCARBON CIRCULATION

1. Startup the paraffin feed stripper column (in the Pacol unit) on total reflux.
2. Inventory the Pacol feed surge drum.
3. Inventory the Pacol stripper column bottom (via Pacol reactor bypass and DeFine bypass).
4. Establish a vacuum on the paraffin column. (Note: depending on the vacuum equipment, it may not be possible to establish a vacuum on the column before filling the accumulator tray with paraffin. For example, it may be necessary to provide recycle paraffin to the vacuum pump for use as seal oil. In that case, the paraffin column vacuum can be established after completion of step #7 below.)
5. Establish a paraffin level in the bottom of the benzene column by bringing in Pacol stripper bottoms via the paraffin/olefin feed control valve (or the #1 (top) bed feed control valve for Split-Feed reactors), through the Detal reactor bypass, filling the mix drum (in units that have a mix drum), and into the benzene column. When a level appears in the bottom of the benzene

column, stop the flow of Pacol stripper bottoms to the unit. Start the benzene column bottoms pump and spill back to the reboiler tub.

6. Inventory the recycle paraffin stripper and paraffin column accumulator trays using the start up filling line from the benzene column bottoms pump. Bring in Pacol stripper bottoms to the benzene column as necessary to keep a level in the bottom of the benzene column. (Some units have a start up filling line coming from the paraffin/olefin feed line which can also be used to fill the accumulator trays.)
7. Start the recycle paraffin stripper and paraffin column overhead pumps and establish a flow of "cold reflux" to the contact condenser in both columns.
8. Bring up the Pacol stripper column on total reflux.
9. Prepare to bring in makeup benzene to the benzene column overhead receiver. Start the benzene feed pump and spill back to the benzene feed storage tank.
10. Start the benzene column overhead condenser and begin heating the benzene column with hot oil to the bottom reboiler. Bring in hydrocarbon from the Pacol stripper bottoms and benzene from the feed tank (or bring in material from the off-spec tank on subsequent startups) to maintain the levels in the benzene column as it heats up.
11. As a liquid level appears in the benzene column overhead receiver, the overhead pump can be started and reflux established to the top of the column. Also, as a level appears on the recycle benzene accumulator tray, the benzene recycle pump can be started and reflux below the accumulator can be established. The benzene recycle and regenerant, and PEP desorbent lines should be kept closed for now. Stabilize the column operation on total reflux.

NOTE: On the initial startup it may seem desirable not to bring any benzene into the unit until after the catalyst is loaded (if possible) because startup maintenance and vessel entry is safer when benzene is not present. However, past operating experience suggests that it will probably be necessary to bring in benzene to stabilize the operation of the column and prevent excessive temperature in the benzene column overhead system.

12. When the pressure in the paraffin column is lined out and the benzene column is running on total reflux, the recycle paraffin stripper and the paraffin column can be started up.

- a) Start up the recycle paraffin stripper. (If there is no recycle paraffin stripper, proceed to start up the paraffin column as described in Step b) below.)

Make sure the cold reflux circulation to the contact condenser is running, then establish the design pressure on the recycle paraffin stripper. Start a small flow of feed from the benzene column bottoms pump to the recycle paraffin stripper through the control valve. Maintain the levels in the benzene column by bringing in feed from the Pacol stripper bottoms (or the off spec tank). When a level appears in the bottom of the recycle paraffin stripper, start the recycle paraffin stripper bottoms pump and spill back to the reboiler inlet. Also start heating up the recycle paraffin stripper by starting a flow of hot oil to the reboiler. When the overhead level accumulator begins increasing, start a flow of hot reflux to the column. Begin sending net overhead liquid from the recycle paraffin stripper to the Pacol feed surge drum as necessary to control the level on the accumulator tray. At first, use the benzene column middle reboiler bypass line to send net recycle paraffin liquid to the Pacol unit. As the heat to the recycle paraffin stripper reboiler is increased (and the heat load on the overhead cooler increases), begin circulating some recycle stripper overhead liquid to the benzene column middle reboiler. Gradually increase the flow circulating liquid flow to the benzene column middle reboiler as the recycle paraffin stripper is heated up. When the flow to the

benzene column middle reboiler exceeds the net overhead flow from the recycle paraffin stripper, close the middle reboiler bypass line and open the normal net overhead line.

- b) Start up the paraffin column.

Start a small flow of feed to the paraffin column through the feed control valve. Maintain the levels in the benzene column by bringing in feed from the Pacol stripper bottoms (or the off spec tank). When a level appears in the bottom of the paraffin column, start the paraffin column bottoms pump and spill back to the reboiler tub. Also start heating up the paraffin column by starting a flow of hot oil to the reboiler. When the overhead accumulator level begins increasing, start a flow of hot reflux to the column. Begin sending net overhead material from the paraffin column to the Pacol feed surge drum as necessary to control the level on the accumulator tray.

13. Continue to establish and stabilize the paraffin circulation.

For Split-Feed reactors, establish paraffin flow through the #2 (middle) and #3 (bottom) reactor bed feed control valves, bypassing the reactors.

Gradually increase the paraffin flow rate to about the design value.

The paraffin circulation at this point should be: Pacol feed surge drum -> Pacol stripper -> Detal reactor bypass -> (alkylation mix drum) -> benzene column -> [recycle paraffin stripper] -> paraffin column -> Pacol feed surge drum.

14. Establish recycle benzene flow through the Detal reactor bypass. Bring in benzene to the unit as necessary (from storage or off-spec).
15. When the proper split (between benzene and paraffin) is achieved in the benzene column, establish a flow of benzene through the regenerant system using the manual valve on the startup reactor bypass line.

16. If LAB is available (in the off-spec tank), the rerun and recycle columns [or LAB and finishing columns] may be started up at this time. If LAB is not available, these columns cannot be started up until after feed is started to the reactors. (The procedure for starting up the rerun and recycle columns [or LAB and finishing columns] is given later in this Section VII - part C. Startup.)
17. During the initial startup, check the operation of the regenerant temperature ramping control system to insure that it is working properly. Do this check while the regenerant is bypassed around the reactors.

B. PIPING & VESSEL DRYOUT

18. During the initial startup, the reactor circuit piping must be dried out before catalyst is loaded. On subsequent startups, it will also be necessary to dryout any piping and vessels that have been opened up and exposed to air or moisture during the shutdown before the hydrocarbon feed is started to the catalyst. If no air or water entered the reactor during the shutdown, the piping and vessel dryout is not necessary.
19. Liquid fill the reactors:
 - a) Make sure all reactor blinds are removed and that the air has been removed from the reactors by nitrogen purging or evacuating/purging with nitrogen. Also make sure that the regenerant, recycle benzene and reactor feed streams are isolated from the reactors.
 - b) Liquid fill one reactor with paraffin from the recycle paraffin stripper overhead cooler or the paraffin column overhead cooler using the reactor filling line. Fill only one bed at a time, starting with the bottom bed. All beds may be filled through the filling line to the bottom bed. Keep the valves open on the sight glass on the relief valve bypass for

each bed as it is filling. When liquid appears in the sight glass, close the relief valve bypass and begin filling the next bed.

- c) After all beds of the first reactor have been filled with paraffin, adjust the reactor valve sequencer so that the first reactor is in the regeneration step and keep the main regenerant flow bypassed around the reactors (through the regenerant manual startup bypass). Then begin displacing the paraffin out of the first reactor to the benzene column (via the regenerant return line) with a slipstream of regenerant benzene using the startup purge valve provided.

NOTE: There is no flow meter on the regenerant being directed to the reactor via the startup purge valve. It may be preferable to purge the paraffin out of the first reactor through the shutdown line (located on the regenerant effluent line) to the benzene column instead of to the normal regenerant return line. In most units, the shutdown line can be directed through the off-spec return flow meter, providing an indication of the purge flow rate. Alternatively, the regenerant bypass can be closed and the regenerant flow control valve and flow meter can be used to control the purge rate, but the regenerant flow rate may have to be reduced to prevent an upset due to purging too fast. This would also require a significant flow through the regenerant pump spillback to maintain minimum flow through the pump.

- d) At the same time the paraffin is being displaced out of the first reactor, begin liquid filling the second reactor with paraffin from the recycle paraffin stripper overhead cooler or the paraffin column overhead cooler using the reactor filling line. (Note: Purging the first reactor and filling the second reactor at the same time will minimize the amount of paraffin that must be exported from the unit.)

20. When the first reactor has been purged with regenerant, close the startup purging line and line up the reactor for regeneration. Adjust the regenerant flow to design and begin heating up the regenerant.

21. When the second reactor has been filled with paraffin, close the filling line. Check to be sure the reactor valve sequencer is set on the proper step. (the first reactor should be in the regeneration mode and the second reactor in the on-line mode). Then gradually re-direct the recycle benzene and paraffin feed (#1 (top) bed feed in Split-Feed reactors) through the reactor and close the bypass. For split-Feed reactors, also re-direct the #2 (middle) and #3 (bottom) bed feed to the reactor and close the bypasses.
22. Adjust flows and temperatures to design and begin the normal daily reactor regeneration switching sequence. Periodically check to be sure there is no accumulation of non-condensables at the top of each reactor bed.
23. Start up and dry out the PEP unit according to the instructions in the PEP operating manual.
24. Perform normal dryout activities:
 - a) Periodically drain low points.
 - b) Switch flow paths so all lines are dried out.
 - c) Periodically flush control valve bypasses.
 - d) Periodically switch pumps.
 - e) Flush all startup lines and bypasses (especially the reactor liquid filling line).
 - f) Monitor moisture analyzer readings.
25. The unit can be considered to be dry when all of the moisture analyzers indicate less than 5 wppm water, and the moisture readings have lined out.
26. When the unit is dry, switch the paraffin feed flow (in Split-Feed units this is the #1 (top) bed feed) from going upstream of the static mixer, to going through the shutdown line. Then bypass the flow of recycle benzene around the reactor but continue feeding the paraffin to the reactor to purge the benzene out of the reactor.

27. Bypass the regenerant flow around the other reactor. Close the double block and bleed valves to isolate the regenerant system from the reactors.
28. When the benzene has been purged from the first reactor, advance the valve sequencer so that paraffin feed (all 3 beds in Split-Feed reactors) goes to the other reactor.

NOTE: As soon as possible, close the gate valve on the top bed feed line to the first reactor just upstream of the nitrogen purge out connection. This will help prevent any benzene (trapped in dead legs) from migrating into the purged reactor.

29. When the benzene has been purged from the second reactor, bypass the paraffin feed around the reactors and isolate the feed from the reactors using the double block and bleed valves. Continue the flow of paraffin, recycle benzene, and regenerant through the bypasses.
30. Use nitrogen to displace the paraffin from the reactors to the benzene column feed line using the shutdown line provided.
31. Remove any residual hydrocarbon from the reactors by nitrogen purging to flare or evacuating/pressuring with nitrogen.
32. Install blinds and prepare the reactor for entry according to standard refinery practice.
33. Load the catalyst in the reactors according to the catalyst loading procedure (which is given in Section XIII – Special Procedures).

NOTE: The hydrocarbon circulation may be maintained during reactor loading, if desired.

C. STARTUP

The following procedure is written for the original plant startup and for any other startup following a catalyst reload (where a catalyst dryout is required). For subsequent re-start operations, some of these steps will not be necessary (such as the catalyst dryout). The procedures for subsequent re-starts can be altered to fit the specific situation.

At this point, it is assumed that the benzene and paraffin circulation loops have been established (Steps 1 – 17 Hydrocarbon Circulation), the reactor circuit piping has been dried out (Steps 18 – 33 Piping & Vessel Dryout), the catalyst has been loaded (Section VIII.A. Reactor Loading), and the benzene and paraffin are still circulating via the reactor bypass.

On subsequent restarts when none of the piping or vessels have been opened, the Piping & Vessel Dryout is not necessary. In this case, it is only necessary to complete Steps 1 – 17 (Hydrocarbon Circulation) before proceeding with the following startup procedures.

34. Liquid fill the reactors:

- a) Make sure all reactor blinds are removed and that the air has been removed from the reactors by nitrogen purging or evacuating/purging with nitrogen. Also make sure that the regenerant, recycle benzene and reactor feed streams are isolated from the reactors.
- b) Liquid fill one reactor with paraffin from the recycle paraffin stripper overhead cooler or the paraffin column overhead cooler using the reactor filling line. Fill only one bed at a time, starting with the bottom bed. All beds may be filled through the filling line to the bottom bed. Keep the valves open on the sight glass on the relief valve bypass for each bed as it is filling. When liquid appears in the sight glass, close the relief valve bypass and begin filling the next bed.

NOTE: It is very important that the paraffin used to fill the reactors does not contain any olefin. Permanent catalyst deactivation can occur if olefin comes in contact with the catalyst without a sufficient amount of benzene present.

- c) After all beds of the first reactor have been filled with paraffin, adjust the reactor valve sequencer so that the first reactor is in the regeneration step and keep the main regenerant flow bypassed around the reactors (through the regenerant manual startup bypass). Then begin displacing the paraffin out of the first reactor to the benzene column (via the regenerant return line) with a slipstream of regenerant benzene using the startup purge valve provided.

NOTE: There is no flow meter on the regenerant being directed to the reactor via the startup purge valve. It may be preferable to purge the paraffin out of the first reactor through the shutdown line (located on the regenerant effluent line) to the benzene column instead of to the normal regenerant return line. In most units, the shutdown line can be directed through the off-spec return flow meter, providing an indication of the purge flow rate. Alternatively, the regenerant bypass can be closed and the regenerant flow control valve and flow meter can be used to control the purge rate, but the regenerant flow rate may have to be reduced to prevent an upset due to purging too fast. This would also require a significant flow through the regenerant pump spillback to maintain minimum flow through the pump.

- d) At the same time the paraffin is being displaced out of the first reactor, begin liquid filling the second reactor with paraffin from the recycle paraffin stripper overhead cooler or the paraffin column overhead cooler using the reactor filling line. (Note: Purging the first reactor and filling the second reactor at the same time will minimize the amount of paraffin that must be exported from the unit.)

35. When the second reactor is liquid full, close the filling line. Make sure that the reactor valve sequencer is still on the proper step (the automatic valves for the first reactor should be set for the regeneration step and the automatic valves for the second reactor should be set for the on-line step). The paraffin feed and recycle benzene should still be bypassing the reactors at this point. Keep the second reactor isolated and filled with paraffin while the catalyst in the first reactor is dried out (in Steps 36 - 40).
36. When the paraffin has been purged from the first reactor (using regenerant per Step 34.c.), line up the main regenerant flow through the reactor and close the manual bypass and startup purge valves. Adjust the regenerant flow through the reactor to the design rate and begin heating up the reactor using the regenerant heater. If necessary, regenerate the reactor according to the normal regeneration procedure.
37. For the initial startup it will be necessary to dry out the fresh catalyst. After starting the regenerant flow through the first reactor, ramp up to the normal regeneration temperature (typically 250°C) and hold at that temperature until the catalyst is dry. This may take several days. Monitor the regenerant effluent moisture analyzer readings. When the moisture in the regenerant effluent drops below about 10 wppm and lines out, the catalyst can be considered dry.

Maximizing the regenerant flow rate during catalyst dryout will minimize the time required for dryout. At the same temperature, the dryout time is inversely proportional to the flow rate. The regenerant temperature is also a very important factor in the time required for dryout, especially toward the end of the dryout. If a trade-off between flow rate and temperature must be made, the best strategy is to maximize flow rate during the first part of the dryout (when the regenerant effluent is above about 40 wppm H₂O), even if that means running 20°C or 30°C lower temperature. Then during the last part of the dryout (when the regenerant effluent is less than 40 wppm H₂O), the temperature should be kept at 250°C, even if that means running at a lower flow rate.

NOTE: Some units are designed with provisions to circulate significantly more than the normal regenerant flow rate during catalyst dryout (usually this "dryout" flow rate is about 200% of the normal regenerant flow rate). This is typically done by running both of the regenerant pumps in parallel. Special attention must be given to the regenerant pumps when both are running at the same time to insure that both pumps have sufficient flow to prevent damage to the pumps. Typically a special flow transmitter on the regenerant pump spillback control loop or a separate spillback for each pump is provided for this case. Also, special dryout lines may be provided on some units to avoid high pressure drop through some of the regenerant exchangers and valves at the higher catalyst dryout flow rate.

38. Adjust the flow rates and temperatures of the recycle benzene and the paraffin feed streams to the desired values (still bypassing the reactors at this time).

NOTE: During the initial dryout, it is recommended that both the recycle benzene and the paraffin feed flow rates be adjusted to 100% design in order to minimize the amount of paraffin displaced from the reactor and to maximize the rate of catalyst dryout. During subsequent startups, it is recommended that the recycle benzene flow rate again be adjusted to 100% design, but that the paraffin feed rate be adjusted to about 75% of design. The higher benzene flow rate will insure sufficient benzene/olefin ratio in case of an upset when the Pacol reactor is started up, and the lower paraffin rate will make it easier to start up the DeFine and Pacol reactors (lower hydrogen losses, smoother DeFine reactor filling, etc.).

39. After the catalyst has been dried out, reduce the regenerant flow back down to the normal design flow rate (if a higher "dryout" flow rate was used). Then use the regenerant heater to adjust the temperature of the first reactor to 5°C above the desired operating temperature. This will heat up the catalyst in preparation for starting feed to the reactor. This step is especially important in subsequent startups in cases where the olefin may not have

been completely removed from the recycle paraffin stream (e.g. an emergency shutdown).

For the Initial Startup, a conservative estimate for the Start-Of-Run (SOR) temperature is about 140°C. So, the regenerant should be adjusted to about 145°C in this step.

For Subsequent Startups, the desired operating temperature will usually be the same reactor temperature that was being used before the shutdown. So, the regenerant should be adjusted to about 5°C higher than the previous reactor temperature in this step.

40. When flows and temperatures are stable, open the manual regenerant bypass around the reactors, and isolate the regenerant flow from the reactors. Also shut off the regenerant heater. Then advance the automatic valve sequencer so that the first reactor is in the on-line mode and the second reactor is in the regeneration mode. The recycle benzene and paraffin feed flows should still be bypassing the reactors at this point. The regenerant should also be bypassing the reactors.
41. Gradually re-direct the recycle benzene and paraffin feed [#1 (top) bed feed] into the first reactor by opening the reactor outlet and inlet valves and closing the bypass valve. The Pacol reactor should still be bypassed at this time. [In Split-Feed reactors, start the #2 (middle) and #3 (bottom) bed feeds to the reactor and close the bypasses at this time.]

NOTE: During the initial startup, depending on how long it takes to dry out the fresh catalyst, it may be desirable to dry out the catalyst in the second reactor before starting feed to the first reactor.

42. At the same time feed is being started into the first reactor, begin purging the paraffin out of the second reactor with a slipstream of regenerant. Use the startup purge valve to avoid heating up the catalyst too quickly. This is

especially important with fresh catalyst in the reactor due to the moisture that may be present on the catalyst.

NOTE: As operational experience is gained, it may be possible to simplify the procedure for starting feed to the first reactor and regenerant to the second reactor.

43. When the paraffin has been purged from the first reactor (using regenerant per Step 42), make sure the regenerant flow rate is adjusted to the design rate, then line up the main regenerant flow through the reactor and close the manual bypass and startup purge valves. Begin heating up the reactor using the regenerant heater. Begin the normal regeneration sequence.
44. For the initial startup it will also be necessary to dry out the fresh catalyst in the second reactor. Use the same procedure to dry out the catalyst in the second reactor as was used to dry out the first reactor (in Step 37):

Ramp up to the normal regeneration temperature and hold at that temperature until the catalyst is dry. Monitor the regenerant effluent moisture analyzer readings. When the moisture in the regenerant effluent drops below 10 wppm and lines out, the catalyst can be considered dry.

45. Line out all flows, temperatures, and pressures. If it has not been done yet, establish a vacuum on the rerun and recycle [or LAB and finishing] columns.
46. Make sure that the PEP unit is running properly. (During the initial startup, the PEP unit must be properly commissioned and dried out according to the procedures in the PEP operating manual). The Pacol product stripper bottoms should be flowing through the PEP adsorbers (not bypassed) and the PEP columns should be lined out.
47. Startup the DeFine reactor.

NOTE: The Detal catalyst will deactivate quickly if the DeFine reactor is not started up before the Pacol reactor. The diolefins in the Pacol reactor product will cause accelerated polymer formation on the Detal catalyst which will cause rapid deactivation of the catalyst. Some of this polymer can be removed during the normal benzene regeneration cycle, but it is typically not possible to remove all of the polymer that is produced during diolefins upset and this will result in some permanent Detal catalyst deactivation. Feed should always be bypassed around the Pacol reactor if the DeFine reactor is not running properly.

48. When the unit operations are stable, start feed to the Pacol reactor.
49. When the Pacol unit begins converting paraffins to olefins, LAB will start being produced in the Detal unit, and the LAB will start to accumulate in the bottom of the paraffin column. Send the paraffin column bottoms to the off-spec tank until the rerun column [or LAB column] is started up.
50. When LAB is available in the paraffin column bottoms, use the startup filling line from the paraffin column bottoms pump to inventory the accumulator tray in the rerun and recycle columns [or LAB and finishing columns]. (In units with a heavy alkylate column, the accumulator tray in that column should also be filled at this time).
51. Start the rerun and recycle column [or LAB and finishing column] overhead pumps and establish a flow of cold reflux to the contact condenser in each column. (Note: bypass the clay treaters during startup. They will be liquid filled later).
52. Begin feeding paraffin column bottoms to the rerun column [or LAB column]. Stop sending paraffin column bottoms to the off-spec tank.

53. As a level appears in the bottom of the rerun column [or LAB column], start a flow of hot oil to the reboiler and begin heating up the column. Also start the rerun column [or LAB column] bottoms pump and spill back to the reboiler tub.
54. When the accumulator level in the rerun column [or LAB column] begins rising, establish hot reflux to the column. Send net overhead product to the off-spec tank as necessary until the LAB quality meets specifications.
55. If the unit has a finishing column, use the following steps to startup the finishing column:
 - a) Confirm that the finishing column accumulator tray has been filled and that cold reflux flow to the contact condenser has been established. Adjust the vacuum control system to give the desired column pressure.
 - b) Begin feeding LAB column net overhead material to the finishing column. As a level appears in the bottom of the finishing column, begin heating the column by starting a flow of hot oil to the reboiler. Also start the bottoms pump and spill back to the reboiler tub.
 - c) As the level rises on the finishing column accumulator tray, establish hot reflux to the top of the packing below the accumulator tray. Establish a flow of net overhead to the benzene column. Send the net bottoms to the off-spec tank as necessary until the finishing column bottoms product meets the LAB quality specifications. The clay treater should remain bypassed until the finishing column operation is stable.
56. If the unit has a recycle column, use the following steps to startup the recycle column:
 - a) Begin feeding rerun column [or LAB column] bottoms to the recycle column. As a level appears in the bottom of the recycle column, begin

- heating the column by starting hot oil to the reboiler. Also start the bottoms pump and spill back to the reboiler tub.
- b) As the level rises in the recycle column accumulator, establish hot reflux in the column. Send the net overhead to the suction of the benzene column bottoms pump. The recycle clay treater should still be bypassed at this point.
 - c) As material accumulates in the bottom of the recycle column, send it to the off-spec tank. When the LAB content of the bottoms product is acceptable, begin sending the recycle column bottoms to HAB storage.
57. If the unit has a heavy alkylate column, use the following steps to startup the heavy alkylate column:
- a) Begin feeding rerun column [or LAB column] bottoms to the heavy alkylate column. As a level appears in the bottom of the heavy alkylate column, begin heating the column by starting hot oil to the reboiler. Also start the bottoms pump and spill back to the reboiler tub.
 - b) As the level rises in the heavy alkylate column accumulator, establish hot reflux in the column. Send the net overhead to storage.
 - c) As material accumulates in the bottom of the heavy alkylate column, send it to the off-spec tank. When the quality of the bottoms product meets HAB specifications, begin sending the heavy alkylate column bottoms to HAB product storage.
58. Liquid fill the clay treaters and put them on line. (During the initial startup, the rerun and recycle columns [or LAB and finishing columns] should be started up with the clay treaters bypassed. These columns must be started up and running in a stable manner before the clay treaters are liquid filled.) Following is a general procedure for filling the clay treaters:

- a) Before liquid filling, the clay treater feed temperature controller should be commissioned and should be controlling the temperature at the desired set point (approximately 70°C to 90°C) with the feed bypassed around the treater. Make sure all blinds that were installed for clay loading have been removed. Make sure that the air has been removed from the clay treater by nitrogen purging per standard refinery practice. Make sure that the clay treater drain lines and the steamout and purging lines are closed. Make sure that the bleeder valves on the double block and bleeds at the inlet and outlet of the treater are closed.
- b) Gradually open the valves on the clay treater effluent line and begin filling the clay treater in an upflow direction. Open the valves on the clay treater relief valve bypass to vent the non-condensables to the flare header. Fill the treater slowly to avoid upsetting the clay bed. On smaller treaters, one of the two block valves on the treater outlet should be a globe valve. On larger treaters, there will be a small 1" line with a globe valve that bypasses one of the effluent block valves. Use the globe valve to control the liquid flow filling the treater. The flow should be adjusted so that it takes about 1 hour to fill the treater.
- c) When liquid appears in the sight glass on the relief valve bypass, close the valves on the relief valve bypass and on the clay treater effluent line.
- d) Gradually open the double block valves on the clay treater inlet line to line up the feed to the treater. Open the valves on the relief valve bypass slightly for a short time to vent off any non-condensable gasses that were present in the clay treater inlet line. Then close the valves on the relief valve bypass.
- e) Open the double block valves on the clay treater effluent line. Then gradually close the valves on the clay treater bypass line.

- f) Carefully watch the operation of the downstream equipment (typically a pump or a column) after lining up the clay treater. If all of the non-condensables were not removed from the treater, they could cause cavitation in a pump, or a pressure upset in a column.
 - g) Make adjustments to the clay treater feed temperature as necessary to optimize the LAB product quality.
59. When column operations have stabilized, close the column bottoms spillbacks to the reboiler tubs.
60. Line out operations and begin normal daily reactor switching. Periodically check to be sure there is no accumulation of non-condensables at the top of each reactor bed.

D. COLD WEATHER OPERATION

The freezing (or solidification) temperature for benzene is about 5°C and for the paraffin recycle stream it is about -15°C. Some special precautions are necessary during cold weather operation of the unit to prevent benzene or paraffin freeze-up.

1. Air Coolers

In cold weather, the fans on the air cooled exchangers (such as the paraffin column overhead cooler) should not all be shut off. This could allow freezing of the material in some of the tubes. At least one fan in each exchanger bay should be kept running and the louvers should be closed to control the cooler outlet temperature. During startup or shutdown, it may be necessary to heat the air below some of the exchangers to prevent solidification of the paraffin or benzene in the exchanger. Some of the exchangers may be provided with steam coils for this purpose. In cold climate locations, an external air recirculation system is typically used on the benzene column overhead condenser to prevent freezing and plugging in this air-cooled exchanger.

2. Pumps

Many of the pumps in the unit are equipped with warm-up lines. Normally these warm-up lines are only used to warm-up the hot service pumps just before starting them up. In cold weather, the warm-up lines on the spare pumps should be kept partially open all of the time to prevent freeze-up of the hydrocarbon in the pump.

Another concern in cold weather is the viscosity of the fluid being pumped. The pump motors are typically sized for the viscosity at operating temperature. In cold weather, the viscosity of most fluids increases and could cause over-amping the motor in some cases. If this is a problem, throttling the discharge valve of the pump may prevent tripping the motor until the fluid reaches its normal operating temperature.

3. Off-Spec Tank

Cold weather could cause freezing of benzene in the off-spec tank. Typically the benzene would freeze around the walls of the tank first. Frozen benzene on the walls of the tank could seriously damage, or even sink, the internal floating roof in this tank.

In cold weather locations, the off-spec tank is equipped with a tank heater. However, in order for this heater to effectively prevent freezing of benzene on the walls of the tank, it is necessary to circulate the contents of the tank (with the off-spec pump) to properly distribute the heat in the tank.





VIII. NORMAL OPERATIONS

A. CALCULATIONS

1. Detal Catalyst Life

The cumulative Detal catalyst life is defined as metric tons of LAB product divided by total kilograms of catalyst loaded into both reactors (MT LAB/kg catalyst). The total cumulative amount of LAB that has been produced (starting with the date that the catalyst was loaded) should be used. One metric ton is equal to 1000 kg.

2. Liquid Hourly Space Velocity (LHSV)

The LHSV is defined as the volume of reactor feed (M^3/hr) divided by the volume of catalyst (M^3) in the reactor. The units for LHSV are 1/hr. The reactor feed includes the recycle benzene plus the paraffin/olefin feed to all three beds. The volume of catalyst is the actual loaded volume in all of the beds of one reactor. The reactor being regenerated is not included in this calculation.

3. Benzene/Olefin Ratio

The overall benzene/olefin ratio is defined as the moles of benzene divided by the moles of olefin in the total reactor feed. The moles of benzene can be calculated from the recycle benzene flow rate, the % benzene in the recycle benzene, and the molecular weight of benzene. The moles of olefin can be calculated from the total flow rate of paraffin/olefin feed to the reactor (include all 3 beds in a Split-Feed reactor), the concentration of each of the olefin species in the paraffin/olefin feed, and the molecular weight of each of the olefin species. (In a Split-Feed reactor, the benzene/olefin ratio in each individual bed can be calculated if desired).

Alternatively, the benzene/olefin ratio can be calculated by analyzing the reactor effluent for alkylate (both LAB and HAB), paraffin, and benzene. The benzene/olefin ratio is the moles of benzene plus the moles of alkylate, divided by

the moles of alkylate. This method is not as accurate as the calculation based on the feed rates and compositions due to the uncertainty of the molecular weight of the HAB. However, it can be performed occasionally as a check.

The benzene/olefin ratio is a very important process variable. A low benzene/olefin ratio will cause an increase in the amount of HAB produced in the reactor (as measured by the HAB/LAB ratio). Low benzene/olefin ratio can also promote side reactions which could increase the bromine index of the LAB product and can cause catalyst deactivation.

4. HAB/LAB Ratio

The HAB/LAB ratio is defined as the weight of HAB product (recovery column net bottoms, or LAB column net bottoms) divided by the weight of LAB product (rerun column net overhead, or finishing column net bottoms). A high HAB/LAB ratio is a sign of poor yield of LAB and indicates either a low benzene/olefin ratio, a fractionation problem or other upstream problems (such as poor performance in the DeFine reactor or the PEP unit).

5. Fresh Paraffin/LAB Ratio

The fresh paraffin/LAB ratio is defined as the weight of fresh paraffin feed to the Pacol unit divided by the weight of LAB product (rerun column net overhead, or finishing column net bottoms). This ratio is an indication of the selectivity of both the Pacol and Detal units.

6. Benzene/LAB Ratio

The benzene/LAB ratio is defined as the net weight of make-up benzene to the Detal unit divided by the weight of LAB product (rerun column net overhead, or finishing column net bottoms). The net make-up benzene is the total make-up benzene flow minus the benzene drag flow. The benzene/LAB ratio is an indication of selectivity of the Pacol and Detal units, and of the performance of the DeFine reactor and the PEP unit.

NOTE: The HAB/LAB, Fresh Paraffin/LAB, and Benzene/LAB ratios are not representative when material from the off-spec tank is being processed in the unit. In order to get meaningful results for these ratios, they should be calculated on a minimum time basis of 24 hours.

7. Bromine Index

The bromine index of various streams is a valuable tool in determining the performance of the catalyst.

- a) An increase in the bromine index of the reactor effluent indicates that olefins are breaking through the reactor. An increase in reactor temperature may be necessary to bring the olefin conversion back up to 100%.
- b) An increase in the bromine index of the paraffin column overhead (recycle paraffin) also indicates that olefins are breaking through the reactor. An increase in reactor temperature may be necessary to bring the olefin conversion back up to 100%.
- c) An increase in the bromine index of the HAB product (recycle column bottoms, or LAB column bottoms) – with no increase in the bromine index of the reactor effluent or the paraffin column overhead – could be an indication of excessive water on the Detal catalyst. The high bromine index in the recycle column bottoms is a sign of more polymer formation, which could be a result of temporary Detal catalyst deactivation due to water.
- d) Bromine index of the LAB product is a key indicator of LAB product quality. Lower bromine index indicates that the LAB will have lower (better) color when it is sulfonated. An increase in LAB bromine index could be caused by poor Detal catalyst performance, poor clay treater performance, or a fractionation problem.

B. DATA SUMMARY SHEETS

Keeping records of the unit operating variables is a valuable troubleshooting tool for both operations and maintenance. At the end of this section there are data summary sheets for recording and reporting the important operating variables in the Detal unit. These sheets include the critical data that should be monitored on a typical Detal unit in order to identify any process-related problems that might occur. It is suggested that the refiner monitor additional data on a regular basis in order to identify equipment-related problems.

DETAL PROCESS DATA SUMMARY SHEET
(Split-Feed Rx, Rerun & Recycle Column)

Refiner: _____ Location: _____

DATE: _____

Calculated Variables

Detal Catalyst Life (MT LAB/kg)
LHSV (/hr)
Benzene/Olefin (mol/mol)
HAB/LAB (wt/wt)
Fresh Paraffin/LAB (wt/wt)
Benzene/LAB (wt/wt)

Flow Rates

Paraffin/Olefin Feed to Top Bed (M³/hr)
Paraffin/Olefin Feed to Middle Bed (M³/hr)
Paraffin/Olefin Feed to Bottom Bed (M³/hr)
Recycle Benzene to Reactor (M³/hr)
Regenerant from Reactor (M³/hr)
Benzene Makeup (M³/hr)
Benzene Drag (M³/hr)
Benzene Column Top Reflux (M³/hr)
LowerReflux (M³/hr)
Paraffin Column Feed (M³/hr)
"Cold" Reflux to Cond. (M³/hr)
"Hot" Reflux (M³/hr)
Paraffin recycle(net ovhd) (M³/hr)
Rerun Column Feed (M³/hr)
"Cold" Reflux to Cond. (M³/hr)
"Hot" Reflux (M³/hr)
LAB Product (net ovhd) (M³/hr)
Clay Treater Effluent (M³/hr)
Recycle Column Feed (M³/hr)
"Cold" Reflux to Cond. (M³/hr)
"Hot" Reflux (M³/hr)
Clay Treater Effluent (net ovhd) (M³/hr)
HAB Product (net btms) (M³/hr)

Temperatures

Paraffin/Olefin Feed to Detal Unit (°C)
Feed from Feed/Product Exch (°C)
Top Bed Olefin Cooler Outlet (°C)
Mid/Btm Bed Olefin Cooler Outlet (°C)
Mixed Bz/Olefin Feed To Top Bed (°C)
Mixed Feed to Middle Bed (°C)
Mixed Feed to Bottom Bed (°C)
Product from Reactor Outlet (°C)
Product from Feed/Product Exch. (°C)
Regenerant to Reactor (°C)
Regenerant from Reactor (°C)
Regenerant to Mix Drum (°C)
Combined Feed to Mix Drum (°C)
Benzene Column Ovhd Vapor (°C)
Ovhd Condenser Outlet (°C)
Liquid from Ovhd Receiver (°C)
Liquid from Accum. Tray (°C)
Tray ____ (°C)
Tray ____ (°C)
Bottoms Vapor (°C)
Paraffin Column Tray 1 (°C)
Tray ____ (°C)
Bottoms Vapor (°C)
Bottoms Liquid (°C)
Liquid from Accum. Tray (°C)
Overhead Cooler Outlet (°C)
Rerun Column Vapor Below Accum. (°C)
Bottoms Vapor (°C)
Bottoms Liquid (°C)
Liquid from Accum. Tray (°C)
Overhead Cooler Outlet (°C)
Clay Treater Inlet (°C)
Recycle Column Vapor Below Acc. (°C)
Bottoms Vapor (°C)
Bottoms Liquid (°C)
Liquid from Accum. Tray (°C)
Overhead Cooler Outlet (°C)
Clay Treater Inlet (°C)

DETAL PROCESS DATA SUMMARY SHEET
(Split-Feed Rx, Rerun & Recycle Column)

Refiner: _____ Location: _____

DATE: _____

Pressures

Reactor (Top) (Kg/cm²)
Alkylation Mixing Drum(Back Press) (Kg/cm²)
Regenerant (Back Pressure) (Kg/cm²)
Benzene Column (Above Accum.) (Kg/cm²)
Paraffin Column (Above Accum.) (mmHg abs)
Rerun Column (Above Accum.) (mmHg abs)
Recycle Column (Above Accum.) (mmHg abs)
Clay Treater (Back Pressure) (Kg/cm²)

Analyzers

Paraffin/Olefin Feed (ppm H₂O)
Recycle Benzene (ppm H₂O)
Reactor Effluent (ppm H₂O)
Regenerant Effluent (ppm H₂O)

Laboratory Analyses

Paraffin/Olefin Feed: (Sp Gr)
Total Normal Olefin (Wt%)
Total Normal Paraffin (Wt%)
Total Non-Normals (Wt%)
Aromatics (Wt%)

Benzene Makeup: (Sp Gr)
Benzene (Wt%)

Reactor Effluent: Bromine Index

Recycle Benzene: (Sp Gr)
Benzene (Wt%)
nC5 / Cyclo C6 (Wt%)
C7+ Paraffin (Wt%)

Recycle Paraffin: (Sp Gr)
LAB (Wt%)
Bromine Index

LAB Product: (Sp Gr)
Bromine Index
Saybolt Color
Sulfonate Color (Klett)
Unsulphonatable material (Wt%)
Non-Linear AB (Wt%)
Paraffin (Wt%)
Light Material (Wt%)
C10 LAB (Wt%)
C11 LAB (Wt%)
C12 LAB (Wt%)
C13 LAB (Wt%)
C14 LAB (Wt%)
Heavies (Wt%)

Rerun Clay Treater
Bromine Index @ Inlet
Bromine Index @ Outlet

Heavy Alkylate Product: (Sp Gr)
LAB (Wt%)
ASTM Color
Bromine Index

Recycle Column Overhead: (Sp Gr)
Bromine Index @ Clay Treater Inlet
Bromine Index @ Clay Treater Outlet
Saybolt Color
Non-Linear AB (Wt%)
C10 LAB (Wt%)
C11 LAB (Wt%)
C12 LAB (Wt%)
C13 LAB (Wt%)
C14 LAB (Wt%)
Heavies (Wt%)

DETAL PROCESS DATA SUMMARY SHEET

Refiner:

Location:

(Single Feed Rx, Recy Pf Stripper, LAB & Finishing Column)

DATE:

Calculated Variables

Detal Catalyst Life	(MT LAB/kg)
LHSV	(/hr)
Benzene/Olefin	(mol/mol)
HAB/LAB	(wt/wt)
Fresh Paraffin/LAB	(wt/wt)
Benzene/LAB	(wt/wt)

Flow Rates

Paraffin/Olefin Feed to Reactor	(M ³ /hr)
Recycle Benzene to Reactor	(M ³ /hr)
Regenerant from Reactor	(M ³ /hr)
Benzene Makeup	(M ³ /hr)
Benzene Drag	(M ³ /hr)
Benzene Column Top Reflux	(M ³ /hr)
LowerReflux	(M ³ /hr)
Recycle Paraffin Stripper Feed	(M ³ /hr)
"Cold" Reflux to Cond.	(M ³ /hr)
"Hot" Reflux	(M ³ /hr)
Net Pf Stripper Ovhd Liquid	(M ³ /hr)
Paraffin Column Feed	(M ³ /hr)
"Cold" Reflux to Cond.	(M ³ /hr)
"Hot" Reflux	(M ³ /hr)
Net Pf Column Ovhd Liquid	(M ³ /hr)
Total Recycle Paraffin to Pacol	(M ³ /hr)
LAB Column Feed	(M ³ /hr)
"Cold" Reflux to Cond.	(M ³ /hr)
"Hot" Reflux	(M ³ /hr)
HAB Product (net btms)	(M ³ /hr)
Finishing Column Feed (from Clay)	(M ³ /hr)
"Cold" Reflux to Cond.	(M ³ /hr)
"Hot" Reflux	(M ³ /hr)
Net Ovhd Liquid (to Benzene Co	(M ³ /hr)
LAB Product (net btms)	(M ³ /hr)

Temperatures

Reactor Top Bed Inlet (Feed)	(°C)
Reactor Top Bed Outlet	(°C)
Reactor Bottom Bed Inlet	(°C)
Reactor Bottom Bed Outlet	(°C)
Reactor Effluent to Benzene Col	(°C)
Regenerant to Reactor	(°C)
Regenerant from Reactor	(°C)
Regen Effluent Htr Outlet	(°C)
Regen Effluent to Benzene Col	(°C)
Benzene Column Ovhd Vapor	(°C)
Ovhd Condenser Outlet	(°C)
Liquid from Ovhd Receiver	(°C)
Liquid from Accum. Tray	(°C)
Tray ____	(°C)
Tray ____ (TIC)	(°C)
Bottoms Vapor	(°C)
Recy Pf Stripper Vapor Below Accum	(°C)
Liquid from Accum. Tray	(°C)
Cold Reflux to Contact Conc	(°C)
Bottoms Vapor	(°C)
Bottoms Liquid	(°C)
Paraffin Column Vapor Below Accum	(°C)
Liquid from Accum. Tray	(°C)
Overhead Cooler Outlet	(°C)
Tray ____ (TIC)	(°C)
Bottoms Vapor	(°C)
Bottoms Liquid	(°C)
LAB Column Vapor Below Accum.	(°C)
Liquid from Accum. Tray	(°C)
Overhead Cooler Outlet	(°C)
Clay Treater Inlet	(°C)
TIC in Lower Packed Section	(°C)
Bottoms Vapor	(°C)
Bottoms Liquid	(°C)
Finishing Column Vapor Below Acc.	(°C)
Liquid from Accum. Tray	(°C)
Overhead Cooler Outlet	(°C)
Bottoms Vapor	(°C)
Bottoms Liquid	(°C)

DETAL PROCESS DATA SUMMARY SHEET

Refiner: _____

Location: _____

(Single Feed Rx, Recy Pf Stripper, LAB & Finishing Column)

DATE: _____

Pressures

Reactor (Top)	(Kg/cm ²)
Reactor Effluent (Back Pressure)	(Kg/cm ²)
Regenerant (Back Pressure)	(Kg/cm ²)
Benzene Column (Ovhd Vapor)	(Kg/cm ²)
Recycle Pf Stripper (Above Accum)	(Kg/cm ²)
Paraffin Column (Above Accum.)	(mmHg abs)
LAB Column (Above Accum.)	(mmHg abs)
Finishing Column (Above Accum.)	(mmHg abs)

Analyzers

Paraffin/Olefin Feed	(ppm H ₂ O)
Recycle Benzene	(ppm H ₂ O)
Reactor Effluent	(ppm H ₂ O)
Regenerant Effluent	(ppm H ₂ O)

Laboratory Analyses

Paraffin/Olefin Feed:	(Sp Gr)
Total Normal Olefin	(Wt%)
Total Normal Paraffin	(Wt%)
Total Non-Normals	(Wt%)
Aromatics	(Wt%)

Benzene Makeup:	(Sp Gr)
Benzene	(Wt%)

Reactor Effluent: Bromine Index

Benzene Column Overhead:	(Sp Gr)
Benzene	(Wt%)
nC5 / Cyclo C6	(Wt%)

Regenerant:

Benzene	(Wt%)
nC5 / Cyclo C6	(Wt%)
C7+ Paraffin	(Wt%)

Recycle Benzene:

Benzene	(Wt%)
nC5 / Cyclo C6	(Wt%)
C7+ Paraffin	(Wt%)

Recycle Paraffin Stripper Overhead:	(Sp Gr)
Bromine Index	

Paraffin Column Overhead:	(Sp Gr)
Bromine Index	
LAB	(Wt%)

LAB Product: (Sp Gr)

Bromine Index	
Saybolt Color	
Sulfonate Color (Klett)	
Unulfonatable material	(Wt%)
Non-Linear AB	(Wt%)
Paraffin	(Wt%)
Light Material	(Wt%)
C10 LAB	(Wt%)
C11 LAB	(Wt%)
C12 LAB	(Wt%)
C13 LAB	(Wt%)
C14 LAB	(Wt%)
Heavies	(Wt%)

LAB Clay Treater

Bromine Index @ Inlet	
Bromine Index @ Outlet	

Heavy Alkylate Product: (Sp Gr)

LAB	(Wt%)
ASTM Color	
Bromine Index	

Finishing Column Overhead: (Sp Gr)

Bromine Index	
Benzene	(Wt%)
Light Material	(Wt%)
LAB	(Wt%)





IX. ANALYTICAL

A. GENERAL LIQUIDS

Sampling liquids normally requires some care to limit personal exposure and release to the environment. Sampling streams containing benzene requires an additional amount of care. Many liquid streams in the Detal unit contain benzene. Personal exposure should be minimized because of the acute poisoning possible from inhaling benzene vapors.

The sample containers can be glass bottles or metal cans. Metal cans have the advantage of not breaking when dropped. Glass bottles have the following advantages:

1. There is no surface adsorption of polar compounds.
2. The sample color can be observed.
3. Less spilling occurs because the level can be observed.

UOP recommends the use of glass bottles except when breakage is likely to occur. It is recommended that the bottles be permanently labeled and used repeatedly for the same sample.

A typical sample connection is shown in Figure IX-1. As shown, the sample connection is piped up around a control valve assembly. The control valve pressure drop provides the driving force to have flow through the $\frac{3}{4}$ inch pipe. One alternative location is to put the flowing sample around a pump. For this case, the flow in the $\frac{3}{4}$ inch pipe would be opposite to that shown, and the positions of the $\frac{3}{4}$ inch gate and globe valves would be reversed.

For hot streams, the sample cooler would be located between the $\frac{3}{4}$ inch gate valve and the $\frac{1}{4}$ inch sample connection.

The length of the ¼ inch pipe should be no more than 300 mm (1 ft.) to limit the amount of liquid which must be drained in order to flush the connection.

The sample circulation loop should only be put in operation when a sample is to be taken. The ¾ inch gate valve should normally be closed except to take a sample. To put the circulation loop in operation:

1. Close the ¼ inch valve.
2. Close the ¾ inch globe valve.
3. Open the ¾ inch gate valve.
4. Throttle the ¾ inch globe valve to regulate the flow.

The ¾ inch globe valve should be adjusted so that the flow through the loop does not affect the operation of the control valve or pump. This is conveniently done during startup. When sample coolers are included, cooling water is normally introduced only when a sample is about to be obtained. When sampling is complete, the cooling water can be drained.

To obtain a sample, the ¼ inch valve is opened to obtain a steady flow of liquid. The sample container should be filled about 25% full a couple of times and rinsed out with material from the sample connection. Finally, the sample container should be filled to about 75% with the sample. The container should not be completely filled so that breakage due to thermal expansion of the liquid does not occur.

Personal equipment required during sampling include:

1. Rubber gloves
2. Protective face cover

For analysis of small concentrations of water, special sample connections, sample containers, and container preparation will be required. The appropriate methods and procedures should be used. Typical instructions include oven-dried containers, rubber stoppers, and a hollow needle sample connection. In this case, rinsing the sample container is not possible.

B. CLOSED FLOWING SAMPLING APPARATUS

On all new units, UOP is designing closed sampling systems to comply with OSHA benzene exposure requirements. Figure IX-2 shows a schematic of a closed flowing sampling apparatus. The sampling procedure is as follows:

1. Normal operation – Valves A, B, C, and D closed. The cylinder is disconnected at the quick-connects.
2. Nitrogen purge the cylinder in the laboratory. Pressure to about 1.0 Kg/cm² (15 psig) in the laboratory. Valves E and F (on the cylinder) are closed.
3. Slowly open valves B and D to depressure the quick-connect lines.
4. Install the cylinder in the apparatus; make sure the cylinder top is properly situated. Attach the quick-connect fittings to the cylinder.
5. Close valve D. Then slowly open valve A and check for leakage at the quick-connect fittings.
6. Slowly open valve C. Use valve C to throttle the sample flow so that the operation of the control valve (or other equipment around which the sample apparatus is connected) is not affected.
7. Open valves E and F. Then close valve B to direct the flow through the sample cylinder.
8. Allow sample to flow through the cylinder for 2-4 minutes. Do not allow sample flow for an excessive period of time.
9. Close valves A, C, E, and F in that order. Then open valves B and D to depressure the quick-connect lines.
10. Remove the sample cylinder via the quick-connect fittings.

11. Provide safe outage in the sample cylinder by draining off a little bit of the liquid from the bottom valve with the cylinder in a vertical position. This will prevent over-pressuring the cylinder due to thermal expansion of the liquid. (Note: Some sample cylinders have dip-tubes inside which automatically keep a vapor space at the top of the cylinder. These dip-tube cylinders must be installed properly in the sample station for the vapor space to be maintained. It is not necessary to drain a bit of liquid out of the dip-tube cylinders if they are used properly.)

Some cylinders are protected by a rupture disc in one of the cylinder valves. Handle these cylinders such that any possible relief of the rupture disc will not endanger personnel.

In all sampling situations, the personnel involved should remain at arm's length from the sample container and situate themselves upwind of the container if possible. This simple precaution will minimize exposure to the hydrocarbon vapors.

After the sample is analyzed in the laboratory, the cylinder's contents should be safely disposed of and the cylinder thoroughly flushed and drained.

C. LABORATORY TEST SCHEDULE**DETAL PROCESS UNIT**

Stream and Test	Test Method Number	Frequency	
		Normal	Startup
Benzene Recycle, Regenerant, and Benzene Column Overhead Impurities (by GC)	ASTM D-2360 or ASTM D-4492	1/D	1/D
Makeup Benzene Feed			
Relative Density	ASTM D-4052 or ASTM D-3505	1/D	1/D
Benzene & Impurities (by GC)	ASTM D-4492	1/D	1/D
Sulfur	UOP 727 or ASTM D-4045	1/W	1/D
Thiophene	ASTM D-1685 or ASTM D-4735	1/W	1/D
Acid Wash Color	ASTM D-848	1/W	1/D
Bromine Index	ASTM D-1492 or ASTM D-5776	1/D	1/D
Water	UOP 481	1/M	1/D
Appearance (Visual Inspection)	ASTM D-3437	1/W	1/D
Color	ASTM D-1209	1/W	1/D
Solidification Point (Freeze Pt)	ASTM D-852	1/W	1/D
Total Nitrogen	UOP 384	1/W	1/D
p-Dioxane	UOP 921	1/W	1/D
Paraffin/Olefin Feed			
Relative Density	ASTM D-4052 or ASTM D-1298	1/D	1/D
Bromine Number	ASTM D-1159	1/D	3/D
Composition (Normal Paraffins)	UOP 411	1/D	1/D
Composition (Paraffins/Olefins)	UOP 688	1/D	1/D
Composition (Diolefins by HPLC)	UOP 869 and UOP 902	1/D	1/D
Composition (Aromatics by HPLC)	UOP 902 (Modified)	1/D	1/D
Sulfur	UOP 727	1/D	1/D
Composition (Carbon No. by MS)	UOP 711 or UOP 687	Occas.	Occas.
Reactor Effluent			
Bromine Index	UOP 304	1/D	3/D
Regenerant Effluent			
Impurities (by GC)	ASTM D-2360 or ASTM D-4492	Occas.	Occas.
Recycle Paraffin Stripper and Paraffin Column Overhead			
Relative Density	ASTM D-4052 or ASTM D-1298	1/D	1/D
Composition (Normal Paraffins)	UOP 411	1/D	3/D
Aromatics	UOP 495	3/W	1/D
Composition (Benzene Content)	UOP 621	Occas.	1/D
Bromine Index	UOP 304	1/D	1/D
Saybolt Color	ASTM D-156	Occas.	1/D
Composition (Carbon No. by MS)	UOP 687	Occas.	Occas.
Recycle Paraffin Stripper Bottoms and Paraffin Column Bottoms			
Bromine Index	UOP 304	1/W	1/D

Stream and Test	Test Method Number	Frequency	
		Normal	Startup
LAB Product (Rerun/LAB Column Overhead, Finishing Column Bottoms)			
Bromine Index	UOP 304	1/D	3/D
Relative Density	ASTM D-4052 or ASTM D-1298	1/D	1/D
Composition (Isomer Distribution)	UOP 673 or UOP 698	1/D	1/D
Mol. Wt. (by Calculation)	UOP 673 or UOP 698	1/D	1/D
Non-Linear Alkylbenzene Content	UOP 698	1/D	1/D
Saybolt Color	ASTM D-156	1/D	1/D
Composition (Paraffin Content)	UOP 621	1/W	1/D
Composition (Trace Benzene Content)	UOP 863	1/W	1/D
Distillation	ASTM D-86	1/W	1/D
Percent Unsulfonatable	UOP 429	Occas.	Occas.
Biodegradability of Sulfonated LAB	ASTM D-2667	Occas.	1/W
Doctor Test	UOP 41	Occas.	Occas.
Composition (HCBN Types by MS)	UOP 672	Occas.	Occas.
Dialkyltetralins (by HPLC)	UOP 929	Optional	
HAB Product (Rerun/LAB or Recycle Column Bottoms)			
Relative Density	ASTM D-4052 or ASTM D-1298	1/D	1/D
Composition	UOP 696	1/D	1/D
ASTM Color	ASTM D-1500	1/D	1/D
Bromine Number or Index	ASTM D-1159 or UOP 304	3/W	1/D
Distillation	ASTM D-1160	Occas.	1/D
Viscosity	ASTM D-88	Occas.	Occas.
Composition (HCBN Types by MS)	UOP 672	Occas.	Occas.
Clay Treater Inlet & Outlet			
Bromine Index	UOP 304	3/W	1/D
Recycle Column Overhead or Finishing Column Overhead			
Relative Density	ASTM D-4052 or ASTM D-1298	1/D	1/D
Composition by GC	UOP 698, UOP 621, or UOP 863	1/D	1/D
Non-Linear Alkylbenzene Content	UOP 698	1/D	1/D
Saybolt Color	ASTM D-156	1/D	1/D
Bromine Index	UOP 304	3/W	1/D
Distillation	ASTM D-86	Occas.	Occas.
Composition (HCBN Types by MS)	UOP 672	Occas.	Occas.
Off-Spec Tank			
Relative Density	ASTM D-4052 or ASTM D-1298	Occas.	Occas.
Composition (Bz, Pf Content)	UOP 621	Occas.	Occas.
Bromine Number	ASTM D-1159	Occas.	Occas.

3/D: Three times per day
 1/D: Once per day
 3/W: Three times per week

1/W: Once per week
 1/M: Once per month
 Occas. Occasionally

NOTE: UOP 672, UOP 687 and UOP 711 require the use of a mass spectrophotometer and normally must be sent to an outside laboratory.

D. SAMPLES FOR UOP ANALYSIS

If it is necessary to send samples to UOP for laboratory analysis, please contact your UOP representative who can provide the proper up-to-date shipping procedures for the samples. This will prevent delays and other problems that could be caused if the correct procedures are not followed.

When taking samples for UOP analysis always label the samples with the refinery name, location, unit from which the sample was taken (e.g. Detal), sample description, and the date sample was taken. Include any pertinent information that is useful to the laboratory and your UOP representative.

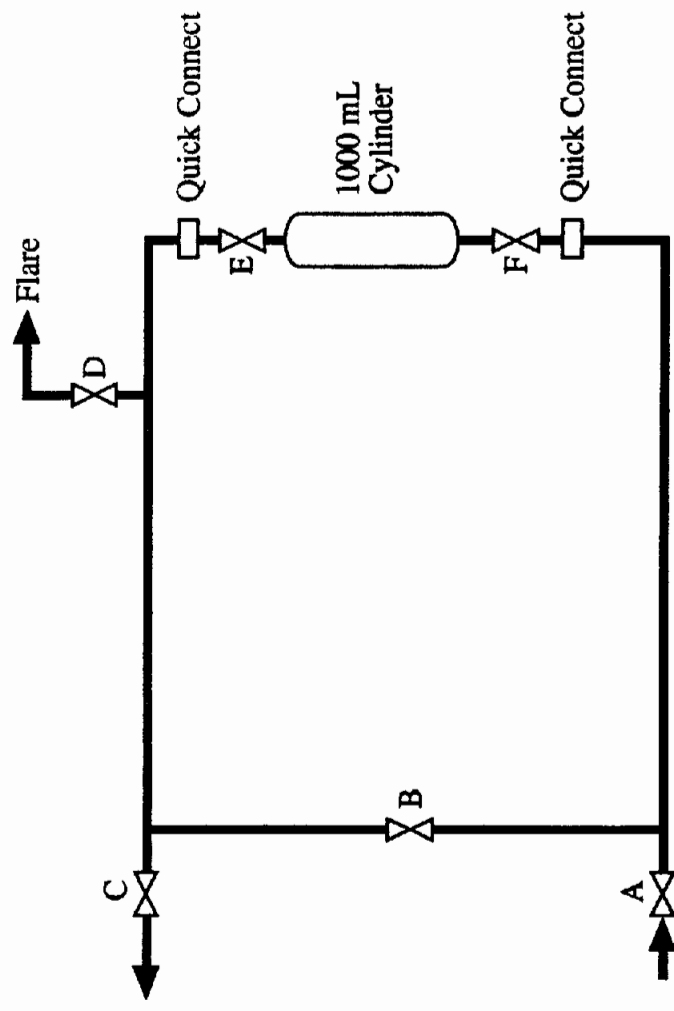


Figure IX - 1



DTL-R01-12

Figure IX - 2
Closed Flowing Sampling Apparatus



DTL-R01-13

X. TROUBLESHOOTING

Corrosion in the Detal unit should be far less than would be expected in the HF Detergent Alkylation process.

Information on catalyst poisons is included in Section III (Process Variables).

Due to the relatively new nature of the Detal process, there have not been a large number of problems or operational incidents from which a comprehensive troubleshooting guide can be assembled. The following examples are some incidents that may be of interest.

1. Problem: After loading catalyst in the Detal reactors, loose catalyst was found in the outlet line of one of the reactor beds when the blinds were being removed.

Discussion: In this case, the reactor switching valves were being tested after the catalyst was loaded. One of the outlet lines must have still been pressured up with air or nitrogen from the shutdown operation. When the switching valves were opened, the catalyst bed was upset by the sudden flow of gas in an upward direction through the catalyst bed. This allowed some of the catalyst to mix down into the bed of support balls. The slots in the outlet basket are not small enough to prevent catalyst from falling through into the outlet line. It was necessary to completely unload the bed and reload the support balls and the catalyst in that bed of the reactor. Great care must be taken not to upset the catalyst bed after the reactors are loaded. Also, the reactor switching valves should be checked out before catalyst is loaded.

2. Problem: Higher temperature is required on one of the Detal reactors in order to get the same performance (same Br index at the reactor outlet) as the other reactor.

Discussion: This has been experienced on more than one unit. Temperature differences of 2-5°C have been necessary in some units. One possible cause is poor flow distribution in one of the reactors due to a physical upset such as described in Troubleshooting Example 1 above. Another possible cause is sudden permanent deactivation of the catalyst in one of the beds due to diolefin breakthrough from the DeFine reactor.

3. Problem: During the Detal reactor switching procedure, the regenerant pump suddenly cavitates or surges for a couple of minutes.

Discussion: This problem was occurring at the point in the switching sequence when the regenerant was first lined up to the previously on-line reactor. The regenerant system operates at a significantly higher pressure than the normal reaction pressure. In general, liquid-filled systems are very quickly pressured up because liquids are relatively incompressible. However, liquid benzene is actually slightly compressible and therefore it takes a few minutes to pressure up the reactor to the regenerant pressure. The regenerant flow controller is located on the regenerant outlet line so when the reactor is suddenly lined up to the higher-pressure regenerant system, the flow through the regenerant pump is suddenly uncontrolled. This is very stressful to the high-head regenerant pump and can cause serious pump damage and possibly catastrophic mechanical failure. A pressure-up valve is now included on the regenerant inlet line to each reactor which opens to allow a small flow through a restriction orifice that slowly pressures up the reactor before the main regenerant inlet valve is opened. The pressure-up timer must be set to allow sufficient time for the reactor to pressure-up. Note that unusually long pressure-up times (and possibly regenerant pump cavitation or surging) can be caused by non-condensables in the reactor system. If non-condensables are present, they will typically accumulate in the top of the reactor beds where they can be vented through a sight flow indicator on the PSV bypass line.

4. **Problem:** During the Detal reactor switching procedure, the recycle benzene flow rate is suddenly upset (possibly causing a Detal reactor shutdown due to low flow) and the feed rate to the benzene column suddenly increases, upsetting the benzene column operation.

Discussion: This problem was occurring at the point in the switching sequence when the freshly-regenerated reactor was first lined up to the previously on-line reactor in preparation for displacement of the regenerant. The regenerant system operates at a significantly higher pressure than the normal reaction pressure. In general, liquid-filled systems are very quickly pressured up because liquids are relatively incompressible. However, liquid benzene is actually slightly compressible and therefore it takes a few minutes to depressure the regenerated reactor to the reaction system pressure. The recycle benzene flow controller is located upstream of the reactors and the reactor back-pressure control valve is located downstream of the reactors. So when the higher pressure regenerated reactor is suddenly lined up to the reactor system, a couple of things can happen. If the back-pressure valve cannot respond fast enough, the recycle benzene flow can temporarily be backed out of the reactor due to the sudden increase in reactor pressure, and this can trip the low flow shutdown of the paraffin/olefin feed to the Detal reactors. If the back-pressure controller does respond fast enough, there is a sudden large increase in the flow rate of feed to the benzene column, upsetting the column. The depressuring timer must be set to allow sufficient time for the reactor to depressure. Note that unusually long depressuring times can be caused by non-condensables in the reactor system. If non-condensables are present, they will typically accumulate in the top of the reactor beds where they can be vented through a sight flow indicator.

5. **Problem:** Poor separation of LAB and HAB. The operation of the rerun or recovery column may be unstable. The column may show signs of flooding at design feed and reflux rates.

Discussion: Particles of rust and scale can plug the small holes in the liquid distributor trays in these columns. This will cause poor liquid flow distribution across the packing, resulting in poor column performance. The liquid flux in both of these columns is very low and in order to maintain a sufficient number of holes in the liquid distributor trays to get proper flow distribution, the size of the holes is typically very small (less than 6 mm diameter). Small holes are easy to plug. Basket strainers are installed on the feed and reflux streams to the rerun and recycle column to reduce the potential for tray plugging. Many of the column internal parts are stainless steel to minimize the amount of scale generated downstream of the basket strainers. The carbon steel vessel walls of the rerun and recycle column should be cleaned very well before the initial start-up to reduce the possibility of tray plugging.

6. Problem: Excessive rust and scale accumulation on the start-up screens in the suction of the paraffin column bottoms pumps. The pump suction strainers had to be cleaned every 2 to 4 hours during startup.

Discussion: The paraffin column and internals (including trays and packing) are carbon steel. Significant amounts of rust and scale can form on the carbon steel if it is exposed to water and air. In this case, the column was water flushed, and then sat open to the atmosphere for more than a month before the unit was started up. On this particular startup, the rerun and recycle columns were thoroughly steamed out and flushed with condensate shortly before starting them up in order to prevent similar problems.

7. Problem: Liquid benzene is accumulating in the flare knockout drum. The benzene column overhead condenser outlet temperature is normal.

Discussion: Any pentane that escapes from the PEP unit generally goes to the Detal unit either via the paraffin/olefin feed, or the desorbent return stream. This pentane will accumulate in the overhead receiver of the benzene column. If the pentane concentration in the benzene column overhead receiver rises above about 5%, the vapor pressure of the liquid will get high enough to cause a small net flow of hydrocarbon vapor from the

overhead receiver to the flare line. Although the pentane is the component that is causing the vaporization, it will carry a large amount of benzene with it. As this vent gas travels through the flare line, the temperature will typically decrease by 5 to 10°C, and this is enough to condense a large portion of the vapor. The portion that condenses is primarily benzene. So even though the cause of the vent gas is pentane, the liquid that collects in the flare knockout drum is primarily benzene.

The solution to the problem is to return the pentane in the benzene column overhead receiver back to the PEP unit by recycling a flow of benzene drag back to the PEP fractionation system. All Detal units are designed to do this. This recirculated benzene drag stream must somehow be dried to prevent water on the PEP sieve, and all units are designed to do this as well. Also, the PEP operation should be adjusted to minimize the amount of pentane that is escaping to the Detal unit, so the concentration of pentane in the benzene column overhead receiver is minimized.

8. Problem: Liquid benzene is accumulating in the flare knockout drum. In this case, the benzene column overhead condenser outlet temperature is higher than normal.

Discussion: In hot weather, the benzene column overhead condenser may have difficulty condensing all of the benzene column overhead vapor. Check to be sure the louvers on this air-cooled exchanger are fully open and the fans are all operating properly with the fan pitch control set to maximize air flow. Check to see if the fins on the exchanger tubes are bent, damaged, or plugged with dirt or other foreign material. These fins should be cleaned during each turnaround. Check to be sure the top reflux flow rate – if it is significantly above the design flow rate, reduce the total reboiler heat to the column. If the heat is reduced, it may be necessary to reduce the recycle benzene flow rate to maintain a good split between paraffin and benzene in the column. Minimize the pentane concentration in the overhead receiver using the benzene drag to the PEP unit.

9. Problem: High HAB/LAB ratio.

Discussion: Check to see if the HAB/LAB ratio is low. The primary process variable that affects the HAB/LAB ratio is the benzene/olefin ratio. Make sure none of the recycle benzene is bypassing the reactor. Check the aromatics and diolefin content in the paraffin/olefin feed from the PEP unit. Aromatics and diolefins tend to make HAB. Check the LAB content of the HAB. If the LAB fractionation columns are not running correctly, there could be excessive LAB in the HAB product.

10. Problem: High bromine index of the LAB product and the recycle paraffin, and an increase in the Detal reactor temperature does not reduce the bromine index of either stream.

Discussion: A tube leak in the paraffin/olefin feed - reactor effluent exchanger or a leaking reactor bypass valve will allow olefins to bypass the Detal reactor. This will obviously cause olefins in the recycle paraffin, increasing the bromine index of that stream. Less obviously, it will also tend to cause higher bromine index in the LAB product. Most of the olefins that bypass the reactor will end up being fractionated overhead in the paraffin column and leave the Detal unit with the recycle paraffin. But, some of the olefins will polymerize in the benzene column reboiler to form heavier olefins. These heavier olefins can then thermally crack to smaller olefin and paraffin molecules. So, leaking olefin into the reactor effluent typically results in the formation of a wide boiling range of olefins. The concentration of olefins in the LAB boiling range is relatively low, but even low concentrations of olefin in the LAB product are very significant with respect to bromine index. The leaking exchanger example described here is actually from a HF Detergent Alkylation unit. Such an exchanger leak is much less likely in a Detal unit due to the lower potential for corrosion in a Detal unit. However, careful attention should be given to prevent leakage through the Detal reactor bypass lines. Double block and bleed valve arrangements are provided on the Detal reactor bypass lines to insure that there is no leakage.

11. Problem: Unstable bromine index of the LAB product. The bromine index appears to change for no visible reason.

Discussion: In this case, the laboratory was using the wrong method for analyzing the bromine index of the LAB product. UOP recommends using the UOP 304 laboratory method for bromine index of the LAB product. This has proven to be the most reliable method for accurately analyzing LAB product.

Refiners often try to use the ASTM D-1492 method instead of UOP 304. The problem with ASTM D-1492 is that the solubility of LAB in the electrolyte solution is very poor. This tends to give falsely low results for bromine index because not all of the LAB is dissolved in the electrolyte where it can be properly titrated. The UOP 304 method uses a different electrolyte solution which is more compatible with the LAB sample.

The ASTM D-1159 method for bromine number works fine for the paraffin/olefin feed stream but does not give accurate results for LAB because it is intended for samples with a much higher bromine number.

ASTM D-2710 could possibly be used for analyzing the bromine index of LAB, but most refiners do not have the necessary equipment or prefer not to use this "dead-stop" titration method.

12. Problem: Higher than normal LAB Br index. The reactor outlet Br index and recycle paraffin Br index are normal.

Discussion: The normal Br index at the reactor outlet and on the recycle paraffin suggests that the problem is not in the Detal reactors. Check the LAB fractionation columns to see if there could be some HAB slipping up into the LAB product. (The best way to do this is to make sure the concentration of LAB in the HAB product is normal). Check the Clay treater operation. Make sure the Br index reduction across the clay treater is normal. If not, try increasing the clay temperature.

Fresh clay contains an excess of water. It will take a few days to fully dry out the clay. During this time it may be necessary to run the clay temperature a little higher than the expected start-of-run temperature because the excess water inhibits the clay activity. When the clay is dried out, the clay temperature may be reduced to the normal start-of-run temperature. The clay treater outlet sample may also appear cloudy during the first few days following the clay change-out. This is normal and is caused by the water which is not very soluble in the LAB. This water is not a problem because it will be removed from the clay treater effluent in the overhead of the column that the effluent is sent to (e.g. Finishing column, benzene column, or paraffin column).

13. Problem: Higher than normal LAB Br index. In this case, the reactor outlet Br index and recycle paraffin Br index are higher than normal.

Discussion: The high Br index at the reactor outlet and in the recycle paraffin suggests incomplete reaction in the Detal reactor. There are many possible causes:

- a) Low reactor temperature. Check to be sure the reactor temperature control system is operating properly and the reactor temperatures are at the desired set points.
- b) Feed rate increase. If the feed rate to the Detal reactor increases, a higher reactor temperature will be required to maintain the same reactor performance (same Br index at the reactor outlet).
- c) Water on the Detal catalyst. Water inhibits the Detal catalyst activity. The best indication of the amount of water on the Detal catalyst is to check the moisture analyzer on the regenerant effluent during the high temperature hold of the regeneration (this is when the concentration of water is highest). If the regeneration effluent is higher than normal, it may be necessary to increase the Detal reactor temperature until the catalyst is dried back to the normal level. Check the feed and

recycle streams for possible sources of water. If there is water in the paraffin/olefin feed, the PEP adsorbers may have been exposed to water – check the pentane purge stream in the PEP for water. If there is water in the recycle benzene or the regenerant, check the operation of the benzene column – be sure there is sufficient heat and reflux in the column to properly strip out the water. Check the water level in the benzene column overhead receiver boot (both on the instrument and in the level glass) – refluxing free water to the column will cause water to be pushed down the column into the benzene sidecut.

- d) Aromatics or Diolefins – inhibit Detal catalyst activity. If the aromatics are high check the PEP operation. Adjust the cycle time as necessary to maximize aromatics removal. Remember that the aromatics production in Pacol increases near the end of the Pacol catalyst cycle, so the PEP cycle time will have to be shorter at that time. Check the DeFine operation (H_2 /diolefin ratio, reactor temperature, sulfur injection). Diolefins are especially damaging to the Detal catalyst.
- e) Contaminants in the makeup benzene. Contaminants such as oxygenates (e.g. p-dioxane), organic nitrogen (e.g. n-formyl morpholine), and corrosion inhibitors can be present in the benzene feed and can deactivate the Detal catalyst. Check to see if the source of the purchased benzene has changed.







XI. NORMAL SHUTDOWN

The procedures for any shutdown of the Detal unit depend on the extent of the shutdown and the length of time the unit is to be down. A total shutdown of the unit is usually only necessary for planned maintenance and inspections. The following procedure is for a planned complete shutdown of the unit. The procedures may be modified or shortened for less extensive shutdowns. Prior to any planned shutdown, the contents of the off-spec tank should be rerun through the unit to make room for the plant hydrocarbon inventory which will be sent to the off-spec tank during the shutdown.

1. Bypass the Pacol reactor to stop producing olefin. Continue to circulate paraffin until all of the olefin has been alkylated. Do not shut down the DeFine reactor until after the Pacol reactor has been bypassed.
2. As the LAB production decreases, the net bottoms flow in the paraffin, rerun, and recycle columns [or paraffin, LAB, and finishing columns] will decrease to zero. Open the bottoms pump spillback to the reboiler tub in each of these columns to keep the tub full of liquid. Open the bypass around the clay treaters and close the inlet and outlet valves. (If the clay treaters are to be emptied, this should be done before shutting down the rerun and recycle columns [or LAB and finishing columns]. The procedure is given in the Special Procedures section (XIII) under "Clay Treater Unloading").
3. When there is no more accumulation of LAB, and the net paraffin column bottoms flow drops to zero, block in the rerun column [or LAB column] feed line and the recycle column [or finishing column] net overhead line. Then shut off the hot oil to the reboilers of the rerun and recycle columns [or LAB and finishing columns]. Break the vacuum on the columns, then stop the circulation of cold reflux to the contact condensers. Pump the liquid from the accumulator trays into the columns using the reflux pumps. Open the

reboiler tub drain valves. Pump out the liquid from the column bottoms to the off-spec tank using the bottoms pumps.

4. Shut down the PEP unit according to the instructions in the operating manual for that unit. The PEP unit shutdown can be done at the same time the Detal unit is being shut down. The PEP shutdown should be done after the Pacol reactor is bypassed and before the Detal benzene and paraffin columns are shut down.

NOTE: In the following steps the reactor that was being regenerated at the time the Pacol reactor was bypassed will be referred to as the first reactor, and the reactor that was on-line will be referred to as the second reactor.

5. Continue regenerating the first reactor and continue paraffin circulation and recycle benzene to the second reactor. When the regeneration of the first reactor is complete, and the olefin in the recycle paraffin has all reacted to form LAB, switch the reactors according to the normal reactor switching sequence.
6. Regenerate the second reactor following the normal procedure.
7. If the shutdown duration will be less than one month, it is not necessary to remove the liquid from the reactors. If the reactors are to be kept filled with liquid, the catalyst should be cooled down as much as possible using the regenerant and recycle benzene/paraffin flows, then the reactors should be isolated using the manual valves at the inlet and outlet of the reactors.

If the reactors must be opened for inspection, maintenance, or catalyst reloading, then the liquid must be removed from the reactors as described in Steps 8 – 11.

8. Switch the paraffin feed flow [the #1 (top) bed feed in Split-Feed reactors] from going upstream of the static mixer, to going through the shutdown line. Then bypass the flow of recycle benzene around the first reactor but

continue circulating paraffin through the reactor via the shutdown line. Continue this "hot paraffin wash" until the benzene has been removed from the catalyst.

The temperature of the "hot paraffin wash" should be at least 160°C and preferably about 170°C. If the temperature drops below 150°C before pulling vacuum and beginning to purge with nitrogen, excessive hydrocarbon will be left on the catalyst. Experience shows that heat loss to the atmosphere can easily cause a 10°C to 20°C drop in temperature.

9. When the regeneration of the second reactor is finished and the "hot paraffin wash" has been completed in the first reactor, bypass the flow of regenerant around the reactors. Then advance the reactor valve sequencer to direct the paraffin feed (still flowing via the shutdown line) to the second reactor. Begin a "hot paraffin wash" on the second reactor to remove the benzene from the catalyst.
10. Remove the liquid from the first reactor:

Isolate the inlets to the first reactor. Using the nitrogen purge line at the reactor inlet, purge the paraffin out of the reactors to the benzene column through the shutdown line provided (on the regenerant effluent line from the reactors). When the purging is complete, isolate the reactor outlet.

If reactor vessel entry is required, the reactors can be hydrocarbon-freed by purging with nitrogen to flare, or evacuating and pressuring up with nitrogen. If the reactors are not to be opened, just keep a positive nitrogen pressure on the reactors (after emptying) to prevent air from entering the reactors.

Note – If the catalyst is to be unloaded from the Reactors: In order to minimize the amount of benzene and other hydrocarbons on the catalyst in preparation for catalyst unloading, as soon as the paraffin has been pushed out of the first reactor, and while the catalyst is still hot, align the reactor to the vacuum ejector and pull a vacuum on the reactor. Try to get below 250

mm Hg absolute pressure (-510 mm Hg at sea level); below 150 mm Hg absolute (-610 mm Hg at sea level) is better. Then break the vacuum with nitrogen and pressure up to about 0.4 to 0.7 kg/cm² above atmospheric pressure with nitrogen. Then pull a second vacuum and break with nitrogen again. After breaking the vacuum for the second time, purge the reactor with nitrogen until the catalyst is below 65°C so that it will be cool enough to unload. After the reactors have been purged and cooled, they can be unloaded according to the Reactor Unloading procedure in Section XIII.B.

11. When the benzene has been removed from the catalyst in the second reactor, bypass the paraffin feed around the reactors. Then isolate the inlets to the second reactor and purge the paraffin out of the second reactor with nitrogen using the same procedure as described in step 10.
12. During the shutdown operations, send excess paraffin to the off-spec tank as necessary from the paraffin column bottoms pump. Send excess benzene to off-spec from the recycle benzene pump.
13. Stop the flow of regenerant and reduce the levels in the benzene column, recycle paraffin stripper, and paraffin column.
14. Stop the paraffin feed from the Pacol stripper, then shut off the heat to the reboilers in the benzene column, recycle paraffin stripper, and paraffin column. Continue the circulation of cold reflux to the contact condenser in both the recycle paraffin stripper and the paraffin column.
15. In units that have a mix drum, use nitrogen to pressure the hydrocarbon from the mix drum into the benzene column.
16. Pump the hydrocarbon from the bottom of the benzene column to the recycle paraffin stripper or paraffin column feed.
17. Pump the hydrocarbon from the bottom of the recycle paraffin stripper and paraffin column to the off-spec tank.

18. Break the vacuum on the paraffin column. Then in both the recycle paraffin stripper and the paraffin column; stop the circulation of cold reflux to the contact condenser, pump the level on the accumulator tray down into the column through the hot reflux line, and then pump this material to the off-spec tank as it accumulates in the bottom of the column.





XII. EMERGENCY PROCEDURES

General

Emergencies must be recognized and acted upon immediately. The operators must carefully study, in advance, the steps to be taken in emergency situations. They should also familiarize themselves with the emergency safety factors incorporated with the design of the unit. While some of the emergencies listed in this section may not result in a unit shutdown, they could cause serious trouble if not handled properly. Exact procedures cannot be provided for every type of emergency situation. It is the responsibility of the plant superintendent to recommend safe remedial action for each specific situation. General procedures for the most common types of emergency situations are outlined on the following pages. **The best protection for any plant is an operator who is thoroughly familiar with the process flow and equipment, as well as the effects of the process variables.** In general, operators should try to protect the catalyst from damage while keeping the unit in a safe condition.

Some special considerations regarding the Detal unit in an emergency:

1. The process temperature of some of the paraffin streams can be above the auto-ignition temperature of the paraffin.
2. The unit contains benzene, which is a known health hazard.
3. Some sections of the unit operate under a vacuum. Excessive air leakage into the unit in these sections could create an explosion hazard.

A. UTILITY FAILURES

For general utility failures, it is necessary to know the trouble encountered on each piece of equipment and the action to be taken. Sometimes the failure may entail partial or complete shutdown of the unit according to the normal shutdown

procedures for the unit. However, if there is a utility failure, it will often not be possible to shut down the plant in an orderly fashion. Some of the immediate attention in case of a utility failure is to be given to protection of the catalyst in both the Detal unit and the Pacol unit.

Following is a list of the possible utility failures:

1. Loss of Steam

A steam failure will usually force a shutdown of the unit due to the loss of the following services:

- a. Vacuum system on paraffin, rerun, and recycle columns (for ejector-type vacuum systems).
- b. Hot oil system (due to loss of atomizing steam in fuel oil fired heaters).
- c. Spare steam-driven turbine pumps (if applicable).

2. Electrical Power Failure

All main process pumps will normally be lost in an electrical power failure, and a subsequent shutdown of the unit may be required. In addition, loss of air coolers, electronic instruments, electrical cooling water pumps, etc., will occur. The time required to restart the pumps and cooling fans will determine whether an extended shutdown should be carried out. If the power failure is just a few seconds long, it may be possible to restart the pumps and fans very quickly and resume operation. However, in a more serious power failure, the unit will have to be shut down. In this case, all electrically driven pump and fan switches should be turned off and the heat input to the unit (reboilers, heaters, etc.) should be reduced or shut down.

- If the recycle benzene pump stops, immediate action must be taken to protect the Detal catalyst. The olefin feed must be bypassed around the Detal reactor, and the feed should be bypassed around the Pacol reactor as well.
- If the regenerant pump stops, make sure that the control valve on the hot oil to the regenerant heater and the backpressure control valve on the regenerant from the reactor are closed.

3. Cooling Water Failure

Cooling water failure will usually necessitate a complete shutdown of the unit due to the loss of condensation in the vacuum system on the vacuum columns as well as other critical cooling services in the unit. If the cooling water is not restored quickly, the unit will have to be shut down using an accelerated shutdown procedure:

- a) Bypass olefin feed around the Detal reactors. Make sure the Pacol reactor has been bypassed. Continue benzene recycle flow through the reactor as long as possible to sweep the alkyl benzene product out of the reactor.
- b) Bypass regenerant flow around the other reactor and isolate the reactor to prevent vaporization of the benzene regenerant in the reactor.
- c) Reduce heat input to the unit as necessary to compensate for the loss of cooling water.
- d) Bypass the recycle benzene around the reactor and isolate the reactor to prevent vaporization of the hydrocarbon in the reactor.
- e) Follow the normal shutdown procedure as closely as possible.

If the vacuum can be maintained on the vacuum columns, it may be possible to keep the unit on circulation. However, it will probably be necessary to stop olefin

feed to the reactor due to the loss of cooling on the LAB and/or heavy alkylate product streams.

4. Instrument Air Failure

When instrument air fails, all control valves will assume their fail positions. For each control valve, the fail position is designed to be either open, closed, or locked in position. The fail position depends on the service, and is designed to keep the unit in a safe condition if there is an instrument air failure. In general, flow and heat control valves fail closed; reflux and spillback valves fail open. The operators should know the fail positions for all control valves.

If there is a plant-wide instrument air failure, the chances are that the unit will have to be shut down. This will be the safest course of action. If the reason for the failure can be located quickly, a shutdown may be prevented. For example, if the shutdown is the result of failure of the air drying section of the instrument air system, the air dryers can be bypassed and repaired.

If the instrument air fails to only one or two control valves, operation could possibly proceed using the control valve bypass. This ability depends on the service. Operating with a manual bypass will require constant operator attention. If the manual bypass is used, the control valve should be blocked in - just in case the instrument air is suddenly restored.

If there is an instrument air failure and the air pressure cannot be regained, proceed as follows:

- a) Bypass olefin feed and recycle benzene around the reactor. Isolate the reactor to prevent vaporization of the hydrocarbon in the reactor.
- b) Bypass regenerant flow around the other reactor. Isolate the reactor to prevent vaporization of the hydrocarbon in the reactor.

- c) Make sure that all of the heaters and reboilers are shut off to prevent overheating and overpressuring. For all the control valves that fail closed, make sure the associated block valves are closed. This will prevent loss of control if the air pressure is suddenly restored.
- d) Follow the normal shutdown procedure as closely as possible.

5. Heating Failure (Fired Heaters, Hot Oil System, etc.)

In the event of heating failure, heat input will be lost (or reduced, depending on the extent of failure). If heating failure involves loss of process side flow to a fired heater or hot oil heater, firing to the heater must be discontinued and the shutdown procedures initiated until the nature of the failure is determined. Once flow to the heater can be restored, begin firing the heater and resume unit operations in a safe and normal manner. Normally, a temporary hot oil heater shutdown can be handled without a complete unit shutdown because there is enough residual heat in the hot oil circuit to maintain heat input to the most critical services long enough to restart the heater.

If the heating failure causes a decrease in reactor feed temperature, the olefin feed must be bypassed around the reactor. Low reactor temperature can cause olefin breakthrough from the reactor as well as excessive tar formation in the reactor which could lead to permanent catalyst damage.

If the water stripping section of the benzene column is affected by the heating failure, the recycle benzene (as well as the olefin feed) must be bypassed around the reactor. Also, the regenerant must be bypassed around the other reactor. Poor operation of the water stripping section of the benzene column could cause an increase in the water content of the recycle benzene and regenerant, which could cause catalyst damage.

If the heat cannot be restored quickly, the unit must be shut down. The normal shutdown procedures should be followed as closely as possible.

B. FLANGE AND GASKET LEAKS

Thermal expansion and stress, along with dirty flange faces, are generally the cause of leaks at flanges and gasketed joints. For some of the paraffin streams, the process temperature can be above the auto-ignition temperature, so a leak could ignite even without a spark. Also, there are many streams that contain benzene, which is a known health hazard. A leak of any type is to be considered an emergency situation and these leaks must be repaired as soon as possible.

C. EXPLOSION, FIRE, OR LINE RUPTURE

In the event of explosion, fire, or line rupture, the major considerations are to minimize personnel injury, prevent a fire or prevent the fire from spreading, and to put the fire out. Proceed as follows if at all possible:

1. Stop all feed streams to the unit.
2. Stop all heat input to the unit; block in battery limit fuel supply lines.
3. Isolate the plant into sections. This should be done initially from the control room using control valves, and then in the field using manual valves if possible.
4. Depressure and shut down the unit as circumstances permit.

D. CATALYST PROTECTION

Protection of the Detal catalyst from damage, especially during an upset, is a primary concern in the operation of the unit. The operators should be familiar with the potential causes of catalyst damage so that the proper steps can be taken quickly when a situation arises that could damage the catalyst.

1. Loss of Recycle Benzene

One of the most important considerations in the protection of the Detal catalyst is to always have sufficient benzene present in the reactor to prevent the olefin from forming heavy polymers which will deactivate the catalyst. A stream containing olefins should never be fed to the reactor at any time unless a sufficient amount of benzene is mixed with the olefin to prevent polymerization. As a general rule, the design benzene/olefin ratio should be used as a minimum. This includes operations such as reactor filling, startup, and shutdown as well as normal operations. (Note that pure paraffins do not harm the Detal catalyst as long as the temperature is below about 180°C. It is just the olefins that must be diluted with benzene.)

Any time the recycle benzene flow is lost, the olefin feed to the reactors must be stopped. In addition, the Pacol reactor should always be bypassed any time the olefin feed is not going to the Detal reactor. The paraffin/olefin feed may be bypassed around the Detal reactors to help keep the benzene and paraffin columns operating normally, but the Pacol reactor must be bypassed and the valves on the paraffin/olefin feed to the Detal reactors must be closed.

An automatic shutdown is provided on the paraffin/olefin feed control valves if the recycle benzene flowmeter indicates low or no flow. Whenever a shutdown of this type occurs, the operator should always close the manual valves in the unit to insure that olefin cannot possibly get into the reactor when the recycle benzene flow is lost. If possible, with the Pacol reactor bypassed, the recycle benzene and paraffin feed should be quickly re-started through the Detal reactor bypass to minimize the upset to the benzene and paraffin columns. Once the recycle benzene flow is lined out and dry, it can be sent through the reactor. After the recycle benzene is established to the reactor, the paraffin feed should be sent to the reactor for a reasonable amount of time before starting up the Pacol reactor, to remove the olefins from the recycle paraffin stream.

2. Low Reactor Temperature

Low reactor temperature will cause olefin breakthrough from the reactor (due to incomplete reaction), and can cause excessive tar formation (due to olefin polymerization) which could result in permanent catalyst deactivation.

If reactor temperature control is lost for some reason, the olefin feed should be bypassed around the reactors, and the Pacol reactor bypassed.

Any time olefin feed is started to the Detal reactors, the reactor bed temperature, as well as the reactor feed temperature, should be properly adjusted in order to prevent possible catalyst damage.

3. DeFine Unit Upset

Diolefins in the feed to the Detal unit will cause increased polymer and tar formation which would cause catalyst deactivation. In order to prevent damage to the Detal catalyst, it is essential that the DeFine unit be operating properly to remove the diolefins.

If the DeFine unit is upset for any reason that would allow diolefins to be fed to the Detal reactors, the olefin feed must be bypassed around the Detal reactors, and the Pacol reactor bypassed. Once the DeFine unit is operating properly again, the olefin feed can be re-introduced to the Detal reactors.

4. PEP Unit Upset

Aromatics breakthrough from the PEP unit will inhibit the Detal catalyst activity. The effect should not be permanent over a short term (a few days). The aromatics production per pass through the Pacol reactor is typically less than 0.5%. However, if the PEP unit is bypassed, the aromatics concentration in the recycle paraffin can build up to 3% - 5% over a period of one to two days because only a small portion of the aromatics are removed (by reaction) per pass through the Detal unit. This higher concentration of aromatics would require a Detal reactor temperature about 20°C higher than if the PEP unit were operating properly and removing most of the aromatics.

If the PEP unit has an upset or a mechanical problem, the paraffin/olefin feed can be bypassed around the adsorbers without having a serious long-term effect on the Detal unit. If the PEP adsorbers are bypassed for any reason, the Detal reactor temperature should be increased by about 10°C immediately to compensate for the activity reduction caused by the aromatics in the paraffin/olefin feed. This will help prevent olefin breakthrough from the Detal reactors, which would lead to higher bromine index of the LAB product, and possible permanent deactivation of the Detal catalyst. If the PEP unit remains bypassed for more than a couple of hours, the Detal reactor temperature will probably have to be further increased (to about 20°C above normal) in order to prevent olefin breakthrough (due to the gradual build-up of aromatics in the recycle paraffin). The bromine index of the reactor effluent should be closely monitored in this situation and appropriate adjustments to the reactor temperature and flow rates should be made to prevent Detal catalyst damage and to maintain acceptable LAB product quality. (Remember, as the Detal reactor temperature increases, the non-linears in the LAB product increase).

5. Heavy Hydrocarbons in the Regenerant

Heavy hydrocarbons (such as paraffins, olefins, LAB, HAB, and aromatics from a PEP unit) will permanently damage the Detal catalyst if they are present in the regenerant. These same hydrocarbons, in reasonably small quantities, are not harmful in the recycle benzene. The regenerant is at a much higher temperature than the recycle benzene, and these heavy hydrocarbons tend to crack and polymerize, resulting in "tar" deposits on the surface of the catalyst at regeneration conditions. These tar deposits can cause permanent deactivation of the Detal catalyst. Small amounts (<0.5%) of the lighter paraffins should not cause significant damage to the catalyst.

If the benzene column is upset, or some other condition causes heavy hydrocarbons to get into the regenerant, the regenerant should be bypassed around the reactor until the problem is solved.

6. Water

Water is a poison to the Detal catalyst. The catalyst must be loaded in a manner which minimizes exposure to moisture. During operation of the unit, the reactor feed streams (including the regenerant) must be kept below 1 wppm water. At low concentrations (less than 10 ppm in the feed), water tends to act as a temporary poison; the activity of the catalyst will normally be restored after the water is desorbed off of the catalyst. Contact with water in the liquid aqueous phase will permanently damage the catalyst by removing the fluoride and must not be allowed to happen.

During normal operation of the unit, water is removed from the paraffin/olefin feed in the Pacol stripper. Water is removed from the make-up benzene in the top stripping section of the benzene column. When a PEP unit is included in the complex (on the paraffin/olefin feed to the Detal unit), it must be properly dried out before being brought on-line. The make-up pentane to the PEP unit must be dried before being brought into the unit.

Moisture analyzers are located on various streams in the unit (such as the paraffin/olefin feed, benzene recycle or regenerant pump discharge, regenerant effluent, and reactor effluent streams) to monitor the water concentration in these streams. If a sudden increase in water concentration is indicated in any of these streams, immediate action must be taken to minimize the amount of water going to the reactor.

If the water upset is just in the paraffin/olefin feed stream, the paraffin/olefin feed should be bypassed around the reactors and the Pacol reactor should be bypassed. If the recycle benzene and regenerant streams are still dry, they should continue to be fed to the reactors to help dry off the catalyst. When the paraffin/olefin feed stream is less than 1 wppm, and the reactor effluent moisture analyzer indicates that the catalyst moisture level is back to normal, the paraffin/olefin feed can be fed to the reactor.

If there is a water upset in the recycle benzene/regenerant stream, the regenerant should be bypassed around the reactor immediately. Then the paraffin/olefin feed should be bypassed around the other reactor (and the Pacol reactor should also be bypassed), and finally the recycle benzene should be bypassed around the reactor. Never stop the flow of recycle benzene to the reactor until after the olefin feed has been stopped. When the moisture level in the recycle benzene/regenerant stream has returned to less than 1 wppm, begin sending the regenerant and recycle benzene through the reactor again. Continue to bypass the paraffin/olefin feed around the reactor until the reactor effluent moisture analyzer indicates that the moisture level on the catalyst is back to normal.

Note that it is very important for the top stripping section of the benzene column to operate properly in order to keep the recycle benzene and the regenerant dry. An upset to the benzene column can cause a loss of water stripping in the column which will result in water being fed to the reactors. Due to the large liquid inventory in the reactors, a change in the ratio of benzene to paraffin/olefin in the reactors can cause the overhead receiver of the benzene column to go completely full or empty. This would definitely cause a water upset to the unit. Whenever the benzene column is upset, close attention should be given to the recycle benzene/regenerant moisture analyzer to prevent water in the feed to the reactors.

Following any shutdown in which equipment in the unit has been opened up to the atmosphere, it will be necessary to dryout the unit (with the reactors bypassed) before feed is started to the Detal reactors.





XIII. SPECIAL PROCEDURES

A. REACTOR LOADING

The initial catalyst loading in the Detal unit is done in two parts. First, the catalyst support balls are loaded into the bottom of each reactor bed prior to the dryout of the unit. This allows the support balls to be dried out along with the rest of the unit. Second, after the unit dryout is finished, the Detal catalyst and top hold-down balls are loaded.

For subsequent catalyst re-loads, the catalyst withdrawal sleeve in the bottom of each bed allows removal of only the catalyst and top hold-down balls, so it is not necessary to re-load the support balls in the bottom of each reactor bed.

1. Preparation and General Loading Guidelines

- a) Before the reactor is opened up for loading, all inlet and outlet lines must be blinded per standard refinery practice.
- b) Before any personnel enter the reactor, the proper confined space entry permit must be issued. Anyone entering the reactor must be equipped with the appropriate safety equipment and follow the standard confined space entry procedures.
- c) An inventory of catalyst, support balls, and any reactor internals that have been stored outside of the reactors should be made well in advance of the loading.
- d) All catalyst loading equipment should be checked for availability and operability. This includes things such as loading hoppers, loading socks or hoses, hoists or crane, fork lift, tape measure, lights, ladders, and safety equipment.

- e) The hoppers and funnels used for the catalyst loading must be clean to prevent contamination of the Detal catalyst. The loading socks or hoses should be new. Of particular concern is equipment that has previously been used to load other types of catalyst. The metals on many types of catalyst are poisons to the Detal catalyst, even in very low concentrations.
- f) When the loading is scheduled to take place during night-time hours, adequate lighting should be provided in the working area.
- g) Placement of the catalyst drums, crane, and empty drums in the unit area should be planned in advance to prevent confusion while the reactor loading is taking place.
- h) Sometimes, the personnel doing the work involved in loading the reactors do not understand the operation of the unit or know why certain loading details must be followed exactly as designated. These personnel should be properly informed as to the reasons and requirements for correct catalyst loading. In addition, the loading procedure should be continuously supervised by someone familiar with the Detal process.
- i) An inspection of the reactors and internals should be completed prior to loading. Before the initial loading, the reactor and internals should be checked for conformity to the project specifications. On subsequent loadings, the mechanical condition of the vessel and internals should be checked.
- j) Do not leave any extraneous material in the reactor. Loading personnel and supervisors should remove any objects from their pockets that might fall into the reactor. Take special care with tools and hard hats as these are often accidentally dropped into the reactor during loading. Aside from the embarrassment of unloading a reactor to retrieve an object that was overlooked during the loading period,

serious operational problems can occur due to a foreign object being left inside a reactor.

- k) Water is a poison to the Detal catalyst. Very small amounts of water in vapor form (such as the moisture in the ambient air) will only cause temporary deactivation of the catalyst and increase the time required for catalyst dryout, so limited contact with the moisture in the ambient air can be tolerated, but should be minimized. However, contact with liquid water will permanently damage the catalyst, and must not be allowed to happen.
- l) The catalyst loading must not take place during rainy, snowy, or other wet weather, in order to prevent exposure of the catalyst to moisture.
- m) Every effort should be made to minimize the time each reactor is open to the atmosphere. A dry instrument air purge or blanket should be maintained on each reactor as it is loaded to prevent ambient air from entering the reactor. All reactor manways and nozzles should be covered or closed except during actual loading of that reactor bed.

NOTE: When personnel entry into the vessel is necessary, the instrument air purge should be stopped and disconnected from the vessel. Then the vessel should be checked for proper air content (at several points in the vessel) and a new safe entry permit should be issued before personnel are allowed to enter. Many instrument air systems automatically switch to nitrogen or some other inert gas if the air compressors shut down. During the relatively short period of time when vessel entry is required during catalyst loading, a small flow of fresh ambient air through the catalyst (such as that caused by a fresh air eductor or "air mover") will not cause a problem. The exposure to fresh air may cause a minor increase in the time required to dry out the catalyst, but no permanent catalyst damage will occur.

- n) Try not to allow a natural draft of air to flow up through a reactor bed. A flow of ambient air for a prolonged period of time may cause temporary damage to the catalyst due to the moisture in the air. A natural draft can occur when a nozzle at the bottom of the reactor is open to the atmosphere at the same time as the top manway. The warm catalyst inside the reactor creates a "chimney effect" and draws air up through the catalyst bed. This effect can also work in the reverse direction when the catalyst is cooler than the ambient air. To prevent this, never allow more than one vessel nozzle to be open to the atmosphere at one time. It is recommended that all nozzles except the manway be blanked off or blinded during loading, except for nozzles being used to supply the dry instrument air purge.
- o) A detailed record should be kept of the catalyst loaded into each reactor. The lot number, drum number, and drum weight of each drum loaded into each reactor bed should be recorded along with the total weight of catalyst loaded into each bed. All support material and catalyst level outages and dimensions should be recorded. For reactor beds with manways on the side of the vessel (instead of the top), outage measurements should be made from a convenient reference point inside the reactor instead of the top manway flange. A clear note of the location of the reference point inside the vessel should be made on the loading diagram.
- p) After each bed is loaded, the loaded catalyst weight and the catalyst level dimensions should be used to calculate a loaded catalyst density.

2. Loading the Bottom Support Balls

For the initial reactor loading, the bottom support balls will be loaded, and the unit will be dried out before the catalyst is loaded. For subsequent loadings, the catalyst withdrawal nozzles are designed so that the bottom support balls remain in place and do not have to be reloaded.

It is not essential that the bottom support balls be absolutely dry for the initial loading, because they will be dried out along with the rest of the unit. (Note: It is best not to load balls that are "dripping wet" because this will unnecessarily extend the time required to dry out the unit.) However, on subsequent loadings where the bottom support balls were unloaded and a dryout will not be performed, it is important that the balls be dry in order to avoid damage to the catalyst.

- a) Make sure that the reactor is properly blinded and a safe entry permit has been issued. Any personnel entering the reactor must be equipped with the proper safety equipment.
- b) Clean the inside of the reactor. Remove any loose scale from the walls and internals of the reactor and remove any foreign material from the reactor.
- c) Install the slotted metal outlet basket in the bottom of the reactor.
- d) Install a removable catalyst support plate in each of the catalyst unloading nozzles. Install a coil of 13 mm ceramic fiber rope on top of each support plate. The purpose of the coil of ceramic fiber rope is to prevent the support balls or catalyst from wedging between the support plate and the nozzle wall, making it difficult to remove the support plate for unloading. After the support plate has been installed, install a blind flange (with gasket) on each unloading nozzle.
- e) Install the extension sleeve in the catalyst withdrawal nozzle. Some reactor beds may not have the extension sleeves. Check the reactor specification drawings to determine where extension sleeves are to be installed.
- f) Fill the catalyst unloading nozzles and extension sleeves with 6 mm catalyst support balls. The top 100 mm of the extension sleeves should be filled with 3 mm catalyst support balls.

- g) Using a piece of chalk or lumber crayon, mark the walls of the reactor to represent the depth of the 19 mm, 6 mm, and 3 mm catalyst support balls according to the project specifications. Use a carpenter's level and a tape measure to locate these marks.
- h) Begin loading the 19 mm support balls. A funnel and a long loading sock may be used to load the balls. The balls should not be allowed to "free-fall" into the reactor because they will break as they hit the bottom shell of the reactor.
- i) Load enough 19 mm balls so that the leveled surface of the balls comes up to the marks that were made on the walls of the reactor. The surface of the balls should be leveled with a flat board.
- j) Load the layers of 6 mm and 3 mm balls in a similar manner. Each layer of balls should be carefully leveled. The outage dimension for each layer should be recorded for documentation purposes. The loading personnel should be careful not to disturb the layers of 6 mm and 3 mm balls. Catalyst containment problems could result from poor loading of the support balls.
- k) When the loading of the support balls is complete, remove all loading materials from the reactor. Install the reactor bed thermowells and any other reactor internals that have not yet been installed. Then close up the reactor to prepare for the dryout of the unit.

3. Catalyst Loading

Due to the sensitivity of the Detal catalyst to water, the piping and equipment in the unit must be dry before the catalyst is loaded. Prior to the initial catalyst loading, a hydrocarbon circulation is established to dry out the unit. For subsequent loadings, if the unit is already dry, this dry out circulation may not be necessary.

- a) Before loading the catalyst, the unit should be dried out, and the bottom support balls should be properly loaded. The reactor should be cool and should not contain any hydrocarbon.
- b) Make sure that the reactor is properly blinded. Establish a purge of dry instrument air into the reactor to displace the nitrogen and prevent ambient air from entering the reactor. The amount of ambient air allowed to enter the reactor should be minimized because prolonged exposure to the moisture in the ambient air can cause temporary damage to the catalyst and increase the time required for dryout.

NOTE: When personnel entry into the vessel is necessary, the instrument air purge should be stopped and disconnected from the vessel. Then the vessel should be checked for proper air content (at several points in the vessel) and a new safe entry permit should be issued before personnel are allowed to enter. Many instrument air systems automatically switch to nitrogen or some other inert gas if the air compressors shut down. During the relatively short period of time when vessel entry is required during catalyst loading, a small flow of fresh ambient air through the catalyst (such as that caused by a fresh air eductor or "air mover") will not cause a problem. The exposure to fresh air may cause a minor increase in the time required to dry out the catalyst, but no permanent catalyst damage will occur.

- c) Open the reactor top manway. Make sure a safe entry permit has been issued. Any personnel entering the reactor must be equipped with the proper safety equipment and follow the standard confined space entry procedures.
- d) A "short sock" loading scheme should be used. This will prevent the need to have a person inside the reactor during the entire loading process.

- e) Prepare the inside of the reactor for loading. Make sure that the bottom support balls have not been disturbed. If necessary, remove any internals in the top of the reactor that will interfere with the loading. Make sure that the reactor bed thermocouples have been installed. Cover any openings (including distributors and nozzles) in the top of the reactor with plastic and tape to prevent catalyst from spilling into them.
- f) Install the catalyst loading funnel in position just outside the reactor manway. The loading funnel should be large enough so that the contents of an entire drum of catalyst can be poured into the hopper without spilling catalyst. The loading funnel should be properly supported to prevent personnel injury and catalyst loss. The funnel should be positioned so that there is enough open space in the manway to allow loading personnel to visually check the loading inside the vessel. The manway flange should be protected to prevent damage to the flange face.
- g) Attach the loading sock or hose to the catalyst loading funnel. The sock or hose must be long enough to extend into the centerline of the reactor but short enough so that the end of the sock or hose will be just above the final height of the catalyst bed. A cone-shaped distributor ("coolie hat") should be attached to the end of the loading sock or hose to evenly distribute the flow of catalyst. The end of the sock should be securely positioned in the center of the reactor near the top. The catalyst loading hopper must be positioned so that the loading sock or hose is never at an angle greater than 45 degrees from vertical, or else the catalyst will not flow through the sock or hose. The catalyst loading hopper and the cone-shaped distributor should be properly grounded to the reactor to prevent electrical sparks. The grounding wire on the cone-shaped distributor should also serve as a retaining wire to prevent loss of the distributor in case it tears loose from the sock or hose.

NOTE: An alternative loading method is to use an open-ended loading sock or hose without the cone-shaped distributor. In this method, the loading sock or hose should be cut to the same length as described above. One of the loading personnel must continuously swing the end of the hose around inside the reactor in order to evenly distribute the catalyst as it is loaded. This can normally be done from outside the reactor.

- h) At ground level, two large transfer hoppers (which will hold 4 to 6 drums of catalyst in each) should be positioned so that a crane can easily lift them up to the loading funnel which has been positioned at the reactor manway. Scaffolding should be set up around the transfer hoppers on the ground to provide a platform high enough for loading personnel to open the catalyst drums and dump them into the hoppers. A fork lift should be used to move the drums of catalyst up on to the scaffold platform.

During the loading operation, the loading personnel can be filling one of the transfer hoppers with catalyst from the drums, while the crane is lifting the other hopper up to the loading funnel to be loaded into the reactor. The loading can be done with only one transfer hopper, but it will be a little slower because the crane will have to wait with the hopper at the ground while it is filled with catalyst from the drums.

Alternatively, the loading can be performed without a transfer hopper at all. The drums may be lifted one at a time up to the loading funnel and dumped directly into the funnel. However this method will take much more time than if transfer hoppers are used to lift the catalyst.

- i) The seal on each catalyst drum should not be broken until just before the drum is opened. The number of drums that are open at any given time during the loading should be minimized to prevent catalyst damage due to contact with moisture.

- j) As each drum is dumped into the transfer hopper (or into the reactor), record the catalyst lot number and drum number on a catalyst drum summary sheet. If a composite catalyst sample is desired, take a small, equal size sample of catalyst from each drum as the drums are emptied into the transfer hopper.
- k) When the transfer hopper has been filled with catalyst, the hopper (or drum) should be lifted by crane to a position just above the loading funnel at the reactor manway. The transfer hopper should have a short sock or hose attached to the bottom outlet of the hopper which extends into the loading funnel to prevent catalyst spillage from overfilling the funnel. Excessive catalyst spillage is likely to occur if a sock or hose is not used and the catalyst flow from the transfer hopper to the loading funnel is only regulated by a slide valve. If individual drums are lifted by the crane instead of transfer hoppers, the loading funnel must be large enough to hold an entire drum of catalyst without spillage.
- l) As the catalyst is loaded, periodic visual checks should be made from the reactor manway to make sure the loading is proceeding evenly. Adequate lighting must be provided to allow the person directing the catalyst flow to observe the bed height so that he may raise the catalyst level evenly. Raising the catalyst level evenly is considered to be the most important consideration in the loading step. If the bed is raised unevenly, catalyst will roll down the "hills" that are created, resulting in an uneven loaded density across the reactor. This could result in uneven flow distribution in the reactor and poor performance of the unit.

The surface of the catalyst bed should be checked several times as the catalyst is loaded (approximately every 1000 mm of bed depth). The surface should be reasonably flat and level. If it is not, an adjustment in the loading technique should be made. The position of the cone-shaped distributor can be changed, or the method of

swinging the sock or hose (for an open-sock loading) can be changed. If severe mounding of the catalyst occurs, vessel entry may be required. The proper precautions must be taken for any vessel entry.

During the actual loading of catalyst into the reactor, a lot of dust is generated inside the reactor, making it difficult to see the catalyst level. It will probably be necessary to stop loading for a short time to allow the dust to settle in order to visually check the catalyst level. If the dry instrument air purge is hooked up to a nozzle near the top of the reactor it will tend to allow the dust to settle faster than if it is hooked up to a nozzle near the bottom. However, purging air through a nozzle near the top does not provide a flow of air through the reactor and special care should be taken in this case if it is necessary for someone to go inside the reactor.

- m) While the first 200-300 mm of catalyst is being loaded, care should be taken to prevent "cratering" or upsetting the layer of 3 mm support balls due to the impact of the falling catalyst.
- n) Load the catalyst up to the proper level according to outage dimensions calculated from the reactor drawing in the project specifications. Smooth and level the surface of the catalyst bed. Measure and record the distance between the top surface of the catalyst and the reference point (outage measurement). Also record the number of drums and total weight of catalyst loaded into the reactor.
- o) The loaded volume of the catalyst bed can be calculated from the actual loading dimensions, and using the total weight of catalyst loaded, a loaded density or Average Bulk Density (ABD) can be calculated for the reactor bed. This actual loaded density should be compared to the design loaded density to make sure that the proper amount of catalyst was loaded.

- p) After the catalyst has been loaded and the top surface smoothed off, load the layers of 6 mm and 19 mm hold down balls on top of the catalyst bed according to the dimensions given in the reactor drawing in the project specifications. Be careful not to disturb the surface of the catalyst bed as the hold down balls are loaded. Smooth off the top surface of each layer of the hold down balls.
- q) Remove the loading funnel and hose from the manway. Remove any plastic or other material that was used to cover the openings in the top of the reactor. Re-install any reactor internals that were removed in order to perform the loading. Check to make sure that the feed distributor is positioned properly and has not been plugged by catalyst. Remove all tools and foreign materials from the reactor.
- r) Make a final check inside the reactor to be sure nothing has been left inside and that all internals are in place. Clean the manway flange face and close the manway. Close up the rest of the reactor. Pressure up the reactor to about 0.5 kg/cm² with dry instrument air, then discontinue the dry instrument air purge. Maintain this positive pressure in the reactor until the other two reactor beds in the stack have been loaded.
- s) Load the other reactor beds in both of the reactor stacks in a similar manner.
- t) When all three reactor beds in a stack have been loaded, remove the blinds, then evacuate and purge the reactors with nitrogen to the flare to air-free the reactors and associated piping. Pressure the reactor section up with nitrogen to about 1.0 kg/cm² and maintain a positive pressure on the reactors until the unit is ready for startup. Check the flanges that were opened during the loading procedure for leaks before starting up.

B. REACTOR UNLOADING

During normal operation of the unit, the Detal catalyst gradually deactivates. Eventually it will become necessary to replace the catalyst in the reactors. The following is only a suggested catalyst unloading procedure, intended as a guideline for a very broad range of conditions. This procedure should be adjusted as necessary to fit the particular set of conditions and regulations that exist at the given unit.

1. Shutdown, hot paraffin wash, empty, and nitrogen purge the reactors according to the Normal Shutdown procedure given in this manual. The hydrocarbon must be adequately removed from the reactors and catalyst before the catalyst is unloaded.

In order to unload the Detal catalyst without special protective clothing, it is necessary to remove the benzene from the catalyst. The purpose of the "hot paraffin wash" (which is described in the Normal Shutdown procedure) is to remove benzene from the catalyst.

2. Install blinds as necessary to isolate the reactors for unloading.
3. Pull a vacuum on each reactor at least two times to help remove hydrocarbon from the catalyst. After each time a vacuum is pulled, pressure up with nitrogen to a positive pressure.
4. Cool the catalyst beds to less than 65°C using a nitrogen purge. Continue a small purge of nitrogen to the reactor as it is opened and unloaded in order to prevent air from entering the reactor. The top manway can be opened prior to unloading catalyst, but should not be left open during the unloading procedure. Do not allow a natural draft of air to pass through the reactor.
5. Remove all combustible materials from the area. Several CO₂ fire extinguishers should be readily available in the unloading area.

6. Remove the blankoff flange and attach a catalyst unloading spout to the catalyst unloading nozzle on the reactor. The unloading spout should have a full opening, positive shutoff valve (such as a slide valve or a ball valve).
7. Position a metal drum or metal hopper under the unloading spout. Use a hose to establish a small nitrogen purge into the drum to prevent combustion of any residual hydrocarbon on the catalyst. Alternatively, a piece of dry ice (frozen CO_2) can be placed at the bottom of each drum during unloading, to purge the drum. Use only metal drums or hoppers for unloading in case combustion occurs. Combustion of residual hydrocarbon on the catalyst inside the metal drums is normally not serious and can quickly be extinguished with CO_2 or nitrogen.
8. In addition to the usual catalyst unloading precautions, special attention should be paid to the possibility of residual benzene on the catalyst. During the unloading procedure, the concentration of benzene in the working area should be checked frequently, and proper precautions must be taken to prevent excessive personnel exposure to benzene.

Catalyst unloading should not be done in rainy, snowy, or other wet weather because the catalyst can become very corrosive when exposed to significant quantities of water. The catalyst drums should be kept dry to prevent corrosion of the drums.

9. Open the slide valve on the unloading spout and remove the catalyst support plate inside the catalyst unloading nozzle on the reactor. Stop the catalyst flow in order to retrieve the support plate from the drum if necessary. Then open the slide valve and continue unloading the catalyst into the drum.
10. Close the slide valve when the drum is full. The drums should be filled to within 10-15 cm of the top of the drum. Place the lid on the drum and seal it. Put the nitrogen hose into the next drum and begin unloading catalyst into that drum.

11. If the catalyst bridges in the unloading nozzle, or is not free-flowing, break the plug with a blast of nitrogen. Do not allow air to be drawn into the reactor.
12. A small amount of spent catalyst will be left in the bottom of the reactor because surface of the support material is flat and the catalyst will be sloped towards the unloading nozzle. The remaining catalyst may be removed by opening the top manway and using a long-handled "rake" to sweep the catalyst towards the unloading spout. Personnel performing this "raking" procedure must stay well outside the reactor opening to prevent being overcome by the nitrogen inside the reactor. The nitrogen purge on the reactor should be minimized during this "raking" procedure to prevent accumulation of nitrogen outside the reactor around the personnel at the top of the reactor. Care should be taken not to disturb the support balls in the bottom of the reactor. It should not be necessary to unload the bottom support balls in the reactor when changing out the catalyst.
13. After the spent catalyst has been unloaded, the catalyst unloading spout should be removed and the catalyst support plate installed in the unloading nozzle. A coil of 13 mm ceramic fiber rope should be installed on top of the support plate in the unloading nozzle. The unloading nozzle and catalyst withdrawal sleeve should then be filled with the appropriate size of support balls.
14. Fresh catalyst can then be loaded into the reactor according to the Reactor Loading Procedure in this manual.

C. CLAY TREATER LOADING

The rerun and recycle clay treaters should be loaded after the initial line flushing is finished and before the dryout of the unit begins. The clay treater vessels should be clean and dry before loading the clay. The clay is supported on layers of various mesh sand and ceramic balls. The graduated layers of sand prevent the clay from escaping from the vessel. See Figure XIII-1 for a typical clay treater loading diagram. The clay treaters should be loaded in accordance with the appropriate vessel drawing provided in the UOP Project Specification.

The support material in the treaters must be loaded carefully so that the clay will not escape from the vessel. The clay must also be loaded carefully to avoid the possibility of uneven flow distribution or channeling through the bed. When the clay is loaded, a canvas sock or other suitable method must be used to distribute the clay evenly across the entire bed area, avoiding a mound (or hill) of clay at any point in the bed. Particles that roll down a hill to the final resting point tend to pack to a lower density than particles that land directly in their final resting point. Also, larger particles tend to distribute toward the outer periphery of the mound. Such uneven loading patterns can cause uneven flow distribution in the bed leading to poor clay performance and short clay life.

The procedure for an initial loading is given below. The clay treaters are designed so that the clay can be removed through the side manway without disturbing the support material. Subsequent loadings are similar except that it is not necessary to rebuild the layers of support material.

The loading procedure is as follows:

1. Install the clay treater support screen at the bottom of the vessel. This screen is sometimes called the "elephant stool" because of its shape. The bottom of this stool has a plate which is used for centering. The clay and the clay support material hold the screen firmly in place. It is not necessary to fasten it to the outlet.
2. Mark the sides of the vessel with chalk indicating the various levels of clay support material. Mark the walls in at least 3 places so that the beds may be leveled properly. Only load those materials that meet the required sizing criterion and other requirements as given in the UOP Project Specification.
3. Load the ceramic balls. The balls should be passed through the side unloading manway still in the shipping bags. Open the bags in the vessel and distribute the balls manually inside the clay treater. Do not load the balls with a long loading sock through the top manway. If the sock should break under the weight of the ceramic balls, injury could result to the personnel

inside the vessel. Excessive breakage of the ceramic balls will occur if they are allowed to free-fall from the top manway or if a short loading sock is used.

Care must be taken while loading the ceramic balls so that the bottom screen is not displaced with the heavy balls during loading. If the bottom screen is displaced, the clay treater will lose its load of clay to the downstream equipment.

4. After the ceramic balls have been loaded and leveled, start loading the sand. The sand may be loaded with the normal hopper and loading sock. First load the 2-4 mesh, then the 4-8 mesh, 10-20 mesh, and 20-30 mesh. Refer to the loading diagram shown in the Project Specification vessel drawing.
5. Close the side manway and prepare the vessel for clay loading. The rest of the procedure is the same for a reload of clay as for the initial load.
6. Set up the loading hopper at the top of the vessel.
7. Load clay through a loading sock. Move the sock from side to side to distribute the clay evenly. Add the amount of clay shown in the Project Specification. Level the top surface for accurate measurement of the clay and also for proper distribution.
8. Check the inlet distributor to verify that it has been correctly installed.
9. Pressure test the vessel with nitrogen by taping all the flanges and pressuring to 50 psig (3.5 kg/cm²g). Check the flanges for leaks.
10. Purge the vessel thoroughly with nitrogen to free it of oxygen. The purge should be downflow through the vessel to flare. Continue the purge until the oxygen content of the gas leaving the vessel is less than 0.3 percent. After the purging is complete the vessel is ready for hydrocarbon filling.

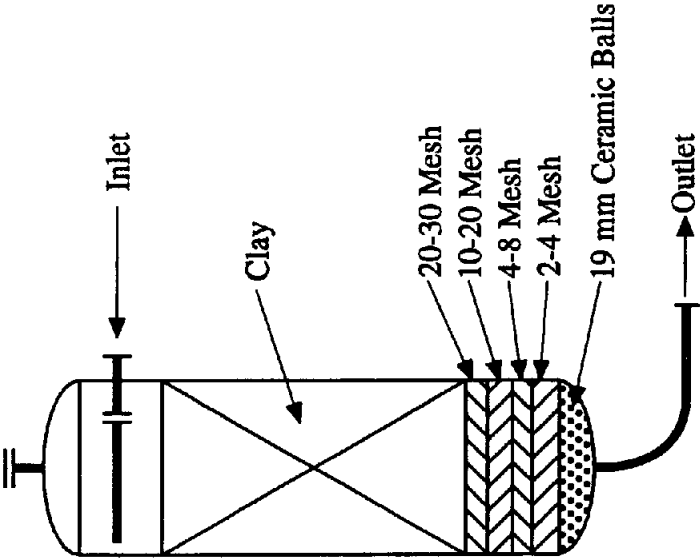
D. CLAY TREATER UNLOADING

The clay will adsorb some of the heavy olefinic compounds that pass through it and eventually the clay will have to be replaced. The clay can be replaced while the unit is running. The procedure for unloading the clay is as follows:

1. Bypass the feed around the clay treater by opening the clay treater bypass and closing the double block valves on the inlet and outlet lines of the clay treater. Open the bleeders between the double block valves on the inlet and outlet lines and install blinds between the valves if possible. If blinds cannot be installed, the bleeders must be locked open during the unloading procedure.
2. Using the nitrogen line provided, pressure the hydrocarbon contained in the treater out through the drain line to the off-spec tank. There is a sight flow indicator on this line to determine when the treater is empty. Close the valve on the drain line when the treater is empty. Do not allow an excessive quantity of nitrogen to go to the off-spec tank, because the nitrogen could cause an upset of the internal floating roof of the tank.
3. Line up the clay treater drain line to the relief header. Make sure the valve in the branch of the drain line going to the off-spec tank is closed. Using the steam line provided, begin steaming the clay treater to the relief header. Periodically drain the condensate from the low points and make sure that steam is passing through the treater (not just condensate).
4. Continue steaming out the treater for at least 24 hours to remove the hydrocarbon from the clay. After the hydrocarbon level on the clay is acceptable, stop the steam. Do not allow a vacuum to be created in the treater as the steam condenses. Use nitrogen to keep a slight positive pressure on the treater. If desired, the clay may be cooled by purging with nitrogen to the relief header.

5. Open the top manway on the treater. Remove all but 4 bolts on the side manway. If desired, install an unloading chute on or under the side manway. Position a large hopper under the side manway to catch the clay as it is unloaded.
6. Loosen the remaining 4 bolts on the side manway. Allow the clay to flow out of the side manway using the manway cover to regulate the flow.
7. When the clay no longer flows freely, purge the vessel with air. The unloading personnel must enter the vessel to unload the remaining clay and level the surface. Care must be taken not to disturb the layers of support sand under the clay bed. It is recommended that a layer of clay remain in the vessel to ensure that the support material is not disturbed.
8. Remove the unloading chute. Clean the side manway flanges and close up the side manway.
9. Load new clay into the vessel according to the previously described Clay Treater Loading procedure.

Figure XIII - 1
Clay Tower







XIV. SAFETY

The information and recommendations contained in this manual have been compiled from sources believed to be reliable and accurate. However, no guarantee, expressed or implied, is made by UOP as to the correctness or completeness of this information. Each refiner should determine the suitability of the following material for his own particular situation.

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.

NOTE: There are NIOSH (www.cdc.gov/niosh), OSHA (www.osha.gov), and API (www.api.org) publications pertaining to safety and chemical hazards that are useful references. It is the individual refiner's responsibility to make sure that the latest publications of this type are obtained as those on hand will become outdated. In addition, there may be local rules about which UOP has no knowledge that are the refiner's responsibility to keep up with and conform to. Information, and in some cases even access to the publications, is available via the internet.

A. GENERAL SAFETY PROCEDURES

Safe practices in operating and maintenance are necessary in the Detal Unit. In general, the normal refinery safety rules pertaining to the handling of hydrocarbons under pressure and moderately high temperatures also apply to the Detal Unit. Rigid rules must be developed, particularly for use during maintenance, when there is the greatest likelihood of personnel coming in contact with benzene and other hydrocarbons.

All personnel working or coming within the battery limits are required to wear protective clothing as prescribed by the foreman in charge of the unit.

No persons are allowed within the battery limits without the knowledge of the unit foreman, particularly maintenance personnel. A safety work permit is required before any work can be done. The work permit lists personnel assigned and the specific work to be done on designated equipment. The foreman must certify in the permit that all sources of pressure are blocked and blinded, that equipment to be worked on has been depressurized and purged (by unit personnel), the type of protective clothing which must be worn, and what special precautions are to be taken. Whenever any piece of equipment is initially opened, the opening must be done as though it were under pressure.

The foreman of the personnel assigned to do the work also must certify in the permit that he had a conference with the unit foreman on the hazards peculiar to the specific job, what was required of him and that the two were in agreement on how to go about the work, and that he had thoroughly briefed his own crew on potential hazards.

B. SAMPLING

Sampling liquids requires some care to limit personnel exposure and release to the environment. Sampling of streams containing benzene requires additional care. Closed sample stations are provided for benzene. Further information on sampling is included in Section IX – Analytical.

C. HYDROCARBON FREEING

Following a shutdown, each piece of equipment can be freed of the remaining traces of hydrocarbon either by steaming or by evacuating and purging with nitrogen.

For those parts of the unit which are steamed, the following guides are suggested:

1. Make maximum use of the steamout connections to introduce dry steam.
2. Supplement the above steam sources with steam hoses connected to vents and drains.
3. Each vessel should have a net steam flow from the bottom to the top.
4. Each line should have a net steam flow to purge the line.
5. Dry steam plumes should be observed at each open vent and drain.
6. Nitrogen or dry instrument air can be used to maintain a slight positive pressure, if desired, when the flow of steam is stopped. Use a compound pressure gauge.

NOTE: If nitrogen is not used to maintain a positive pressure, sufficient vents and drains must be opened to prevent pulling a vacuum on the equipment when the flow of steam is stopped.

Condensate should be drained as much as possible. Testing for combustible mixtures is recommended to make sure that adequate steaming took place.

Steam should not be used to hydrocarbon-free the reactors. Nitrogen purging should be used instead.

D. NITROGEN ATMOSPHERE

Safely Entering Confined Spaces

Nitrogen is non-toxic – 79% of the normal air we breathe is nitrogen, and 21% is oxygen. However, in vessels or areas where there is a high concentration of nitrogen, there is a deficiency of oxygen for breathing. Breathing an atmosphere deficient in oxygen will rapidly result in dizziness, unconsciousness or death depending on the time of exposure. At an oxygen concentration of 18-19%, the effects of oxygen deficiency begin to appear (nitrogen narcosis, dizziness, etc.). At 16-17% oxygen, severe oxygen deficiency reactions can take place, including loss of consciousness, coma, and death.

Do not enter or even stick your head into a vessel containing a high concentration of nitrogen and therefore deficient in oxygen. Do not stand close to bleeders where nitrogen is being bled out of equipment at a high rate which might temporarily cause a deficiency in oxygen close to the bleeder. Anyone entering a vessel which contains an inert or contaminated atmosphere must follow all prescribed standard safety precautions and regulations which apply.

E. EMERGENCY AND SAFETY EQUIPMENT

Prior to starting up any equipment in the unit, the following items must be checked for safe operation. In addition, specific inspection and maintenance programs for these safety features should be established.

1. Fire monitor and fire extinguisher systems
2. Safety showers and eyewash stations
3. Personnel communication system
4. Safety relief valves (proper rating and setting)
5. Car sealed open valves
6. Blow down facilities (vent tank and flare systems)
7. Pumpout facilities

8. Sewer system (closed H/C and open drains)
9. Instrument alarms (process and utility)
10. Shutdown instruments (automatic)

F. 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 the U.S. refiners to inform all contractors of the hazardous chemicals the contractor's employees may be exposed to while performing their work, and any suggestions for appropriate protection measures. It is then the responsibility of the contractor 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 its 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 employees should 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 file of MSDS's, labeling requirements and their meaning, and an introduction to toxicology.

The OSHA standard states that contractors performing work in these refineries are required to train their people before they enter the refinery. However, it is the responsibility of U.S. refiners to inform contractors of the specific hazardous chemicals to which contractors' employees may be exposed. The contractor should comply 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.

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 Sheets (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.

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.

G. POTENTIAL CHEMICAL HEALTH HAZARDS

Detailed information concerning all potential hazards to health caused by aromatic hydrocarbons has not been included. UOP considers this should be more properly handled by the refiner's safety department.

1. Benzene

Benzene is widely used in the manufacture of petrochemicals and has been found to be safe if proper precautions are used. However, benzene is a known carcinogen and all employees should be advised of the hazards involved and precautions to be taken when working with benzene.

Benzene is a clear, colorless liquid with a characteristically pleasant odor at low concentrations and a disagreeable odor at higher concentrations. It forms a highly flammable and explosive mixture with air at concentrations ranging from 1.4 to 6.8% benzene by volume. Pure benzene burns with a yellow, luminous, smoky flame.

Benzene is relatively insoluble in water (0.08 gram in 100 ml of water at 20°C) but is relatively soluble in all proportions with alcohol, ether, acetic acid, chloroform, carbon disulfide, carbon tetrachloride, and similar organic solvents. Commercial benzene is practically never pure and usually contains traces of impurities such as toluene, paraffinic hydrocarbons, thiophene, phenol, or similar substances. Benzene as produced by Sulfolane operations is relatively pure (99.9%) and has a freeze point of about 5.45°C (41.8°F).

Chemically, benzene is the simplest of the aromatic hydrocarbons. It is relatively stable, but is capable of a variety of substitution reactions such as chlorination, nitration, sulfonation, and alkylation. Benzene is an excellent solvent for most organic substances.

Acute exposure to benzene produces rapidly increasing symptoms of tightening of leg muscles, dizziness, excitation, and pallor, followed by flushing, weakness, headache, breathlessness, apprehension of death, and constriction in the chest. The victim may lose consciousness and pass into a coma, or may develop acute mania and delirium. Convulsions are fairly frequent. Death may occur almost at once or several hours following exposure.

Skin contact with benzene results in defatting of the skin, and leads to the development of erythema, dry scaling and, in some cases, the formation of vesicular papules. Prolonged exposure may produce lesions resembling first or second degree burns. The production of systemic benzene poisoning by skin absorption has been included in several reports and when accompanied by inhalation (which is almost always the case) is difficult to distinguish from inhalation alone. Certain simple precautions can minimize any chances of benzene contact. If benzene is spilled on clothing, including gloves, immediately remove the clothing.

Wash the skin areas exposed to benzene with soap and water. Take a complete bath if the body area is wetted with benzene. Do not wear clothing that has been wet with benzene until the garment has been washed and dried. Wearing clothing that has been wet with benzene almost assures that the person will inhale benzene vapors over a long period of time with serious hazard to health.

Avoid draining benzene to the ground or into the sewers where it can vaporize and create a health hazard. If benzene is accidentally spilled, flush it from the concrete and sewer catch basin with large quantities of cold water. Do not use hot water or steam which causes the vaporization of benzene. If you must enter an area of high benzene vapor concentration resulting from a spill, wear appropriate respiratory protection, such as a self-contained breathing apparatus.

A large benzene spill may result in a health hazard from benzene vapor at the waste water separator. The separator must be skimmed as completely as possible and all slop pumped out of the sump to the refinery recycle (or slops) system.

Though not specifically a health hazard, problems can result from benzene entering the sewer because benzene is more soluble in water than other hydrocarbons. This places an extra load on the effluent treating system.

In general, operating and laboratory personnel involved in obtaining samples should wear chemical-type safety goggles or shield, protective apron or laboratory coat, solvent-resistant gloves, and approved respiratory protective equipment where ambient concentration exceeds allowable limits. This protective equipment is not, however, a substitute for safe working conditions, proper ventilation, good personnel practices, and proper maintenance of both operating and safety equipment. In all cases, skin contact (especially eyes) and inhalation must be minimized.

Medical Attention

The U.S. NIOSH/OSHA Guidelines for benzene give recommendations for medical monitoring of personnel working in environments where exposure to these materials can occur. UOP recommends that the NIOSH/OSHA guidelines for medical monitoring be considered in development and implementation of an occupational health monitoring program for employees who may be exposed to benzene.

OSHA has recommended the following medical monitoring for employees who may be exposed to benzene:

Preplacement and quarterly examinations, including a history which includes past work exposures to benzene or any other hematologic toxins, a family history of hematological neoplasms, a history of blood dyscrasias, bleeding abnormalities, abnormal function of formed blood elements, a history of renal or liver dysfunction, a history of drugs routinely taken, alcoholic intake and systemic infections; complete blood count including a differential white blood cell count; and additional tests, where, in the opinion of the examining physician, alterations in the components of the blood are related to benzene exposure. (42 Federal Register 22516, 1977.)

UOP recommends that these guidelines be considered in development and implementation of an occupational health monitoring program for employees who may be exposed to benzene. All new or current employees should be alerted to the early signs and symptoms resulting from exposure to benzene, and any workers experiencing such symptoms should seek professional medical attention. In addition to the above, all employees should be advised of the hazards involved and precautions to be taken when working with benzene.

2. n-Paraffins

These compounds are only mildly toxic and do not have the destructive effect on blood forming organs as does benzene. Avoid breathing vapors and if clothing

becomes wet with n-paraffins, immediately remove the clothing, bathe and put on fresh clothing.

3. Catalyst

Information on safely loading and unloading the catalyst for the Detal Unit is contained in Section XIII – Special Procedures. An example MSDS for the Detal catalyst is included at the end of this Section XIV.

4. Clay

Information on safely loading and unloading the Clay Treater is contained in Section XIII – Special Procedures. An MSDS for the clay should be obtained from the clay supplier.

H. TOXICITY INFORMATION

Toxicity and safe handling information for benzene can be found in the following reference:

NIOSH criteria for a recommended standard – Occupational Exposure to Benzene

The above NIOSH publication is available through the Department of Health, Education, and Welfare of the United States of America from the following address:

NIOSH Publications
Robert A. Taft Laboratories
4676 Columbia Parkway
Cincinnati, Ohio 45226
United States of America

OSHA has established stringent maximum exposure levels for various chemicals, some of which may be found in UOP catalysts. These exposure limits can be found in the Code of Federal Regulations, **Title 29, Chapter 17, Subpart Z, Section 1910.1000**. This information is also contained in NIOSH/OSHA Pocket Guide to Chemical Hazards, **DHEW (NIOSH) Publication No. 78-210**.

I. SUMMARY OUTLINE – PERMANENT OSHA BENZENE STANDARD

1. Complete initial monitoring to determine the airborne concentrations of benzene to which employees may be exposed by April 12, 1978 (30 days from the effective date of this standard) to comply with Section 1910.1026(e)(2).
2. Subsequent employee exposure monitoring is to be performed at the following frequencies to comply with Section 1910.1028(e)(3) and (4):
 - a. Quarterly, for employees whose airborne exposure to benzene is in excess of the action level (0.5 ppm overage for 8.0-hour workday), but below the permissible exposure limit (1.0 ppm average for 8-hour workday with ceiling level of 5.0 ppm for any 15-minute period);
 - b. Monthly, for employees whose exposure exceeds the permissible exposure limit (1.0 ppm 8-hour average with 15-minute ceiling of 5.0 ppm); or
 - c. Whenever there have been changes in the process, etc., or leaks, etc., occur which may result in new or additional exposures to benzene.
3. Provide affected employees an opportunity to observe any measuring or monitoring of employee exposure, as explained in Section 1910, 1028(m).

4. Notify employees in writing within five (5) working days after the receipt of exposure measurement results, as required by Section 1910, 1028(3)(5). (Keep a copy of this notification in permanent file.)
5. Control and maintain employee exposures to airborne concentrations of benzene at or below the permissible exposure limits by the methods described in Section 1910, 1028(f) and (g). These methods are to be administered as follows:
 - a. Institute engineering and work practice controls, except to the extent that these controls are not feasible. Feasible engineering and work practice controls are to be used to reduce exposure to the lowest practicable level, even though this level is above the permissible exposure limit, and shall be supplemented by the use of respirators.
 - b. Respirators are to be used in the following circumstances:
 - (1) During the time period necessary to implement feasible engineering and work practice controls;
 - (2) Whenever feasible engineering and work practice controls are not sufficient to reduce exposure to or below the permissible exposure limit;
 - (3) During maintenance and repair work for which engineering and work practice controls are not feasible; or
 - (4) In emergencies.
6. Regulated Areas
 - a. Establish at each work location regulated areas where airborne benzene concentrations are in excess of the permissible airborne

- exposure limit, and limit access to regulated areas to authorized persons, as required by Section 1910.1028(d)(1) and (2).
- b. Provide a report to the OSHA Area Office within 30 days following the establishment of a regulated area. This report shall contain the information required by Section 1910.1028(d)(3).
- 7. Provide and require employees to wear protective clothing and appropriate equipment where eye or skin contact with liquid benzene may occur. Section 1910.1028(h).
 - 8. Provide a medical surveillance program for the following employees to comply with Section 1910.1028(i):
 - a. Employees who are or may be exposed to airborne concentrations of benzene at or above the action level (0.5 ppm average for an 8-hour workday); or
 - b. Employees who are exposed to benzene in an emergency, i.e., a massive release of benzene.
 - 9. Provide a training program at the following times for employees assigned to work areas where benzene is present to comply with Section 1910.1028(j):
 - a. Initially by June 11, 1978, which is 90 days from the effective date of the standard;
 - b. At the time employees are initially assigned to work at locations where benzene is present.
 - c. At least annually thereafter.
 - 10. Post signs in "regulated areas", as required by Section 1910.1028(k)(1).

11. Place labels on all containers of benzene and of products containing any amount of benzene to comply with Section 1910.1028(k)(2), (3), and (4).
12. Record Keeping
 - a. Establish records for all exposure measurements and medical surveillance to comply with Section 1910.1028(1).
 - b. Maintain these records for each affected employee at least 40 years or the duration of employment plus 20 years, whichever is longer. See Section 1910.1028(1)(1)(iii), and (2)(iii).
 - c. Records are to be made available for examination and copying upon request of the affected employee, physician designated by the affected employee, Assistant Secretary for OSHA, or Director of NIOSH. See Section 1910.1028 (1)(3).

J. MATERIAL SAFETY DATA SHEETS (MSDS)

UOP has included copies of the following Material Safety Data Sheets (MSDS's) for illustrative purposes only. These MSDS's may not contain exact information relating to the materials used in or resulting from the operation of the unit. In addition, the MSDS's may not be current. These MSDS's are not a substitute for safety information provided by the supplier. UOP assumes no responsibility for the use of any information contained in these MSDS's.

1. Detal Catalyst DA-114



SAFETY DATA SHEET

1. PRODUCT AND COMPANY IDENTIFICATION

Product Name: DA-114 DETAL CATALYST

Product Use: Catalyst

UOP LLC
25 E. Algonquin Road
Des Plaines, IL 60017-5017
USA
Tel: +1-847-391-3189
Fax: +1-847-391-2953

UOP Ltd.
"Liongate", Ladymead
Guildford, Surrey GU1 1AT
UK
Tel: + 44-1483-304-848
Fax: + 44-1483-466-336

Emergency Assistance - 24 hour Emergency Telephone Numbers:

EUROPE (BIG) : + 32-14-58.45.45
USA (UOP LLC) : + 1-847-391-2123
USA (CHEMTREC) : + 1-703-527-3887

2. COMPOSITION/INFORMATION ON INGREDIENTS

<u>INGREDIENT & CAS No</u>	<u>% WEIGHT</u>	<u>EC SYMBOL</u>	<u>EC R-PHRASES</u>	<u>EC Exposure Limit mg/m³</u>
Aluminosilicate 1327-36-2	>95	N.E.	N.E.	N.A.
Aluminum fluoride oxide 13596-12-8	<5	N.E.	N.E.	N.E.

Note: A list of abbreviations is compiled in Section 16.

3. HAZARD IDENTIFICATION

No hazard classification.

4. FIRST AID MEASURES

- 4.1 Eye contact:** Flush immediately with plenty of water for at least 15 minutes. If eye irritation persists, consult a physician.
- 4.2 Skin contact:** Wash off with soap and plenty of water. If skin irritation persists, call a physician.
- 4.3 After inhalation:** Remove the victim into fresh air. If symptoms persist, call a physician.
- 4.4 After ingestion:** Do not induce vomiting. Obtain medical attention.

5. FIRE FIGHTING MEASURES

- 5.1 Suitable extinguishing media:** Non-combustible. Use extinguishing media for surrounding fire.
- 5.2 Unsuitable extinguishing media:** N.A.
- 5.3 Fire and explosion hazards:** The product itself does not burn. The used product can retain material of a hazardous nature. Identify that material and inform the fire fighters.
- 5.4 Special protective equipment:** In the case of respirable dust and/or fumes, use self-contained breathing apparatus and dust impervious protective suit.

6. ACCIDENTAL RELEASE MEASURES

- 6.1 Personal protection:** See 8.2
- 6.2 Environmental precautions:** No special environmental precautions required.
- 6.3 Clean-up:** Sweep, shovel or vacuum spilled product into appropriate containers (do not use a vacuum if material has contacted a hydrocarbon material). Pick-up and arrange disposal without creating dust. Never use spilled product.
Spilled product should be disposed of in accordance with all applicable government regulations.

7. HANDLING AND STORAGE

- 7.1 Handling:** Handle and open container with care. Avoid formation of dust particles. Avoid contact with skin and eyes. Provide an electrical ground connection during loading and transfer operations to avoid static discharge in an explosive atmosphere and to prevent persons handling the product from receiving static shocks.
- 7.2 Storage:** Store in original container. Keep in a dry place.

8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

- 8.1 Engineering measures:** Where natural ventilation is inadequate, especially in confined areas, use mechanical ventilation, other engineering controls or respiratory protection to prevent inhalation of product dust.
- 8.2 Personal protection equipment:** Handle in accordance with good industrial hygiene and safety practice.
- Eye protection:** Safety glasses or goggles.
- Hand protection:** Protective gloves.
- Skin and body protection:** Dust impervious protective suit.
- Respiratory protection:** In case of insufficient ventilation wear suitable respiratory equipment with filter classification: N-95 or if oil/liquid aerosols are present P-95 (42 CFR 84).
- 8.3 Exposure limit(s):** International Exposure Limits (mg/m³)

<u>INGREDIENT & CAS No</u>	<u>% WEIGHT</u>	<u>UK OES/MEL</u>	<u>Germany MAK</u>	<u>The Netherlands MAC</u>	<u>France VME</u>
Aluminosilicate 1327-36-2	>95	N.E.	N.E.	N.E.	N.E.
Aluminum fluoride oxide 13596-12-8	<5	N.E.	N.E.	N.E.	N.E.

9. PHYSICAL AND CHEMICAL PROPERTIES

These data do not represent technical or sales specifications.

9.1 Form:	Spheres
9.2 Color:	White
9.3 Odor:	None
9.4 pH:	5.7 (10% AS)
9.5 Boiling point/range:	N.A.
9.6 Melting point/range:	N.A.
9.7 Flash point:	N.A.
9.8 Autoignition temperature:	N.A.
9.9 Bulk density:	35.5-39.5 lbs/ft ³ (0.57-0.63 g/cc)
9.10 Explosion limits:	N.A.
9.11 Vapor pressure:	N.A.
9.12 Relative density/Specific Gravity:	N.A.
9.13 Vapor density:	N.A.
9.14 Viscosity:	N.A.
9.15 Water solubility:	Insoluble
9.16 Solubility:	N.D.

10. STABILITY AND REACTIVITY

10.1 Stability:	Stable.
10.2 Hazardous decomposition products:	No decomposition if used as directed. Hydrocarbons and other materials that contact the product during normal use can be retained on the product. It is reasonable to expect that decomposition products will come from these retained materials of use. If the product is subject to extreme temperatures or chemical conditions, decomposition may occur and the product will include the oxides shown in Section 2.
10.3 Conditions/Materials to avoid:	Exposure to water may release trace amounts of fluorides.

11. TOXICOLOGICAL INFORMATION

11.1 Acute toxicity:

LD50/oral/rat = No data available.

LD50/dermal/rabbit = No data available.

LC50/inhalation/rat = No data available.

11.2 Chronic toxicity: **Classification of Ingredients**

EC Carcinogenic: Not listed.

EC Mutagenic: Not listed.

EC Toxic for Reproduction: Not listed.

Carcinogenicity (ACGIH): Not listed.

IARC classification: Not listed.

11.3 Routes of exposure: Exposure may occur via inhalation, contact with skin and eyes.

11.4 Irritation:

Skin (rabbit): No data available.

Eye (rabbit): No data available.

11.5 Additional product information:

Avoid repeated exposure.

12. ECOLOGICAL INFORMATION

- 12.1 Mobility:** No data available.
- 12.2 Biodegradation:** No data available.
- 12.3 Bioaccumulation:** No data available.
- 12.4 Aquatic toxicity:** No data available.
- 12.5 Further Information:** In a 10% slurry with water, the pH was 5.7 with 0.013 wt% fluoride after 2 days in the slurry.

13. DISPOSAL INFORMATION

- 13.1 Provisions relating to waste:** EC Directives 75/442/EC; 91/156/EC; 91/689/EC and 94/31/EC.
- 13.2 Disposal information:** Where possible recycling is preferred to disposal or incineration. Can be landfilled or incinerated, when in compliance with the local regulations. Materials of a hazardous nature that contact the product during normal use may be retained on this product. The user of the product must identify the hazards associated with the retained material in order to assess the waste disposal options.

14. TRANSPORT INFORMATION

- 14.1 Proper shipping name:** Not applicable.
- 14.2 UN-No.:** N.A.
Packing group: N.A.
- 14.3 ADR/RID:** Not regulated.
Danger Code: N.A.
Danger labels: N.A.
- 14.4 IMO:** Not regulated.
EmS: N.A.
MFAG: N.A.
Marine pollutant: No
- 14.5 ICAO - IATA:** Not regulated.
Instruction "passenger": N.A.
Instruction "cargo": N.A.
- 14.6 Further Information:**
Not classified as hazardous or dangerous in the meaning of transport regulations.

15. REGULATORY INFORMATION

The product is classified and labelled in accordance with the EC Directives and Regulations.

The product does not need to be labelled in accordance with EC-Directives.

Symbol(s): N.A.

Contains: N.A.

Risk Phrases: None.

Safety phrases: None.

European Inventory of Existing Commercial Chemical Substances:

All components of this product are included in EINECS/ELINCS.

WGK Classification (VwVwS - Germany, June 1999): 3

16. OTHER INFORMATION

Summary of changes: New MSDS
Supersedes: None.
Prepared by: UOP Health, Safety & Environmental Department

Abbreviations:

N.A.	- Not applicable
N.E.	- None established
N.D.	- Not determined
OES/MEL	- Occupational Exposure Standard / Maximum Exposure Limit
TWA	- Time-Weighted Average
STEL	- Short-Term Exposure Limit
MAK	- Maximale Arbeitsplatz-Konzentration
MAC	- Maximale Arbeidsplaats Concentratie
VME	- des Valeurs limites de Moyenne d'Exposition
EC	- European Community
IARC	- International Agency for Research on Cancer
SC	- Soluble Compounds
FD	- Fine Dust
TD	- Total Dust
FuD	- Fume and Dust
Fu	- Fume
I	- Inhalable
R	- Respirable
AS	- Aqueous slurry
IS	- Insoluble

For additional information concerning this product, contact the following:

**For health, safety and environmental information,
please contact:**

**For technical or
purchasing information,
please contact:**

Product Stewardship Manager
UOP LLC
25 E. Algonquin Road
Des Plaines, IL 60017-5017
USA
Tel: +1 (847) 391 3189
Fax: +1 (847) 391 2953

Product Safety Steward Europe
UOP N.V.
Noorderlaan 147
B-2030 Antwerpen
Belgium
Tel: +32 (3) 5409 971
Fax: +32 (3) 5417 806

Guildford Marketing Dep.
UOP Ltd.
"Liongate", Ladymead
Guildford, Surrey
GU1 1AT, England
Tel: +44 (1483) 304848
Fax: +44 (1483) 304863

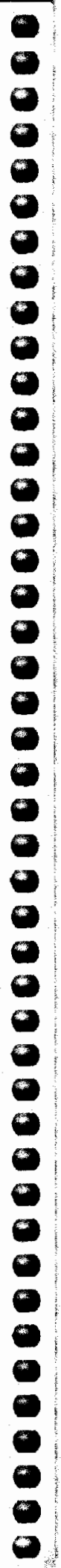
PRODUCT EMERGENCIES

If you have a product-related emergency, resulting in an incident such as a spill or release of product or human exposure and need assistance from UOP, please contact the following number :

24-Hour EMERGENCY NUMBER (BIG) : + 32 - 14 - 58.45.45

The data and recommendations presented in this data sheet concerning the use of our product and the materials contained therein are believed to be accurate and are based on information which is considered reliable as of the date hereof. However, the customer should determine the suitability of such materials for his purpose before adopting them on a commercial scale. Since the use of our products by others is beyond our control, no guarantee, express or implied, is made and no responsibility assumed for the use of this material or the results to be obtained therefrom. Information on this form is furnished for the purpose of compliance with Government Health and Safety Regulations and shall not be used for any other purposes. Moreover, the recommendations contained in this data sheet are not to be construed as a license to operate under, or a recommendation to infringe, any existing patents, nor should they be confused with state, municipal or insurance requirements, or with national safety codes.





XV. EQUIPMENT EVALUATION

Typically, shortly after the initial startup of the unit, a performance test is conducted in order to demonstrate that the unit is capable of meeting the performance guarantees. Such a performance test is often an excellent opportunity to perform a complete equipment evaluation. A good equipment evaluation has several uses:

1. It provides baseline performance data for all of the equipment in new condition. This makes it possible to compare the future performance of any piece of equipment to the original performance. Things like exchanger fouling, column flooding, column tray damage, pump damage, and hydraulic problems are much easier to identify when good records of baseline performance are available.
2. It allows the refiner to assess the full potential of the unit.
3. It helps identify potential bottlenecks for future expansion.
4. It provides data to assist in the optimization of the unit operation.

A. PERFORMANCE TEST

In order to get good results from the equipment evaluation, it is generally necessary to conduct a performance test at the same time. There is a lot of work required to conduct a good performance test. The quality of the data obtained during the performance test will be better when the following criteria are met:

1. The unit must weight balance. The weight balance must be between 98 and 102 wt% for the data to be considered reliable.
2. All operations must be steady, including quality of charge stock, quality of product, flow rates, temperatures, and pressures.

3. Samples should be taken according to the startup sampling schedule (see Section IX – Analytical). Sufficient sample containers and laboratory manpower and facilities must be available.
4. Sufficient technical and operations manpower must be available to gather the extra data and take the extra samples required.
5. All mass balance flow meters and any other critical instrumentation must be calibrated just prior to the test.
6. All flow meter correction factors should be clearly documented.
7. All data should be taken as an average if possible. This is normally possible if a distributed control system (DCS) is used to control the unit. If analog controllers are used, flow readings should be averaged from the strip charts, and the pressure and temperature readings should be taken as spot readings every 4 hours as a minimum. Generally a performance test must be a minimum of 2 to 3 days long in order to insure the unit is lined out and running smoothly.
8. The following information should be gathered during the performance test:
 - a) Flows: All process flows into and out of the unit (mass balance flows), as well as flows of intermediate streams such as recycle streams, column feeds and reflux. Also, utility flows such as steam, hot oil, boiler feed water, instrument and plant air, cooling water, fuel gas and fuel oil, electric power consumption, etc.
 - b) Temperatures: All process temperatures including local gauges.
 - c) Pressures: All process pressures including local gauges. Include suction and discharge of all pumps as well as inlet and outlet of reactors, adsorbers, and other loaded vessels.

- d) Levels: The levels of the process vessels should be recorded because they may help explain a high or low mass balance. Feed and product storage tank levels should always be recorded as a check against the flow meters. Also, record the tank levels of any chemicals injected into the process.
- e) Samples: Samples should be taken and analyzed according to the startup analytical schedule given in Section IX. It is especially important to get unit feed and product samples as well as samples of any treater inlet and outlet and all column product streams.

B. EQUIPMENT EVALUATION

The equipment evaluation will require detailed information to be collected on columns, heat exchangers, pumps, control valves, and hydraulics of piping. It is suggested that the equipment evaluation be performed along with a performance test.

In general, spot readings are taken for equipment evaluation (instead of averages, like in the performance test). Many of the readings will be from local gauges or special temporary gauges.

It is not necessary to obtain all of the detailed information at exactly the same time. As long as the unit operation is stable and consistent, the detailed data can be collected any time during the performance test (or period of lined-out operation). Sometimes it is more convenient to collect the detailed information for the equipment evaluation during a period of lined-out operation just before or after a performance test. However, all of the data for a particular piece of equipment or pressure survey should be collected at about the same time – not spread over several hours or days.

In addition to the information collected in the performance test, the following information should be collected for equipment evaluation:

1. General

General description of the unit and any unusual features.

Any comments on the data collection procedures or circumstances.

Measurement standards system that was used (e.g. Metric, English-USA, Imperial) and definition of any uncommon units. Standard conditions for liquid volume measurement and for gas flow measurement should be clearly defined.

Ambient air conditions:

Temperature

Relative humidity

Barometric pressure

Wind velocity and direction (shown on sketch of plot plan)

2. Columns

Flow rate, specific gravity, and composition of all streams entering or leaving the column.

Temperature of all streams entering or leaving the column as well as the overhead system and reboiler.

Single gauge pressure survey of the column. This should include the pressure of the overhead vapor line at the top of the column and the vapor space below the bottom tray (for total tray delta P) as a minimum.

3. Exchangers

Flow rate, specific gravity, and composition of the streams on both sides of the exchanger. Calculate the mass flow rate.

Temperature in and out on both sides of exchanger. Also between shells and bundles if there are multiple exchangers.

Pressure in and out on both sides of the exchanger if possible.

For air coolers; air temperature in and out, calculated air flow rate, motor amps, variable pitch fan position, louver position, note any belt slippage, etc.

Calculate the amount of heat exchanged and the overall heat transfer coefficient.

4. Pumps

Flow rate, specific gravity, and composition of the fluid being pumped.

Suction and discharge pressure.

Temperature of fluid being pumped.

Motor amps (actual) and voltage.

5. Control Valves

Flow rate, specific gravity, and composition of the fluid passing through the control valve.

Control valve position at flow rate stated above.

Up stream and down stream pressure.

Temperature of the stream.

6. Hydraulic (Pressure) Survey

A hydraulic survey should be performed on the reactor circuit, fractionation columns (including the overhead system), and any other important flow paths in the unit.

The purpose of the hydraulic survey is to identify any hydraulic bottlenecks and to provide a baseline for future reference. The baseline will assist in identifying problems such as plugging and fouling.

A sketch of the piping and equipment included in the survey should be made and the pressures should be written directly on the sketch.

A single pressure gauge should be used to measure the pressure at every point in the survey system. Using a single gauge will eliminate any error due to calibration of different gauges. After the pressure at all points in the survey system has been measured, the pressure at the first one or two points should be measured again to confirm that the calibration of the pressure gauge has not changed.

All flow rates and control valve positions in the survey system should be recorded.

7. Utilities

Utility consumption and production data from the following systems:

- Steam (for each pressure steam header)
- Hot Oil
- Air (Plant and Instrument)
- Nitrogen
- Cooling Water
- Boiler Feed Water
- Condensate (Steam and Process)
- Utility Water

The following Summary Sheets may be used as a guideline for gathering Equipment Evaluation data.

COLUMN SUMMARY

page _____

date

Item No.: _____ by _____

Service: _____

Type of Operation: _____

No. of Trays: _____ Reflux Ratio: _____

Type of Trays: _____

[illegible]

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

Weight balance _____ Heat balance _____

Deviations from UOP Specifications: _____

HEAT EXCHANGER SURVEY

page _____
date _____
by _____

Item No.: _____ by _____
 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: _____

		Pressure	Temperature
Shell Side	Inlet		
	Outlet		
Tube Side	Inlet		
	Outlet		
Q (calc.) Shell side			
Q (calc.) Tube side			

[illegible]

Deviations from UOP Specification: _____

CENTRIFUGAL PUMP SURVEY

page _____

date _____

Item No.: _____ by _____

Service: _____

Manufacturer: _____

Type, Model: _____

No., Size and Style (Mfgs. Designation) _____

	Pressure	Temperature	Flow Rate
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: _____

CONTROL VALVE SUMMARY

page _____
date _____
Item No.: _____ by _____
Service: _____
Description of Valve: _____ Design CV: _____
Mfr. and Catalog No.: _____
Positioner? _____

	Actual	Design
Percent open (valve position)	_____	_____
Flow rate:	_____	_____
Upstream pressure:	_____	_____
Downstream pressure:	_____	_____
Flowing temperature:	_____	_____

Deviations from UOP Specification: _____

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.

REACTOR SECTION PRESSURE SURVEY (sketch of survey system)

page _____
date _____
by _____

REACTOR SECTION PRESSURE SURVEY
(pressures)

page _____
date _____
by _____

[illegible]