



# **Pacol Enhancement Process**

## **General Operating Manual**

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## UOP PACOL ENHANCEMENT PROCESS

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The operating conditions mentioned within do not necessarily represent exact operating conditions or guarantees.

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# I. INTRODUCTION

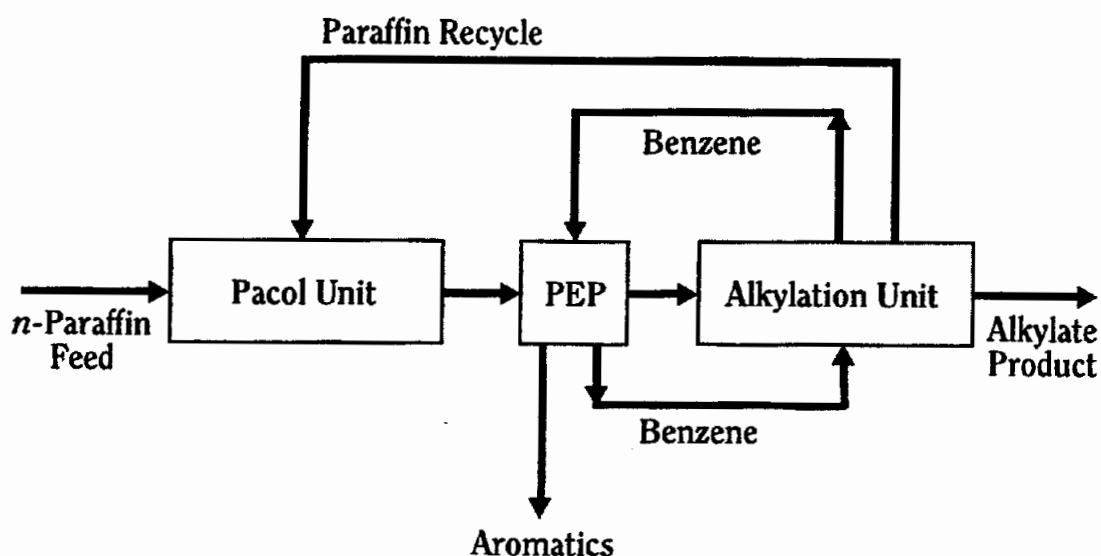
## A. PURPOSE

As the world demand for biodegradable detergents has increased dramatically in the past decade, petrochemical producers have been looking for efficient methods to improve the yield and product quality of Linear Alkyl Benzene (LAB). The removal of aromatics from the detergent alkylation unit feed is a logical step to do this.

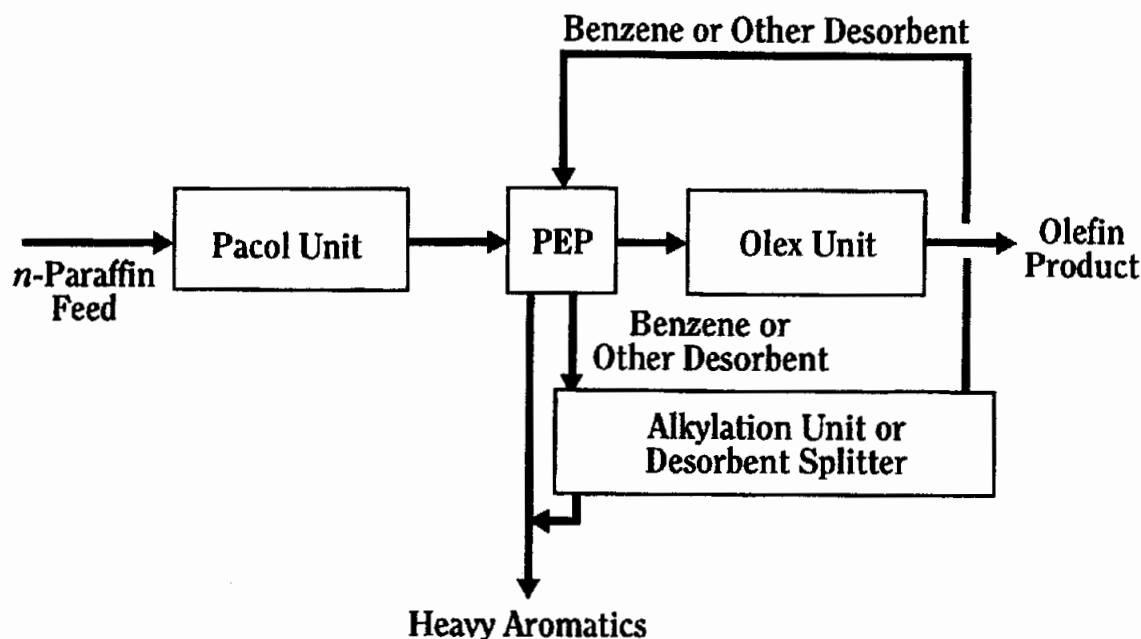
The UOP Pacol Enhancement Process (PEP) is a fixed bed adsorption unit for the selective removal of aromatic components from the Pacol product stream. Using this proprietary adsorption technology, removal of aromatics to a level less than 1% may be expected.

Normally, the PEP unit will be located downstream of the Pacol unit and upstream of a detergent alkylation or an Olex unit.

*Figure I-1*  
*Pacol Enhancement Process in LAB Complex*



**Figure I-2**  
**Pacol Enhancement Process for Olex Unit**



The primary sources of aromatics in the Pacol product are:

- A. Aromatics in the fresh n-paraffin feed to the Pacol unit
- B. Aromatics produced in the Pacol reactor
- C. Light alkylate in the recycle paraffin (from the alkylation unit)

As the aromatics build up in the recycle paraffin stream, the LAB yield will drop off and the product quality will also decline. The removal of aromatic compounds from the alkylation unit feed will reduce the production of heavy alkylate by 60-75% and increase the yield of LAB product by 3-5%. Improvement of the LAB product quality is also possible.

The reduction of aromatics in the Pacol product will have a significant impact on unit operation. If the detergent alkylation unit is an HF Alkylation process, the reduction of aromatics will increase the HF acid purity (at a given regeneration

rate), increasing the extraction of color bodies in the acid phase, which will improve the color of the LAB product. If the detergent alkylation unit is a Detal process, the reduction of aromatics will increase the activity of the catalyst, allowing the reactor temperature to be reduced by 10-15°C (18-27°F). This will increase the linearity of the LAB product by about 1.5%.

If the unit following the PEP is an Olex process, the aromatics removal from the feed will reduce the aromatics content in the Olex extract by about 80%, allow operation at higher Pacol conversion, and give a small increase in the extract purity.

## **B. PROCESS DESCRIPTION**

The Pacol Enhancement Process (PEP) is designed to remove aromatic compounds from the C<sub>10</sub> to C<sub>14</sub> paraffin/olefin product from the Pacol process. The unit is typically designed to remove about 80% of the aromatics that are normally present in the Pacol product, with minimal loss of paraffins and olefins.

The PEP unit has two sections: the adsorption section and the fractionation section. The adsorption section consists of six adsorbers loaded with molecular sieve which remove the aromatics from the Pacolate. The fractionation section typically consists of two columns which are used to recover the purge material and the desorbent.

The adsorbers cycle through a series of different steps during normal operation. Each adsorber will cycle through an adsorption step, a purge step, and a desorption step. At any given time there are typically four adsorbers on the adsorption step, one on the purge step, and one on the desorption step.

Aromatics in the feed are adsorbed into the pores of the molecular sieve during the adsorption step. In the desorption step, aromatics are displaced from the molecular sieve by the desorbent (usually benzene). Because the desorbent and the adsorbed aromatics boil at different temperatures, they can be separated by fractionation. In order to prevent the loss of paraffin/olefin feed due to mixing with

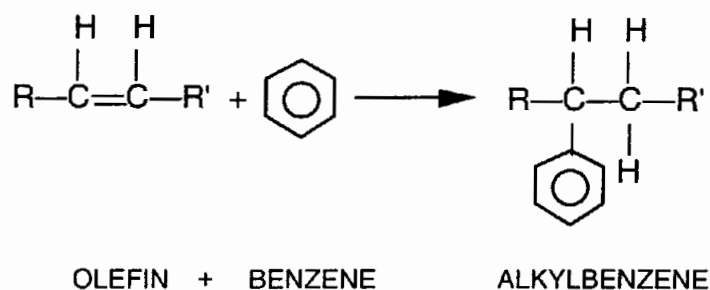
the desorbed aromatics, a purge step is necessary. During this step, a purge flow (typically n-pentane) is sent through the adsorbers to displace the paraffin/olefin product out of the adsorber before the desorbent comes in. The aromatics cannot be separated from the paraffin/olefin feed by fractionation because they boil in the same temperature range.

## II. PROCESS PRINCIPLES

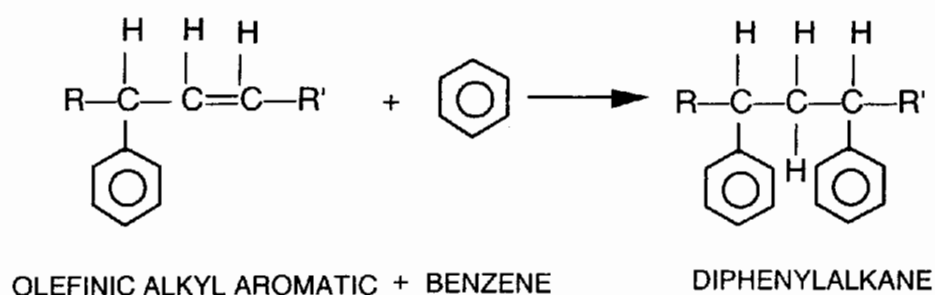
### A. CHEMISTRY

There are no chemical reactions that should take place in the PEP unit. The purpose of the PEP unit is to remove aromatics by adsorption, which does not involve any chemical reaction. However, it is helpful to understand a little bit of the chemistry involved with the detergent alkylation process in order to understand why it is necessary to remove aromatics from the Pacol product.

The desired reaction in the detergent alkylation unit is the alkylation of benzene with a linear olefin to form linear alkylbenzene:

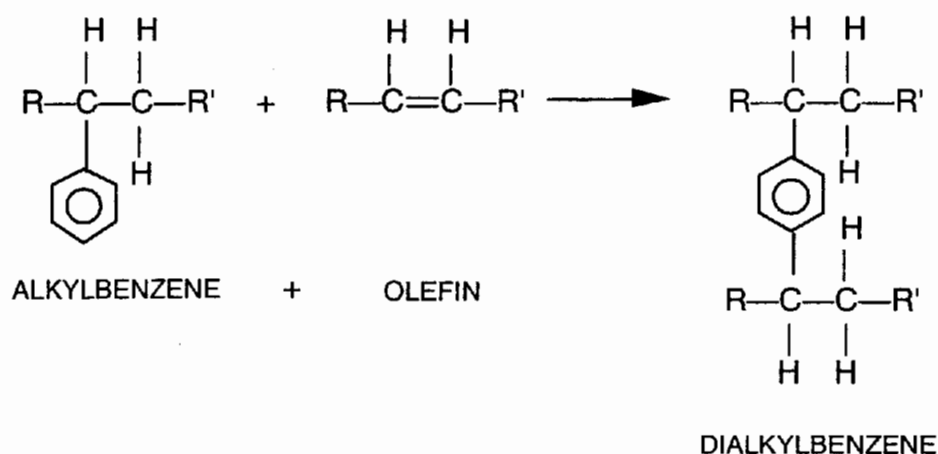


The Pacol product consists of linear paraffins and olefins in the  $\text{C}_{10}$  to  $\text{C}_{14}$  range. Only about 12% of the product is olefin, so there is a large recycle paraffin stream between the Pacol unit and the alkylation unit because the paraffins do not react in the alkylation unit. Aromatic compounds in the  $\text{C}_{10}$  to  $\text{C}_{14}$  range tend to build up in the recycle paraffin stream between the detergent alkylation unit and the Pacol unit. As these aromatics pass through the Pacol unit, a certain percentage of them undergo dehydrogenation (similar to the paraffins), resulting in an olefinic bond on the alkyl chain of the aromatic compound. This olefinic alkyl aromatic will then react with benzene in the detergent alkylation unit to form a diphenyl compound that boils in the same range as the desired linear alkylbenzene product.



These diphenyl compounds are not as biodegradable as the linear alkylbenzene and so they are undesirable.

Alkyl aromatics can also react with straight chain olefins in the detergent alkylation unit to form dialkyl benzene compounds which boil in the heavy alkylate range.



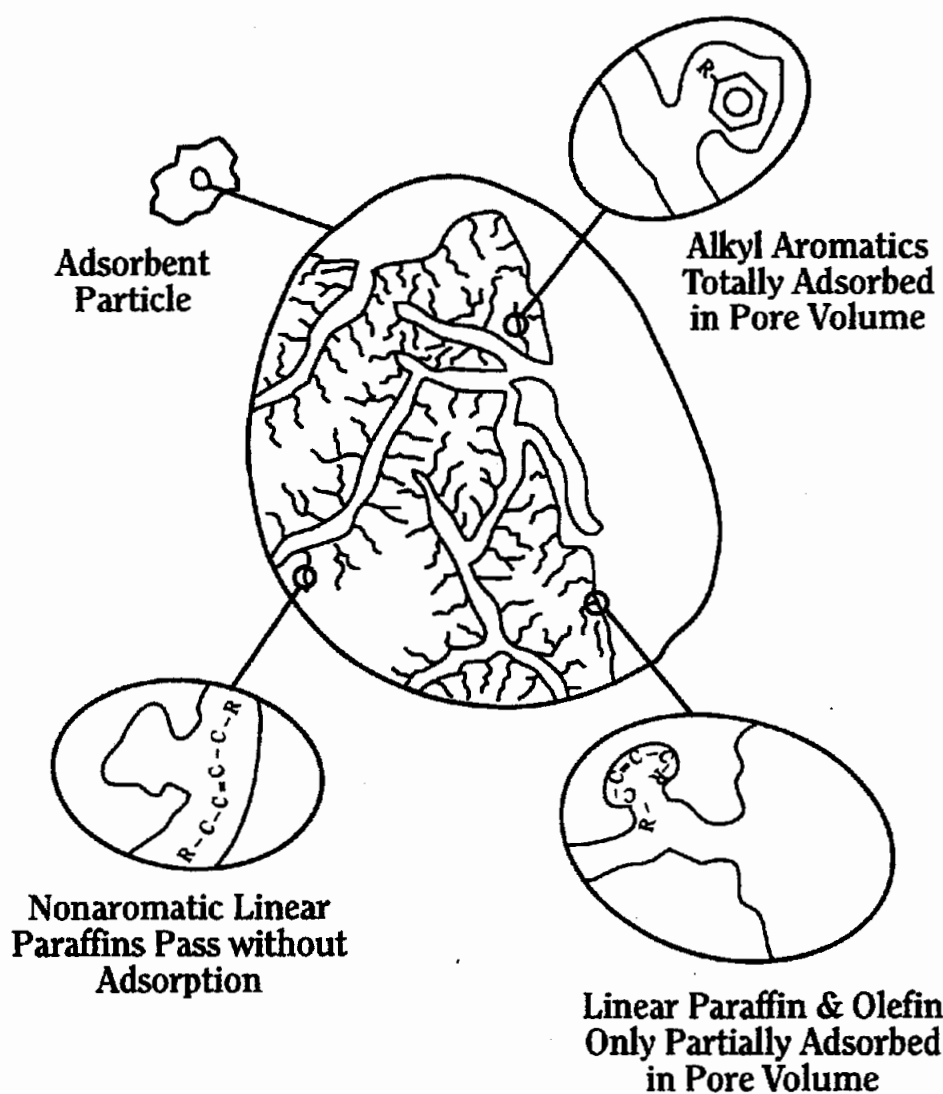
This reaction reduces the yield of LAB so it is undesirable.

## B. ADSORPTION

Adsorption is the adhesion of a very thin layer of molecules to a solid surface. So in order for adsorption to take place, there must be a solid surface, and a force or attraction to cause the adhesion of the thin layer of molecules. The molecular sieve that is used in the PEP unit adsorbers provides both the solid surface and part of the attractive force, and thus is called the adsorbent.

The molecular sieve is made up of synthetic crystals of zeolite, bound together with clay to form pellets or beads. The molecular sieve adsorbent contains a myriad network of tiny pores (similar to a sponge) as shown in Figure II-1. These tiny pores provide the enormous amount of surface area required to adsorb the aromatic compounds from the paraffin/olefin stream passing through the adsorbent. One gram of molecular sieve can contain as much as 500 square meters (5000 sq. ft.) of surface area.

**Figure II-1**  
**PEP Adsorbent**



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The molecular sieve is specially formulated to attract "polar" molecules. Some molecules, such as aromatics (and water), have slightly positive charges on one end of the molecule and slightly negative charges on the other end, and are called "polar molecules". The attractive force between the polar charge on the molecule and the specially formulated molecular sieve causes the adsorption of polar molecules onto the surface of the molecular sieve.

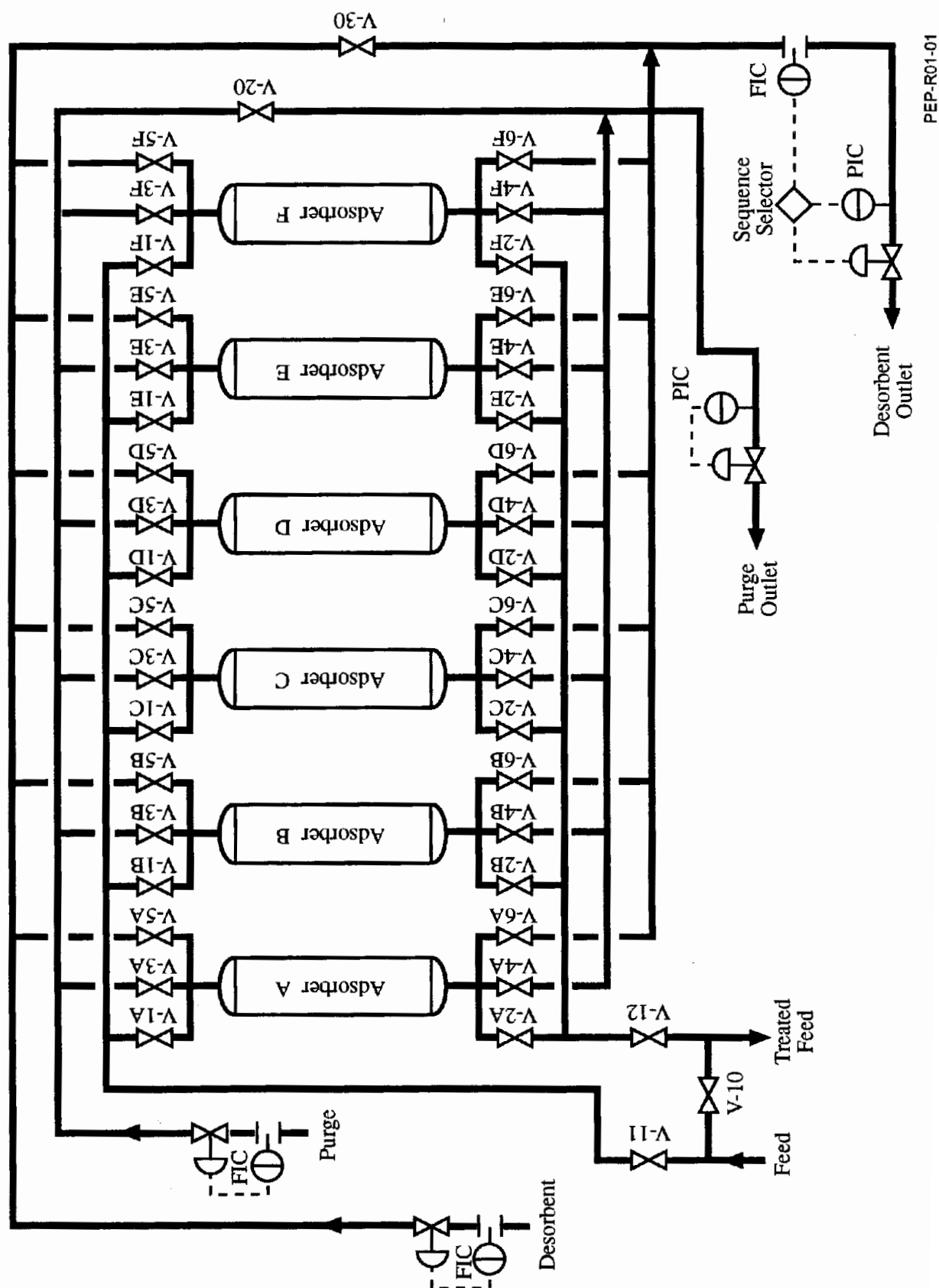
Paraffins and olefins are relatively "non-polar" so they are not strongly adsorbed on the molecular sieve. Therefore, when a mixture of paraffins/olefins and aromatics pass through a bed of adsorbent, the aromatics will be adsorbed on the molecular sieve, and the paraffins/olefins will pass through the bed. Strongly polar molecules will be more strongly adsorbed on the sieve. Benzene is more strongly polar than the alkyl aromatic compounds that are being removed from the Pacolate. Therefore, benzene can be used to desorb the alkyl aromatic compounds off the sieve.

### C. ADSORBER OPERATION

In order to apply adsorption technology to a continuous process flow, it is necessary to use multiple adsorber vessels which operate in a cyclic manner. Each adsorber will cycle through a series of steps. The sequence of steps is the same for each adsorber, but the timing of the steps is arranged so that each adsorber is at a different point in the cycle at any given time. The steps are controlled by the position of a series of remote operated valves (ROV's) on the inlet and outlet manifolds of the adsorbers (see Figure II-2). Due to the complexity of the step sequence, a valve sequence controller is used to control the opening and closing of the ROV's.

There are three main steps that are required; adsorption, purge, and desorption. In between each of these three steps there is a displacement step. There are also some other minor steps which will not be covered in this section because they do not have a significant impact on the theory of operation.

## Figure II-2 PEP Adsorption Section



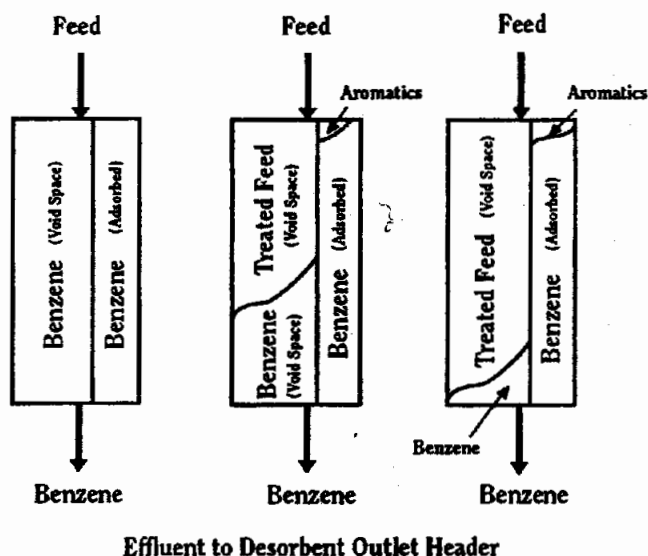
In order to understand the six basic steps that the adsorbers cycle through, we will take a look at what happens in just one adsorber over the course of an entire cycle. A full detailed description of the adsorber sequence will be included in Section IV - Process Flow.

## 1. Desorbent Displacement Step

At the beginning of the Desorbent Displacement Step the void space in the adsorber (as well as the adsorption sites on the surface of the molecular sieve) are full of desorbent (benzene). Feed is then introduced into the adsorber, and the benzene in the void space is displaced to the desorbent column. As the feed begins passing through the adsorber, the aromatics in the feed start to adsorb on the molecular sieve. Just before feed breaks through to the adsorber outlet, the Desorbent Displacement Step ends and the Adsorption Step begins.

(Note: The term "aromatics" as used here means aromatics in the feed from the Pacol unit. Even though benzene is an aromatic compound, it is not normally present in the feed from the Pacol unit and will not be included in our definition of "aromatics" in this manual.)

**Figure II-3.1**  
**Desorbent Displacement Step**



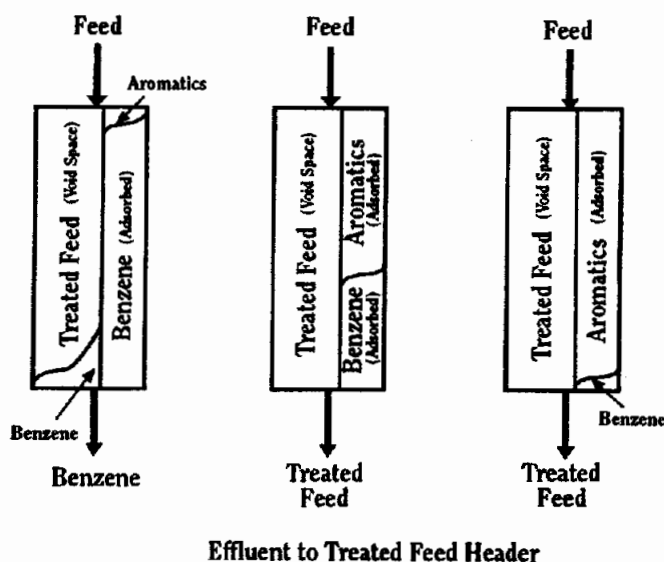
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## 2. Adsorption Step

Following the completion of the Desorbent Displacement Step, feed continues to go to the adsorber inlet, but the outlet is directed to the treated feed product line. The small amount of benzene that was left in the void space is displaced with feed. As aromatics are adsorbed on the molecular sieve, benzene is displaced from the sieve. The low concentration of benzene in the void space of the adsorber allows the adsorbed benzene to be displaced by the aromatics. This benzene flows along with the treated feed to the alkylation unit where it is recovered into the recycle benzene stream.

In order to prevent aromatics from breaking through to the adsorber outlet, the Adsorption Step must end just before the aromatics have completely filled the adsorption sites on the molecular sieve. The feed to the adsorber is stopped and the adsorber proceeds to the next step.

*Figure II-3.2*  
*Adsorption Step*

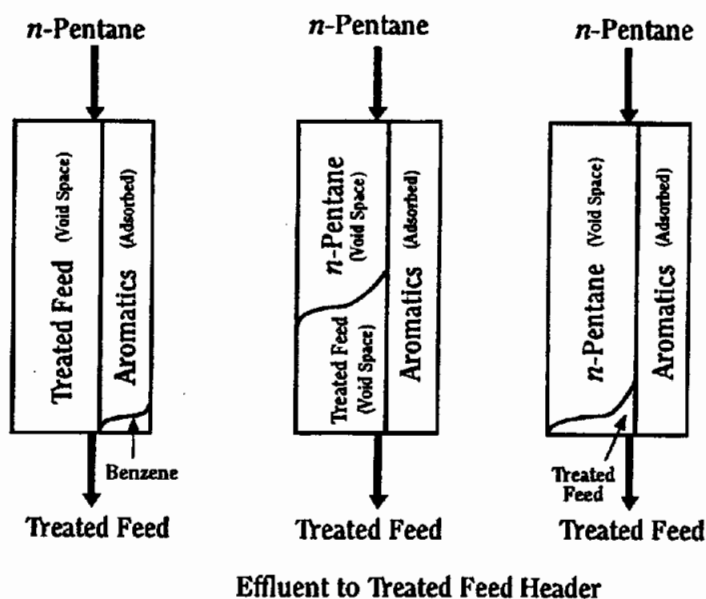


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### 3. Treated Feed Displacement Step

In order to prevent the loss of treated feed, a light paraffin (n-pentane) is fed to the top of the adsorber to displace the treated feed in the void space. During the Treated Feed Displacement Step, the adsorber effluent goes to the treated feed product line. The Treated Feed Displacement Step should end just before n-pentane breaks through to the outlet of the adsorber. Any n-pentane that leaves the unit with the treated feed will be lost.

**Figure II-3.3**  
**Treated Feed Displacement Step**

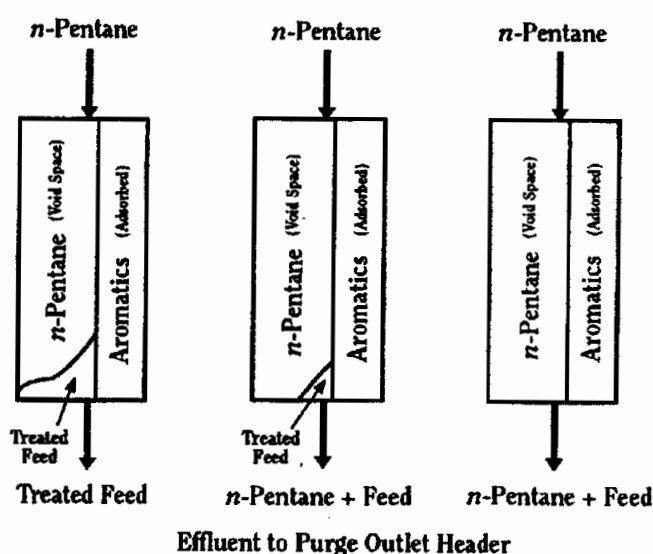


#### 4. Purge Step

The purpose of the Purge Step is to create a buffer between the feed and desorbent (benzene), which prevents contamination and/or excessive loss of the feed. If the adsorber inlet was switched directly from feed (paraffin/olefin) to desorbent (benzene), the desorbed aromatics would start coming out of the adsorber before all of the treated feed came out. This would cause a significant amount of treated feed to be lost in the desorbent column bottoms, or a significant amount of aromatics in the treated feed, depending on when the adsorber outlet valves were switched.

The n-pentane purge removes nearly all of the treated feed from the adsorber, but does not desorb any of the aromatics from the molecular sieve. During the Purge Step, the adsorber effluent goes to the depentanizer column where the n-pentane is recovered overhead, and the treated feed and benzene are sent to the alkylation unit. The purge step does not take much time and the purge flow bypasses the adsorber for some time before the Pentane Displacement step begins. The reason for bypassing the purge flow is to prevent desorbing some of the aromatics during this step.

**Figure II-3.4**  
**Purge Step**



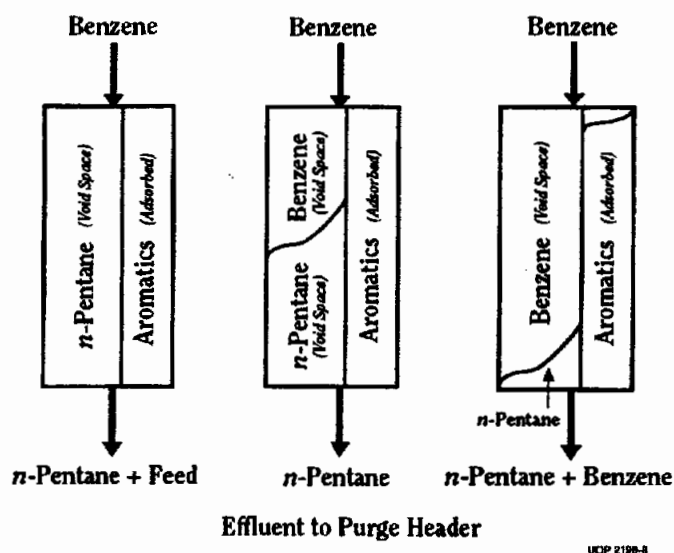
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## 5. Pentane Displacement Step

During the Pentane Displacement Step, benzene is introduced to the top of the adsorber displacing the n-pentane contained in the void space. The adsorber effluent is still sent to the depentanizer column. When most of the n-pentane has been displaced, and before the benzene desorbs a significant amount of aromatics from the sieve, the Pentane Displacement Step ends and the adsorber effluent is switched from the depentanizer to the desorbent column.

Any aromatics that go to the depentanizer column will pass on to the alkylation unit with the feed, which is undesirable. Any n-pentane that goes to the desorbent column should be recovered because the desorbent column overhead goes to the depentanizer column.

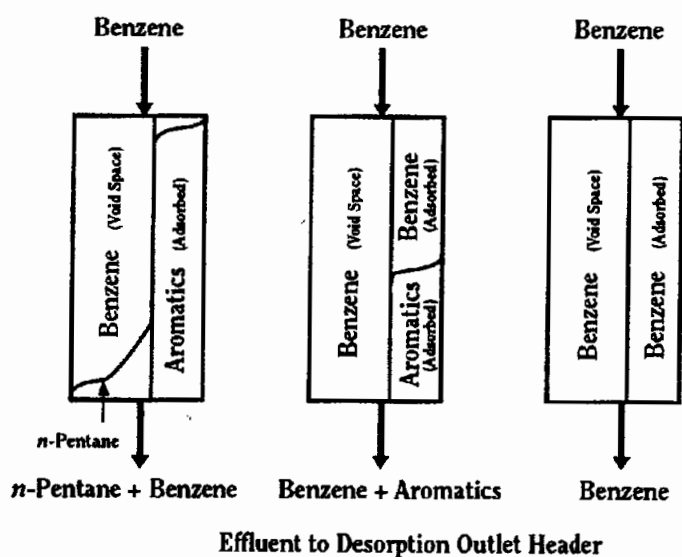
**Figure II-3.5**  
***Pentane Displacement Step***



## 6. Desorption Step

In the Desorption Step, benzene is used to remove the aromatics that are adsorbed on the molecular sieve. The adsorber effluent goes to the desorbent column where the benzene is recovered overhead and the aromatics are taken off as the bottoms product from the column. Nearly all of the aromatics are removed from the sieve during this step. At the end of the Desorption Step, the adsorber is full of benzene and the cycle returns to the Desorbent Displacement Step.

*Figure II-3.6*  
*Desorption Step*



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### III. PROCESS VARIABLES

#### A. ADSORBER TEMPERATURE

The adsorbers are designed to operate at a constant temperature. The design operating temperature of the adsorbers is set by the characteristics of the sieve and the operating pressure, and is typically about 130°C (266°F). Higher temperatures tend to reduce the capacity of the molecular sieve. Lower temperatures tend to reduce desorption efficiency. Small changes (+/- 10°C) in adsorber temperature do not have a significant effect on the process. The adsorbers should always be operated at the design temperature unless there are unusual circumstances.

The adsorbers should never be operated at a temperature which would cause vaporization of the hydrocarbons. The lightest hydrocarbon stream is the n-pentane purge, so conditions in all adsorbers should be kept so that n-pentane will stay in the liquid phase. Typically it is recommended to keep the operating temperature at least 28°C (50°F) below the vaporization temperature of the n-pentane at the operating pressure of the adsorbers.

The adsorber temperature is controlled by independently controlling the temperature of the three different streams feeding the adsorbers (feed, desorbent, and purge). The temperature of the feed, desorbent, and purge streams going to the adsorbers should always be equal to prevent large temperature swings as the adsorbers step through the sequence. Sudden, large temperature swings can cause thermal shock to the sieve, or vaporization in the adsorber which could lead to sieve damage or channeling in the sieve bed.

## B. ADSORBER PRESSURE

The adsorbers are designed to operate at a constant pressure. The design pressure is set to fit the hydraulics of the upstream and downstream units, and to keep the hydrocarbons in the liquid phase. The pressure has very little effect on the performance of the adsorbers as long as the hydrocarbons stay in the liquid phase.

Typically, there is no pressure controller on the adsorbers when they are in the adsorption step. The pressure of the adsorbers in this step is typically determined by the discharge pressure of the upstream pump (usually the Pacol stripper bottoms pump), the flow control valve on the treated feed coming out of the adsorbers (usually located in the detergent alkylation unit), and the pressure drop of the system. In order to minimize pressure swings during adsorber valve switching, the back-pressure controllers for the desorbent and purge should be set so that the pressure of the adsorbers in the desorption and purge steps are about the same as the adsorbers in the adsorption step. Of course, the back-pressure controllers should never be set above the design pressure of the vessels.

As stated above, the desorbent and purge back-pressure controllers should be set to keep the pressure of all 6 adsorbers as close to equal as possible to minimize pressure upsets during switching and to minimize the amount of any leakage across the switching valves. However, to prevent the loss of pentane, the purge pressure can be set slightly less than the adsorption and desorption pressures. That way, if there is a leak across one of the switching valves, the pressure gradient will push benzene or paraffin/olefin into the purge inlet/outlet header instead of pushing pentane out. Benzene and paraffin/olefin will be recovered properly in the PEP fractionation system if either leaks into the purge outlet line. An excessive leak of benzene into the purge inlet line could desorb some of the aromatics during the purge step and this would cause reduced aromatics removal efficiency. An excessive leak of paraffin olefin into the purge inlet header could result in a loss of paraffin/olefin because some of this paraffin/olefin will remain in the adsorber at the end of the purge step. However, either of these two consequences are not as bad as the problem of lost pentane if the pressure gradient was reversed such that the pentane is pushed out of the purge header.

Also, to prevent the loss of paraffin/olefin, the desorption pressure can be set slightly higher than the adsorption pressure. That way, if there is a leak across one of the switching valves, the pressure gradient will push benzene into the adsorption inlet/outlet header instead of pushing paraffin/olefin out. Any benzene that leaks into the adsorption outlet header will simply mix with the paraffin/olefin feed to the Detal unit where it will be used and recovered along with the rest of the benzene in the Detal reactor feed. An excessive leak of benzene into the adsorption inlet header could desorb some of the aromatics from the sieve, thus reducing the apparent aromatics removal capacity of the sieve per cycle. The cycle time could be reduced to compensate for this. In any case, this is a much better situation than if the pressure gradient was reversed such that the paraffin/olefin was pushed into the desorbent header where it would ultimately wind up in the rejected aromatics stream that is removed from the PEP fractionation system.

In summary, the desorbent and purge back-pressure controllers should be set so the adsorber pressures are as follows:

$$\text{Purge Pressure} \leq \text{Adsorption Pressure} \leq \text{Desorption Pressure}$$

### C. FEED FLOW RATE

The flow rate of the paraffin/olefin feed from the Pacol unit is typically controlled in the downstream Detergent Alkylation unit. This flow will normally be kept at a constant rate, but could change depending on the conditions in the Detergent Alkylation unit.

Even if the feed flow rate changes, the purge and desorbent flow rates should be kept at the design settings. The unit is designed to allow for changes in the feed flow rate, keeping the desorbent and purge flow rates constant. If the purge or desorbent flow rates are changed from the design settings, the adsorbers will be over (or under) purged and desorbed, causing poor aromatics removal or increased losses of feed or pentane. Therefore, the set points of the purge, desorbent, and desorbent mixing drum effluent flow meters should always be set at the design flow rate.

#### D. PURGE FLOW RATE

The unit is designed to operate with a constant purge flow rate. The purge flow should be set at the design rate regardless of the paraffin/olefin feed rate to the other adsorbers. However, the purge flow rate must be less than or equal to the total paraffin/olefin feed rate to avoid problems during the displacement step. Normally this is not a problem because the design paraffin/olefin feed rate is 4 times greater than the design purge flow rate.

If the purge flow is too low, some treated feed will be left in the adsorber at the end of the step and that feed will be lost during the subsequent desorption step. If the purge flow is too high, a small amount of aromatics may desorb from the molecular sieve during the purge step.

In order to prevent desorbing some of the aromatics due to over-purging, the purge step must be stopped about halfway through the adsorption cycle. However, in order to prevent operational upsets due to large swings in the flow rate (and composition) of the feed to the depentanizer column, the purge flow to the column must be maintained constant for the entire cycle. For this reason, there is an idle step in the sequence where the purge flow bypasses the purge adsorber for a portion of the cycle.

#### E. DESORBENT FLOW RATE

The unit is designed to operate with a constant desorbent flow rate. The desorbent flow should be set at the design rate regardless of the paraffin/olefin feed rate to the other adsorbers.

In order to prevent problems during the displacement step, the purge and desorbent flow rates should always be equal. In addition, the flow controller on the desorbent mixing drum effluent should be set at the same flow rate as the purge and desorbent flow controllers. Proper adjustment of the purge and desorbent flow rates will prevent sudden changes in the feed rates to the depentanizer and desorbent columns.

If the desorbent flow rate is too low, it is possible that the aromatics will not be fully desorbed from the molecular sieve. This will reduce the capacity of the molecular sieve on the next adsorption cycle, and could lead to aromatics breakthrough during the adsorption step. If the desorbent flow rate is too high, aromatics could be desorbed during the pentane displacement step and these aromatics would go to the alkylation unit via the depentanizer column bottoms.

## F. TIMER SETTINGS

There are three timers that are used to control the switching of the adsorber valves. The three timers run sequentially (when the Phase I timer ends and the valves change position, Phase II starts; when the Phase II timer ends and the valves change position, Phase III starts; and when the Phase III timer ends and the valves change position, Phase I starts again). The exact setting of the timers may be adjusted in the field during startup.

As mentioned earlier, the purge and desorbent flow rates should never be changed from the design flow rate, but if they are changed for some reason, all of the timer settings should also be adjusted to compensate for the change in these flow rates. Changes in the feed flow rate will not require a change in the timer settings.

### 1. Phase I Timer (Displacement)

The Phase I timer controls the length of all three displacement steps (desorbent displacement, treated feed displacement, and pentane displacement). This time will typically be about 5 minutes at the design flow rates. This is the time necessary to displace the liquid from the void space in the adsorber and piping after the inlet valves to the adsorber are switched. The approximate Phase I timer setting can be calculated, but true plug-flow does not occur in the adsorbers, so the exact timer setting should be optimized in the field.

If the displacement time is too long:

- a) Treated feed will be sent to the desorbent column during the Desorbent Displacement Step, causing excessive feed loss.
- b) n-Pentane will be sent to the treated feed outlet header during the Treated Feed Displacement Step, causing excessive loss of n-pentane.
- c) Benzene will be sent to the depentanizer column during the Pentane Displacement Step. This would not cause a benzene loss because the benzene would be recovered in the depentanizer bottoms and returned to the Detergent Alkylation unit.

If the displacement time is too short:

- a) Benzene will be sent to the treated feed outlet header during the Adsorption Step. This would not cause a benzene loss because the benzene would be sent to the Detergent Alkylation unit with the paraffin/olefin feed.
- b) Treated feed would be sent to the depentanizer column during the Purge Step. This would not cause a feed loss because the treated feed would be sent to the Detergent Alkylation unit with the benzene from the bottom of the depentanizer.
- c) n-Pentane would be sent to the desorbent column during the Desorption Step. This would not cause a n-pentane loss because the n-pentane would go overhead in the desorbent column and be returned to the depentanizer column.

When adjusting or optimizing the displacement timer setting, it is usually better for the displacement time to be a little bit too short rather than a little too long.

## 2. Phase II Timer (Purge)

The Phase II timer controls the length of the purge step. This timer will typically be set at about 15 minutes. This will give a total of about 20 minutes (Phase I + Phase II) that the n-pentane purge is flowing into an adsorber. If the n-pentane were allowed to flow through the adsorber during the entire step, a small amount of the aromatics would be desorbed, reducing the aromatics extraction efficiency of the unit. However, the purge flow rate cannot be reduced because it must remain equal to the desorbent flow rate so the unit is not upset during the displacement (Phase I) step. Therefore, the purge flow is bypassed around the adsorber at the time set by the Phase II timer.

If the purge time is too short, some treated feed will be left in the adsorber at the end of the step and that feed will be lost during the subsequent desorption step. If the purge time is too long, a small amount of aromatics may desorb from the molecular sieve during the purge step. Usually it is better for the purge step to be a little bit too long rather than a little bit too short.

## 3. Phase III Timer (Idle)

The Phase III timer controls the length of the idle step. This timer will typically be set at about 20 minutes. This will give a subcycle time (which is the same as the desorption time) of about 40 minutes (Phase I + Phase II + Phase III). The total of the three timer settings will be the amount of time that desorbent is flowing into an adsorber. Sometimes the Phase III timer is called the desorption timer because it indirectly controls the desorption time.

For a given adsorber feed rate and composition, a longer subcycle time will increase the loading of aromatics on the molecular sieve. If the subcycle time is too long, the molecular sieve will be overloaded and aromatics will break through into the treated product. A shorter subcycle time increases the number of steps in a given amount of time, which increases the loss of treated feed and n-pentane which occurs to a small extent each time the adsorber goes through a cycle. Furthermore,

if the subcycle time is much too short, the aromatics may not be completely removed during the desorption step.

The amount of aromatics in the feed to the PEP unit will vary significantly depending on several factors such as; amount of aromatics in the Pacol fresh paraffin feed, conversion in the Pacol reactor, Pacol catalyst life, and amount of LAB in the recycle paraffin. If the amount of aromatics in the PEP feed is not sufficient to "load-up" the molecular sieve in the adsorbers in the given subcycle time, a small amount of olefin may be adsorbed on the sieve. In order to minimize the loss of olefin, it may be desirable to adjust the PEP adsorber subcycle time based on the amount of aromatics in the feed. The Phase III timer can be adjusted to give a longer subcycle time when the amount of aromatics in the feed is lower.

The best way to determine if the subcycle time is too long is to analyze the treated Pacolate product from the PEP unit for aromatics content. If the subcycle is too long, the aromatics content of this stream will increase.

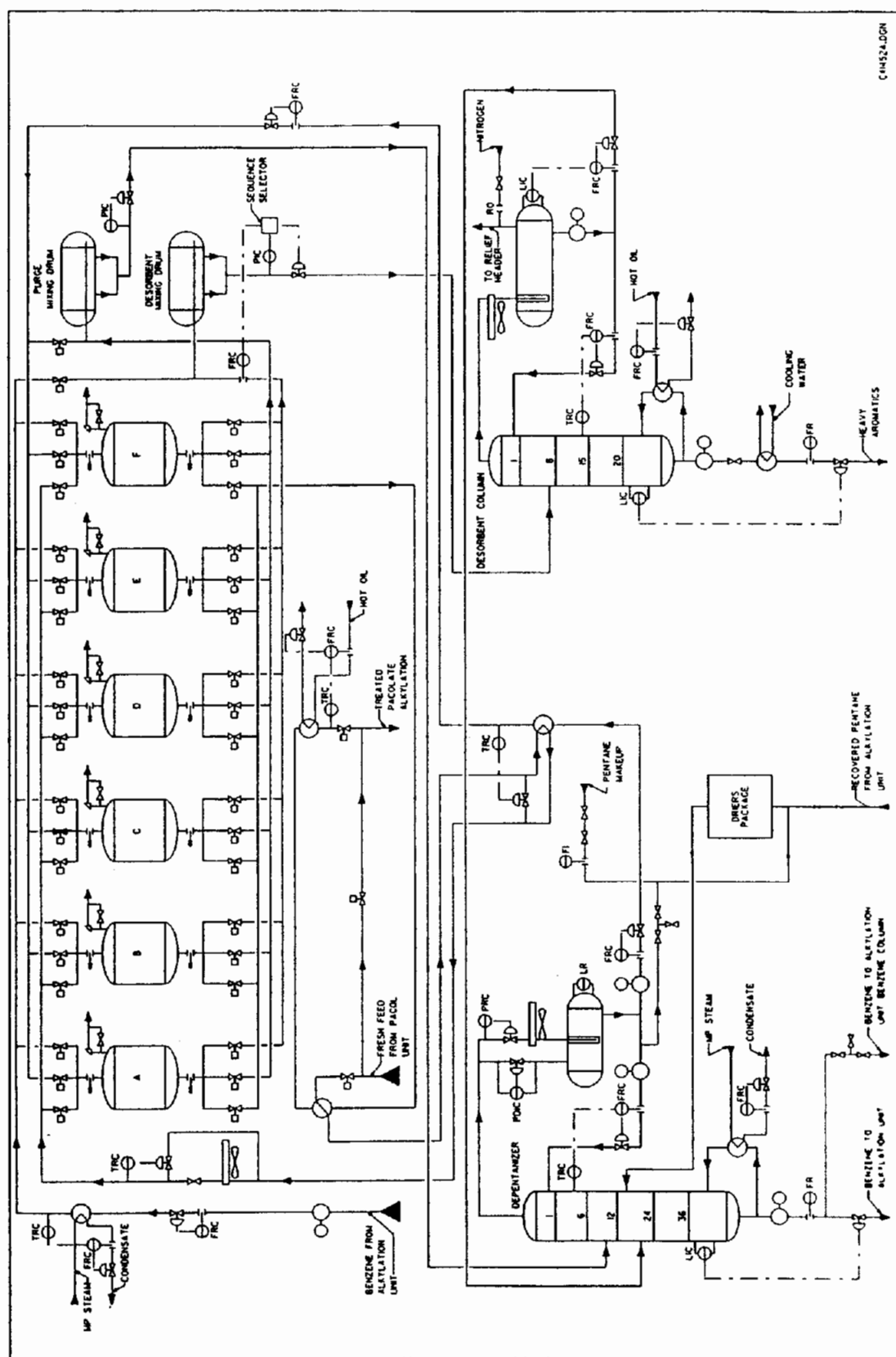
The best way to determine if the subcycle is too short is to do a complete analysis of the desorbent column bottoms for paraffins, mono-olefins, diolefins and aromatics. However, this analysis can be difficult to do. Although it is a little less accurate, the Bromine Number (Br N) of the desorbent column bottoms is a very good tool for adjusting the PEP cycle timers. The Br N is an indicator of how much olefin is adsorbed on the molecular sieve at the end of the adsorption cycle. The order of strength of adsorption (strongest to weakest) is: Benzene - Aromatics - Olefins - Paraffins. So, during an adsorption cycle, olefins are expected to be held on the adsorption sites until the aromatics displace them. Typically, a target value of 8 to 10 Br N for the desorbent column bottoms is recommended to optimize the sub-cycle time. If the Br N is more than 10, there was too much olefin left on the sieve at the end of the cycle - so the Desorption Time should be increased. If the Br N is less than 8, then there was almost no olefin left on the sieve at the end of the cycle, and the aromatics could be breaking through - so the Desorption Time should be decreased.

## IV. PROCESS FLOW and CONTROL

### A. GENERAL

The basic process flow diagram of a typical PEP unit is shown in Figure IV-1. The feed to the PEP unit is typically stripped C<sub>10</sub> to C<sub>14</sub> linear paraffins and olefins from a Pacol unit. The feed enters the unit, passes through the adsorbers where most of the aromatic compounds are removed, and the treated feed then leaves the unit and typically goes to a detergent alkylation unit. Benzene (desorbent) comes into the unit, passes through the adsorber on the desorption cycle, and goes to the desorbent column. The desorbed heavy aromatics are sent to storage off the bottom of the desorbent column. The overhead of the desorbent column goes to the depentanizer column, where pentane is taken overhead and benzene goes out the bottom, returning to the detergent alkylation unit. Pentane (from the depentanizer overhead) passes through the adsorber on the purge cycle and returns to the depentanizer column.

**Figure IV-1**  
**Pacol Enhancement Process**



## B. ADSORBER SECTION

The adsorber section typically has 6 adsorber vessels loaded with molecular sieve. Each adsorber has an inlet and an outlet manifold with switching valves so that it can cycle through the adsorption, purge, and desorption steps. A logic controller is used to control the sequencing of the switching valves. A more complete discussion of the valve switching sequence and equipment is included later in this section.

The fresh feed to the PEP unit typically comes from the Pacol stripper column bottoms pumps. The feed typically passes through one or more heat exchangers to cool the feed to the desired adsorber temperature. The exact configuration of the heat exchange train will vary from one unit to another.

The feed is distributed to 4 of the 6 adsorbers via the adsorber feed header and switching valves. The feed enters the top of the adsorbers and should be distributed evenly with 25% of the total flow going through each of the four adsorbers in the adsorption step. There are not any flow meters or control valves to split the feed to the adsorbers, therefore the inlet and outlet piping must be arranged so that there is an equal pressure drop (and therefore an equal flow) through each of the adsorbers. Inside the adsorbers, aromatics are removed from the feed by adsorption onto the molecular sieve. The treated feed leaves the bottom of the adsorbers, flows into the treated feed header via the switching valves and proceeds to the detergent alkylation unit.

One of the 6 adsorbers will be in the purge step. n-Pentane is taken from the depentanizer overhead receiver on flow control to a heat exchanger where the n-pentane is adjusted to the desired adsorber temperature. The n-pentane then goes into the top of the adsorber in the purge step via the purge inlet header and switching valve. The n-pentane displaces the paraffin and olefin feed components from the pores of the molecular sieve in the adsorber. The purge adsorber outlet goes to the purge mixing drum via the switching valve and the purge outlet header. The mixing drum runs liquid full. The purpose of the mixing drum is to smooth out composition changes that occur during the adsorber sequence which could upset the operation of the depentanizer column. A back pressure control valve on the

mixing drum controls the pressure of the adsorber in the purge step. The purge mixing drum effluent goes to the depentanizer column.

The last of the 6 adsorbers will be in the desorption step. Benzene is typically pumped from the detergent alkylation unit on flow control to a heat exchanger where the benzene is adjusted to the desired adsorber temperature. The benzene then goes into the top of the adsorber in the desorption step via the desorbent inlet header and switching valve. The benzene desorbs (removes) the aromatics from the molecular sieve in the adsorber. The desorption step effluent goes to the desorbent mixing drum via the switching valve and the purge outlet header. The mixing drum runs liquid full. The purpose of the mixing drum is to smooth out composition changes that occur during the adsorber sequence which could upset the operation of the desorbent column. A back pressure control valve on the mixing drum controls the pressure of the adsorber in the desorption step. The desorbent mixing drum effluent goes to the desorbent column. During the displacement step, the back pressure control valve on the mixing drum is reset by a flow controller instead of the pressure controller. This is necessary because the flow rate of paraffin/olefin feed to the adsorber during this step is not controlled upstream of the adsorber.

### C. DESORBENT COLUMN

The feed to the desorbent column is the effluent from the desorbent mixing drum. This stream contains mostly benzene along with the aromatics that are desorbed from the molecular sieve during the desorption step. At certain times in the adsorber sequence, there will also be some n-pentane in this stream due to imperfect separation between the pentane displacement step and the desorption step. The mixing drum reduces the upsetting effect of the sudden changes in the concentration of n-pentane and aromatics that occur during the adsorber sequence. This helps prevent upsets in the operation of the desorbent column.

In the desorbent column, the benzene and n-pentane are taken overhead and the aromatics go out the bottom of the column. The desorbent column reflux is reset by a temperature controller located on a tray at an intermediate point in the column. The desorbent column net overhead flow is reset by the level in the overhead receiver. The net overhead goes to the depentanizer column where the n-pentane is separated from the benzene. The desorbent column net bottoms flow is reset by the bottoms level. The net bottoms is typically pumped through a cooler to a storage tank. This stream consists of the aromatics that have been removed from the paraffin/olefin feed.

The paraffin/olefin feed to the PEP unit is heavier than benzene. Any paraffin/olefin feed that slips into the desorbent column feed will be lost out the bottom of the column with the aromatics. Therefore it is important to minimize the amount of paraffin/olefin feed that goes to the desorbent column in order to minimize the loss of this material.

#### **D. DEPENTANIZER COLUMN**

There are two feed streams to the depentanizer column. The first is the net overhead from the desorbent column which is mostly benzene along with a little n-pentane. The second is the effluent from the purge mixing drum. This stream contains mostly n-pentane along with the treated feed that is displaced from the pores of the molecular sieve during the purge step. At certain times in the adsorber sequence, there will also be some benzene in this stream due to imperfect separation between the pentane displacement step and the desorption step. The purpose of the mixing drum is to smooth out the sudden changes in the concentration of treated feed and benzene that occur during the adsorber sequence. This helps prevent upsets in the operation of the depentanizer column. The two different feed streams enter the column at different trays because the composition of the streams are much different, therefore the optimum feed point is different.

In the depentanizer column, the n-pentane is taken overhead and the benzene and treated feed go out the bottom of the column. The depentanizer column reflux is reset by a temperature controller located on a tray at an intermediate point in the column. The net overhead stream is the purge to the adsorber section. The net overhead flow controller is set to provide the desired purge flow rate. The overhead receiver acts as the surge drum for n-pentane in the unit so there is no controller on the liquid level. The depentanizer column net bottoms flow is reset by the bottoms level. The net bottoms consists of the benzene that has been used as desorbent in the unit along with some treated feed. This stream is typically pumped back to the detergent alkylation unit reactor section.

Any aromatics that slip into the depentanizer column feed will go out the bottom of the column with the benzene and on to the detergent alkylation unit. Therefore it is important to minimize the amount of desorbed aromatics that go to the depentanizer column in order to maximize the aromatics removal efficiency of the PEP unit.

#### **E. ADSORBER SEQUENCE CONTROLLER**

The six adsorbers must run through a cycle of adsorption, purge, and desorption steps in order to continuously remove the aromatics from the paraffin/olefin feed. The adsorber sequence controller (or logic controller) automatically opens and closes the valves on the adsorber inlet and outlet manifolds to perform these steps.

The total cycle time for the entire sequence is typically 240 minutes. During the total cycle, each adsorber will spend 160 minutes in the adsorption step, 40 minutes in the purge/idle step, and 40 minutes in the desorption step.

The total cycle is divided into 6 subcycles which are typically 40 minutes each. During each subcycle, one of the adsorbers will complete a purge/idle step, and one adsorber will complete a desorption step. The other four adsorbers will be in the adsorption step.

Each subcycle is divided into three phases:

Phase I typically lasts about 5 minutes. During Phase I, three of the adsorbers will undergo a displacement step:

- a. The adsorber switching from the desorption step to the adsorption step will undergo a desorbent displacement step. Treated feed is used to displace the desorbent from the void space into the desorbent outlet header.
- b. The adsorber switching from the adsorption step to the purge step will undergo a treated feed displacement step. n-Pentane is used to displace the treated feed from the void space into the treated feed outlet header.
- c. The adsorber switching from the purge step to the desorption step will undergo a pentane displacement step. Desorbent is used to displace the n-pentane from the void space into the purge outlet header.
- d. The other three adsorbers will remain on the adsorption step.

Phase II typically lasts about 15 minutes. During Phase II, one adsorber will start a desorption step and one adsorber will complete a purge step:

- a. The adsorber that just completed a desorbent displacement step will start an adsorption step. The adsorber will remain on the adsorption step for 4 subcycles.
- b. The adsorber that just completed a treated feed displacement step will start a purge step. The purge step will end at the end of Phase II. (The total purge time is Phase I + Phase II = typically 20 minutes).
- c. The adsorber that just completed a pentane displacement step will start a desorption step. The desorption step will last until the end of Phase III. (The total desorption time is Phase I + Phase II + Phase III = typically 40 minutes).
- d. The other three adsorbers will remain on the adsorption step.

Phase III typically lasts about 20 minutes. During Phase III, the adsorber that started a desorption step will finish that step, and the adsorber that completed a purge step will be idle.

- a. The adsorber that started an adsorption step in Phase II will remain on the adsorption step.
- b. The adsorber that started a purge step in Phase II will be bypassed and will be idle during Phase III.
- c. The adsorber that started a desorption step in Phase II will remain on the desorption step until the end of Phase III.
- d. The other three adsorbers will remain on the adsorption step.

The details of the logic controller operation may differ slightly from one unit to another. A general description of the logic 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 valve sequence based on the times set by the Phase I, II, & III timers. 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 Phase I, II & III timers will not cause the sequence to advance in this mode. All of the appropriate valve position verifications and interlocks are fully operational in this mode.
  - c) In the Manual mode, each individual valve can be opened or closed using the logic controller. The main purpose of this mode is for dryout operations and maintenance. In order to switch from the Manual mode

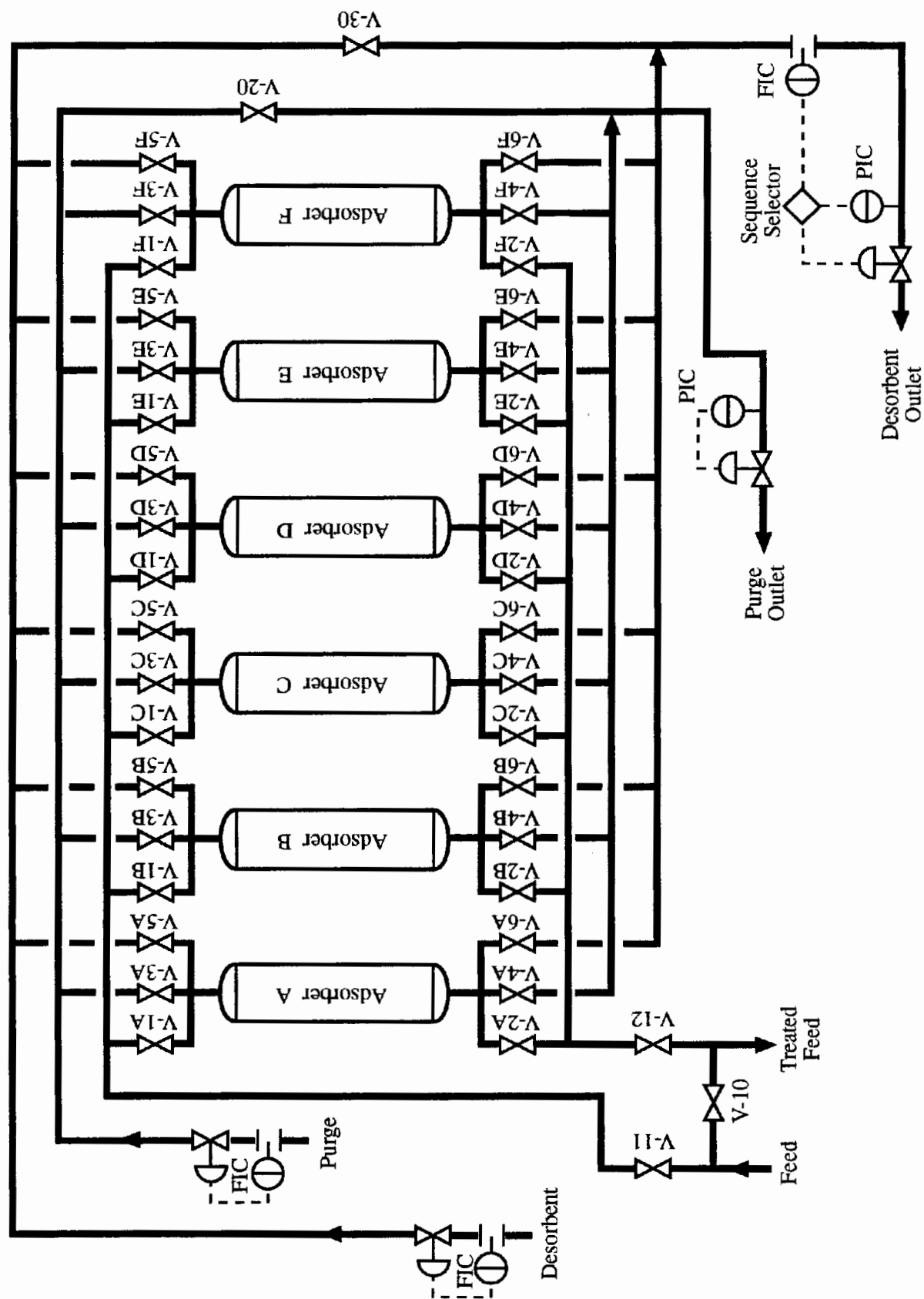
to Semi-automatic or Automatic, all of the valve positions must correspond to the current sequence step on the controller. Manual operation of the valves should not be possible when the controller is in the Automatic or Semi-automatic modes.

2. Each remote operated valve will have limit switches to indicate when the valve is fully open or fully closed. The logic 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 logic controller will have a valve verification and interlock system that monitors the valve limit switches and stops the sequence if a valve does not open or close properly. The interlock system will not allow the valves to change position unless all of the other valves are in the correct position. The controller will bypass flow around the adsorber section in some cases where the valve position does not properly verify, in order to prevent an unsafe condition. A valve transition timer is used to allow a given time (usually about 15 seconds) for the valves to open or close before a verification alarm occurs.
3. The logic controller will typically have 6 timers:
  - a) Phase I Timer: Controls the displacement step time in the Automatic mode. Typically set at about 5 minutes.
  - b) Phase II Timer: Controls the purge step time in the Automatic mode. Typically set at about 15 minutes.
  - c) Phase III Timer: Controls the idle and desorbent step time in the automatic mode. Typically set at about 20 minutes.
  - d) Valve Transition 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 15 seconds.

- e) Cycle Timer: Sets the time allowed for a subcycle to be completed. This timer is usually set one or two minutes more than the expected subcycle time. The expected subcycle time is typically about 40 minutes. If the subcycle is not completed within the timer setting, an alarm will be given.
  - f) System Total Cycle Timer: Sets the time allowed for an entire cycle to be completed. This timer is usually set a few minutes more than the expected total cycle time. The total expected cycle time is typically about 240 minutes. If the total cycle is not completed within the timer setting, an alarm will be given.
- 4. During Phase I, the controller will send a signal to the selector switch on the control valve at the outlet of the desorbent mixing drum so that the control valve position will be set by the flow rate controller. During Phase II & III, the control valve position will be set by the back pressure controller.
  - 5. The timers should stop timing, but not reset to zero, when the controller is put in the manual or semi-automatic mode. The timers should not reset to zero when there is a power failure either. This will allow the adsorbers to start back up at the same step in the sequence. The controller should not allow the sequence to automatically restart when power is restored following a loss of power.
  - 6. The logic controller should have a feed bypass switch that opens the feed bypass and closes the main valves on the feed inlet and treated feed outlet lines.

A schematic of a typical adsorber section and all of the remote operated valves is given in Figure IV-2. A typical valve sequence table is shown in Figure IV-3.

**Figure IV-2**  
**PEP Adsorption Section**



### Figure IV-3

| ADSORBER STEP STATUS        |                    |
|-----------------------------|--------------------|
| <input type="checkbox"/> A  | ADSORPTION         |
| <input type="checkbox"/> P  | PURGE              |
| <input type="checkbox"/> PH | PURGE HOLD         |
| <input type="checkbox"/> D  | DESORPTION         |
| <input type="checkbox"/> DF | DISPLACE FEED      |
| <input type="checkbox"/> DP | DISPLACE PURGE     |
| <input type="checkbox"/> DD | DISPLACE DESORBENT |

Valve closed

- Valve opens in this step
- Valve closes in this step
- Timer starts in this step
- = Timer finishes (times out)
- = Timer starts in this step and

## V. PROCESS EQUIPMENT

### A. VESSELS

#### 1. Adsorbers

There are typically 6 identical adsorber vessels in the PEP unit. The adsorbers (Figure V-1) are vertical vessels loaded with molecular sieve adsorbent. The adsorbent bed is supported by two layers of ceramic/alumina balls at the bottom of the vessel. The outlet distributor (profile wire screen) in the bottom of the vessel prevents adsorbent and broken support balls from getting into downstream equipment. A floating hold-down screen is installed on top of the adsorbent bed to prevent the hold-down balls from sinking down into the adsorbent bed. The ceramic/alumina hold-down balls on top of the adsorbent bed help prevent movement of the adsorbent particles and reduce the void space in the vessel. The inlet screen distributor helps to evenly distribute the flow in the adsorber. A manway is provided on the side of the vessel near the bottom of the bed to unload the adsorbent.

#### 2. Desorbent and Purge Mixing Drums

The two mixing drums (Figure V-2) are very similar. The purpose of the mixing drums is to smooth out variations in composition in the feed streams to the desorbent and depentanizer columns. Sudden variations in feed composition would upset the column operation. There are eductor nozzles on the inlet distributors inside the mixing drums which promote good mixing. The mixing drums run liquid full. Vent lines are provided on the top of the mixing drums to remove non-condensibles.

### **3. Desorbent Column and Overhead Receiver**

The purpose of the desorbent column is to remove the heavier aromatics from the desorbent effluent stream. Typically there is no pressure controller on the column. The overhead receiver is open to the relief header so the column pressure rides on the relief header pressure. Net overhead liquid product is taken from the overhead receiver on level control. Circulating hot oil is typically used as the heating medium for the reboiler.

### **4. Depentanizer Column and Overhead Receiver**

The purpose of the depentanizer column is to recover the n-pentane from the purge effluent and desorbent column net overhead. The column pressure is typically controlled by a hot vapor bypass around the overhead condenser. The overhead receiver acts as the surge vessel for the n-pentane in the unit. Circulating hot oil is typically used as the heating medium for the reboiler. The net column bottoms is typically sent to the alkylation unit.

## **B. DEPENTANIZER DRIER**

The depentanizer drier is typically a vendor-supplied package unit that uses automatically regenerated molecular sieve to remove water from the make-up n-pentane and from the small stream coming from the benzene column overhead receiver in the alkylation unit. The drier is also used during start-up to help dry out the unit.

## **C. EXCHANGERS**

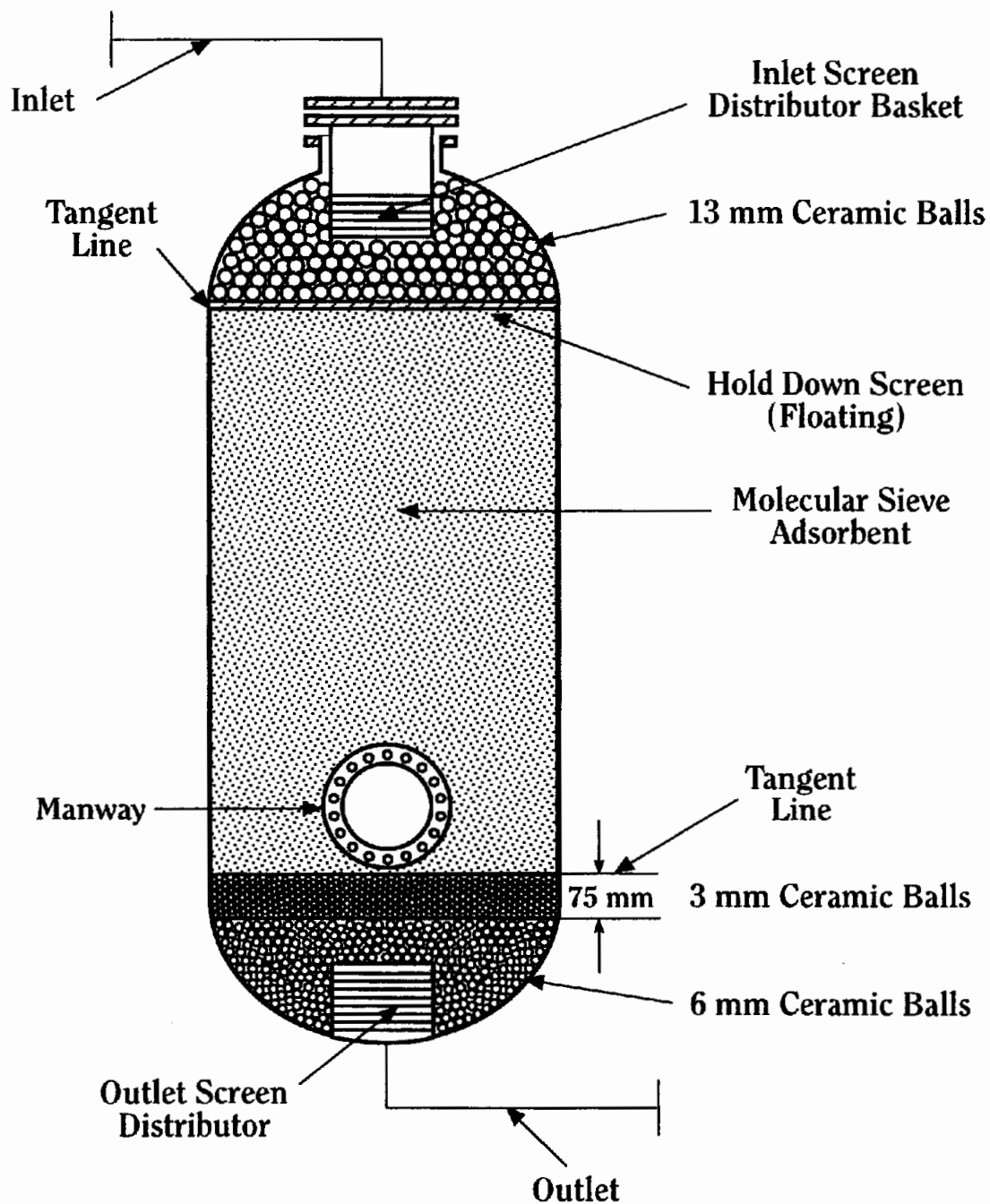
1. Feed/Adsorber Effluent Exchanger
2. Feed Cooler
3. Adsorber Effluent Exchanger
4. Feed/ Purge Heater

5. Desorbent Heater
6. Depentanizer Condenser
7. Depentanizer Reboiler
8. Desorbent Column Condenser
9. Desorbent Column Reboiler
10. Desorbent Column Bottoms Cooler

#### **D. PUMPS**

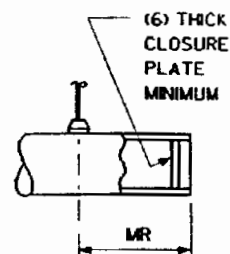
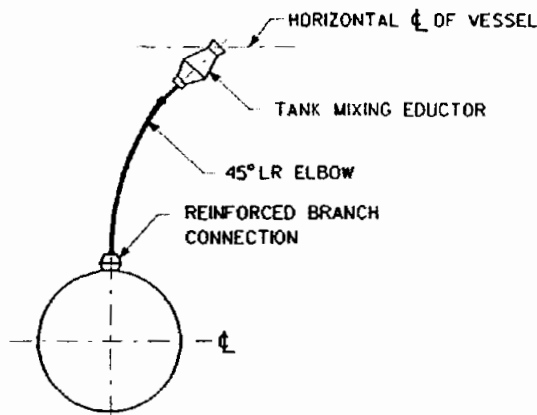
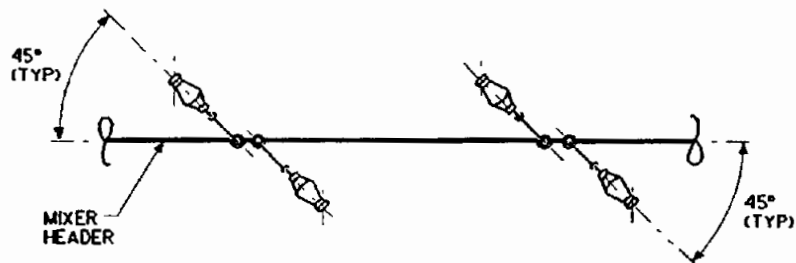
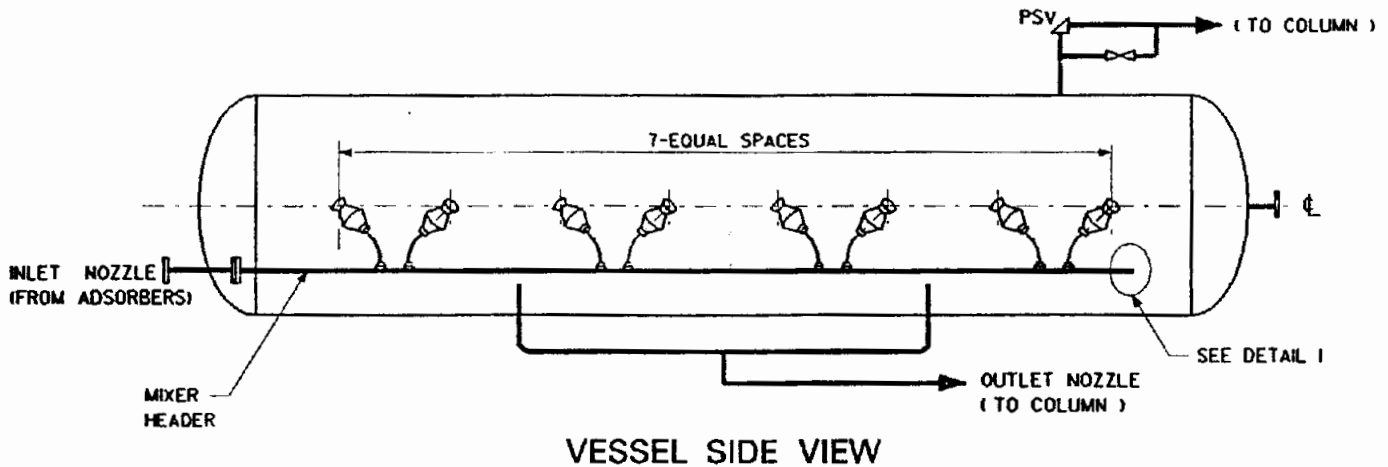
1. Desorbent Pumps
2. Depentanizer Bottoms Pumps
3. Depentanizer Reflux Pumps
4. Purge Pumps
5. Desorbent Column Bottoms Pumps
6. Desorbent Column Overhead Pumps

**Figure V-1**  
**Detail of a PEP Adsorber**



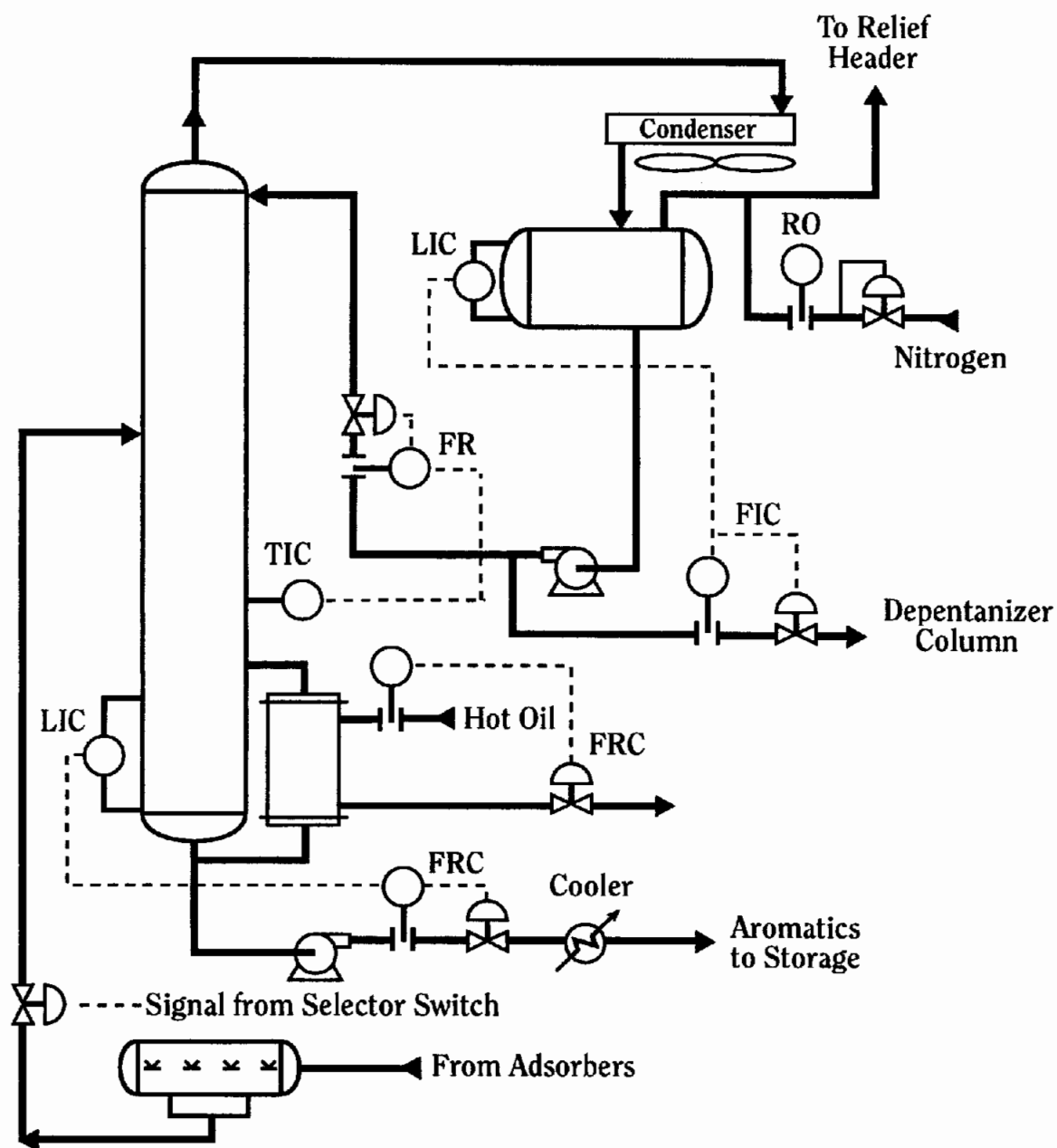
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**Figure V-2**  
**Desorbent and Purge Mix Drums**



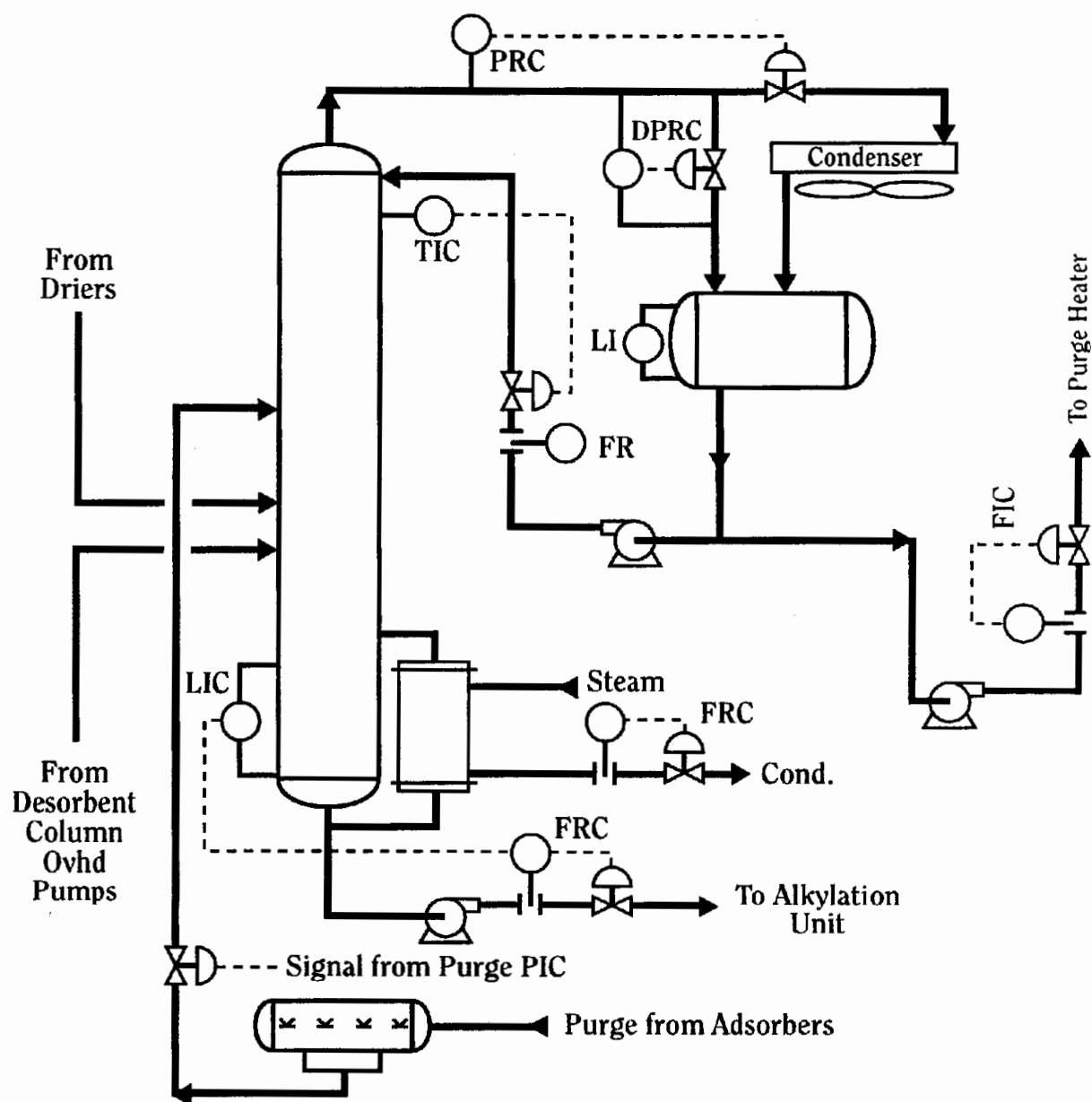
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*Figure V-3*  
*Desorbent Column*



UOP 2198-14

**Figure V-4**  
**Depentanizer Column**



UOP 21.1.1

## 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 load molecular sieve in the adsorbers.

- To dry out the unit (initial).

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. Testing of adsorber valve sequence controller.
9. 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.

The adsorber valve sequence controller should be fully checked out before startup. A loop check should be done on each remote operated valve (ROV) and the limit switches adjusted if necessary. The sequence should be checked to insure the valves follow the proper sequence.

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. VESSEL LOADING**

### **1. Adsorbers**

The adsorbers should be loaded after the initial line flushing (with water) of the unit is finished and before hydrocarbon is brought into the unit. The adsorber vessels should be clean and dry before the adsorbent is loaded. Also, any water in the piping around the adsorbers should be drained and the lines blown dry with air. This will help reduce the time required for dryout. The procedure for loading the adsorbers is given in Section XIII (Special Procedures).

## G. 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 60 psig (4 kg/cm<sup>2</sup>) 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.

## VII. NORMAL STARTUP

### A. 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. Steaming followed by nitrogen purge and blanketing is generally the technique recommended by UOP for air freeing a unit prior to startup. A properly performed steamout will help dry out the unit. However, the loaded adsorbers should not be steamed out because the molecular sieve will adsorb moisture from the steam. Nitrogen should be used to purge the adsorber section and the relief and flare system.

The easiest way to assure that all piping is air-free is to divide the unit into sections (off-sites, adsorbers, columns, etc.) and purge each part separately. Normally, mechanical flow diagrams will be posted in the control room so that all piping can be marked as the lines are purged and sampled for O<sub>2</sub>.

A final unit leak test should be performed after all hydrotesting blinds have been removed. The leak test can often be performed as part of the air freeing procedure.

Steaming or nitrogen purging are the most common methods to air free a system. The following procedure is recommended for air freeing the unit.

#### 1. Adsorption Section

The adsorption section should not be steamed out. The molecular sieve will adsorb water, requiring a longer dryout time. Nitrogen purging should be used to air free the adsorption section.

- a. Isolate the fractionation section from the adsorption section at the column feed control valves (downstream of the mixing drums).
- b. Introduce nitrogen into the adsorber vessels and lines and bring the system pressure up to 7 kg/cm<sup>2</sup> (100 psig) (or available nitrogen pressure). While the system is under full nitrogen pressure, soap test all flanges and screwed connections for leaks. (Note: Do not exceed design pressure limitations when air freeing vessels.)
- c. Depressure the vessels and lines to about 0.3 kg/cm<sup>2</sup> (5 psig). The system should be depressured on the opposite end from which it is filled in order to maximize the removal of oxygen. Vent the nitrogen at each available low and high point vent.
- d. Pressure-up and depressure with nitrogen two or three times.
- e. Obtain an analysis of the gas at the most downstream bleed points of each line and vessel being purged to determine the percentage of oxygen remaining in the system. If the oxygen content is greater than 0.5%, then repeat the nitrogen displacement procedure. If the oxygen content is less than 0.5%, the air freeing step may be considered complete.

Upon completion of the air freeing operation, leave all vessels and lines under a positive nitrogen pressure of 0.7 kg/cm<sup>2</sup> (10 psig).

All lines to and from offsite storage tanks and the unit battery limits must be air-freed. This includes the feed lines, desorbent line, n-pentane makeup lines, depentanizer column bottoms line to the Alkylation unit, adsorber effluent line to the Alkylation unit, and aromatic rundown lines. By using nitrogen, the lines can also be blown dry prior to bringing in hydrocarbon.

## 2. Fractionation Section

The fractionation section may be air freed by either steaming out or nitrogen purging. If a steam out procedure is used, be sure not to allow steam or condensate to enter the adsorption section because this would increase the time required to dry out the unit.

### 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 adsorbers 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/cm<sup>2</sup>g (30-45 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/cm<sup>2</sup>g (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 0.5%). If not, follow the nitrogen purging procedure below.
9. Start cooling water to all exchangers and condensers and turn on all fans. Use nitrogen to maintain 0.7 kg/cm<sup>2</sup> (10 psig) as the system cools to prevent creating a vacuum in the system.

### **Nitrogen Purging**

It is also acceptable to use nitrogen purging without steamout for air freeing the unit prior to startup; however, this may 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/cm<sup>2</sup>g (10 psig) and maintain this blanketing pressure until startup.

## B. UNIT DRYOUT AND STARTUP

Water is considered harmful to the PEP adsorbent. If the adsorbent is exposed to water, the adsorbent capacity will be reduced. Therefore the system should be dried out properly to remove any residual water left from the hydrotesting or line flushing operation.

This procedure is for the original plant startup. For subsequent startups and dryout operations, alterations to these procedures may be necessary. It is highly advisable to conduct the PEP unit dryout operation along with the Alkylation unit dryout.

Prior to hydrocarbon introduction the following items must be completed:

- The unit must be air free.
- All pressure relief safety valves must be installed and in service.
- All instruments must be installed and ready for operation.
- All pumps should be equipped with temporary fine mesh strainers. The fine mesh strainers should be removed when the dryout is completed.
- All utility systems must be in service (such as hot oil, steam, cooling water, etc.).
- The adsorbers should be loaded with the proper molecular sieve. The inlet and outlet block valves on all of the adsorbers should be closed.
- Start cooling water to all coolers and condensers and start fans on all air-cooled exchangers. In cold weather, make sure that water, benzene, or paraffin does not get cold enough to solidify or freeze.
- Check all low point drains for water.
- Make sure that dry benzene and paraffin is available from the upstream and downstream units.

During the dryout period, constant attention will be required by the operators because it will be harder to hold column levels and temperatures than when the unit is running normally.

Before starting the dryout, a final check of all low point drains (including piping, vessels, pumps, exchangers, and instruments) should be made to make sure there is no free water left in the unit. As the columns are being started up and the hydrocarbon circulation is established, the low point drains should be checked and the water drained frequently in places where water is likely to collect (such as overhead receivers and pumps).

During this dryout circulation, it is very likely that the pump suction strainers 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, as well as for any unusual noises or vibrations. Flush and cooling water must be in service to the pumps that require it.

The dryout and startup operations must be coordinated with the Pacol and alkylation unit operations. If possible, it is best to perform the PEP unit dryout along with the alkylation unit dryout.

The following procedure is for the original startup for a typical PEP unit. For subsequent startups, the procedures may change depending on the extent of the shutdown. The operating conditions of the upstream or downstream units could also require changes from this procedure.

1. Make sure all of the adsorber inlet and outlet manual block valves are closed (there is one inlet and one outlet manual block valve on each adsorber). Put the sequence controller in the "Manual" mode. Close all of the inlet and outlet remote operated valves (ROV's) for each adsorber (there are 6 ROV's on each adsorber). Open the purge and desorbent bypass ROV's. Make sure that the fresh feed and treated Pacolate ROV's are closed and the feed bypass ROV is open. The feed from the Pacol unit should be bypassing the adsorbers at this time. (Note: It may be necessary to put the valve interlock switch in the bypass (dryout) position for some of the dryout operations.)

2. Before starting the dryout circulation in the PEP unit, it is recommended to bypass the reactors in the alkylation unit in order to avoid possible water upsets. As the PEP unit dryout circulation is established, there is a chance that some water will be sent to the alkylation unit. Water upsets will cause the acid to become more corrosive in an HF alkylation unit and will cause catalyst deactivation in a Detal alkylation unit. (Note: Normally it will be necessary to bypass the Pacol reactor before bypassing the alkylation reactors.)
3. Start up the desorbent column:
  - a) Start a flow of benzene into the unit (typically from the alkylation unit) bypassing the adsorbers (through the desorbent bypass ROV), and into the desorbent mixing drum. Do not introduce benzene into any of the adsorbers yet.
  - b) When the mixing drum is liquid full, continue importing benzene and establish a liquid level in the bottom of the desorbent column. Make sure that the overhead condenser is running, then start heating the desorbent column reboiler. Do not allow the reboiler level to rise above the bottom tray because this could cause damage to the trays. It may be necessary to briefly interrupt the flow of benzene into the unit as the column is started up to avoid overfilling the reboiler.
  - c) When a liquid level is established in the overhead receiver, start the reflux pump and establish reflux to the column. Continue increasing the heat input to the reboiler and stabilize the column operation.
  - d) Continue to import benzene to the unit. As the desorbent column overhead receiver level increases, begin sending some net overhead to the depentanizer column.

4. Start up the depentanizer column and establish purge circulation:

- a) When a liquid level is established in the bottom of the depentanizer (due to accumulation of the feed from the desorbent column overhead), start heating the depentanizer reboiler. Do not allow the reboiler level to rise above the bottom tray because this could cause damage to the trays. It may be necessary to briefly interrupt the flow of benzene into the unit as the column is started up to avoid overfilling the reboiler.

(Note: Try to avoid sending depentanizer bottoms to the alkylation unit until the depentanizer column operation is stable; there could be some moisture in the depentanizer bottoms if the column is not running properly. As mentioned earlier, the alkylation reactors should be bypassed until the column operation is stable to avoid water upsets.)

- b) As the depentanizer is warming up, begin bringing in pentane from storage, through the pentane dryer, and into the depentanizer.

(Note: On some units, a startup nitrogen line is provided on the overhead receiver. This should only be used for air-freeing or to prevent flashing of pentane when it is first brought into the unit. The depentanizer overhead is a totally condensing system, and any nitrogen that is brought in will eventually have to be vented to flare to avoid over-pressuring.)

(Note: Be careful not to allow pentane to slump into the bottom of the column. Any pentane that is sent to the alkylation unit with the depentanizer net bottoms will probably be lost. The only chance to recover the pentane is by taking a benzene drag stream from the alkylation unit, through the pentane drier, and into the depentanizer.)

- c) When a liquid level is established in the overhead receiver, start the reflux pump and establish reflux to the column. Continue increasing the heat input to the reboiler and stabilize the column operation on total reflux.
- d) When reflux has been established and there is a good level of pentane in the overhead receiver, start the purge pumps and commission the pump spillback loop. Open the purge bypass ROV around the adsorbers and start a flow of pentane from the purge pumps to the purge mixing drum. Commission the purge mixing drum back pressure controller. Liquid fill the mixing drum by venting the non-condensibles to the depentanizer column. Do not introduce pentane into any of the adsorbers yet.
- e) As soon as the purge mixing drum is full, stop bringing pentane into the unit. Check the composition of the depentanizer overhead by laboratory analysis and only add more pentane if necessary. It is usually better to start with too little pentane than too much. If too much pentane is brought in, some of it will probably be lost from the bottom of the column.

(Note: Theoretically, it is possible to start up the depentanizer on total reflux with just the benzene from the desorbent column. This would help minimize pentane losses due to operational upsets that will likely occur during the startup of the column. However, it is recommended to bring in pentane as soon as the depentanizer is warmed up and reflux is established because: a) there is a big difference in specific gravity between n-pentane and benzene – this could cause over-amping of the depentanizer reflux and purge pumps as well as causing inaccurate reflux and purge flowmeter readings, b) the solubility of water is about 5 times greater in benzene than in n-pentane at the conditions in the depentanizer overhead receiver – this extra water would be carried back to the adsorbers, extending the dryout time required. Also, in cold weather, special care must be taken to avoid the solidification of benzene in the depentanizer overhead system because it has not been designed for benzene. Pentane must be brought in to the column before benzene if the ambient temperature is expected to drop below 5°C (40°F).)

5. When the depentanizer column operation is stable, and there is no pentane in the bottom of the column, establish a benzene circulation back to the alkylation unit. The benzene circulation will be as follows: benzene from alkylation unit => desorbent pump => desorbent bypass (around the adsorber section) => desorbent mixing drum => desorbent column => desorbent overhead receiver and pump => depentanizer column => depentanizer bottoms pump => benzene return to the alkylation unit.
6. During the dryout, when n-pentane is not being brought into the unit, a slipstream of depentanizer reflux should be directed through the makeup pentane drier. This will help remove the water which tends to concentrate in the depentanizer overhead. Whenever it is necessary to bring in more n-pentane from storage it will be necessary to temporarily stop the slipstream of depentanizer overhead to the drier. It is very important to regularly drain any free water that collects in the depentanizer overhead receiver and piping.
7. Fill the adsorbers with feed hydrocarbon. This may be done before, during, or after the desorbent and depentanizer columns are being started up.

A cool paraffin stream should be used for liquid-filling the adsorbers. This is especially important for the initial filling of an vessel loaded with fresh adsorbent. The exotherm (heat generation) when the fresh adsorbent first comes in contact with the hydrocarbon (or water) is much greater than during subsequent re-filling operations where the adsorbent has been previously exposed to hydrocarbon or water vapors.

Benzene should NOT be used to liquid fill the adsorbers because the heat of adsorption for benzene is much greater than for paraffin. This could cause excessive adsorber temperatures if benzene is used for the initial liquid filling.

The Pacol reactors must be bypassed for the PEP adsorber hydrocarbon filling procedure. If there is more than 5% olefin in the hydrocarbon used to fill the adsorbers, some polymerization of the olefin could occur as a result of the heat of adsorption, causing damage to the sieve. It is recommended to keep the olefin content of the hydrocarbon as low as possible during the liquid filling step.

The concentration of both benzene and olefins should be minimized in the liquid used for filling the adsorbers. The filling liquid will be acceptable if the olefin concentration is less than 5% and the benzene concentration is less than 0.5%. Alternatively, the filling liquid will be acceptable if the benzene concentration is less than 5% and the olefin concentration is less than 0.5%.

- a) Make sure that the adsorbers have been loaded, that the air has been removed from the adsorbers, and that each adsorber has a positive nitrogen pressure.
- b) If it has not already been done, establish paraffin circulation through the Pacol unit and alkylation unit according to the procedure given in the manuals for those units. Make sure that the Pacol reactor is bypassed and that there are no olefins in the circulating paraffin. Make sure that the ROV's for the feed to the adsorber section and the treated feed from the adsorber section are closed and the paraffin circulation is passing through the feed bypass ROV.
- c) Make sure all of the inlet and outlet ROV's for each adsorber are closed. Also make sure that the manual valves on the inlet and outlet of each adsorber are closed and that the manual valves on the feed to the adsorber section and the treated feed from the adsorber section are closed. Start the fans on the feed cooler. Then open the ROV on the feed to the adsorber section and slowly open the manual valve on the feed to the adsorber section. Next, open the valve in the adsorber feed manifold start up bypass line so that feed can pass through the feed cooler into the treated feed header on the adsorber outlets. On

units where the feed cooler is downstream of the bypass line, do not open the ROV or the manual valve on the treated feed outlet from the adsorber section. This will insure that hot feed from the bypass line cannot back into the adsorbers during the filling step.

- d) Open the ROV on the first adsorber treated feed outlet line to the treated feed header. Then crack open the manual valve on the first adsorber outlet line to allow hydrocarbon to start slowly flowing into the bottom of the adsorber. Do not open the bypass line around the adsorber relief valve yet. Monitor the temperature of the hydrocarbon flowing into the adsorber and try to keep the temperature below 65°C (150°F). Allow the adsorber pressure to increase and equalize with the incoming feed. Then open the gate valve in the adsorber relief valve bypass line and crack open the globe valve. Fill the adsorber slowly to avoid upsetting the bed. (It should take at least 60 minutes to fill the adsorber with liquid.) Continue filling until liquid appears in the sight flow indicator. Then close both valves in the adsorber relief valve bypass.
- e) Liquid fill the rest of the adsorbers in the same manner, one at a time. Be careful not to upset the adsorber beds with sudden surges of flow or by filling too quickly.

(Note: During the initial filling of the adsorbers, there is a significant exotherm as hydrocarbon is adsorbed on the sieve. Typically, the hydrocarbon is first adsorbed from the vapor phase just above the liquid level as the adsorber is being filled. At the point in the bed where hydrocarbon vapor is being adsorbed on the sieve above the liquid level, a significant temperature rise (as much as 100° to 150°C) occurs because there is no liquid present to absorb the heat being generated. However, as soon as the liquid level rises to that point, the bed is immediately cooled down to within about 10°C of the original temperature.)

The next step is to establish flow through the adsorbers for dryout. The best method for establishing flow through the adsorbers will depend on the operation of the other associated units (Pacol and alkylation) and what is contained in the adsorbers. The following procedure assumes the adsorbers are filled with paraffin and the Pacol and alkylation units are on paraffin circulation with the reactors bypassed. It is also assumed that the desorbent and purge circulations have been established through the adsorber section bypasses.

8. When all 6 adsorbers have been liquid filled with paraffin, establish paraffin circulation through 4 of the adsorbers.
  - a) Close the ROV's and the manual valves on the inlet and outlet of the last two adsorbers. Purge and desorbent flow will be established through these adsorbers later.
  - b) Open the feed ROV's at the inlet of the first four adsorbers. Then open the manual valves on the inlet to the first four adsorbers.
  - c) Check to be sure the manual valves on the outlet of the first 4 adsorbers are still open (from the filling step). Also make sure that the treated feed ROV's at the outlet of the first 4 adsorbers are still open.
  - d) Check to make sure that the main ROV and the manual valve on the feed to the adsorber section is open (if not, open them). Close the manual valve in the adsorber feed manifold start up bypass line. Then open the main ROV and the manual valve on the treated feed from the adsorber section.
  - e) Close the ROV in the adsorber section bypass line. This will force feed to flow through the first four adsorbers.
  - f) Adjust the adsorber feed temperature to the design value.

9. Establish purge circulation through one adsorber and desorbent circulation through one adsorber. This may be done either before or after the paraffin circulation is established through the other four adsorbers.

The most important thing during this step is to prevent the loss of n-pentane. Care must be taken not to allow any pentane to slip into the treated feed outlet header or the bottoms of either the depentanizer or the desorbent column.

It is usually desirable to avoid paraffin losses. Paraffin would be lost if it goes into the desorbent outlet header and is fed to the desorbent column. Therefore, it is recommended that the paraffin in the adsorber (from the liquid filling step) be displaced into the depentanizer column before lining the adsorber up to the desorbent outlet header. The following procedure includes such a displacement step.

- a) Open the purge inlet and outlet ROV's on the last adsorber. Then open the manual valves (first the inlet, then the outlet) on that adsorber.
- b) Close the purge bypass ROV. This will force the purge flow through the last adsorber.
- c) Continue the purge flow through the last adsorber long enough to displace the paraffin out of the adsorber. Then open the purge bypass ROV and close the purge inlet and outlet ROV's on the last adsorber.
- d) Open the desorbent inlet ROV on the last adsorber, then open the desorbent outlet ROV on the last adsorber. Then close the desorbent bypass ROV to force the desorbent flow through the last adsorber. Bring the adsorber up to the design temperature.

- e) Open the purge inlet and outlet ROV's on the fifth adsorber. Then open the manual valves (first the inlet, then the outlet) on that adsorber.
  - f) Close the purge bypass ROV. This will force the purge flow through the fifth adsorber. Bring the adsorber up to the design temperature.
10. At this point, four of the adsorbers should be on the adsorption step, one on the purge step, and one on the desorption step. Keeping the valve sequence controller in the manual mode, adjust the step counter to the step which matches with the current position of the adsorber valves. Then switch the valve sequence controller into the semi-automatic mode. (Note: If the controller will not go into the semi-automatic mode, make sure the step counter has been set to the correct step and that all the ROV's are in the correct position. The sequence controller will not pass from manual to semi-automatic mode unless all the valves match the current step on the valve sequence controller.)
11. Switch the valve interlock bypass switch (dryout switch) to the "normal" position so that the valve interlock system is operational. Check all of the timer settings to make sure they are set properly. For the initial startup, they should be set at the normal value listed in the equipment specification for the valve sequence controller.
12. When the column operation and adsorber temperatures have stabilized, switch the valve sequence controller to the automatic mode. Put the run/stop switch in the run position. The adsorbers should now be running through the normal sequence.

13. 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.
  - f) Monitor moisture analyzer readings.
14. Continue the circulation (with the Pacol and alkylation unit reactors bypassed) until the treated feed product is dry. If the downstream alkylation unit is a Detal unit, the PEP unit dryout should continue until the moisture analyzer shows less than 5 wppm water in the circulating paraffin from the PEP unit, and the moisture readings have lined out. If the downstream alkylation unit is an HF unit, the PEP unit may be considered dry when there is no free water accumulation at any of the low points in the PEP unit.
15. After the PEP unit has been dried out, the Pacol and alkylation reactors can be started up when those units are ready.

### **Alternate Dryout Procedure**

In the case where the Pacol and alkylation units have been started up and are already running, it may be desirable to dry out the PEP unit without keeping the Pacol and alkylation reactors bypassed. In this case it is still strongly recommended that the Pacol and alkylation reactors be bypassed when the PEP columns are first started up and when the adsorbers are liquid filled. However, it is possible to dry out the adsorbers with just the desorbent flow while the Pacol and alkylation units are running. The following is a procedure for doing this.

Steps 1 through 7 should be performed as previously described. At this point it is assumed that the adsorbers are liquid filled with paraffin and the Pacol and alkylation units are on paraffin circulation with the reactors bypassed. It is also

assumed that the desorbent and purge circulations have been established through the adsorber section bypasses.

8. Close all of the inlet and outlet ROV's on each adsorber. Open the feed bypass ROV around the adsorber section and close the fresh feed and treated Pacolate ROV's. Also close the manual valves on the fresh feed and treated Pacolate lines to and from the adsorber section to prevent any leakage of wet hydrocarbon out of the PEP unit. The Pacol and alkylation reactors can then be re-started according to the normal procedure for those units.

It is important to prevent the loss of n-pentane. Care must be taken not to allow any pentane to slip into the treated feed outlet header or the bottoms of either the depentanizer or the desorbent column.

It is usually desirable to avoid paraffin losses. Paraffin would be lost if it goes into the desorbent outlet header and is fed to the desorbent column. Therefore, it is recommended that the paraffin in the adsorber (from the liquid filling step) be displaced into the depentanizer column before lining the adsorber up to the desorbent outlet header. Typically the best way to do this is to use the purge stream to displace the paraffin to the depentanizer column.

The purge stream comes from the depentanizer overhead liquid, which will probably contain a significant concentration of dissolved water early in the startup. The purge stream should not be used to displace the paraffin from the adsorbers until there is no accumulation of free water in the depentanizer overhead receiver or pump suction. As mentioned previously, a slipstream of depentanizer reflux should be directed through the makeup pentane drier whenever possible to help dryout the depentanizer overhead receiver.

9.
  - a) Open the purge inlet and outlet ROV's on the first adsorber. Then open the manual valves (first the inlet, then the outlet) on that adsorber.
  - b) Close the purge bypass ROV. This will force the purge flow through the first adsorber.
  - c) Continue the purge flow through the first adsorber long enough to displace the paraffin out of the adsorber. Then open the purge bypass ROV and close the purge inlet and outlet ROV's on the first adsorber.
10.
  - a) Open the desorbent inlet ROV on the first adsorber, then open the desorbent outlet ROV on the first adsorber. Then close the desorbent bypass ROV to force the desorbent flow through the first adsorber. Bring the adsorber up to the design temperature.
  - b) Dry out the first adsorber with desorbent in this manner until there is no accumulation of free water in the overhead of either the desorbent or depentanizer columns. If a moisture analyzer is available, the dryout should continue until the adsorber outlet is less than 5 ppm water (if the alkylation unit is a Detal unit). This dryout flow should be maintained for a minimum of 12 hours.
  - c) When the adsorber is considered dry, open the desorbent bypass ROV and close the desorbent inlet ROV and desorbent outlet ROV on the first adsorber.
11. Repeat steps 9 and 10 for each of the other adsorbers, one at a time. As the adsorbers are being dried out, perform the 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.
  - f) If a portable moisture analyzer is available, monitor the moisture analyzer readings.

12. For the first four adsorbers, after step 10 is completed, displace the desorbent from the adsorber with paraffin/olefin feed as described below. This must be done one adsorber at a time.
  - a) Close the manual valve at the outlet of the adsorber. Then open the ROV and the manual valve on the fresh feed to the PEP unit. Next, open the feed inlet and desorbent outlet ROV's on the adsorber.
  - b) Carefully open the manual valve at the outlet of the adsorber and begin displacing the desorbent to the desorbent column. Use the flowmeter on the desorbent outlet header to determine the displacement flow rate and adjust the flow rate by throttling the manual valve at the outlet of the adsorber. The flow rate should be adjusted so that the operation of the Pacol and alkylation units is not upset.
  - c) Stop the displacement flow ( by closing the manual valve at the outlet of the adsorber) as soon as the quantity of flow is sufficient to displace the desorbent from the void space in the adsorber. Excessive displacement flow will cause fresh feed to be lost from the bottom of the desorbent column.
  - d) Close the feed inlet and desorbent outlet ROV's on the adsorber. Then close the ROV and the manual valve on the fresh feed to the PEP unit.
13. For the fifth adsorber, after step 10 is completed, displace the desorbent from the adsorber with pentane purge and continue the purge flow through the adsorber as follows:
  - a) Open the purge inlet and outlet ROV's on the adsorber. Make sure that the manual valves on the adsorber inlet and outlet are open.
  - b) Close the purge bypass ROV. This will force the purge flow through the fifth adsorber.
  - c) Adjust the purge flow to the design rate and temperature.

14. For the sixth adsorber, do not open the desorbent bypass ROV and do not close the desorbent inlet ROV or desorbent outlet ROV as described in part c) of step 10. Keep the desorbent flowing at the design rate and temperature through the sixth adsorber.
15. For units where the downstream alkylation unit is a Detal unit, it is highly recommended that the Pacol and Detal reactors be bypassed at this point. It is possible that a water upset could occur as the PEP adsorbers are brought on line due to a pocket of water that was missed during dryout. For units where the downstream alkylation unit is an HF unit, it is probably not necessary to bypass the Pacol or HF Detergent Alkylation reactors because the amount of water that could come off the PEP unit at this point should not be enough to cause significant problems in the HF Alkylation unit.

(Note: If the Pacol reactor was operating during the desorbent displacement described in step 12, there will be olefins in the adsorbers. This will be important if the Pacol and alkylation reactors are shut down when the PEP adsorbers are brought on line because olefins will get into the circulating paraffins. This must be considered when re-starting the Pacol and alkylation reactors.)

16.
  - a) Open the feed ROV's at the inlet to the first four adsorbers. Also open the treated feed ROV's at the outlet of the first four adsorbers. Make sure the manual valves at the inlet and outlet of the adsorbers are all open.
  - b) Open the manual valve and the ROV on the feed to the adsorber section. Then open the manual valve and the ROV on the treated feed from the adsorber section.
  - c) Close the ROV in the adsorber section bypass line. This will force feed to flow through the first four adsorbers.
  - d) Adjust the adsorber feed temperature to the design value.

17. At this point, four of the adsorbers should be on the adsorption step, one on the purge step, and one on the desorption step. Keeping the valve sequence controller in the manual mode, adjust the step counter to the step which matches with the current position of the adsorber valves. Then switch the valve sequence controller into the semi-automatic mode. (Note: If the controller will not go into the semi-automatic mode, make sure the step counter has been set to the correct step and that all the ROV's are in the correct position. The sequence controller will not pass from manual to semi-automatic mode unless all the valves match the current step on the valve sequence controller.)
18. Switch the valve interlock bypass switch (dryout switch) to the "normal" position so that the valve interlock system is operational. Check all of the timer settings to make sure they are set properly. For the initial startup, they should be set at the normal value listed in the equipment specification for the valve sequence controller.
19. When the column operation and adsorber temperatures have stabilized, switch the valve sequence controller to the automatic mode. Put the run/stop switch in the run position. The adsorbers should now be running through the normal sequence.
20. If the Pacol and alkylation unit reactors were bypassed, continue the circulation until the treated feed product is dry. If the downstream alkylation unit is a Detal unit, the PEP unit dryout should continue until the moisture analyzer shows less than 5 wppm water in the circulating paraffin from the PEP unit, and the moisture readings have lined out. If the downstream alkylation unit is an HF unit, the PEP unit may be considered dry when there is no free water accumulation at any of the low points in the PEP unit.
21. After the PEP unit has been confirmed to be dry, the Pacol and alkylation reactors can be started up when those units are ready.

### C. STARTUP WITHOUT DRYOUT

After the initial dryout and startup of the unit, it should not be necessary to perform an adsorber dryout each time the unit is started up or shut down. As long as water is kept out of the adsorbers, a dryout is not necessary. However, there is a possibility for the columns to accumulate some moisture during a shutdown so they should be started up and the depentanizer overhead should be dried out using the makeup pentane drier before starting up the adsorber section.

If air has been allowed to enter the adsorbers, or if new adsorbent has been loaded, it will be necessary to perform a dryout. If the adsorbers were kept under a dry nitrogen positive pressure or kept full of hydrocarbon, a dryout should not be necessary.

The following is a procedure for starting up the unit after a shutdown in which the hydrocarbon has been removed from the unit and the adsorbers were kept under a dry nitrogen positive pressure. This procedure does not cover all possible shutdown cases, but it does serve as a good general guideline for restarting the unit after a shutdown. Depending on the extent of the shutdown, all of the steps may not be necessary. (For example; if the adsorbers were kept full of hydrocarbon, the liquid filling step would not be necessary.)

1. If air was allowed into the unit, the proper air freeing operations must be carried out as described earlier in this section.
2. All utilities must be in service. All equipment should be ready for operation. Start cooling water to all coolers and condensers and start fans on all air-cooled exchangers. In cold weather, make sure that water, benzene, or paraffin does not get cold enough to solidify or freeze. Check all low point drains for water.
3. Make sure all of the adsorber inlet and outlet manual block valves are closed (there is one inlet and one outlet manual block valve on each adsorber). Put the sequence controller in the "Manual" mode. Close all of the inlet and

outlet remote operated valves (ROV's) for each adsorber (there are 6 ROV's on each adsorber). Open the purge and desorbent bypass ROV's. Make sure that the fresh feed and treated Pacolate ROV's are closed and the feed bypass ROV is open. The feed from the Pacol unit should be bypassing the adsorbers at this time. (Note: It may be necessary to put the valve interlock switch in the bypass (dryout) position for some of the startup operations.)

4. Start up the desorbent column:

- a) Start a flow of benzene into the unit (typically from the alkylation unit) bypassing the adsorbers (through the desorbent bypass ROV), and into the desorbent mixing drum.
- b) When the mixing drum is liquid full, continue importing benzene and establish a liquid level in the bottom of the desorbent column. Make sure that the overhead condenser is running, then start heating the desorbent column reboiler. Do not allow the reboiler level to rise above the bottom tray because this could cause damage to the trays. It may be necessary to briefly interrupt the flow of benzene into the unit as the column is started up to avoid overfilling the reboiler.
- c) When a liquid level is established in the overhead receiver, start the reflux pump and establish reflux to the column. Continue increasing the heat input to the reboiler and stabilize the column operation.
- d) Continue to import benzene to the unit. As the desorbent column overhead receiver level increases, begin sending some net overhead to the depentanizer column.

5. Start up the depentanizer column and establish purge circulation:

- a) When a liquid level is established in the bottom of the depentanizer (due to accumulation of the feed from the desorbent column overhead), start heating the depentanizer reboiler. Do not allow the reboiler level to

rise above the bottom tray because this could cause damage to the trays. It may be necessary to briefly interrupt the flow of benzene into the unit as the column is started up to avoid overfilling the reboiler.

(Note: Try to avoid sending depentanizer bottoms to the alkylation unit until the depentanizer column operation is stable; there could be some moisture in the depentanizer bottoms if the column is not running properly.)

- b) As the depentanizer is warming up, begin bringing in pentane from storage, through the pentane dryer, and into the depentanizer.

(Note: Be careful not to allow pentane to slump into the bottom of the column. Any pentane that is sent to the alkylation unit with the depentanizer net bottoms will probably be lost. The only chance to recover the pentane is by taking a benzene drag stream from the alkylation unit, through the pentane drier, and into the depentanizer.)

- c) When a liquid level is established in the overhead receiver, start the reflux pump and establish reflux to the column. Continue increasing the heat input to the reboiler and stabilize the column operation on total reflux.
- d) When reflux has been established and there is a good level of pentane in the overhead receiver, start the purge pumps and commission the pump spillback loop. Open the purge bypass ROV around the adsorbers and start a flow of pentane from the purge pumps to the purge mixing drum. Commission the purge mixing drum back pressure controller. Liquid fill the mixing drum by venting the non-condensibles to the depentanizer column.

- e) As soon as the purge mixing drum is full, stop bringing pentane into the unit. Check the composition of the depentanizer overhead by laboratory analysis and only add more pentane if necessary. It is usually better to start with too little pentane than too much. If too much pentane is brought in, some of it will probably be lost from the bottom of the column.
6. When the depentanizer column operation is stable, and there is no pentane in the bottom of the column, establish a benzene circulation back to the alkylation unit. The benzene circulation will be as follows: benzene from alkylation unit => desorbent pump => desorbent bypass (around the adsorber section) => desorbent mixing drum => desorbent column => desorbent overhead receiver and pump => depentanizer column => depentanizer bottoms pump => benzene return to the alkylation unit.
7. During the startup, when n-pentane is not being brought into the unit, a slipstream of depentanizer reflux should be directed through the makeup pentane drier. This will help remove the water which tends to concentrate in the depentanizer overhead. Whenever it is necessary to bring in more n-pentane from storage it will be necessary to temporarily stop the slipstream of depentanizer overhead to the drier. It is very important to regularly drain any free water that collects in the depentanizer overhead receiver and piping.
8. Fill the first four adsorbers with feed hydrocarbon. This may be done before, during, or after the desorbent and depentanizer columns are being started up.
- a) Make sure that the adsorbers have previously been loaded and wetted with hydrocarbon, and that each adsorber is under a positive nitrogen pressure.

(Note: It is essential that the adsorbent has previously been wetted with hydrocarbon. If this has been done, there will still be hydrocarbon in the pores of the adsorbent even if the hydrocarbon has been removed from the adsorbers and they are blanketed with nitrogen. With hydrocarbon already in the pores of the adsorbent, the exotherm during the filling step will be minimal and it will be possible to fill the adsorbers with paraffins that contain olefin or with benzene. If a hot nitrogen purge were used to remove the hydrocarbons from the adsorbers, there is a possibility that some of the hydrocarbon was removed from the pores of the adsorbent and it is recommended that only paraffin be used to liquid fill the adsorbers as previously described in the procedure for the initial startup of the unit.)

- b) If it has not already been done, establish paraffin circulation through the Pacol unit and alkylation unit according to the procedure given in the manuals for those units. The Pacol and alkylation reactors may be running at this point if so desired. Make sure that the ROV's for the feed to the adsorber section and the treated feed from the adsorber section are closed and the paraffin circulation is passing through the feed bypass ROV.
- c) Make sure all of the inlet and outlet ROV's for each adsorber are closed. Also make sure that the manual valves on the inlet and outlet of each adsorber are closed. Start the fans on the feed cooler. Then open the ROV on the feed to the adsorber section and slowly open the manual valve on the feed to the adsorber section. Next, open the valve in the adsorber feed manifold start up bypass line so that feed can pass through the feed cooler into the treated feed header on the adsorber outlets. On units where the feed cooler is downstream of the bypass line, do not open the ROV or the manual valve on the treated feed outlet from the adsorber section. This will insure that hot feed from the bypass line cannot back into the adsorbers during the filling step.

- d) Open the ROV on the first adsorber treated feed outlet line to the treated feed header. Then crack open the manual valve on the first adsorber outlet line to allow hydrocarbon to start slowly flowing into the bottom of the adsorber. Do not open the bypass line around the adsorber relief valve yet. Monitor the temperature of the hydrocarbon flowing into the adsorber and try to keep the temperature below 65°C (150°F). Allow the adsorber pressure to increase and equalize with the incoming feed. Then open the gate valve in the adsorber relief valve bypass line and crack open the globe valve. Fill the adsorber slowly to avoid upsetting the bed. (It should take at least 20 minutes to fill the adsorber with liquid.) Continue filling until liquid appears in the sight flow indicator. Then close both valves in the adsorber relief valve bypass.
  - e) Liquid fill the rest of the adsorbers in the same manner, one at a time. Be careful not to upset the adsorber beds with sudden surges of flow or by filling too quickly.
9. Fill the fifth adsorber with pentane and establish a purge flow through that adsorber.
- a) Make sure the manual block valves on the inlet and outlet of the fifth adsorber are closed. Next, open the purge outlet ROV on the fifth adsorber. Then crack open the manual valve on the fifth adsorber outlet line to allow pentane purge to start slowly flowing into the bottom of the adsorber. Do not open the bypass line around the adsorber relief valve yet. Monitor the temperature of the hydrocarbon flowing into the adsorber and try to keep the temperature as low as possible. Allow the adsorber pressure to increase and equalize with the incoming pentane purge. Then open the gate valve in the adsorber relief valve bypass line and crack open the globe valve. Fill the adsorber slowly to avoid upsetting the bed. (It should take at least 20 minutes to fill the adsorber with liquid.) Continue filling until liquid appears in the sight flow indicator. Then close both valves in the adsorber relief valve bypass.

- b) Open the purge inlet ROV on the fifth adsorber and make sure the purge outlet ROV is still open. Then open the manual valves (first the inlet, then the outlet) on that adsorber.
  - c) Close the purge bypass ROV. This will force the purge flow through the fifth adsorber. Bring the adsorber up to the design temperature.
10. Fill the sixth adsorber with benzene and establish a desorbent flow through that adsorber.
- a) Make sure the manual block valves on the inlet and outlet of the sixth adsorber are closed. Next, open the desorbent outlet ROV on the sixth adsorber. Then crack open the manual valve on the sixth adsorber outlet line to allow desorbent (benzene) to start slowly flowing into the bottom of the adsorber. Do not open the bypass line around the adsorber relief valve yet. Monitor the temperature of the hydrocarbon flowing into the adsorber and try to keep the temperature as low as possible. Allow the adsorber pressure to increase and equalize with the incoming desorbent. Then open the gate valve in the adsorber relief valve bypass line and crack open the globe valve. Fill the adsorber slowly to avoid upsetting the bed. (It should take at least 20 minutes to fill the adsorber with liquid.) Continue filling until liquid appears in the sight flow indicator. Then close both valves in the adsorber relief valve bypass.
  - b) Open the desorbent inlet ROV on the sixth adsorber and make sure the desorbent outlet ROV is still open. Then open the manual valves (first the inlet, then the outlet) on that adsorber.
  - c) Close the desorbent bypass ROV. This will force the desorbent flow through the sixth adsorber. Bring the adsorber up to the design temperature.

11. Establish paraffin circulation through the first four adsorbers.
  - a) Open the feed ROV's at the inlet of the first four adsorbers. Then open the manual valves on the inlet to the first four adsorbers.
  - b) Check to be sure the manual valves on the outlet of the first 4 adsorbers are still open (from the filling step). Also make sure that the treated feed ROV's at the outlet of the first 4 adsorbers are still open.
  - c) Check to make sure that the ROV and the manual valve on the feed to the adsorber section is open (if not, open them). Close the manual valve in the adsorber feed manifold start up bypass line. Then open the ROV and the manual valve on the treated feed from the adsorber section.
  - d) Close the ROV in the adsorber section bypass line. This will force feed to flow through the first four adsorbers.
  - e) Adjust the adsorber feed temperature to the design value.
12. At this point, four of the adsorbers should be on the adsorption step, one on the purge step, and one on the desorption step. Keeping the valve sequence controller in the manual mode, adjust the step counter to the step which matches with the current position of the adsorber valves. Then switch the valve sequence controller into the semi-automatic mode. (Note: If the controller will not go into the semi-automatic mode, make sure the step counter has been set to the correct step and that all the ROV's are in the correct position. The sequence controller will not pass from manual to semi-automatic mode unless all the valves match the current step on the valve sequence controller.)
13. Switch the valve interlock bypass switch (dryout switch) to the "normal" position so that the valve interlock system is operational. Check all of the timer settings to make sure they are set properly.

14. When the column operation and adsorber temperatures have stabilized, switch the valve sequence controller to the automatic mode. Put the run/stop switch in the run position. The adsorbers should now be running through the normal sequence.

## **D. COLD WEATHER OPERATIONS**

The freezing (or solidification) temperature for benzene is about 5°C (41°F) and for the paraffin feed stream it is about -15°C (5°F). Some special precautions are necessary during cold weather operation of the unit to prevent benzene or paraffin freeze-up.

### **1. Adsorbers**

In cold weather, benzene or paraffin could solidify inside the adsorber if the adsorber was left blocked in for an extended time with benzene or paraffin inside. This should be avoided because it will be very difficult to restart flow through the adsorber. Even if the flow can be restarted there is a high likelihood that the bed will be upset enough to cause flow distribution problems.

If the adsorbers must be shut down in cold weather, the hydrocarbon (especially benzene) should be removed and a positive nitrogen pressure should be kept on the adsorbers. If the temperature is expected to drop below the freezing point of benzene (5°C, 41°F), but not below the freezing point of the paraffin (-15°C, 5°F), the adsorbers could be kept full of paraffin. In this case, any benzene in the adsorbers should be displaced with paraffin.

### **2. Air Coolers**

In cold weather, the fans on the air cooled exchangers (such as the desorbent column overhead condenser) 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.

### **3. Pumps**

A special 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.

Some of the pumps in the unit may be equipped with warm-up lines. Normally the warm-up lines should be used in both hot and cold weather to gradually warm-up the hot service pumps before starting them. This will prevent excessive stress on the pump due to uneven thermal expansion of the metal parts. In cold weather, the warm-up lines can also serve to heat up the hydrocarbon in the pump so the viscosity is not too high when the pump is started. Heat tracing can also be used to help reduce the fluid viscosity before the spare pump is started.

### **4. Heat Tracing**

Heat tracing (either steam or electric) is used on many of the lines in the unit to prevent solidification of the benzene or paraffin. Special attention must be given to maintaining proper operation of the heat tracing in cold weather to insure safe operation of the unit. This is especially important on relief valve piping and some of the bypass lines.

In general, UOP only specifies heat tracing on non-flowing lines. Lines that always have flow during normal operation are not specified to be heat traced. Therefore, in a shutdown or other unusual operation, care must be taken in cold weather to prevent solidification of benzene or paraffin in the un-traced normally flowing lines. During shutdown, the usual procedure is to empty these lines when the flow is stopped.

## VIII. NORMAL OPERATIONS

### A. CALCULATIONS

#### 1. Adsorbent Life

The cumulative PEP adsorbent life is defined as metric tons of LAB product (from the Alkylation unit) divided by total kilograms of adsorbent loaded into all six of the adsorbers (MT LAB/kg adsorbent). The total cumulative amount of LAB that has been produced (starting with the date that the adsorbent was loaded) should be used. One metric ton is equal to 1000 kg.

#### 2. Aromatics Removal

The aromatics removal is defined as the percentage of the aromatics contained in the feed that are removed in the PEP unit. It is difficult to calculate this number exactly, because there is usually not a flow meter on both the feed and product lines to and from the PEP unit. Assuming the amount of aromatics in the feed is relatively small, the feed flow rate will be very nearly equal to the product flow rate. Therefore, in order to simplify the calculation, the aromatics removal will be defined as simply:

$$\frac{\text{wt\% Aromatics in Feed} - \text{wt\% Aromatics in Product}}{\text{wt\% Aromatics in Feed}} \times 100\%$$

#### 3. Feed Loss

The feed loss from the PEP unit is defined as the amount of paraffin and olefin in the feed to the PEP unit that does not end up going to the downstream alkylation unit. The only stream leaving the PEP unit that does not go to the downstream

alkylation unit is the desorbent column bottoms. Therefore, the feed loss should be calculated as follows:

$$\frac{(\% \text{ Paraffin \& Olefin in Desorbent Col. Btms}) \times (\text{Desorbent Col. Btms Flow Rate})}{\text{Feed Flow Rate}} \times 100\%$$

(Note: If the feed flow rate is not available, use the product flow rate.)

#### 4. Adsorbent Capacity

One way to monitor the performance of the adsorbent is to calculate the adsorbent capacity periodically based on flow rates and cycle times. The amount of material that comes off the adsorbent during one desorption cycle can be calculated by taking the desorbent column bottoms flow rate multiplied by the time that each adsorber is in the desorption step (which is the total time on the three PEP timers: Displacement, Purge, and Desorption). The equation looks like this:

$$\text{M3/Desorption} = Q \cdot (d/60 + P + D) / 60$$

where:

Q=Desorbent Btms Flow Rate (M3/hr)

d=displacement time (seconds)

P=Purge time (minutes)

D=Desorption time (minutes)

This method of calculating adsorbent capacity does not always give an accurate measurement of the adsorbent capacity. If the cycle time is longer than necessary, the adsorbent will not be completely loaded with aromatics, and the calculated capacity will be lower than the actual capacity. The M3/Desorption will typically increase during the course of a Pacol catalyst cycle due to the fact that the aromatics production at the start of the Pacol catalyst cycle is lower and so the PEP sieve will typically not be fully loaded at the beginning of the Pacol catalyst cycle. However, if the M3/Desorption is monitored over many Pacol catalyst cycles, it can give an indication of the capacity of the adsorbent over a long period of time and this can be used to determine if the adsorbent has been damaged or deactivated.

## **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 PEP unit. These sheets include the critical data that should be monitored on a typical PEP 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.

## PEP PROCESS DATA SUMMARY SHEET

Refiner: \_\_\_\_\_ Location: \_\_\_\_\_

DATE: \_\_\_\_\_

## Calculated Variables

|                           |           |
|---------------------------|-----------|
| Cumulative Adsorbent Life | MT LAB/kg |
| Aromatics Removal         | (wt%)     |
| Feed Loss from PEP Unit   | (wt%)     |

## Flow Rates

|                                    |                      |
|------------------------------------|----------------------|
| Paraffin/Olefin Feed to Adsorbers  | (M <sup>3</sup> /hr) |
| Desorbent to Adsorbers             | (M <sup>3</sup> /hr) |
| Purge to Adsorbers                 | (M <sup>3</sup> /hr) |
| Adsorb Effluent to Desorb Mix Drum | (M <sup>3</sup> /hr) |
| Benzene Drag to DeC5 Dryer         | (M <sup>3</sup> /hr) |
| Depentanizer Reflux                | (M <sup>3</sup> /hr) |
| Fresh Pentane from Storage         | (M <sup>3</sup> /hr) |
| Ovhd Liquid Recirc to Dryer        | (M <sup>3</sup> /hr) |
| Net Bottoms to Alky Unit           | (M <sup>3</sup> /hr) |
| Cond/Hot Oil from Reboiler         | (M <sup>3</sup> /hr) |
| Desorbent Column Reflux            | (M <sup>3</sup> /hr) |
| Net Overhead to Depentanizer       | (M <sup>3</sup> /hr) |
| Net Bottoms                        | (M <sup>3</sup> /hr) |
| Cond/Hot Oil from Reboiler         | (M <sup>3</sup> /hr) |

## Temperatures

|                                     |      |
|-------------------------------------|------|
| Paraffin/Olefin Feed to PEP Unit    | (°C) |
| Feed Cooler Outlet (to Adsorbers)   | (°C) |
| Adsorbent Effluent (to Detal/Alky)  | (°C) |
| Desorbent Htr Outlet (to Adsorbers) | (°C) |
| Purge Heater Outlet (to Adsorbers)  | (°C) |
| Depentanizer Ovhd Vapor             | (°C) |
| Ovhd Condenser Outlet               | (°C) |
| Tray _____                          | (°C) |
| Tray _____                          | (°C) |
| Bottoms Liquid                      | (°C) |
| Desorbent Column Ovhd Vapor         | (°C) |
| Ovhd Condenser Outlet               | (°C) |
| Tray _____                          | (°C) |
| Bottoms Liquid                      | (°C) |
| Bottoms Cooler Outlet               | (°C) |

## Pressures

|                                    |                       |
|------------------------------------|-----------------------|
| Adsorber Inlet (Adsorption Cycle)  | (Kg/cm <sup>2</sup> ) |
| Adsorber Inlet (Purge Cycle)       | (Kg/cm <sup>2</sup> ) |
| Adsorber Inlet (Desorption Cycle)  | (Kg/cm <sup>2</sup> ) |
| Purge Mixing Drum                  | (Kg/cm <sup>2</sup> ) |
| Desorbent Mixing Drum              | (Kg/cm <sup>2</sup> ) |
| Depentanizer Overhead              | (Kg/cm <sup>2</sup> ) |
| Depent Ovhd Hot Vap Bypass delta P | (Kg/cm <sup>2</sup> ) |
| Desorbent Column Overhead          | (Kg/cm <sup>2</sup> ) |

## Valve Cycle Timer Settings

|                              |       |
|------------------------------|-------|
| Phase I Timer (Displacement) | (sec) |
| Phase II Timer (Purge)       | (min) |
| Phase III Timer (Idle)       | (min) |

## PEP PROCESS DATA SUMMARY SHEET

Refiner: \_\_\_\_\_ Location: \_\_\_\_\_

DATE: \_\_\_\_\_

## Laboratory Analyses

Paraffin/Olefin Feed to PEP Unit: (Sp Gr)

Bromine Number

Total Normal Olefin (Wt%)

Total Normal Paraffin (Wt%)

Total Non-Normals (Wt%)

C7+ Aromatics (Wt%)

Treated Feed from PEP Unit: (Sp Gr)

Bromine Number

Total Normal Olefin (Wt%)

Total Normal Paraffin (Wt%)

Total Non-Normals (Wt%)

C7+ Aromatics (Wt%)

iC5 / nC5 / cyC5 (Wt%)

Benzene (Wt%)

Desorbent from Alky Unit: (Sp Gr)

iC5 / nC5 / cyC5 (Wt%)

Total C6 (iC6, nC6, cyC6) (Wt%)

Benzene (Wt%)

C7+ Paraffin (Wt%)

Desorbent Column Overhead: (Sp Gr)

iC5 / nC5 / cyC5 (Wt%)

Total C6 (iC6, nC6, cyC6) (Wt%)

Benzene (Wt%)

C7+ Paraffin/Aromatics (Wt%)

Desorbent Column Bottoms: (Sp Gr)

Bromine Number

C6- (Wt%)

Benzene (Wt%)

C7+ Paraffin (Wt%)

C7+ Aromatics (Wt%)

Depentanizer Overhead: (Sp Gr)

iC5 (Wt%)

nC5 (Wt%)

cyC5 (Wt%)

Total C6 (iC6, nC6, cyC6) (Wt%)

Benzene (Wt%)

C7+ Paraffin/Aromatics (Wt%)

Depentanizer Bottoms: (Sp Gr)

iC5 / nC5 / cyC5 (Wt%)

Total C6 (iC6, nC6, cyC6) (Wt%)

Benzene (Wt%)

C7+ Paraffin/Aromatics (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 PEP 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  $\frac{1}{4}$  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  $\frac{3}{4}$  inch gate valve should normally be closed except to take a sample. To put the circulation loop in operation:

1. Close the  $\frac{1}{4}$  inch valve.
2. Close the  $\frac{3}{4}$  inch globe valve.
3. Open the  $\frac{3}{4}$  inch gate valve.
4. Throttle the  $\frac{3}{4}$  inch globe valve to regulate the flow.

The  $\frac{3}{4}$  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  $\frac{1}{4}$  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/cm<sup>2</sup> (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

## PEP PROCESS UNIT

| Stream and Test                                                           | Test Method Number | Frequency |         |
|---------------------------------------------------------------------------|--------------------|-----------|---------|
|                                                                           |                    | Normal    | Startup |
| Depentanizer Bottoms<br>Composition                                       | UOP 621            | 1/D       | 1/D     |
| Depentanizer Overhead<br>Composition                                      | UOP 621 or UOP 551 | 1/D       | 1/D     |
| Desorbent Column Bottoms<br>Composition                                   | UOP 621            | 1/D       | 1/D     |
| Bromine Number                                                            | ASTM D 1159        | 1/D       | 1/D     |
| Desorbent Column Overhead<br>Composition                                  | UOP 621            | 1/D       | 1/D     |
| Fresh Desorbent from Detal Unit<br>Composition                            | UOP 621            | 1/D       | 1/D     |
| Pacol Product (Stripper Bottoms) to PEP<br>Composition (Normal Paraffins) | UOP 411            | 1/D       | 1/D     |
| Composition (Paraffins/Olefins)                                           | UOP 688            | 1/D       | 1/D     |
| Composition (Aromatics by HPLC)                                           | UOP 902 (Modified) | 1/D       | 1/D     |
| Composition (Carbon No. by MS)                                            | UOP 687            | Occas.    | Occas.  |
| Treated Pacolate Product from PEP<br>Bromine Number                       | ASTM D 1159        | Occas.    | Occas   |
| Composition (Normal Paraffins)                                            | UOP 411            | Occas.    | Occas   |
| Composition (Paraffins/Olefins)                                           | UOP 688            | Occas.    | Occas   |
| Composition (Aromatics by HPLC)                                           | UOP 902 (Modified) | 1/D       | 1/D     |
| Composition (Diolefins by HPLC)                                           | UOP 869 & UOP 902  | Occas.    | Occas.  |
| Sulfur                                                                    | UOP 727            | Occas.    | Occas   |

1/D: Once per day

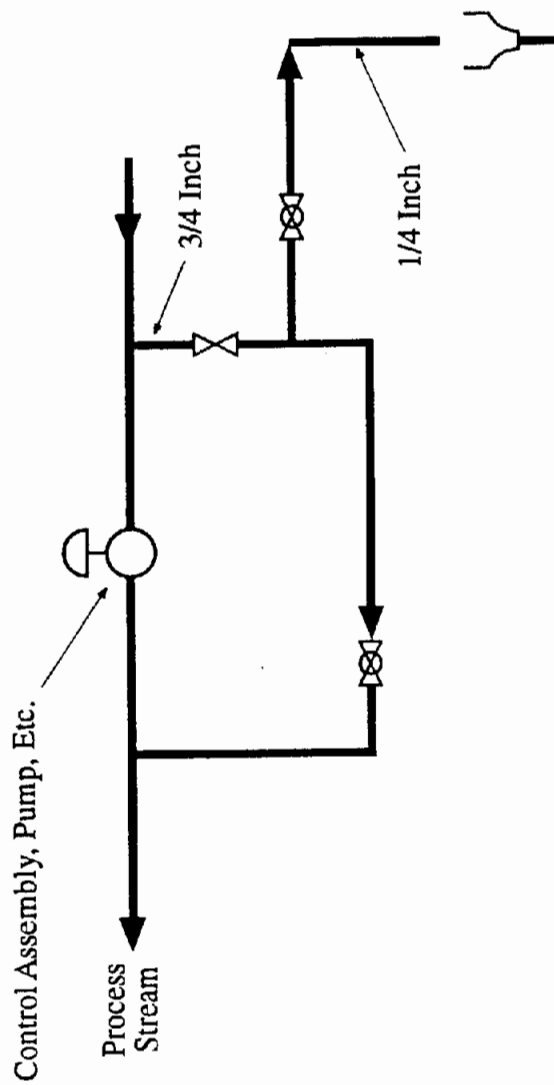
Occas. Occasionally

#### **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.

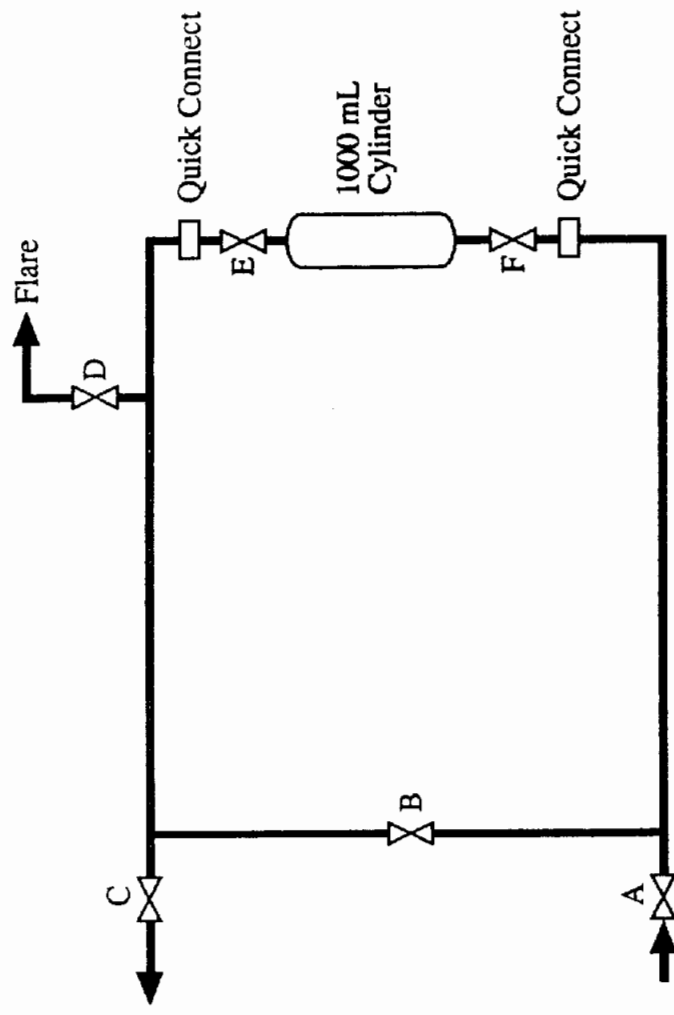
When taking samples for UOP analysis always label the samples with the refinery name, location, unit from which the sample was taken (e.g. PEP Unit), sample description, and the date sample was taken. Include any pertinent information that is useful to the laboratory and your UOP representative.

**Figure IX - 1**  
**Liquid Sample Connection**



DTL-R01-12

**Figure IX - 2**  
**Closed Flowing Sampling Apparatus**



DTL-R01-13

## X. TROUBLESHOOTING

Corrosion in the unit should be minimal due to the lack of corrosive chemicals or water in the unit.

Information on adjusting unit performance is included in Section III (Process Variables).

Due to the relatively new nature of the PEP process, there is not much operating experience from which a troubleshooting guide can be assembled. The following examples are some incidents that may be of interest.

1. Problem: Aromatics breaking through the PEP unit even though the aromatics concentration in the feed is normal and the cycle time is normal.

Discussion: Water on the adsorbent will reduce the capacity of the adsorbent to remove aromatics. Water can enter the unit in an upset, if the makeup pentane (benzene drag) driers are overloaded, or if the steam reboiler in the depentanizer column has a tube/tubesheet leak. The water tends to accumulate in the top of the depentanizer column, and continues to recycle the water back to the adsorbers via the purge stream. The makeup pentane driers should periodically be aligned to dry a slipstream of depentanizer overhead liquid in order to prevent water from building up in the purge stream.

2. Problem: Laboratory analysis shows aromatics in the PEP outlet even though the Bromine Number of the desorbent column bottoms is high.

Discussion: Typically, a high Bromine Number of the desorbent column bottoms stream indicates n-olefins remaining on the adsorbent at the end of the cycle because the adsorbent is not being fully loaded with aromatics. So, this is typically an indication that the cycle time is short enough to prevent

aromatics breakthrough. However, in this particular case, there was a problem in the paraffin column in the Detal unit that caused an excessive amount of LAB in the recycle paraffin returning to Pacol. When LAB passes through the Pacol reactor, some of the alkyl chains on the LAB will be dehydrogenated resulting in an olefin bond on the alkyl side chain on the LAB. This "olefinic LAB" will be adsorbed in the PEP unit because it is an aromatic molecule and will end up in the desorbent column bottoms along with the rest of the aromatics. However, the "olefinic LAB" also has an olefin bond that reacts with bromine, so it will increase the Bromine Number of the desorbent column bottoms, causing a misleading indication that the cycle time is too short (because it appears there are more n-olefins in the desorbent column bottoms). In this case, the cycle time can be reduced to give more capacity for aromatics removal until the problem with LAB in the recycle paraffin is fixed.

## XI. NORMAL SHUTDOWN

The procedures for any shutdown of the PEP 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 inspection. The following procedure is for a planned complete shutdown of the unit. The procedures may be modified or shortened for less extensive shutdowns.

This procedure will be written assuming the Pacol and alkylation units will continue to operate. If the Pacol and alkylation units will also be shut down, the Pacol reactor should be bypassed before the PEP unit is bypassed in order to remove the olefins from the PEP unit.

If the adsorbent is to be unloaded from the adsorbers, the adsorber temperature should be reduced to below 65°C (150°F) if possible, before bypassing flow around the adsorbers. The Pacol and alkylation reactors may have to be bypassed in order to prevent problems in the alkylation unit due to such a low PEP operating temperature.

1. Switch the sequence controller from the "Automatic" to the "Semi-Automatic" mode. Then switch the feed bypass from the "Normal" position to the "Bypass" position. This will open the adsorber section bypass ROV and close the ROV's on the feed to the adsorber section and the treated feed from the adsorber section. Immediately shut off the heat to the adsorber effluent heater (note: not all units have this heater). Block in the manual valves on the feed to the adsorber section and the treated feed from the adsorber section to prevent leakage of feed into the PEP unit. (Note: If the ambient temperature is less than 0°C, shut off the fan on the feed cooler. In cold ambient temperatures, the appropriate actions must be taken to prevent solidification of benzene and paraffin in the piping and equipment.)

2. Continue the desorbent flow through the adsorber that was on the desorption step for 40 minutes to remove the aromatics from the adsorbent. (Less than 40 minutes may be acceptable depending on how long the adsorber had been in the desorption step prior to the shutdown.) Then reduce or shut off the heat to the desorbent heater and the purge heater.

The purpose of continuing the desorbent flow is to prevent sending a slug of aromatics to the alkylation unit as the hydrocarbon is removed from the adsorber and to prepare the adsorber for the re-start. Failure to fully desorb the aromatics prior to shutdown should not damage the adsorbent in case it is not possible to continue the desorbent flow as described above.

3. Make a note of which adsorbers are in the adsorption, purge, and desorption steps. When the unit is re-started, it is recommended to put the adsorbers back into the same step that they were in before the shutdown.
4. Switch the sequence controller to the "manual" mode and open the desorbent bypass ROV and the purge bypass ROV. Then close all of the inlet and outlet ROV's on each adsorber.

Note: For extended shutdowns or if the ambient temperature will be low enough to solidify the benzene or paraffin contained in the adsorbers, the hydrocarbon should be removed from the adsorbers. Otherwise, the hydrocarbon can be left inside the adsorbers - this will not harm the adsorbent.

This shutdown procedure includes removal of the hydrocarbon from the adsorbers. If the hydrocarbon is to be left in the adsorbers, close the manual valves at the inlet and outlet of each adsorber at this point, and proceed to empty the mixing drums and shut down the columns according to the appropriate following shutdown instructions. In order to prevent lifting a relief valve due to thermal expansion of the liquid, some of the hydrocarbon should be drained out of each adsorber. When the liquid is left in the adsorbers, the adsorbers must be started back up in the same step as when they were shut down, or else there could be an upset (poor product quality, pentane losses, etc.) when the unit is re-started. It is important to maintain a

good record of which adsorbers were in the adsorption, purge, and desorption steps so the unit can be re-started smoothly.

5. Prepare the pentane pumpout line (from the depentanizer overhead reflux pump to the pentane storage tank) for service. Reduce the level of pentane in the depentanizer overhead receiver to a low level to make room for the pentane that will be displaced out of the adsorber that was on the purge step.
6. Make sure all of the inlet and outlet ROV's are closed on all of the adsorbers. Close the manual valve at the inlet of each adsorber. Keep the manual valve at the outlet of each adsorber open at this time.
7. Open the purge outlet ROV on each adsorber so that the outlet of all of the adsorbers are lined up to the purge mixing drum. Make sure the heat is shut off to the desorbent heater and the purge heater. Then slowly reduce the set point of the back pressure control valve on the outlet of the purge mixing drum. Gradually reduce the set point to 0 Kg/cm<sup>2</sup>. Then put the control valve on manual control and keep the valve 100% open.
8. If necessary, reduce the purge flow rate (which will reduce the back pressure upstream of the nozzles in the purge mix drum) so that the adsorber pressure is low enough so that nitrogen can be used to displace the hydrocarbon out of the adsorbers.
9. At this point, the pressure in all of the adsorbers should be equalized with the purge mixing drum at a pressure low enough to allow the use of nitrogen to displace the hydrocarbon out of the adsorbers. Close the manual valves on all of the adsorbers. This will prevent gravity flow of hydrocarbon (due to differential in static head) from one adsorber to another during the hydrocarbon displacement.

10. Nitrogen will be used to displace the hydrocarbon out of each adsorber, through the purge mixing drum, and into the depentanizer. Use the following procedure for each adsorber, working on one adsorber at a time. It is recommended that the adsorber that was on the purge step at the time the adsorbers were bypassed be displaced first so that the pentane is recovered and pumped out to storage before the other adsorbers are emptied. This will minimize the loss of pentane if there is a problem during the shutdown operation.
  - a) Using threaded or flanged steel piping, attach a check valve to the  $\frac{3}{4}$ " vent/drain connection just inside the manual block valve on the adsorber inlet. Make sure the nitrogen will be flowing into the top of the adsorber so that the hydrocarbon will be displaced out the bottom of the adsorber. (Note: The primary purpose of hard piping this check valve to the process line is to prevent the serious safety hazard of a large hydrocarbon leak in case the nitrogen hose ruptures. In addition, it will help prevent the contamination of the nitrogen header (with hydrocarbon) in case of a pressure reversal. This hard-piped check valve should be used even if there is already a check valve on the nitrogen supply header (which would only prevent contamination of the header). In any case there must be at least one check valve in the nitrogen line to prevent backing hydrocarbon into the nitrogen header.
  - b) Attach a temporary nitrogen hose to the check valve on the adsorber inlet  $\frac{3}{4}$ " connection described above.
  - c) Open the manual valve on the adsorber outlet.
  - d) Make sure the nitrogen pressure is higher than the adsorber pressure and the adsorber is lined up to the purge mixing drum. Then slowly open the nitrogen line and begin displacing the hydrocarbon out of the adsorber, through the purge mixing drum, and into the depentanizer.

- e) Continue to run the depentanizer as normally as possible during the displacement of hydrocarbon from the adsorbers to minimize the loss of pentane. As pentane accumulates in the overhead receiver, use the pentane pumpout line to send the excess pentane to the pentane storage tank. It is recommended to continue running the desorbent column as normally as possible during this time. The desorbent column overhead stream coming into the depentanizer will help stabilize the depentanizer column operation and reduce the chance of losing pentane due to poor fractionation.
  - f) When all of the hydrocarbon has been displaced out of the adsorber, close the purge outlet ROV and the manual valve at the outlet of the adsorber, then stop the flow of nitrogen into the adsorber.
  - g) Disconnect the nitrogen hose and check valve from the adsorber and leave the adsorber under a positive nitrogen pressure.
11. After the hydrocarbon has been displaced from all of the adsorbers, make sure all of the inlet and outlet ROV's are closed on all of the adsorbers. Also make sure all of the manual valves on the inlet and outlet of each adsorber are closed.
12. Make sure that the heat to the desorbent heater has been shut off. Gradually reduce and then stop the flow of desorbent into the PEP unit. Shut off the desorbent pump and block it in. Adjust the heat to the reboilers of the desorbent column and depentanizer column to keep them running.
13. Use nitrogen to pressure the contents of the desorbent mixing drum (and as much of the associated upstream piping as possible) into the desorbent column. The valves on the mixing drum non-condensable vent line (from the top of the mixing drum to the liquid outlet line) must be closed in order to displace the liquid out of the mixing drum. Adjust the back pressure control valve on the outlet of the desorbent mixing drum as necessary to reduce the mixing drum pressure below the nitrogen supply pressure. If the desorbent

mixing drum pressure drops below the vapor pressure of the hydrocarbon (benzene) in the mixing drum, some flashing may occur. Such flashing should not be a significant problem at this point. Continue to run the desorbent column so that the benzene is recovered overhead and sent to the depentanizer.

14. When the desorbent mixing drum is empty, close and block in the back pressure control valve at the outlet of the desorbent mixing drum. Decrease the level in the desorbent column overhead receiver as much as possible. Then shut off the heat to the desorbent column reboiler. Stop the reflux to the column and shut off the overhead pump. As the column cools, pump the bottoms to storage, then shut off the bottoms pump and block it in. If necessary, re-start the overhead pump long enough to pump any liquid that accumulates in the overhead receiver to the depentanizer. Then shut off and block in the overhead pump.
15. Make sure the heat to the purge heater has been shut off. If it has not already been done, stop the purge flow. Shut off the purge pump and block it in. Adjust the heat to the depentanizer reboiler to keep the column running so that pentane can be taken overhead.
16. Use nitrogen to pressure the contents of the purge mixing drum (and as much of the associated upstream piping as possible) into the depentanizer column. The valves on the mixing drum non-condensable vent line (from the top of the mixing drum to the liquid outlet line) must be closed in order to displace the liquid out of the mixing drum. Continue to run the depentanizer so that the pentane is recovered overhead. Send pentane from the overhead receiver to the storage tank as necessary.
17. When the purge mixing drum is empty, close and block in the back pressure control valve at the outlet of the purge mixing drum. Recover as much pentane as possible in the depentanizer overhead receiver and send it to the pentane storage tank through the pentane pumpout line. Decrease the level in the depentanizer overhead receiver as much as possible. Then shut off

the heat to the depentanizer reboiler. Stop the reflux to the column and shut off the overhead pump. As the column cools, pump the bottoms to the alkylation unit, then shut off the bottoms pump and block it in. If necessary, re-start the overhead pump long enough to pump out any liquid that accumulates in the overhead receiver. If the accumulated liquid is mostly pentane, pump it out to the pentane storage tank, if it is mostly benzene or heavier, pump it back into the column via the reflux line and then out the bottom of the column to the alkylation unit. Then shut off and block in the overhead and bottoms pumps.

## 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 into the design of the unit. While some of the emergencies listed in this section may not result in a 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, during any emergency, the operators should try to prevent losing or damaging the adsorbent while keeping the unit in a safe condition. During an emergency shutdown, care should be taken to ensure that no large pressure changes occur in the adsorbent beds that could result in lifting the beds or vaporizing the hydrocarbon in the adsorbers. Also, contamination of the adsorbent with water and the loss of pentane should be avoided if possible. Typically, the first response to an emergency should be to bypass and block in the adsorbers to protect them from pressure upsets. This can normally be done without upsetting the operation of the upstream or downstream units. Some other special considerations regarding the PEP unit in an emergency are:

1. The normal process temperature of some of the paraffin/olefin streams can be above the auto-ignition temperature.
2. The unit contains benzene, which is a known health hazard.

## **A. OPERATIONAL UPSETS**

### **1. Loss of Feed**

- a) Bypass the paraffin/olefin feed around the adsorber section. Switch the valve sequence controller to the "semi-automatic" mode to hold the sequence in the current step.
- b) Shut off the heat to the adsorber effluent heater. (Note: not all units have this heater.)
- c) Block in the adsorbers that were on the adsorption step.
- d) Maintain the desorption and purge flows if possible. The purge flow should bypass the adsorber to prevent desorption of the aromatics into the depentanizer column.
- e) Maintain the depentanizer and desorbent column operation.
- f) When feed flow is restored, the adsorption section may be restarted on the same step as when it was bypassed.

### **2. Loss of Desorbent**

If the desorbent was lost due to failure of the desorbent pump, try to restart the pump (or the spare) as quickly as possible and continue normal operation.

If the desorbent flow cannot be quickly restored:

- a) Shut off the desorbent pump. Shut off the heat to the desorbent heater.
- b) Switch the valve sequence controller to the "semi-automatic" mode to hold the sequence in the current step.

- c) Block in the adsorber on the desorption step.
- d) Maintain the feed flow through the adsorbers. Aromatics may load up and break through the adsorbers but this will not damage the adsorbent and will be no worse for the product quality than bypassing the feed around the adsorber section. Increase the Detal reactor temperature as necessary to compensate for the aromatics breaking through the PEP unit.
- e) Maintain the purge flow (bypassing the adsorber) if possible.
- f) Keep the desorbent column running on total reflux.
- g) When desorbent supply is restored, the adsorption section may be restarted on the same step as when it was bypassed.

### 3. Loss of Purge

If the purge flow was lost due to failure of the purge pump, try to restart the pump (or the spare) as quickly as possible and continue normal operation.

If the purge flow cannot be quickly restored:

- a) Shut off the heat to the purge heater.
- b) Switch the valve sequence controller to the "semi-automatic" mode to hold the sequence in the current step.
- c) Block in the adsorber on the purge step.
- d) Maintain the desorbent and feed flow to the adsorbers. Aromatics may load up and break through the adsorbers but this will not damage the adsorbent and will be no worse for the product quality than bypassing the feed around the adsorber section. Increase the Detal reactor temperature as necessary to compensate for the aromatics breaking through the PEP unit.

- e) Keep the depentanizer running to recover the pentane and send the desorbent back to the alkylation unit. Try to avoid losing pentane out the bottom of the depentanizer.
- f) When the purge flow can be restored, the adsorption section may be restarted on the same step as when it was bypassed.

## **B. 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 utility failure is to be given to protection of the catalyst in the upstream and downstream units as well as the adsorbent beds in the PEP unit.

Following is a list of possible utility failures:

### **1. Electrical Power Failure**

All main process pumps will normally be lost in an electrical power failure and a shutdown of the unit may be required. In addition, the loss of other equipment such as air cooler fans, electrical cooling water pumps, and electronic instruments will occur. The time required to restart the pumps and cooling fans will determine whether a full 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.

If a full shutdown is required:

- a) Bypass the adsorbers and block them in.

- b) Stop all heat input into the unit (shut off all reboilers, heaters, etc.).
- c) Try to prevent the loss of pentane from the depentanizer bottoms.
- d) Turn off all electrically driven pump and fan switches to prevent the unexpected re-start of the pumps and fans.

## **2. Cooling Water Failure**

For a partial loss of cooling water it may be possible to keep the unit running by simply reducing the heat input to the columns. It may also be possible to bypass the adsorbers and run the columns on total reflux, if the overhead condensers on the fractionation columns are air-cooled exchangers.

If there is a complete loss of cooling water to the unit, a full shutdown will probably be necessary. This is true even if there are no water-cooled exchangers in the PEP unit because cooling water is still required for pumps and seals, as well as the upstream and downstream units.

If a shutdown is required:

- a) Bypass the adsorbers and block them in.
- b) Shut down the fractionation columns in an accelerated manner, following the normal shutdown procedure as closely as possible. Try to minimize the loss of pentane.

## **3. 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 of 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 valve 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 unexpectedly restored.

If there is a total instrument air failure and the air pressure cannot be restored quickly, the unit should be shut down:

- a) Bypass the adsorbers and block them in.
- b) Shut down the fractionation columns in an accelerated manner, following the normal shutdown procedure as closely as possible.
- c) Make sure that the adsorbers are blocked in by closing the manual valves at the inlet and outlet of each adsorber.
- d) Make sure that all of the heaters and reboilers are shut off. 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.

#### **4. Heating Failure (Steam, Hot Oil System, etc.)**

If the hot oil heater or the steam boiler shuts down, heat input to the unit will be lost (or reduced depending on the extent of the failure). Often a heater or boiler

shutdown will be caused by some kind of upset such as loss of flow in the heater or boiler tubes. In this case, the heater or boiler can usually be restarted as soon as flow is restored through the tubes. Normally, a temporary hot oil heater shutdown can be handled without a complete shutdown because there is enough residual heat in the hot oil circuit to maintain heat input into the most critical services long enough to restart the heater.

If the heat cannot be restored quickly, the unit must be shut down:

- a) Bypass the adsorbers and block them in.
- b) Shut down the fractionation columns in an accelerated manner, following the normal shutdown procedure as closely as possible.
- c) Make sure to close the control valves on the heaters and reboilers in case the hot oil or steam is suddenly restored.

### **C. 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.

### **D. EXPLOSION, FIRE, OR LINE RUPTURE**

In the event of an 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 into the unit. (The paraffin/olefin feed can be stopped by bypassing the adsorbers).
2. Stop all heat input to the unit (such as hot oil and steam).
3. Stop all pumps if possible.
4. Block in the adsorbers and isolate the plant into sections. This should be done initially from the control room using control valves and ROV's, and then in the field using manual valves if possible.
5. Depressure and shut down the unit as circumstances permit.

## **E. ADSORBENT PROTECTION**

Protection of the PEP adsorbent, especially during an upset, is a special concern in the operation of the unit. The operators should be familiar with the potential causes of adsorbent damage or loss so that the proper steps can be taken quickly when a situation arises that could cause damage or loss of the adsorbent. Fortunately, there are not many things that are likely to damage the adsorbent.

### **1. Physical Upset of the Adsorbent Bed**

The biggest concern is to prevent upsetting the adsorbent bed especially in the event of a sudden pressure upset. An adsorbent bed can be physically upset by things like:

- a) A high flow rate passing upward through the adsorber
- b) Vaporization of the hydrocarbon in the adsorber due to a sudden decrease in pressure
- c) A large bubble of gas passing upward through an adsorbent bed filled with hydrocarbon.

Such upsets tend to fluidize the adsorbent bed and this leads to a variety of problems such as:

- a) Loss of adsorbent out of the adsorber. Not only does this cause a loss in adsorber capacity to remove aromatics, but the lost adsorbent will cause valve damage and plugging of downstream equipment.
- b) Poor flow distribution or channeling in the adsorber bed following the upset. This would effectively reduce the aromatics removal capacity of the adsorber.
- c) Increased pressure drop across the adsorbent bed following the upset. When the adsorbent is fluidized, the individual adsorbent particles tend to break up into smaller pieces as they crash into each other. The smaller particle size will cause a higher pressure drop.
- d) If the hold down screen on top of the adsorbent bed is dislodged, the top hold down balls will sink if the adsorbent bed is fluidized. This would leave the top of the adsorbent bed uncovered which will cause the small adsorbent particles to be disturbed by the inlet flow. This disturbance would tend to break up the adsorbent particles creating a fine crust on top of the bed which would increase the pressure drop and could cause flow distribution problems.

The unit is designed so that, during normal operation, it is not likely that the adsorbent beds will be upset. However, during startup, shutdown, upset, and emergency operations, care must be taken to prevent conditions which would upset the adsorber beds. Typically this is most easily done by blocking in the adsorbers. In most cases, the hydrocarbon can be left in the adsorbers without any problem.

(Note: In cold weather operation, benzene or paraffin could solidify inside the adsorber if the adsorber was left blocked in for an extended time with benzene or paraffin inside. This should be avoided because it will be very difficult to restart flow through the adsorber. Even if the flow can be restarted there is a high likelihood that the bed will be upset enough to cause flow distribution problems.)

Extremely high flow rates passing downflow through the adsorber (which causes a high pressure drop through the adsorbent bed) can cause crushing of the adsorbent particles. The typical design is for a maximum of 1 Kg/cm<sup>2</sup> (15 psi) across the adsorbent bed itself. However, actual experience shows that adsorbent crushing would not be expected to occur unless the pressure drop across the bed exceeds about 4 Kg/cm<sup>2</sup> (60 psi). It is very unusual for adsorbent crushing to occur.

## 2. Water

Water does not permanently damage the adsorbent. However, water is rather strongly adsorbed and therefore significantly reduces the adsorbent capacity for aromatics. Therefore, the feed, purge and desorbent streams should be very dry. If water does get into the adsorbent, it can be removed by passing a dry hydrocarbon stream over it. Procedures for drying the adsorbent are outlined in the dryout instructions in the Normal Startup section.

If the down stream unit is a Detal unit, water in the treated product from the PEP unit can cause Detal catalyst deactivation. Water in the PEP unit feed can obviously lead to water in the product, but water in the purge or desorbent streams can also lead to water in the product because these streams can lay water down on the adsorbent and as the adsorber cycle progresses, the water will desorb into the product stream.

Typically the PEP feed from the bottom of the Pacol stripper column will be very dry as long as the Pacol stripper is running properly. Also, the desorbent from a Detal unit will be very dry as long as the Benzene column is running properly (even if the column is running on total reflux). However if either of these columns are upset, it is usually best to bypass the PEP adsorbers until the columns are running properly in order to insure dry streams coming into the PEP unit. The purge is the only stream going to the adsorbers from the overhead of a column (depentanizer). As long as the adsorbent has been properly dried out and the other feeds to the unit are dry, there should not be any accumulation of water in the depentanizer overhead. However, the depentanizer overhead is where any water that comes into the unit will tend to accumulate. If the depentanizer overhead does become wet, a line has

been provided to send a slipstream of depentanizer overhead liquid (via the reflux pump) through the pentane drier and back to the depentanizer column in order to dry out the depentanizer overhead.

### 3. Olefin Polymerization

At unusually high temperatures, olefins can react to form large polymers which can coat the adsorbent particles and plug the pores of the adsorbent. This is not a problem at normal operating temperatures. This type of polymerization reaction does not occur unless the temperature exceeds about 350°C (660°F). The only time this should be a concern is during the initial hydrocarbon filling of the adsorbers with fresh adsorbent.

During the initial hydrocarbon filling of the adsorbers, there is a significant exotherm as hydrocarbon is adsorbed on the sieve. Typically, the hydrocarbon is first adsorbed from the vapor phase just above the liquid level as the adsorber is being filled. At the point in the bed where hydrocarbon vapor is being adsorbed on the sieve above the liquid level, a significant temperature rise occurs because there is no liquid present to absorb the heat being generated. However, as soon as the liquid level rises to that point, the bed is immediately cooled down to within about 10°C of the original temperature.

The Pacol reactors must be bypassed for the initial PEP adsorber hydrocarbon filling procedure (with fresh adsorbent). If there is more than 5% olefin in the hydrocarbon used to fill the adsorbers, some polymerization of the olefin could occur as a result of the heat of adsorption in the vapor phase, causing damage to the sieve. It is recommended to keep the olefin content of the hydrocarbon as low as possible during the initial liquid filling step.

Benzene should NOT be used to liquid fill the adsorbers (with fresh adsorbent) because the heat of adsorption for benzene is much greater than for paraffin. This could cause excessive adsorber temperatures if benzene is used for the initial liquid filling.

## XIII. SPECIAL PROCEDURES

### A. ADSORBENT HANDLING

**NOTE:** Protect yourself and others. Read and understand this section thoroughly before loading fresh molecular sieves. Observe all shipping container precaution labels.

In their fresh, unused state, molecular sieves are not flammable. When exposed to water, however, they can get quite hot. When first wetted, molecular sieves can heat to the boiling point of water. For this reason, you should not put molecular sieves into your mouth or allow them to touch your eyes.

Molecular sieve dust may irritate your nose, throat, eyes, lungs and skin. Since some dust may be present when you load fresh molecular sieves, you should wear eye protection, dust masks, gloves, and clothing that covers your body.

Molecular sieves are normally shipped in 55-gallon sealed drums. A vacuum may exist inside the removable-top drums. If you force the lid off, air may rush in and spray the particles about. First, loosen the vent screw on the lid and break the vacuum. Then remove the top.

Take care when moving molecular sieve containers. Full drums weigh more than 300 pounds. During loading, the containers must normally be lifted to the top of the vessel, typically more than 30 feet above ground level. Sometimes, several drums are dumped into a large hopper. The hopper is then lifted to the top of the vessel. Prior to hoisting any containers, rope off the area under the lifting path. Warn all personnel to stand clear.

Occasionally, pneumatic trucks are used to blow molecular sieves into the vessel through a hose. Keep the hose outlet pointed away from personnel when there is air pressure on the storage tanks. The transfer and loading hoppers should be

electrically grounded to help dissipate the static charge built up as the molecular sieves travel through the system. If flammable vapors such as hydrocarbons are present in the loading area, grounding is especially important to avoid discharge of static electricity that could cause an explosion.

**If you must enter the vessel, take the following precautions to prevent asphyxiation:**

1. Install blind flanges on all inlet and outlet piping to prevent accidental release of hazardous fluids into the vessel. Before each entry, have safety personnel analyze the atmosphere in the vessel to make sure it is safe to breathe.
2. Use atmospheric air to vent the vessel. Never use a plant air supply to provide air for ventilation. The air supplied might be contaminated, or the supply could, by mistake, be piped to a gas other than air.

Only properly trained and equipped workers should enter the vessel. A safety harness and rope should always be attached to that worker. Two properly trained and equipped co-workers standing outside the entry port should constantly observe the worker in the vessel. The co-workers should use the safety rope to prevent falls from the ladder used to enter or leave the vessel. Also, if disabled, the worker can be removed using the rope.

#### **Key Safety Precautions for Loading Fresh, Unused Molecular Sieves**

1. Prevent Possible Burns – Do not put molecular sieves into your mouth or allow them to touch your eyes. Whenever you handle molecular sieves, wear eye protection, a dust mask, gloves, safety shoes, and clothing that completely covers your body.

2. Avoid Flying Molecular Sieve Particles
  - (a) When you open molecular sieve drums, loosen the vent screw before you remove the lid.
  - (b) When loading from pneumatic trucks, keep the hose outlet pointed away from personnel.
3. Avoid Possible Falling Molecular Sieve Containers – Do not allow anyone into the lifting area; no one should stand or walk under the containers while they are being lifted or transported on the hoist. Before making the initial lift, rope off the lifting area and warn all personnel to stand clear.
4. Avoid Igniting Combustible Vapors – If the molecular sieves are loaded pneumatically, ground the equipment to avoid excessive static electricity. Sparks from static electricity can ignite any combustible vapors present.
5. Prevent Possible Asphyxiation and Falls – Before entering the vessel:
  - (a) Install blind flanges on all inlet and outlet piping.
  - (b) Test the atmosphere inside the vessel to be sure it is safe for entry.
  - (c) Attach a safety rope to the properly trained and equipped worker who will enter. The worker should use the safety harness and rope to prevent falls from the ladder used to enter or leave the vessel. Also, if disabled, the worker can be removed using the rope.
  - (d) Station two properly trained and equipped co-workers outside the entry port. The co-workers must constantly observe the worker inside the vessel. The co-workers should use the safety rope to prevent falls from the ladder used to enter or leave the vessel. Also, if disabled, the co-workers can remove the worker in the vessel using the rope.

## **B. ADSORBER LOADING**

The personnel involved with the loading of the PEP adsorbers should follow all the refinery safety rules and practices. In addition, the workers should wear proper dust masks, eye protection, and gloves. All safety recommendations detailed in the Adsorbent MSDS (Material Safety Data Sheets) should be followed.

### **1. Preparation**

- a. The equipment intended for use in the loading process should be inspected thoroughly. Make sure that all hoppers and accessories are dry and free of contaminants such as oil, water, grease, or other catalyst. If necessary, clean the equipment with steam, then dry the equipment completely. Also, inspect the scaffolding and ladders supplied for the loading operation. Scaffolding should be sturdy enough to support the hopper, a team of men and any other equipment necessary. Ladders should be relatively uncluttered so as not to present tripping hazards to workers.
- b. Verify that the appropriate materials are available to protect empty or partially loaded vessels throughout the loading. Open vessels must be sealed with plastic or other water-proof covering to keep out water.
- c. Install blinds at each adsorber inlet and outlet line. This is necessary to safely load the adsorbent and also to prevent damage to the adsorbent.
- d. The vessel temperature should be less than 30°C (90°F) prior to vessel entry.
- e. Clean dirt and any foreign material from the adsorber. Wire brush the adsorber walls for rust removal if necessary.
- f. Install the adsorber bottom profile wire screen distributor. Be sure the distributor is clean and is not damaged.

- g. The following list of materials is required for the loading of the PEP adsorbers.

1. Ceramic support balls per UOP Schedule A
2. 1000 ml sample bottles with screw tops
3. Sufficient PEP adsorbent as specified in the Schedule A
4. 2 transfer hoppers (approx. 5 m<sup>3</sup> capacity)
5. 1 stationary loading hopper (2-5 m<sup>3</sup> capacity)
6. 1 loading platform
7. 1 forklift
8. 1 crane
9. 1 bucket for loading ceramic support
10. 1m length of new, flexible loading hose
11. 2 or more electric lamps for use inside of the adsorber
12. 15m tape measure
13. Rake for smoothing the surface of the various materials
14. 1 to 2 meter long carpenter's level
15. Ladder up to 10 meters long for entering the reactor
16. Safety harness rope

- h. The loading crew should be organized as follows:

1. A supervisor.
2. A crane operator.
3. Forklift operators, truckers, etc. to keep a steady flow of adsorbent ready to be loaded.
4. Two men on the ground to empty the drums into the transfer hopper(s).
5. Two men on scaffolding to direct the crane operator in positioning the hoppers and control the dumping of adsorbent.

## **2. Loading Adsorbers with Ceramic Support Balls**

- a. Using a tape measure, carpenter's level, and chalk, inscribe circles to represent the depth of the 6 mm and 3 mm ceramic balls.
- b. The ceramic support material should be dry. A couple of retain samples of the support material should be kept aside for future reference. Load the balls into the adsorber with buckets through the bottom manway. The ceramic balls should be spread evenly across the adsorber cross-section. After each layer is loaded, it should be leveled and the dimensions referenced against a point higher up in the adsorber. For a typical PEP adsorber, the top manway flange may be used. The various dimensions should be measured with a tape and recorded (refer to the diagram in Figure XIII-1). The target values should have been pre-recorded using the adsorber design drawings. After all the ceramic support is loaded, the bottom manway should be permanently closed using the proper gasket.

## **3. Loading Adsorbers with Adsorbent**

Due to the high affinity for water and the adverse effects of moisture on the PEP adsorbent, loading should not be done in rainy or snowy weather. If the weather is threatening, the loading crew should be prepared to stop loading and cover all vessels, hoppers, etc. upon direction from the UOP representative on site. The UOP representative should determine when adsorbent loading should begin or stop based on weather condition.

- a. Transfer adsorbent from the drums into transfer hoppers at grade level. This can be accomplished by lifting the drums with a forklift truck onto an elevated platform and then dumping them into the transfer hoppers.

Note: The hoppers and socks used throughout the loading operation must be clean to avoid contaminating the ceramic balls and adsorbents. If necessary, the loading hoppers should be cleaned with steam and then completely dried prior to use. The loading socks should be new.

- b. Lift the transfer hopper to the top of the adsorber. Empty the contents into the loading hopper anchored at an adequate elevation from the adsorber top manway. On the bottom of the loading hopper, install a 1 meter long flexible loading hose. This hose should be fixed to the hopper by bolting to avoid dropping off into the adsorber during the loading.
- c. Swing the flexible rubber hose back and forth in the manway as the adsorbent flows through it. This will eliminate the formation of a mound in the center of the adsorbent bed. The bed will be loaded more evenly and the possibility of flow channeling during normal operation will be reduced. (Note: It is also possible to use a properly designed distributor to evenly load the adsorbent. The UOP representative should be consulted to approve the use of a particular distributor for loading.)
- d. Take a 10 ml sample from each drum of adsorbent loaded and combine them into a 1000 ml bottle. Label the samples to indicate which adsorber they represent. These samples should be held for future use.
- e. Record the lot and drum number printed on each drum. Record the net adsorbent weight in each drum.
- f. After emptying the contents of each drum of adsorbent, replace the foam rubber seal and re-bolt the top back on the drum. The original drums can be used during future handling operations to avoid contamination of the adsorbent. Store the drums with the bottom side up. Do not remove the drums from the site until the final drum count has been completed.
- g. Stop the loading operation periodically. Allow the dust to settle and then lower lights into the adsorber. Observe the surface of the adsorbent bed. The surface should be flat and level. If it is not, an adjustment in the technique of swinging the loading hose must be made so as to eliminate any mounding.

- h. Load the adsorbent according to outage measurements and the adsorbent loading diagram. Smooth and level the surface of the adsorbent bed. This should then be checked with a carpenter's level. Measure, and record on the loading diagram, the distance between the top surface of the adsorbent bed and the reference point.
- i. The loading diagram should serve as the basis for calculating the volume of loaded adsorbent. The loaded ABD can be calculated based on total adsorbent weight loaded.
- j. UOP personnel will observe the vessel loading and provide advice on the details of the operation.

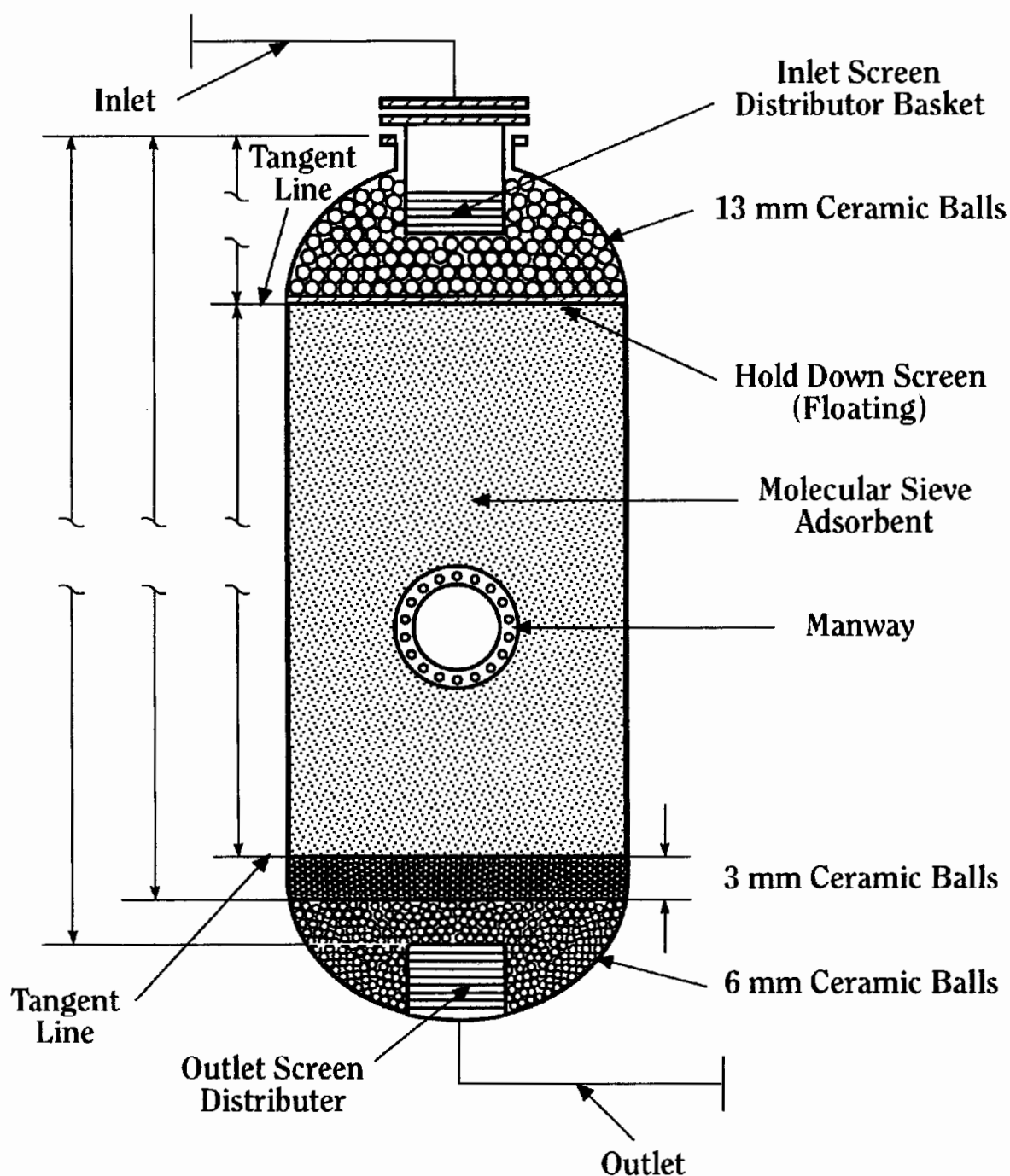
#### **4. Install the Hold Down Screen and Ceramic Support Material**

- a. Install the hold down screen. Be sure the screen is properly positioned on top of the adsorbent bed.
- b. Install the final layer of ceramic support material on top of the hold down screen. The ceramic balls should be leveled and the dimensions referenced against the adsorber inlet manway flange.

#### **5. Install the Top Distributor and Close Up the Vessel**

- a. Install the profile wire screen top distributor. Be sure the distributor is clean and there is no damage.
- b. Be sure there are no tools or foreign matter in the adsorber.
- c. Clean the surface of the manway flange and close the manway.
- d. Pressure each adsorber to 1.0 kg/cm<sup>2</sup> (15 psig) with dry nitrogen. Maintain this positive nitrogen pressure until the plant is ready for startup. Do not remove the blinds at the inlet and outlet of the adsorbers until the unit is ready for startup.

**Figure XIII-1**  
**Loading Diagram for a PEP Adsorber**



UOP 2198-12

Figure XIII-2

**uop****Adsorber Loading Summary**

Technical Service Department

**REACTOR LOADING SUMMARY**

| REFINER |         |                       |         |        |         |                       |         | LOCATION                                                                                        |         |                       |         |           |  |  |  |
|---------|---------|-----------------------|---------|--------|---------|-----------------------|---------|-------------------------------------------------------------------------------------------------|---------|-----------------------|---------|-----------|--|--|--|
| UNIT    |         |                       |         |        |         |                       |         |                                                                                                 |         |                       |         |           |  |  |  |
| REACTOR |         |                       |         | DATE   |         |                       |         | WEATHER                                                                                         |         |                       |         | TABLE NO. |  |  |  |
| DRUM    | LOT NO. | DRUM NO. OF LOT TOTAL | NT. WT. | DRUM   | LOT NO. | DRUM NO. OF LOT TOTAL | NT. WT. | DRUM                                                                                            | LOT NO. | DRUM NO. OF LOT TOTAL | NT. WT. |           |  |  |  |
| ___ 1   |         |                       |         | ___ 36 |         |                       |         | ___ 71                                                                                          |         |                       |         |           |  |  |  |
| ___ 2   |         |                       |         | ___ 37 |         |                       |         | ___ 72                                                                                          |         |                       |         |           |  |  |  |
| ___ 3   |         |                       |         | ___ 38 |         |                       |         | ___ 73                                                                                          |         |                       |         |           |  |  |  |
| ___ 4   |         |                       |         | ___ 39 |         |                       |         | ___ 74                                                                                          |         |                       |         |           |  |  |  |
| ___ 5   |         |                       |         | ___ 40 |         |                       |         | ___ 75                                                                                          |         |                       |         |           |  |  |  |
| ___ 6   |         |                       |         | ___ 41 |         |                       |         | ___ 76                                                                                          |         |                       |         |           |  |  |  |
| ___ 7   |         |                       |         | ___ 42 |         |                       |         | ___ 77                                                                                          |         |                       |         |           |  |  |  |
| ___ 8   |         |                       |         | ___ 43 |         |                       |         | ___ 78                                                                                          |         |                       |         |           |  |  |  |
| ___ 9   |         |                       |         | ___ 44 |         |                       |         | ___ 79                                                                                          |         |                       |         |           |  |  |  |
| ___ 10  |         |                       |         | ___ 45 |         |                       |         | ___ 80                                                                                          |         |                       |         |           |  |  |  |
| ___ 11  |         |                       |         | ___ 46 |         |                       |         | ___ 81                                                                                          |         |                       |         |           |  |  |  |
| ___ 12  |         |                       |         | ___ 47 |         |                       |         | ___ 82                                                                                          |         |                       |         |           |  |  |  |
| ___ 13  |         |                       |         | ___ 48 |         |                       |         | ___ 83                                                                                          |         |                       |         |           |  |  |  |
| ___ 14  |         |                       |         | ___ 49 |         |                       |         | ___ 84                                                                                          |         |                       |         |           |  |  |  |
| ___ 15  |         |                       |         | ___ 50 |         |                       |         | ___ 85                                                                                          |         |                       |         |           |  |  |  |
| ___ 16  |         |                       |         | ___ 51 |         |                       |         | ___ 86                                                                                          |         |                       |         |           |  |  |  |
| ___ 17  |         |                       |         | ___ 52 |         |                       |         | ___ 87                                                                                          |         |                       |         |           |  |  |  |
| ___ 18  |         |                       |         | ___ 53 |         |                       |         | ___ 88                                                                                          |         |                       |         |           |  |  |  |
| ___ 19  |         |                       |         | ___ 54 |         |                       |         | ___ 89                                                                                          |         |                       |         |           |  |  |  |
| ___ 20  |         |                       |         | ___ 55 |         |                       |         | ___ 90                                                                                          |         |                       |         |           |  |  |  |
| ___ 21  |         |                       |         | ___ 56 |         |                       |         | ___ 91                                                                                          |         |                       |         |           |  |  |  |
| ___ 22  |         |                       |         | ___ 57 |         |                       |         | ___ 92                                                                                          |         |                       |         |           |  |  |  |
| ___ 23  |         |                       |         | ___ 58 |         |                       |         | ___ 93                                                                                          |         |                       |         |           |  |  |  |
| ___ 24  |         |                       |         | ___ 59 |         |                       |         | ___ 94                                                                                          |         |                       |         |           |  |  |  |
| ___ 25  |         |                       |         | ___ 60 |         |                       |         | ___ 95                                                                                          |         |                       |         |           |  |  |  |
| ___ 26  |         |                       |         | ___ 61 |         |                       |         | ___ 96                                                                                          |         |                       |         |           |  |  |  |
| ___ 27  |         |                       |         | ___ 62 |         |                       |         | ___ 97                                                                                          |         |                       |         |           |  |  |  |
| ___ 28  |         |                       |         | ___ 63 |         |                       |         | ___ 98                                                                                          |         |                       |         |           |  |  |  |
| ___ 29  |         |                       |         | ___ 64 |         |                       |         | ___ 99                                                                                          |         |                       |         |           |  |  |  |
| ___ 30  |         |                       |         | ___ 65 |         |                       |         | ___ 00                                                                                          |         |                       |         |           |  |  |  |
| ___ 31  |         |                       |         | ___ 66 |         |                       |         | Total Catalyst Loaded:<br>_____ Lbs (Kg) _____ Ft <sup>3</sup> (m <sup>3</sup> )<br>_____ Drums |         |                       |         |           |  |  |  |
| ___ 32  |         |                       |         | ___ 67 |         |                       |         |                                                                                                 |         |                       |         |           |  |  |  |
| ___ 33  |         |                       |         | ___ 68 |         |                       |         | Loaded Density:<br>_____ Lb/Ft <sup>3</sup> (Kg/m <sup>3</sup> )                                |         |                       |         |           |  |  |  |
| ___ 34  |         |                       |         | ___ 69 |         |                       |         |                                                                                                 |         |                       |         |           |  |  |  |
| ___ 35  |         |                       |         | ___ 70 |         |                       |         |                                                                                                 |         |                       |         |           |  |  |  |

### C. ADSORBER UNLOADING

All of the precautions and procedures for handling and loading fresh molecular sieve should be followed for unloading the spent molecular sieve. This includes the use of proper personnel protection such as gloves, eye protection, dust masks, safety shoes, and clothing that completely covers the body. In addition, it should be assumed that there is some residual hydrocarbon (possibly including benzene) left on the sieve. Therefore, unloading personnel should not enter the vessel until after the molecular sieve has been unloaded.

Even after nitrogen purging, some hydrocarbon will remain adsorbed on the molecular sieve. Therefore, hydrocarbon (possibly including benzene) fumes may be released during unloading. The atmosphere in the unloading area should be periodically checked for benzene and hydrocarbons to prevent accidental personnel exposure to benzene or fire. Always keep self-contained breathing apparatus and fire hoses available during unloading in case of emergency.

The following procedure may be used for unloading molecular sieve from the adsorbers:

1. Bypass the adsorber section and remove the hydrocarbon from the adsorbers according to the instructions in the Normal Shutdown (Section XI). If desired, the depentanizer and desorbent columns can be kept running. The hydrocarbon only needs to be removed from the adsorbers to be unloaded.

(Note: In order to minimize the amount of benzene on the molecular sieve to be unloaded, any adsorber that contained benzene when the unit was shut down (such as the adsorber in the desorption step) should be displaced and purged with paraffin or pentane before removing the hydrocarbon.)

2. Make sure the manual inlet and outlet valves on each adsorber are closed. Then, swing the spectacle blinds closed on the inlet and outlet of each adsorber to be unloaded.

3. Depressure the adsorber to flare through the PSV bypass line. Remove the hydrocarbon vapors from the adsorber by pressuring up and depressuring the adsorber at least three times with nitrogen. Check to make sure the hydrocarbon content of the adsorber is well below the acceptable LEL (Lower Explosive Limit). Close the PSV bypass line to the flare. Maintain a nitrogen blanket or nitrogen purge on the adsorber until it is unloaded.

(Note: It is more efficient to pressure up the adsorber from the bottom with nitrogen if it is to be depressured from the top. During pressuring, depressuring and purging operations, excessive gas velocities in the upflow direction can upset the molecular sieve bed, hold down screen, and support balls. Care should be taken to limit the gas velocity passing upward through the adsorber.)

4. When the adsorber is ready for unloading, make sure the valves on the PSV bypass line to the flare are closed. Establish a small nitrogen purge up through the vessel and out the vent near the manual inlet valve. Check to make sure the hydrocarbon content of the adsorber is well below the acceptable LEL.
5. To make dumping the molecular sieve easier, the hold down screen should be removed from the top of the adsorbent bed. Remove the top manway from the adsorber. There is a layer of ceramic balls on top of the hold down screen. The screen can be removed without entering the vessel using a harpoon-type tool. After removing the screen, cover the inlet manway with heavy plastic sheeting or re-install the manway cover to prevent air from entering the adsorber during unloading. If the inlet manway is re-installed, a blind must be inserted in the inlet nozzle so that the adsorber is positively isolated from the flare line.

(Note: A new hold down screen will be necessary if the adsorber is to be re-loaded.)

6. Make sure that the adsorber is blinded from all possible hydrocarbon sources. Then 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 molecular sieve as it is unloaded.
7. Loosen the remaining 4 bolts on the side manway. Allow the molecular sieve to flow out of the side manway using the manway cover to regulate the flow.

(Note: If hydrocarbon adsorbed on the molecular sieve starts to burn or react (give off large amounts of heat), stop dumping immediately. Remove all personnel from the area. Properly trained personnel with self-contained breathing apparatus can then return to stop the reaction or put out the fire. To stop a reaction or put out a fire, use water from fire hoses to flood the molecular sieve outside the vessel.)

8. When the molecular sieve no longer flows out freely, remove the remaining sieve using a rake, hoe, shovel, or vacuum truck. Do not enter the vessel to remove the remaining sieve. Care must be taken not to disturb the layers of support balls under the molecular sieve bed. It is recommended that a thin layer of molecular sieve be left in the vessel to ensure that the support balls are not disturbed.
9. After all of the molecular sieve has been removed, remove the unloading chute and clean the side manway flanges.
10. If desired, prepare the atmosphere in the vessel for safe entry. Remove the nitrogen purge line and attach an air mover so that air is expelled out the bottom of the vessel below the bed support. The air will carry any remaining vapors out of the vessel and away from the workers. Leave the air movers on at all times. Do not use a plant air supply to purge the adsorber with air – a plant air supply could become contaminated or hooked up to another source by mistake.

After safety personnel have determined that the atmosphere inside the vessel is safe to enter, a properly trained worker may go in the vessel to inspect the support balls and vessel internals. Be sure to follow all of the proper procedures for entering a confined space.

11. The adsorber may then be re-loaded according to the Adsorber Loading instructions given earlier in this manual (Section XIII).

## **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](http://www.cdc.gov/niosh)), OSHA ([www.osha.gov](http://www.osha.gov)), and API ([www.api.org](http://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 PEP Unit. In general, the normal refinery safety rules pertaining to the handling of hydrocarbons under pressure and moderately high temperatures also apply to the PEP 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 adsorbers. 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. Adsorbent**

Information on safely handling the adsorbent for the PEP Unit is contained in Section XIII – Special Procedures. An example MSDS for the PEP adsorbent is included at the end of this Section XIV.

## 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. PEP Adsorbent ADS-102.



# SAFETY DATA SHEET

## 1. PRODUCT AND COMPANY IDENTIFICATION

**Product Name:** ADS-102, PEP Adsorbent

**Product Use:** Adsorbent

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

UOP M.S. S.p.A.  
Viale Milanofiori  
Strada 1 - Palazzo E1  
20090 Assago Mi, Italy  
Tel : +39-02-892241  
Fax : +39-02-57500145

**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<br/>&amp; CAS No</u> | <u>% WEIGHT</u> | <u>EC SYMBOL</u> | <u>EC R-PHRASES</u> | <u>EC Exposure Limit<br/>mg/m<sup>3</sup></u> |
|------------------------------------|-----------------|------------------|---------------------|-----------------------------------------------|
| Zeolites<br>1318-02-1              | > 70            | N.E.             | N.E.                | 10(I)<br>3(R)                                 |
| Mineral binder<br>999999-99-4      | < 30            | N.E.             | N.E.                | N.E.                                          |
| Quartz<br>14808-60-7               | < 2             | N.E.             | N.E.                | 0.1(R)                                        |

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

## 3. HAZARD IDENTIFICATION

No hazard classification.

When first wetted, the product can heat up to the boiling point of water. Flooding will reduce the temperature. Repeated and prolonged inhalation of crystalline silica in the form of quartz from occupational sources may cause cancer.

## **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:** Immediately give large volume of water to drink. If symptoms persist, call a physician.

## **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. A copy of UOP's booklet, "Precautions and Safe Practices for Handling Molecular Sieves in Process Units", M-100C, can be obtained from your UOP representative at no cost.
- 7.2 Storage:** Store in original container. Keep in a dry place.

## 8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

- 8.1 Engineering measures:** Ensure adequate ventilation, especially in confined areas.
- 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:** Work uniform and gloves to prevent prolonged contact.
- Respiratory protection:** In case of insufficient ventilation wear suitable respiratory equipment. Breathing apparatus with filter: NIOSH classification N-100 or if oil/liquid aerosols are present P-100 (42 CFR 84).
- 8.3 Exposure limit(s):** International Exposure Limits (mg/m<sup>3</sup>)

| <u>INGREDIENT<br/>&amp; CAS No</u> | <u>% WEIGHT</u> | <u>UK<br/>OES/MEL</u> | <u>Germany<br/>MAK</u> | <u>The Netherlands<br/>MAC</u> | <u>France<br/>VME</u> |
|------------------------------------|-----------------|-----------------------|------------------------|--------------------------------|-----------------------|
| Zeolites<br>1318-02-1              | > 70            | 10(I)<br>4(R)         | 4(I)<br>1.5(R)         | 10(I)<br>5(R)                  | 10(I)<br>5(R)         |
| Mineral binder<br>999999-99-4      | < 30            | 10(I)<br>4(R)         | 4(I)<br>1.5(R)         | 10(I)<br>5(R)                  | 10(I)<br>5(R)         |
| Quartz<br>14808-60-7               | < 2             | 0.3(R)                | 0.15(R)                | 0.075(R)                       | 0.1(R)                |

Note : In case no information on national exposure limits is available, the exposure limit for 'general dust' is presented.

## 9. PHYSICAL AND CHEMICAL PROPERTIES

These data do not represent technical or sales specifications.

|                                         |       |
|-----------------------------------------|-------|
| 9.1 Form:                               | Beads |
| 9.2 Color:                              | Tan   |
| 9.3 Odor:                               | None  |
| 9.4 pH:                                 | N.A.  |
| 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:                       | N.D.  |
| 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:                  | N.D.  |
| 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. |
| 10.3 Conditions/Materials to avoid:    | Sudden contact with high concentrations of chemicals having high heats of adsorption such as olefins, HCl, etc. When first wetted, the product can heat up to the boiling point of water. Flood with water to cool material.                             |

## 11. TOXICOLOGICAL INFORMATION

### 11.1 Acute toxicity:

LD50/oral/rat = > 32 000 mg/kg \*

LD50/dermal/rabbit = > 2 000 mg/kg \*

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): A2 (Quartz)

IARC classification: Group 1 (Quartz)  
Group 3 (Zeolites)

### 11.3 Routes of exposure:

Exposure may occur via inhalation, contact with skin and eyes.

### 11.4 Irritation:

Skin (rabbit): Not classified as a skin irritant in animal testing. \*

Eye (rabbit): Moderate eye irritation. \*

### 11.5 Additional product information:

\* The toxicological data has been taken from products of similar composition.

## 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: No information available.

## 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: Can be landfilled after chemical and physical treatment, when in compliance with the local regulations. Store containers and offer for recycling of material according to the local regulations. Contact waste disposal services. 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): nwg

## 16. OTHER INFORMATION

**Summary of changes:** Sections 3, 11, 15 (US)  
**Supersedes:** August 2000  
**Prepared by:** UOP Health, Safety & Environmental Department

### Abbreviations:

|        |                                                           |
|--------|-----------------------------------------------------------|
| N.A.   | - Not applicable                                          |
| N.E.   | - None established                                        |
| N.D.   | - Not determined                                          |
| OESMEL | - 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 Compounds                                     |

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

UOP M.S. S.p.A.  
Viale Milanofiori  
Strada 1 -Palazzo E1  
20090 Assago Mi  
Italy  
Tel: +39 (02) 892241  
Fax: +39 (02) 57500145

### 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: \_\_\_\_\_

|                       | Feed | Reflux | Off Gas | Btms. | Net Ovhd.<br>Liquid | Other |
|-----------------------|------|--------|---------|-------|---------------------|-------|
| Flow Rate, _____      |      |        |         |       |                     |       |
| Temperature, ° _____  |      |        |         |       |                     |       |
| Pressure, _____       |      |        |         |       |                     |       |
| Composition, _____ %  |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
|                       |      |        |         |       |                     |       |
| Avg. Mol. Wt. _____   |      |        |         |       |                     |       |
| Gravity _____         |      |        |         |       |                     |       |
| Distillation, ° _____ |      |        |         |       |                     |       |
| IBP _____             |      |        |         |       |                     |       |
| 5% _____              |      |        |         |       |                     |       |
| 10% _____             |      |        |         |       |                     |       |
| 20% _____             |      |        |         |       |                     |       |
| 30% _____             |      |        |         |       |                     |       |
| 40% _____             |      |        |         |       |                     |       |
| 50% _____             |      |        |         |       |                     |       |
| 60% _____             |      |        |         |       |                     |       |
| 70% _____             |      |        |         |       |                     |       |
| 80% _____             |      |        |         |       |                     |       |
| 90% _____             |      |        |         |       |                     |       |
| 95% _____             |      |        |         |       |                     |       |
| EP _____              |      |        |         |       |                     |       |

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

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

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



## AIR FIN COOLER SURVEY

page \_\_\_\_\_

date \_\_\_\_\_

by \_\_\_\_\_

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

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

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

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

No. of Bundles: \_\_\_\_\_ No. of Passes: \_\_\_\_\_

No. of Tubes per Pass: \_\_\_\_\_ Fans/bundle: \_\_\_\_\_

Tube Size \_\_\_\_\_ ID x \_\_\_\_\_ Gauge x \_\_\_\_\_ Length \_\_\_\_\_

Piping Geometry: \_\_\_\_\_ Type\*: \_\_\_\_\_

Overall Heat Transfer Coefficient: \_\_\_\_\_

|                | Pressure | Temperature |
|----------------|----------|-------------|
| Process Inlet  |          |             |
| Process Outlet |          |             |

| Air | In | Out |  |  |
|-----|----|-----|--|--|
|     |    |     |  |  |

No. fans on \_\_\_\_\_ Pitch control \_\_\_\_\_  
Louver position \_\_\_\_\_

| Leaver position    | Process | Air |
|--------------------|---------|-----|
| Stream Description |         |     |

|                |       |       |
|----------------|-------|-------|
|                | <hr/> | <hr/> |
| Vol. Flow Rate | <hr/> | <hr/> |

Mass Flow Rate

Q (calc.)

Sp.Gr./Avg.Mol.Wt. \_\_\_\_\_

Composition, \_\_\_\_\_ %

Relative Humidity

Deviations from UOP Specification: \_\_\_\_\_

\*Include sketch of piping geometry if different from UOP standard practice types.

**CENTRIFUGAL PUMP SURVEY**

page \_\_\_\_\_

date \_\_\_\_\_

by \_\_\_\_\_

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

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

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

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

No., Size and Style (Mfgrs. 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 (Mfgrs. 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 Mfgrs. pump curve and plot operating point.

Deviations from UOP Specification: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**CONTROL VALVE SUMMARY**

page \_\_\_\_\_

date \_\_\_\_\_

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

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

Description of Valve: \_\_\_\_\_ Design CV: \_\_\_\_\_

Mfgr. 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]