



MOLEX PROCESS

GENERAL OPERATING MANUAL

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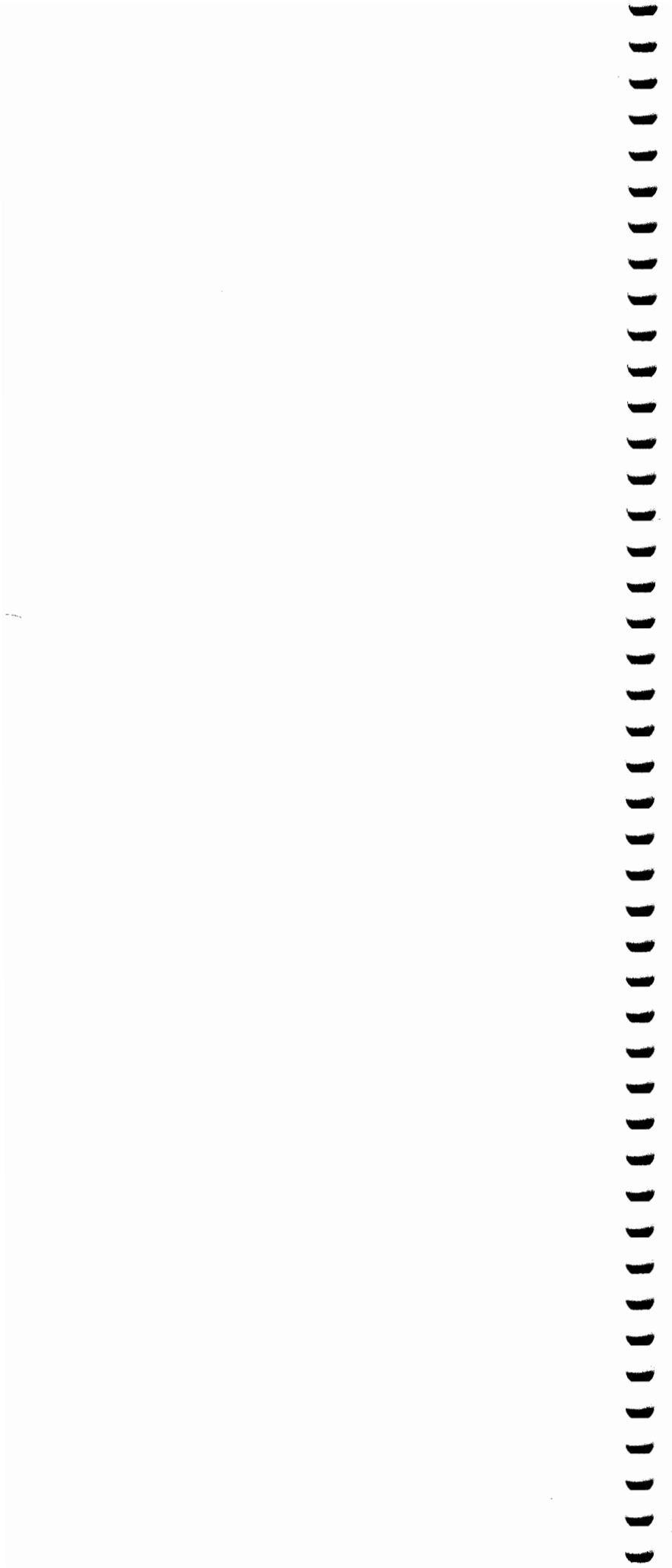
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I. INTRODUCTION

A. THE MOLEX PROCESS

The UOP Molex process is an effective method of continuously separating normal paraffins from a stream of co-boiling hydrocarbons by means of physically selective adsorption. The feedstock, essentially having the same properties of kerosene, is separated into a high purity normal paraffin fraction at high recoveries, and a non-normal fraction.

The process simulates countercurrent contact between a fixed bed adsorbent and the feed stream. It uses a solid adsorbent, liquid desorbent, and a flow directing device called the "rotary valve."

Adsorbent

The key to the UOP Molex process is the proprietary molecular sieve, capable of preferentially adsorbing the narrow, straight chain normal paraffins while excluding the bulkier non-normals. It is this difference in physical shape that allows the separation to take place.

If a vessel were filled with molecular sieve and feed material, given adequate time, a concentration difference of n-paraffins between the material adsorbed by the molecular sieve and the surrounding liquid will develop. The concentration of n-paraffins will be higher in the molecular sieve due to the physical limitation of only n-paraffins being able to enter the selective pores. These selective pores make up approximately 11% of the total volume in the vessel. The remaining larger, non-selective, pores will contain both n-paraffins and non-normal hydrocarbons.

Desorbent

The desorbent is a liquid mixture of a different boiling point than the feed components and is capable of displacing the adsorbed normal paraffins from the pores of the sieve and displacing the non-normal components from the non-selective volume of the adsorbent. Normal pentane (nC_5) and iso-octane (2,2,4 trimethylpentane) both boil much lower than the feed components and therefore can be easily fractionated and recycled.

To remove the non-normals from the non-selective pores, a lighter non-normal material, like iC_8 , can be used to flush the molecular sieve. By flushing enough iC_8 through the molecular sieve, all of the heavier non-normals will be removed out the bottom of the vessel. This will leave only n-paraffins in the selective pores and iC_8 in the larger pores.

With n-paraffins in the selective pores and iC_8 in the remaining volume, the heavy normal paraffins are recovered using a mixture of desorbent, $n-C_5$ and iC_8 . By flushing at a relatively high rate with the desorbent, the $n-C_5$ will displace the heavy normal paraffins from the selective pores. The liquid can then be fractionated to separate the desorbent material from the heavy normal paraffins.

Feed

Paraffinic compounds, those having all saturated, single bonds, in the C_{10} to C_{14} range, are typically fed to the Molex unit. Molex separates the straight chain n-paraffins (nP) from the iso-paraffins and aromatics (nnP).

Products

Molex produces two product streams; the extract, containing a mixture of normal paraffins and desorbent, and the raffinate, containing the non-normals and desorbent. Both streams are sent to columns that remove the recyclable desorbent and produce a high purity normal or non-normal product. The normal paraffins can be sent downstream for further processing in a Pacol Unit for the production of linear n-olefins or sold as high purity n-paraffin product for other petrochemical purposes.





II. PROCESS PRINCIPLES

A. INTRODUCTION

This section will explain the principles underlying the operation of the Molex Unit. The Molex process is one of the many UOP Sorbex processes that use adsorptive chromatography in conjunction with distillation to solve a difficult separation problem. UOP developed an adsorbent/desorbent system that separates normal paraffins from other non-normal paraffins. By incorporating the adsorbent / desorbent system into the Sorbex technology, a continuous operating is obtained. The use of a mechanical system that has been refined and proven for this type of operation provides a high degree of reliability.

B. EQUILIBRIUM

1. Simple Experiments

Equilibrium is the term used to describe a situation where no net change is occurring. Frequently this is confused for a situation where no change is occurring. An example will illustrate the difference:

Take a mixture of fifty of each red and blue marbles. Put them in a box with a divider in the middle such that 50 marbles are on each side of the divider. Label one side of the divider as phase A and the other side as phase B. A good description of the box now is that phase A has 50% red marbles and 50% blue marbles. Phase B has 50% red marbles and 50% blue marbles. Although it could be said that the system is in equilibrium such a static system is usually never reached in a chemical process. This is not what we mean when we say a system is in equilibrium.

To improve the model to be something more useful for our discussion we need to have movement of marbles between the two “phases” we have defined. At the bottom of each phase we will create a hole and set up a conveyor to carry marbles one by one out of each “phase” and drop them into the other “phase”. If the rate at which the marbles are removed from each phase and transferred to the other phase are equal the total number of marbles remains constant in each phase.

Let's assume that there is no difference in the size or weight of the marbles such that they can fall out of each phase with equal ease. If we watch the marbles falling out of phase A and passing to phase B we may see a sequence like: red, blue, red, blue, blue, blue, red, red, blue, red, red, blue, red, blue, red, red, blue, red, blue, blue. Falling out of phase B and passing to phase A we may see: red, blue, red, red, blue, red, blue, red, blue, blue, blue, red, red, blue, red, red, blue, red. So we have seen 10 red balls and 10 blue balls transfer from phase A to B and 11 red balls and 9 blue balls transfer from phase B to A. At the end of the observation Phase A has 26 red balls and 24 blue balls and phase B has 24 red balls and 26 blue balls. Phase A has now 52% red and 48% blue. Phase B has 48% red and 52% blue. Now continue the experiment and allow 20 more marbles to go from each phase to the other and record the amount of each type of marble in each phase you may find phase A has 46% red and 54% blue and phase B now has 54% red and 46% blue. Repeat the experiment many times and we will find that the average composition in each phase is 50% red and 50% blue. At some point the average composition of the experiment becomes stable and we call the composition the equilibrium composition.

One problem with the previous example is that both phases started with the same composition. Start a second example with all of the red marbles in phase A and all of the blue marbles in phase B. Start the marbles moving from each phase to the other again and patiently wait. Eventually you will end up with the same composition of marbles in each phase: 50% red and 50% blue. When this happens we say that the system has reached equilibrium because no matter how long we wait the average composition as time goes by becomes 50% red and 50% blue.

While this is a reasonable description of equilibrium operators of Molex are not trying to mix objects but would rather separate them.

Let's make a change to the experiment. Let's increase the diameter of the blue marbles by 50% and add a second hole and marble pump to the bottom of phase B. The two holes in the bottom of phase B will have two different sizes. One will allow either a red or blue marble out and the other will let only red marbles out. We'll put a slow moving wiper in the bottom of phase B to keep the blue marbles from plugging the smaller hole. The hole in the bottom of phase A will let either red or blue marbles out. The marble pump from phase A is set for 60 marbles a minute and each of the marble pumps for phase B are set for 30 marbles a minute.

When we start the experiment again with 50% of each red and blue marbles in each of phase A and B what we see is that both red and blue marbles can escape phase A equally but that red marbles move more easily from phase B to phase A. If we let the experiment continue for a long time the average composition of each phase again becomes stable.

Regardless of the starting points for the two phases they always reach the same mixture of blue and red marbles at the end. If Phase A starts out as all red or all blue or a 50% mixture of each the final result will be the same. We can say that a certain separation exists between the two phases during equilibrium. We express this as an enrichment factor between the two phases.

2. Enrichment Factors

The relationship between the concentration of component *i* in two different phases is defined as K_i , the equilibrium constant. For the simple experiment we have just discussed, $K_i = A_i/B_i$, where A_i is the concentration of component "i" in the A phase and B_i is the concentration of a component "i" in the B phase.

For the last experiment:

$$K_{\text{red}} = \frac{A_{\text{red}}}{B_{\text{red}}} = \frac{75}{25} = 3 \quad K_{\text{blue}} = \frac{A_{\text{blue}}}{B_{\text{blue}}} = \frac{25}{75} = \frac{1}{3}$$

For a system that only contains two components the use of the equilibrium constant may appear to be an unnecessary complication. In most real world systems many components are present but the concept of equilibrium constants can still be applied.

To get an indication of the relative ease of separating these two components the ratio of the two equilibrium constants provides the enrichment factor which we can arbitrarily call the Gamma Factor (g):

$$\gamma_{\text{red, blue}} = \frac{K_{\text{red}}}{K_{\text{blue}}} = \frac{3}{1/3} = 9$$

As the enrichment factor becomes larger the separation between the two gets easier to make.

C. ADSORBENT THEORY

It is convenient to visualize the adsorbent as a porous solid having certain characteristics. When the solid is immersed in a liquid mixture, the pores become filled with liquid. At equilibrium, the composition of the liquid in the pores will be different from that of the liquid surrounding the particles. The adsorbent is said to be physically selective for the component that is more concentrated in the pores than in the surrounding liquid.

Adsorbents have been produced which differ widely from each other in physical properties. This is particularly the case for adsorbents based on the synthetic crystalline zeolites designated generically as molecular sieves.

The adsorbent employed in Molex is a specially prepared molecular sieve with selective pores. From the structures of the various hydrocarbons depicted in Figure II-1, it may be seen that the normal paraffin (n-paraffin) has a much smaller maximum diameter (in a plane normal to the carbon-carbon bonds) than the other species present. The pore diameter of the sieve is selected so that the n-paraffins can pass through the pores and into the cavities within the crystal structure, while the other species are excluded because of their size; see Figure II-2.

The non-adsorbed branched and cyclic paraffins, referred to as non-normals, may become entrained in the large sieve voids but are easily removed by washing the adsorbent with a non-desorptive hydrocarbon, such as iso-octane (iC_8). The iC_8 effectively flushes away the non-normals while leaving the adsorbed n-paraffins intact.

To displace the n-paraffins from the selective pores, a short linear chained paraffin such as normal pentane (nC_5) must be used. By virtue of its short length and small diameter, the nC_5 is extremely mobile and can easily pass into the selective pores of the sieve and displace the larger C_{10} - C_{14} n-paraffins.

D. ADSORPTION vs. DISTILLATION

When the adsorbent is immersed in a liquid mixture of paraffins, the pores fill with liquid. After an equilibrium has been established the composition of the liquid in the pores will be different from that of the bulk liquid surrounding the adsorbent particles. The compositions are related by enrichment factor. By definition, the adsorbent is more selective for the component that is more concentrated in the pores than in the bulk liquid.

The equilibrium constant K_i describes the relationship between the concentrations of a component "i" in the bulk liquid and the adsorbed (or vapor) phase. Refer to Figure II-3.

For a vapor-liquid phase equilibrium:

$$K_i = \frac{y_i}{x_i}$$

where:

y_i is the concentration of component "i" in the vapor phase

x_i is the concentration of component "i" in the liquid phase

In an analogous manner, for an adsorption system such as Molex:

$$K_i = \frac{z_i}{x_i}$$

where:

z_i is the concentration of component "i" in the adsorbed phase

x_i is the concentration of component "i" in the bulk liquid phase

For vapor-liquid distillation operations, the enrichment factor is the relative volatility, α , which is defined as the ratio of the K 's for two different components **a** and **b** (refer to Figure II-3):

$$K_a = \frac{y_a}{x_a} \quad \text{and} \quad K_b = \frac{y_b}{x_b}$$

$$\alpha = \frac{K_a}{K_b} = \frac{y_a / x_a}{y_b / x_b}$$

For the adsorption system in Molex, the enrichment factor or selectivity, β (beta), is similarly defined as the ratio of the K's (refer to Figure II-4):

$$K_a = \frac{z_a}{x_a} \quad \text{and} \quad K_b = \frac{z_b}{x_b}$$

$$\beta = \frac{K_a}{K_b} = \frac{z_a / x_a}{z_b / x_b}$$

For the Molex adsorption system the enrichment factor, or selectivity, between normal paraffin and the other non-normal paraffins is very high.

To separate normal paraffin from non-normal paraffins it is possible to use either fractional distillation or a series of countercurrent adsorption stages as in Molex. The enrichment factors for each system are as follows:

System	Enrichment Factors	Value
Distillation	α	1
Molex	β	>10

Since α , or relative volatility, is close to 1.0, separation by distillation is extremely difficult. A relative volatility of 1.0 means that the component has no tendency to concentrate in either the liquid or vapor phases. The closer α is to 1, the more trays, reflux, and heat input will be required to effect the separation. This represents an increase of both capital and operating costs. The distillation of normal paraffin from ethylbenzene would require uneconomically large equipment and high energy consumption.

E. THEORETICAL STAGES

To illustrate the importance of the selectivity, β , consider one equilibrium stage in which the normal paraffin content of the liquid is 25%. The composition of the liquid in the adsorbent's micropores is calculated as follows:

$$\beta = \frac{z_a * x_b}{z_b * x_a}$$

where:

z_a = normal paraffin concentration in the liquid present in the adsorbent micropores

x_a = normal paraffin concentration in the liquid present in the bulk liquid phase

z_b = concentration of undesired components in the adsorbent micropores

x_b = concentration of undesired components in the bulk liquid phase

$$z_a + z_b = 1.0$$

$$x_a + x_b = 1.0$$

given:

$$x_a = 0.25 \text{ and } x_b = 0.75$$

$$10 = \frac{z_a * 0.75}{(1 - z_a) * 0.25}$$

Similarly, the concentration of the liquid after further stages of separation can be calculated. The results are as follows:

Ideal Stages	Wt% Normal Paraffin
1	92.4%
2	96.0%
3	97.6%
4	98.5%
5	99.0%

These are the normal paraffin concentrations calculated for an ideal system. A larger number of stages would be required in a real system due to inefficiencies.

F. MOVING BED SYSTEM

When distillation was first applied to separation problems, it was done in a batchwise manner. Liquid was charged to a vessel and components were sequentially boiled out of the liquid and collected as separate products. Continuous distillation works easily as a result of the countercurrent flow that naturally develops in a distillation column. The liquid that flows down a column gradually become concentrated in heavy components as the light components enter the rising phase. In distillation, at each theoretical stage the two phases are brought into contact with each other and then are separated to be sent to the next stages. The vapor and liquid mix on a tray and the vapor escapes to the next stage above and the liquid passes to the next stage below. The challenge was to accomplish this with a solid adsorbent.

A sketch of a hypothetical moving-bed system is shown in Figure II-5. The adsorbent circulates continuously as a dense bed, in a closed cycle, and moves up the adsorbent chamber from bottom to top. Liquid streams flow down through the bed, countercurrent to the solid. For simplicity, the feed is assumed to be a binary

mixture of A and B, with component A being more selectively adsorbed relative to B. Feed is introduced to the bed as shown.

Desorbent, D, is introduced to the moving bed model at a point above the extract location. The desorbent is a liquid of a higher boiling point than the feed components and having a high adsorbent selectivity. This means that the desorbent can desorb the feed components from the adsorbent and in downstream fractionation can be separated from the feed components.

Raffinate product, consisting of the less strongly adsorbed component B mixed with desorbent is withdrawn as shown from a position below the feed entry. Extract product, consisting of the more strongly adsorbed component A mixed with desorbent is withdrawn from the chamber above the feed point. Only a portion of the flowing liquid in the bed is withdrawn, and the remainder continues to flow in a closed loop.

The positions of introduction and withdrawal of net streams divide the bed into four main zones, each of which performs a different function. The zones are described below:

Zone 1 (I) is defined as the section between the feed and raffinate points. The primary function of Zone 1 is to adsorb A from the liquid. The solid entering the bottom of this zone carries only B and D in its pores. As the liquid stream flows downward, countercurrent to this solid, component A is transferred from the liquid stream into the pores of the solid. At the same time, some of components B and D are desorbed from the pores due to concentration driving forces and selectivity differences. This means it is transferred from the pores to the liquid stream making room for A in the pores. Zone 1 is the zone in which normal paraffin is adsorbed from the liquid phase. Thus, it is referred to as the adsorption zone.

Zone 2 (II) is defined as the section between the extract and feed points. The primary function of Zone 2 is to remove B from the pores of the solid. When the solid arrives at the feed point, the pores will contain the quantity of A that was adsorbed in Zone 1. However, the pores will also contain a large quantity of B,

because the solid does not make a perfect separation. The liquid entering the top of Zone 2 contains no B – only A and D. As the solid moves upward, B is gradually displaced from the pores and is replaced by A and D. Thus, when the solid arrives at the top of Zone 2, the pores will contain only A and D. By proper regulation of the liquid rate in Zone 2, B can be desorbed almost completely from the pores. This can be done without simultaneously desorbing all of A, because A is more strongly adsorbed than B. Zone 2 is the zone in which normal paraffin is purified. Thus, it is referred to as the purification zone.

Zone 3 (III) is defined as the section between the desorbent and extract points. The function of this zone is to desorb A from the pores. The solid entering the bottom of the zone carries A and D in the pores; the liquid entering the top of the zone consists of pure D. As the solid rises, A in the pores is displaced by D. Zone 3 is the zone in which normal paraffin is desorbed from the solid. Thus, it is referred to as the desorption zone.

Zone 4 (IV) is defined as the section between the raffinate and desorbent points. The purpose of Zone 4 is to keep component B, which is at the bottom of Zone 1, from entering Zone 4 and flowing through Zone 4 to Zone 3 where it can contaminate the extract material. If the flow rate is set so that desorbent flows up in Zone 4, raffinate material would be prevented from gaining access to Zone 3 where it would contaminate the purified extract stream. This means that the main function of Zone 4 is to separate Zone 3 from Zone 1 and as a result it is referred to as the buffer zone.

The liquid-phase composition profile shown in Figure II-5 serves to illustrate the above actions taking place.

For the liquid-solid system each stage has to mix the solid with the liquid and subsequently separate the two phases after equilibrium is reached. The liquid and solid can then be passed on to the next stages. To make another comparison with distillation, the liquid could be seen as passing up through a column with trays that the solid is passing down through. At each tray the solid would be pushed across. The solid would fall to the tray below and the liquid would pass to the tray above.

G. PROBLEMS WITH CIRCULATING SOLIDS

Literature indicates one option that was considered was to use a series of fluidized beds. In this arrangement the liquid overflows from one stage to the next and the solids are transferred as a dense fluidized phase from one stage to the next. This method of liquid/solid countercurrent contact proved inefficient. It was very difficult to maintain even flow distribution of the liquid and solid phases. The performance of such a system is seriously impaired by non-uniform flow of either phase. The performance of such a system is also reduced by the efficiency of mass transfer because the number of theoretical stages cannot exceed the relatively small number of physical stages that would need to be built. Such a unit would also be very large to accommodate the fluidized beds. Erosion would also prove to be a significant problem.

In contrast to the fluidized bed model plug flow through a series of dense fixed beds as is practiced in liquid chromatography provides hundreds of theoretical stages in a bed of modest length. To physically move such a solid bed directly as with a screw conveyor would have erosion and attrition problems exceeding those of the fluidized bed system. Attempts to move such densely packed beds by mechanically conveying adsorbents in screen buckets through a flowing liquid are also documented in literature. These attempts were plagued by bypassing of flow around the individual beds due to difficulties in sealing the spaces between the beds. The mechanical systems were also prone to failure for a number of reasons.

In view of these difficulties, Molex was developed using a simulated moving bed concept which retains the advantage of continuous countercurrent flow of dense solid and liquid without introducing the problems associated with the actual circulation of the solids.

In a system in which there is actual circulation of solid adsorbent, the adsorbent moves continuously in a closed circuit past the fixed points at which feed and desorbent are added and extract and raffinate are withdrawn. The same effect can be achieved by keeping the adsorbent bed stationary and periodically moving the points at which the liquid streams are added and withdrawn from the chamber.

Changes in the position at which the liquid is added or withdrawn from the chamber follow the direction of liquid circulation through the chamber and simulate the circulation of solids in the opposite direction.

For ideal simulation of countercurrent flow the liquid addition and withdrawal points should be moved continuously by infinitely small distances. This is technically unfeasible because of the impossibility of having an infinite number of liquid addition and withdrawal points on the adsorbent chambers. It was found that countercurrent flow could be approximated well enough by providing a number of discrete (24) separate beds of adsorbent each of which is provided with its own liquid addition or withdrawal line. The addition or withdrawal points are then switched periodically at a precise interval from one bed to the one below in a continuous and regular cycle. The four liquid lines are then effectively moved through the chamber in discrete steps maintaining a fixed number of adsorbent beds between the net streams.

Such a system is illustrated in Figure II-6. The adsorbent is held stationary in a series of beds of equal height and volume. A pump circulates liquid from the bottom to the top of the chamber.

A device is provided for switching the net liquid streams from one point to the next on the chambers. This specialized device, developed by UOP, was a key factor in making the Sorbex processes so successful. It is known as the **rotary valve** and it functions using the same principle as a multiport stopcock valve, directing the net streams to different bed feed lines in a fixed sequence.

H. SIMULATED SOLIDS CIRCULATION

In the moving-bed system of Figure II-5, solid is moving continuously, in a closed circuit, past fixed points of introduction and withdrawal of liquid. The same process results can be obtained by holding the bed stationary and periodically moving the positions at which the various streams enter and leave. A shift in the positions of the liquid feed and withdrawal, in the direction of fluid flow through the bed, simulates the movement of solid in the opposite direction.

It is, of course, impossible to move the liquid feed and withdrawal positions continuously. However, approximately the same effect can be produced by providing multiple adsorbent beds and liquid-access lines to the chamber, and sequentially switching each net stream from one bed to the next bed.

Therefore, the major liquid access positions can be moved around the bed in discreet steps, always maintaining the same distance between the various net streams.

A model of this concept is shown in Figure II-6. Here, the adsorbent is maintained as a stationary bed. A liquid circulating pump is provided to pump liquid from the bottom to the top of the adsorbent chamber. A fluid-directing device known as a "rotary valve" is provided. This functions on the same principle as a multiport stopcock, directing each of several streams to different lines. At the right-hand of the valve, the net streams to and from the process are continuously fed and withdrawn. At the left-hand side of the valve, a number of lines are connected, which terminate in distributors within the adsorbent bed.

At any particular moment, only four lines between the rotary valve and the adsorbent chamber are active. In this example, Figure II-6 shows the flows at a time when lines 2, 7, 16 and 23 are active. When the rotating element of the rotary valve is moved to its next position, each net flow is transferred to the next adjacent line. Thus, desorbent will enter line 3 instead of line 2, extract will be drawn from 8 instead of 7, feed will enter 17 instead of 16, and raffinate will be drawn from 24 instead of 23.

From the moving-bed operation of Figure II-5, it can be seen that the liquid flow rate in each of the five zones is different, because of the addition or withdrawal of the various net streams. In the simulated moving-bed of Figure II-6, the liquid rate is controlled by the circulating pump. This is commonly called the pumparound pump. At the position shown in Figure II-6, the pump is between the raffinate and desorbent ports and, therefore, should be pumping at a rate appropriate for Zone IV. However, after the next two switches in position of the rotary valve, the pump will be between the feed and raffinate ports and should, therefore, be pumping at a rate appropriate for Zone I. Since these rates are different, a sequential flow control system must be employed. The requirements for the pumparound controller will be described later. Stated briefly, the pumparound flow controller must be programmed to pump at different rates. The control setpoint will be altered each time that a net stream is transferred from line 24 to line 1. See Figure II-7 for an illustration of how the pumparound pump flow changes with different rotary valve positions.

To complete the simulation, it is necessary that the liquid flow rate relative to the solid must be the same in both the moving-bed and simulated-moving-bed operations. Since the solid is physically stationary in the simulated-moving-bed operation, the liquid velocity relative to the vessel wall must be higher than in an actual moving-bed operation.

A minor complication results from the fact that each line between the rotary valve and the bed is employed successively in carrying feed and zone flush to the bed. To avoid having this feed carried out with the extract (causing the product purity to drop), it is necessary to flush the feed from each line before the line is used for injecting the zone flush. Flushing is accomplished with the zone flush liquid, and this ability is included in the mechanical construction of the rotary valve and pumparound flow controller.

Likewise, a flush out line is provided to flush the lines of Extract before the Desorbent enters. If left in the line, some of the Extract would flow with the Desorbent, through Zone IV, and out the Raffinate, causing a loss in product recovery. The flush out line is routed through the rotary valve to the extract column so the n-paraffins can be recovered.

I. DEFINITION OF THE ADSORBENT BEDS

The adsorbent beds are composed of adsorbent particles described earlier. Each piece of adsorbent has a diameter of approximately 0.4-0.6 mm (0.16-0.23 in). Each piece of adsorbent is composed of crystals of zeolite. The zeolite crystals themselves have the consistency of fine sand prior to the forming of the adsorbent particle. The zeolite crystals are mixed with a clay binder and formed into spheres. The spheres are dried to give the adsorbed particles sufficient strength so that they can be handled easily. If the zeolite was not formed into these larger particles, a very low flow rate would produce a very high pressure drop that would force the use of specialized equipment.

As can be seen in Figure II-8, there are interstitial voids between the adsorbent particles. In addition of the interstitial voids between the adsorbent particles, the porous structure of the adsorbent also has a significant voidage. The only openings in the adsorbent that contribute to the separation are the passages through the actual zeolite crystals. This volume in the zeolite crystals is referred to as the selective volume of the adsorbent.

Figure II-9 illustrates the volumes of the adsorbent bed. A container of 1000 cc. is filled with adsorbent particles. Feed normal paraffin are introduced to the vessel until all of the space is occupied by either hydrocarbon or adsorbent. In the next step of the experiment the vessel is drained. The liquid drained is found to be 539 cc. This is taken to be the volume of all of the bed interstitial voids and the porous structure of the zeolite particles that does not adsorb normal paraffin. We say the bed has a non-selective volume of 53.9%.

In the next step of the demonstration we heat the adsorbent bed and drive off the normal paraffin that were adsorbed. We find 111 cc. are collected. We say the selective volume of the adsorbent is 11.1%. The remaining 35% of the volume is assumed to be occupied by the solid of the adsorbent that the hydrocarbon cannot displace.

J. NON-SELECTIVE VOLUME CIRCULATION

The volumes we have defined in the adsorbent bed become significant when we examine what happens when we simulate the motion of the adsorbent bed.

Figure II-10 shows our adsorbent bed that we are going to use to discuss the simulated motion. Actually it is a rectangle where we have defined the amount of adsorbed phase, V_a (selective volume), and the bulk liquid phase, V_w (non-selective volume). We wish to move the solid, actually simulate the motion of the solid, in the upward direction. Let's say the feed is located at the top of the bed at time = 0. After an amount of time, T_{seconds} , we stop the rotary valve and the feed is moved to the bottom of the bed. From the point of view of the feed the relative position of the adsorbent is higher at time = T . In this way the adsorbent motion is simulated in the upward direction relative to the feed point. From the point of view of the feed, it does not matter if the adsorbent actually moves up or not the bed of adsorbent moves from $T = 0$ to $T = T$ from below the feed to above the feed.

One complication introduced by the simulated motion is the movement of the bulk liquid in the bed. As we simulate the motion of V_a in the upward direction we also simulate the motion of V_w in the upward direction. Since we wish to set up a countercurrent process, we want the liquid to move down relative to the feed point as the adsorbent moves up. (The exception is in Zone IV which will be commented on later.) In order to have liquid flow down through the bed when compared to the feed point, we need to have an actual liquid flow equal to the flow of bulk liquid we are simulating in the upward direction in addition to the liquid flow we want relative to the feed point. A numerical example should make the situation clear.

Let the adsorbed phase of the bed in Figure II-10 be equal to 14.5 m^3 , $V_a = 14.5 \text{ m}^3$. Let the bulk liquid phase be equal to 59.5 m^3 , $V_w = 59.5 \text{ m}^3$. At $t = 0 \text{ hr}$, the feed is at the top of the bed. At $t = 1 \text{ hr}$, we move the feed to the bottom of the bed. We say that the simulated adsorbent motion of flow is equal to $V_a/T = A = 14.5 \text{ m}^3/\text{hr}$. The simulated non-selective volume motion or flow is equal to $V_w/T = W = 59.5 \text{ m}^3/\text{hr}$. This flow, W , is cocurrent to the adsorbent flow A . For this example, let's say we want the countercurrent flow of liquid past the feed point equal to $10 \text{ m}^3/\text{hr}$. The

countercurrent flow is referred to as the net flow, L_i' . To determine the actual flow through the adsorbent chamber, L_i , we add the net flow to the simulated non-selective volume flow, $L_i = L_i' + W$. As you can see, the "net" flow is equal to the actual gross flow minus the non-selective flow, $L_i' = L_i - W$. The "i" is used to denote the specific zone flow we are discussing. The net flow for Zone III is L_3' . Usually we do not discuss a single bed of the adsorbent chambers. The adsorbent circulation or selective volume circulation rate is expressed in terms of the total selective volume of the adsorbent chamber and the time it takes for the feed point to make a complete cycle through all the bedlines. Actually we rarely discuss net flows. To put the performance of all the units of different throughputs on the same basis we typically discuss the ratios of the controlled flows: A/F_a , L_2'/A , L_3'/A , etc. See Section VIII on **Normal Operations**.

Since the understanding of the relative flow of liquid is so important, two more examples of relative flow are presented.

K. ESCALATOR

Figures II-11 through II-13 use a set of moving stairs to illustrate the concept. The moving stairs or "escalator" is used to make getting from one level to the other easier and faster. The intended method of using the stairs is to walk in the direction of the motion of the stairs so that your relative speed adds to that of the stairs to move you quickly in the direction that the stairs are moving. This would be cocurrent motion of the stairs and the person on them. What we need to visualize is a countercurrent motion, so the person on the stairs is actually trying to walk down as the stairs attempt to carry him up. We will allow the stairs motion in the upward direction to represent the flow of W in the upward direction and allow the person walking down to represent the liquid flow that is pumped in the downward direction.

In Figure II-11 the person walks down at the same rate that the stairs move up so the stationary observer sees no relative motion of the person on the stairs compared to his position. This case of the gross liquid flow L equaling the nonselective flow W does not normally exist in the Molex Unit.

Figure II-12 illustrates what happens if the person walks faster than the stairs are moving upward. Although he is walking very fast to the stationary observer the person on the stairs moves slowly down the stairs. This example is what occurs in all but zone IV of the chamber. For this $L > W$ and L_i' is said to be positive.

The final case illustrated by Figure II-13 has the person on the stairs walking slower than the stairs are moving upward. Although the person on the stairs is actually walking in the downward direction the stationary observer sees him moving slowly upward. This is the case that occurs in Zone IV. $L_i < W$ and L_i' is said to be negative because the flow appears to move up the adsorbent chamber.

L. DYE SPOT

The analogy with the stairs is good for getting an understanding of relative motion of L and W , but it is not exactly how the process works. Figures II-14 through II-16 demonstrate how the system would look if you could see inside the chamber during operation. In these figures you will see a diagram of the chamber with an eye looking into different bedlines. In all three figures the eye (observer) moves down the adsorbent chamber one bed at a time each T seconds. From the observer's viewpoint if his position is considered stationary the adsorbent in the chamber would appear to move up. After a given time T the adsorbent that was directly in front of him will now be one bed above him so the adsorbent has moved up. This how we simulate the moving adsorbent bed. It is not important how the distance is increased between the observer and the adsorbent bed, if the observer moves down or the bed moves up, at the end of a step the distance and the position is the same between the observer and the adsorbent.

The foregoing analysis of the relative motion of the adsorbent is complicated by the fact that the adsorbent bed has a large amount of open space in the bed (53.9%). By the same analysis given above, the distance between the observer and the liquid in the voids of the bed also increases so it appears that the movement of the liquid in the adsorbent moves in the upward direction. The movement of the liquid associated in with the bed in the upward direction is what we define as the flow W .

The figures demonstrate the different possibilities of the relative quantity of flow between the gross flow rate that is pumped down the chamber (L) and liquid flowing up the adsorbent chamber as seen from the observer's viewpoint (W).

In Figure II-14 the rate at which liquid is pumped down the chamber is equal to the apparent rate the liquid carried by the adsorbent up the chamber. Or in other words the flow of liquid down the chamber is sufficient to keep pace with the observer as he moves down the chamber. This is demonstrated by the dye spot that is injected into the chamber at $t = 0$. Each time the observer moves the dye appears to be directly in front of him so he sees no net movement of liquid in the chamber. For this case $L = W$ and $L' = 0$.

In Figure II-15 the rate at which liquid is pumped down the chamber is higher than the apparent rate the liquid carried by the adsorbent up the chamber. Or in other words the flow of liquid down the chamber moves faster than the observer as he moves down the chamber. This is demonstrated by the dye spot that is injected into the chamber at $t=0$. Each time the observer moves the dye appears to move to a point farther below the observer and there appears to be a net flow of liquid down and away from the observer. For this case $L > W$ and there is a net flow of liquid L' . We say L' is positive since by definition $L' = L - W$.

In Figure II-16 the rate at which liquid is pumped down the chamber is lower than the apparent rate the liquid carried by the adsorbent up the chamber. Or in other words the flow of liquid down the chamber moves slower than the observer as he moves down the chamber. This is demonstrated by the dye spot that is injected into the chamber at $t = 0$. Each time the observer moves the dye appears to move to a point farther above the observer and there appears to be a net flow of liquid up and away from the observer. For this case $L < W$ and there is a net flow of liquid L' . We say L' is negative since by definition $L' = L - W$.

M. LINE FLUSH

A minor complication results from the fact that each line between the rotary valve and the bed is employed successively in carrying feed to the bed and carrying extract from it (see Figure II-7 at $T = 0$ and $T = 360$ seconds). To avoid extract contamination with feed components, it is necessary to flush the feed from each line before the line is used for withdrawing extract. Following a similar logic if the line used for extract is subsequently used for desorbent, the extract that remains in the bedline will be reintroduced to the adsorbent chamber at the point between Zones III and IV. Extracted feed components reintroduced to the adsorbent chamber at this point would follow the cocurrent flow through Zone IV to the raffinate point and contribute to a loss of recovery.

Figure II-17 illustrates the operation of the line flush and the secondary flush from the beginning of each step of the rotary valve to the end of the same step of the rotary valve. The rotary valve is stationary during this time. What we mean is that the rotary valve just finished moving for the top of the diagram and is about to move at the bottom of the diagram. Flushing a bedline is a lot like flushing a bottle. You fill the bottle with flushing material and then empty it out. If the bottle is not clean, you flush it again. We have found a bedline is most efficiently flushed about 2.2 times. How this is done is important to the efficiency of the unit.

The line flush is actually a loop from the adsorbent chamber through the rotary valve, the line flush pump, back through the rotary valve and back to the adsorbent chamber. The flow rate can be set very near one bedline volume during one position of the rotary valve such that desorbent moves out of the adsorbent chambers from a point near the desorbent inlet to displace the extract that remained in the bedline after the extract last used that line. The extract displaced from the one bedline is used to displace the feed left in the bedline that was last used by the feed into the adsorbent chamber near the feed point. The extract can remain in the bedline until the bedline is used by the secondary flush which is located one bed below the extract point. The second flush flow is also set near one bedline volume of the time the rotary valve is in position. This reintroduces the extract in the bedline to the adsorbent chamber one bed below the extract. This

secondary flush ensures the bedline is clean prior to use for extract. The small recycle of extract may also have a positive benefit to the performance of the unit but as a rule this should be minimized.

N. ADSORBENT CHAMBER HEAD FLUSHES

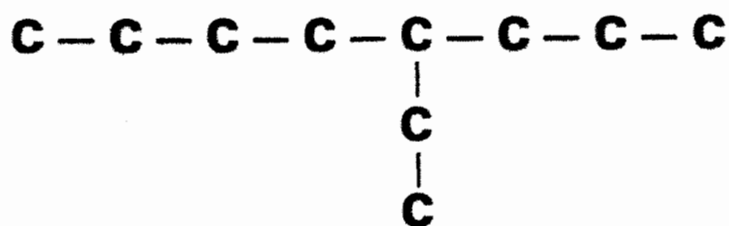
In order to maintain plug and uniform flow in the adsorbent chambers, the adsorbent needs to be loaded to a high efficiency with a constant cross sectional area. This precludes loading the semi elliptical heads of the adsorbent chambers. UOP would prefer an adsorbent chamber with flat heads but this is impractical at the diameters and pressures used for commercial units. The solution was to seal the top and bottom grids such that the heads of the adsorbent chambers are not part of the volume used in the adsorbent/liquid contact in the process. A small vent on the grids provides hydraulic communication between the heads and the adsorbent beds. The heads are kept full of clean desorbent that flows through the vents in the grids into the adsorbent chambers at a low flow rate. This prevents flow from the adsorbent chamber heads.

Figure II-1
Molex Feed Constituents

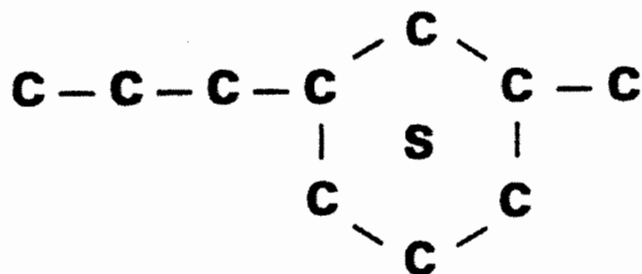
N-PARAFFIN



ISO-PARAFFIN



ALKYLNAPHTHENE



ALKYLAROMATIC

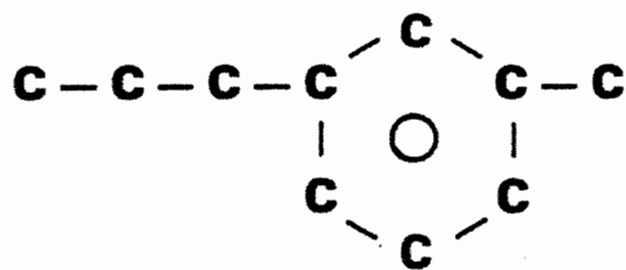


Figure II-2
Molex Adsorbent

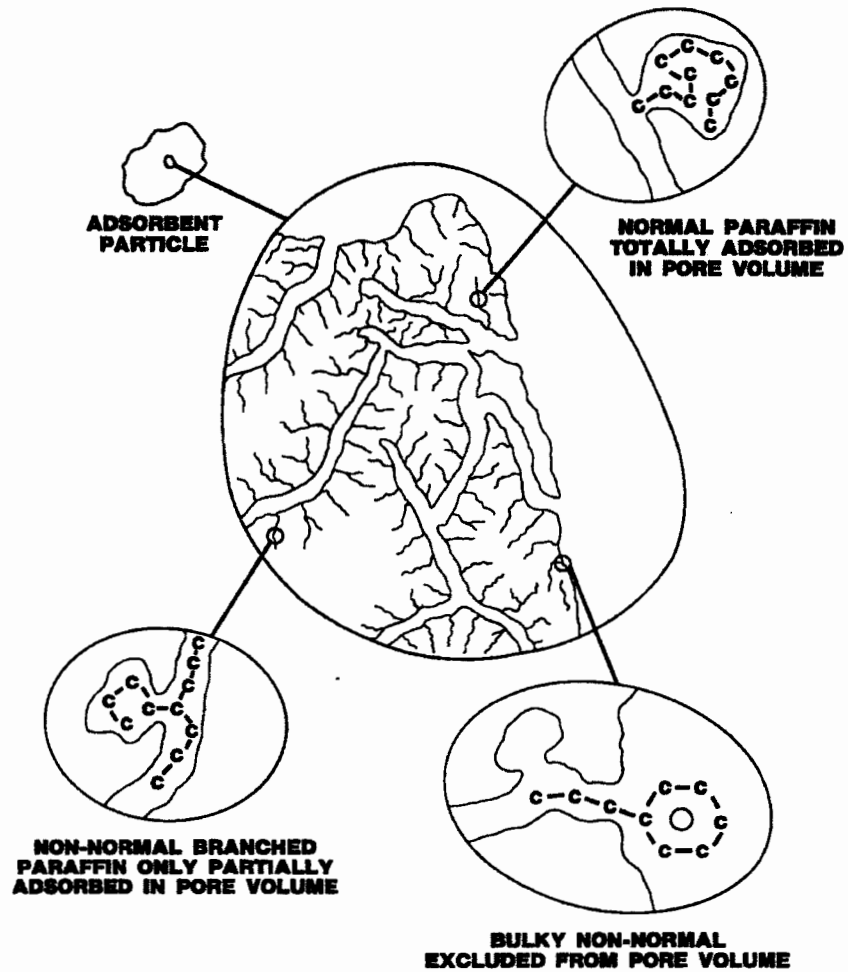


Figure II-3
Enrichment Factors

For Vapor - Liquid Systems:

$$K_a = Y_a / X_a$$

$$K_b = Y_b / X_b$$

where, for component "a":

K_a = equilibrium constant

Y_a = concentration in vapor phase

X_a = concentration in liquid phase

Enrichment Factor (Relative Volatility)

$$\alpha = \frac{K_a}{K_b} = \frac{Y_a * X_b}{X_a * Y_b}$$

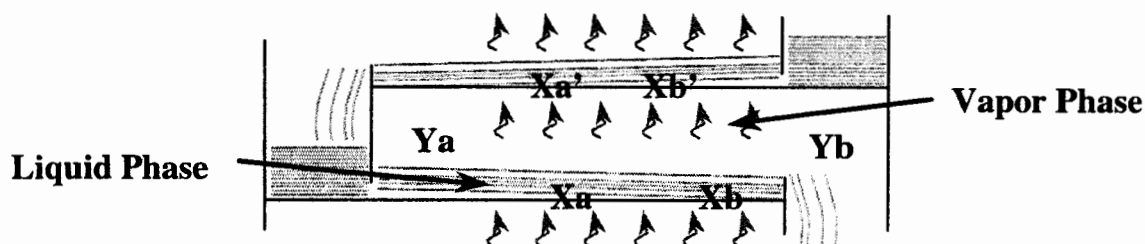


Figure II-4
Enrichment Factors

For Solid - Liquid Systems:

$$K_a = Z_a / X_a$$

$$K_b = Z_b / X_b$$

where, for component "a":

K_a = equilibrium constant

Z_a = concentration in adsorbed phase

X_a = concentration in liquid phase

Enrichment Factor (Selectivity)

$$\beta = \frac{K_a}{K_b} = \frac{Z_a * X_b}{X_a * Z_b}$$

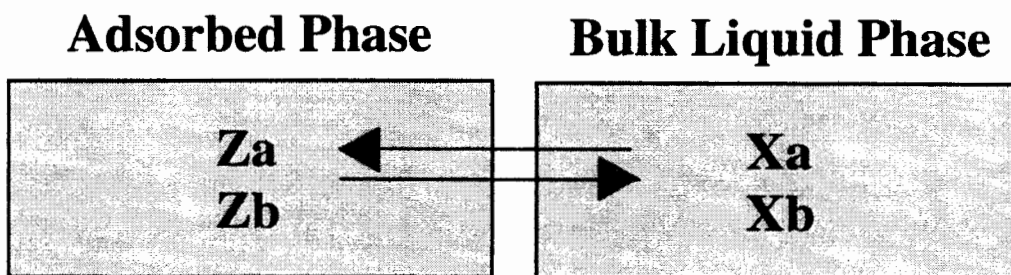


Figure II-5
Adsorptive Separation with Moving Bed

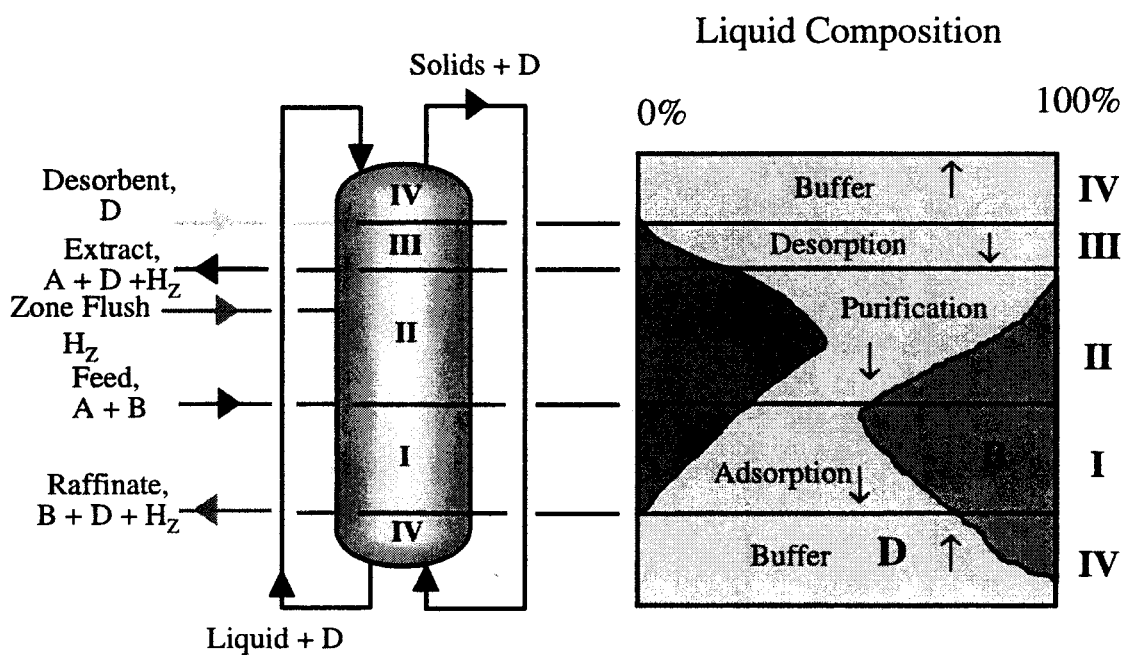


Figure II-6
Simulated Moving Bed Adsorptive Separation

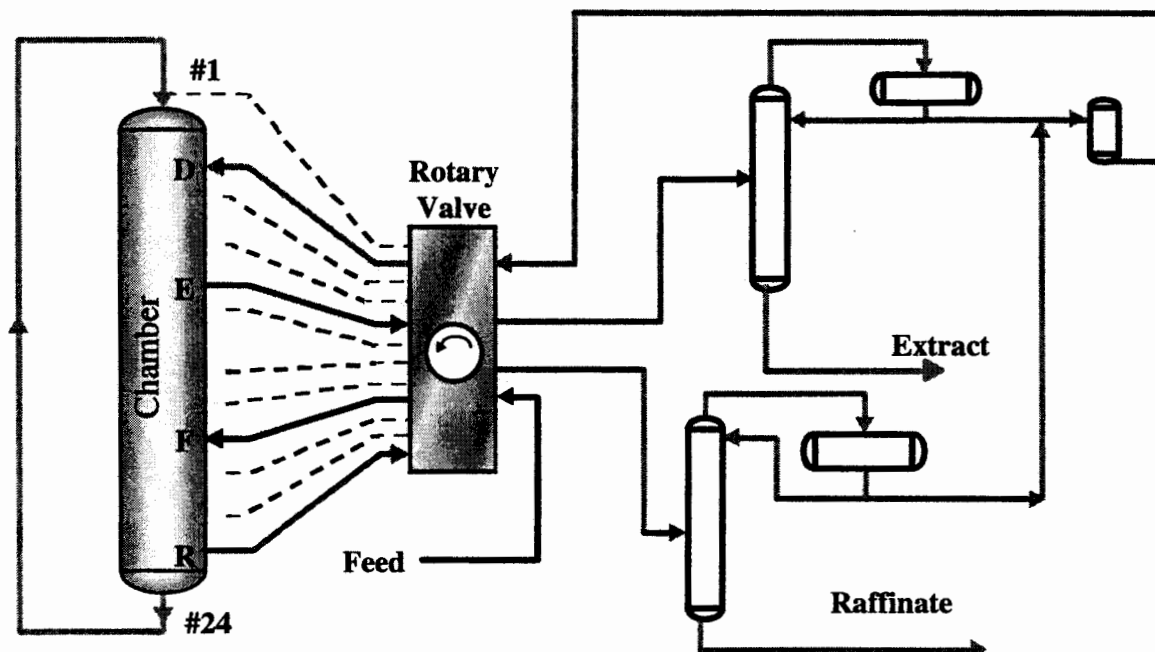


Figure II-7
Molex Simulated Moving Bed Changing PAP Flow

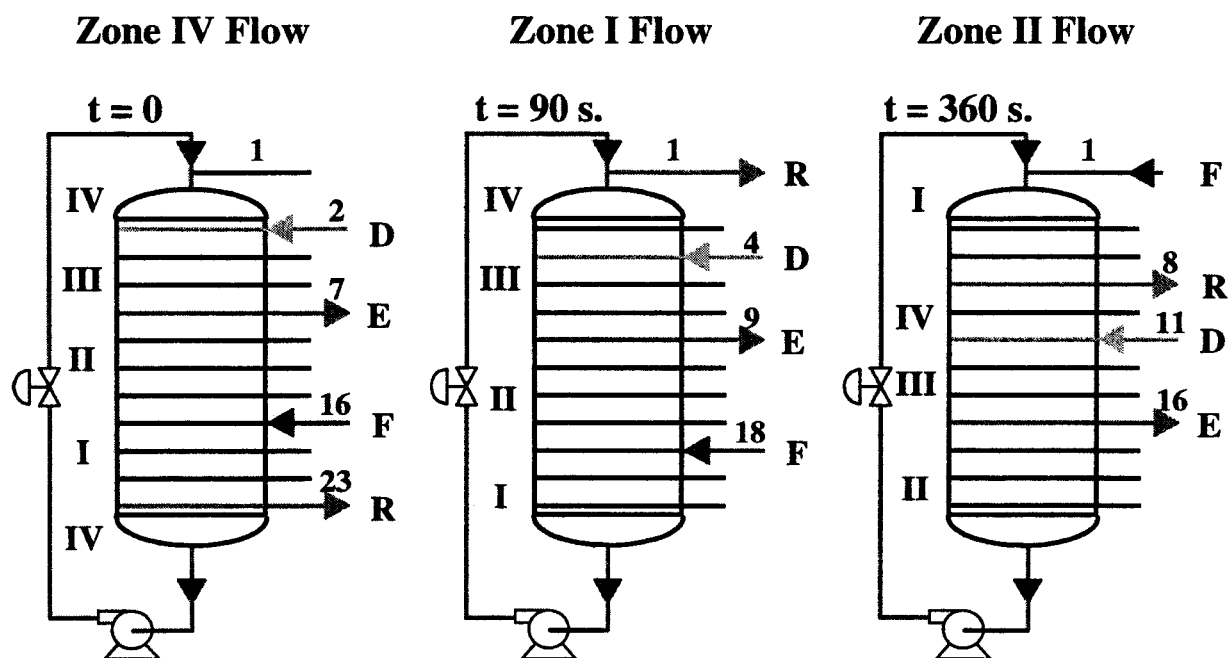


Figure II-8
Adsorbent Volumes

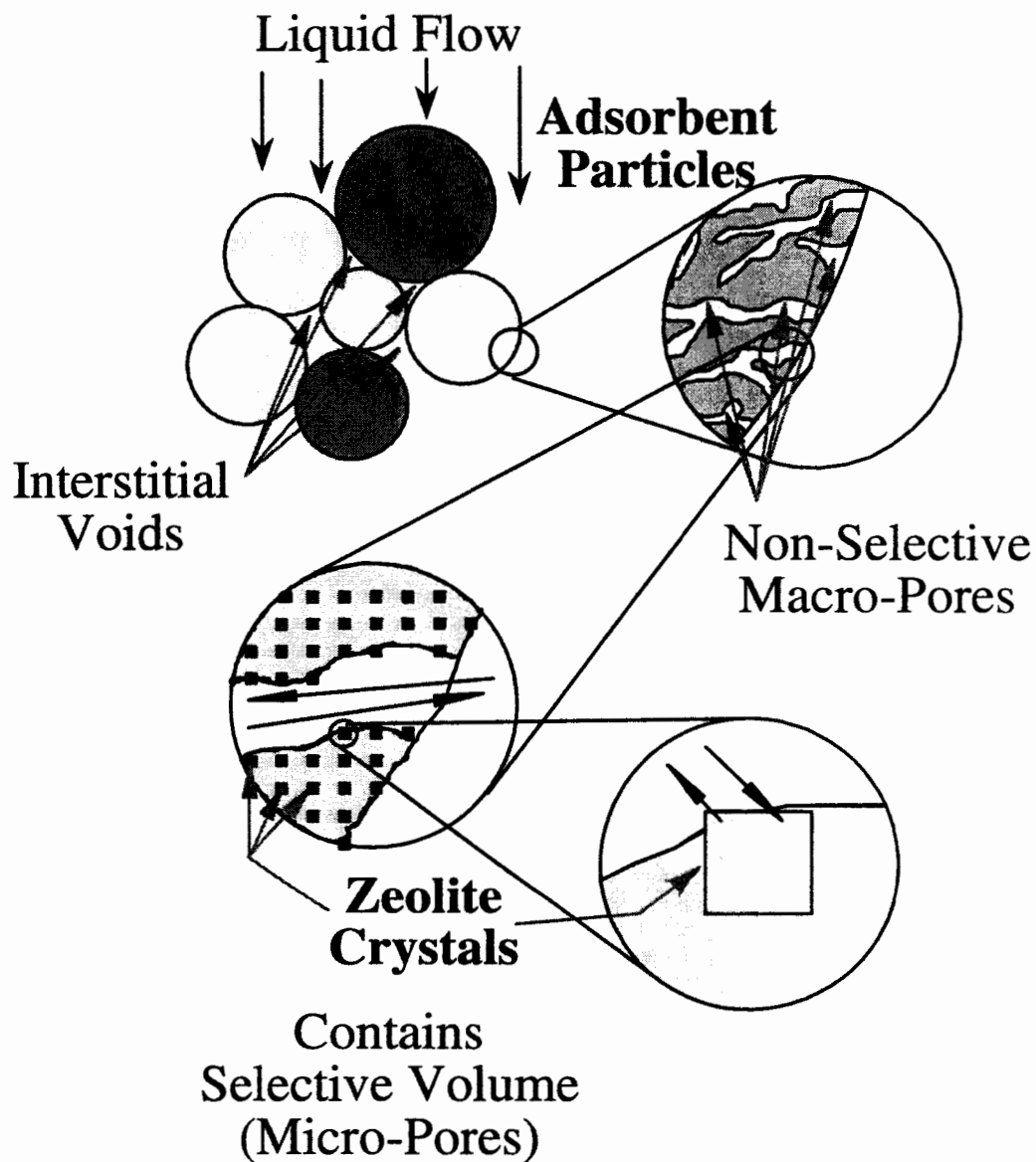


Figure II-9
Representation of Adsorbent Pore Volumes

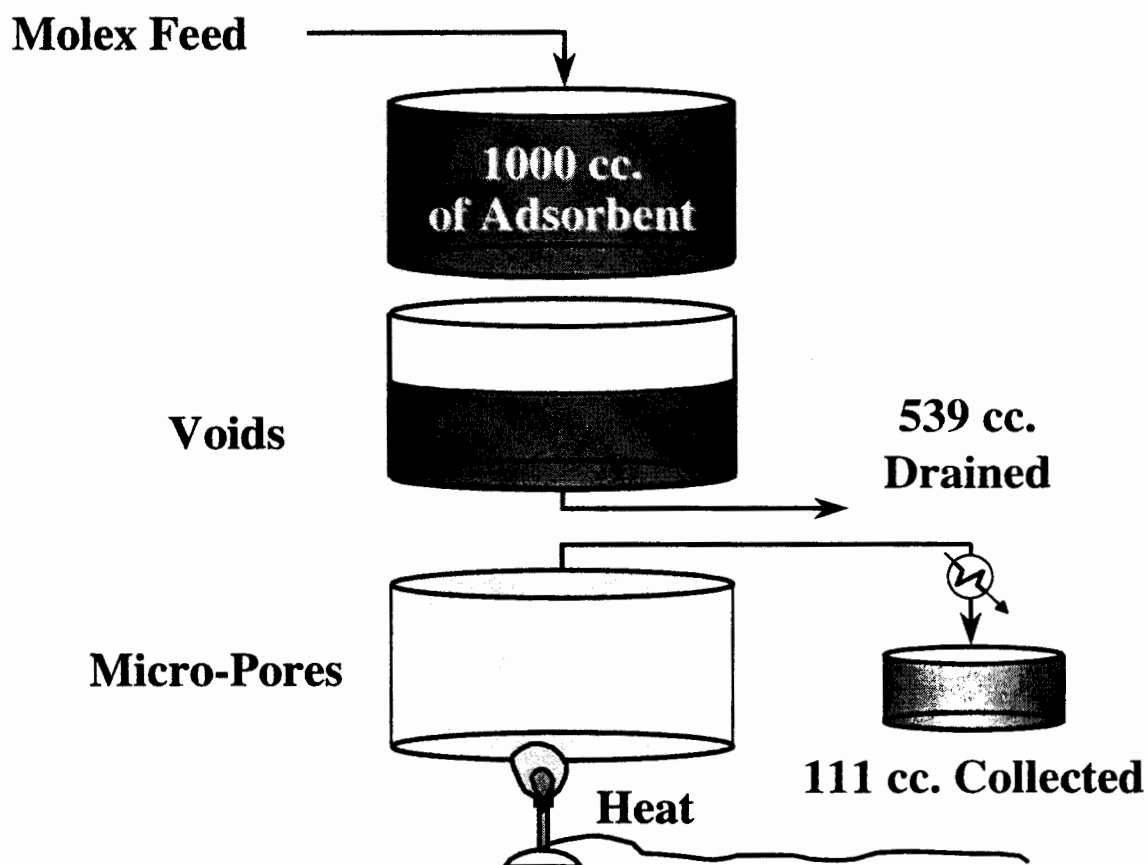


Figure II-10
Net Zone Flow Rate

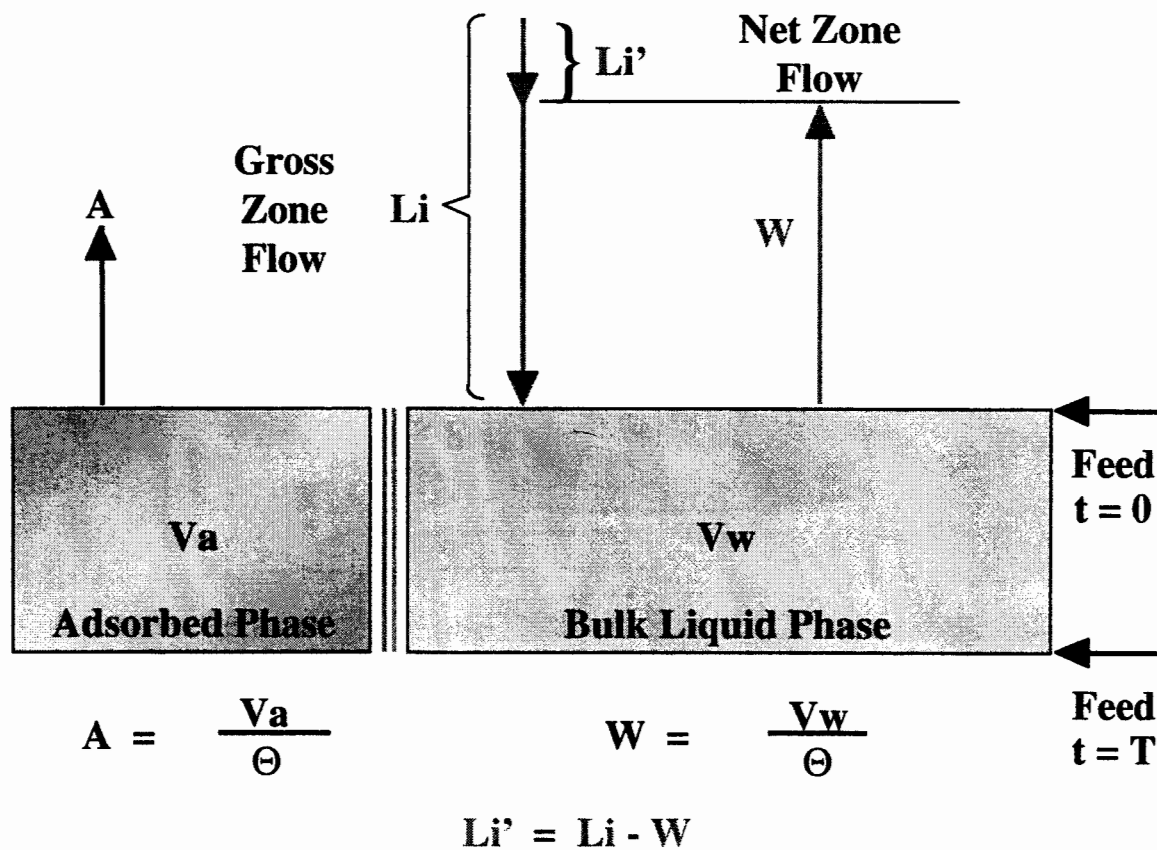
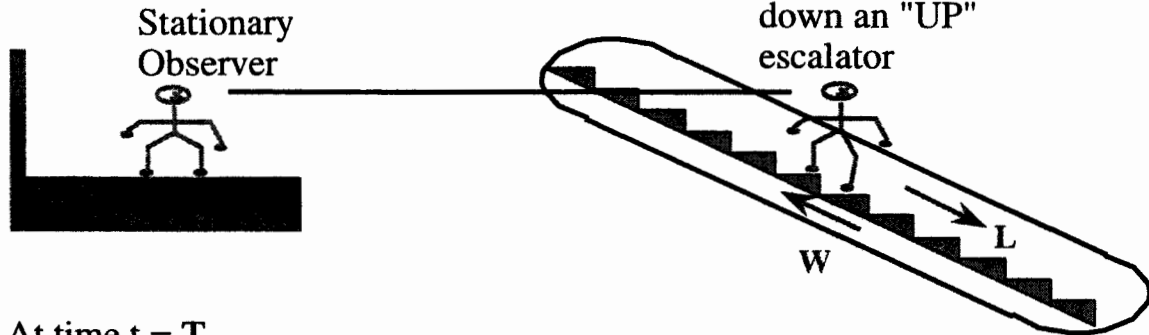
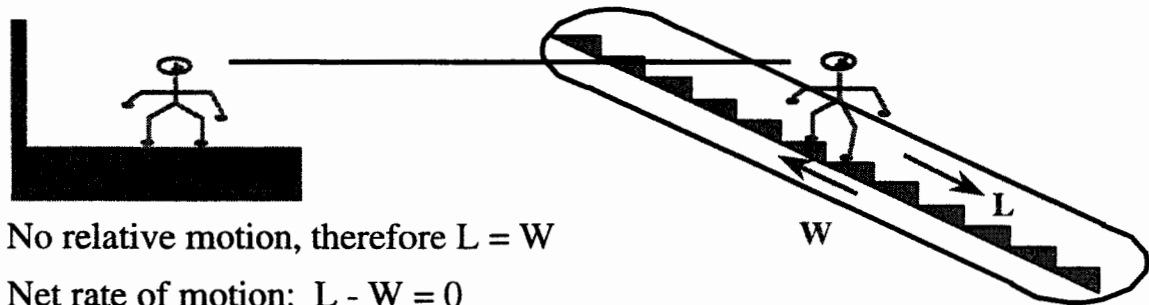


Figure II-11
Illustration of Net Zone Rates (Zero)

At initial time ($t = 0$)



At time $t = T$

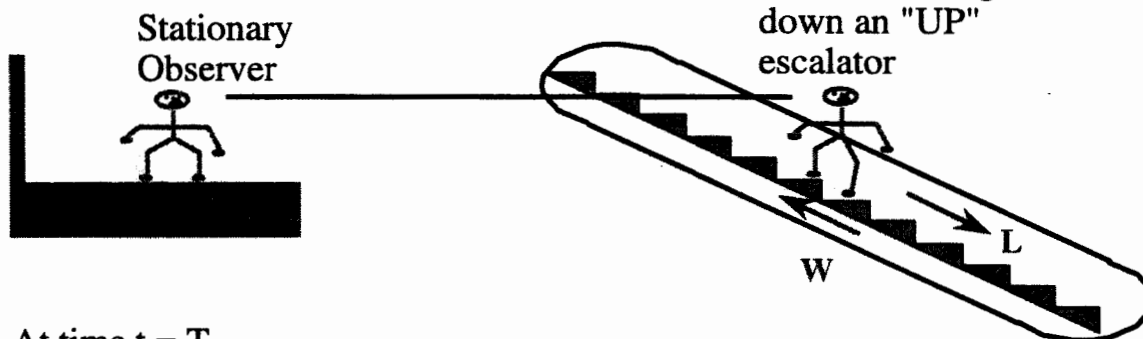


No relative motion, therefore $L = W$

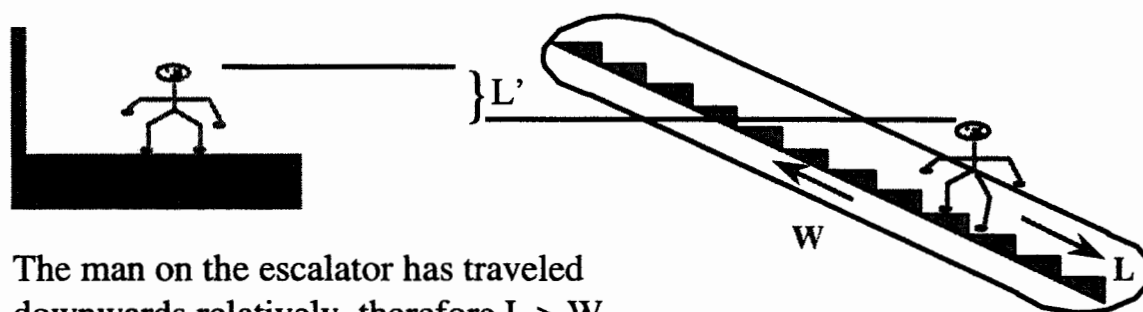
Net rate of motion: $L - W = 0$

Figure II-12
Illustration of Net Zone Rates (Positive)
Zones I, II, III

At initial time ($t = 0$)



At time $t = T$



The man on the escalator has traveled downwards relatively, therefore $L > W$
Net rate of motion: $L - W > 0$

Figure II-13
Illustration of Net Zone Rates (Negative)
Zones 2b and IV

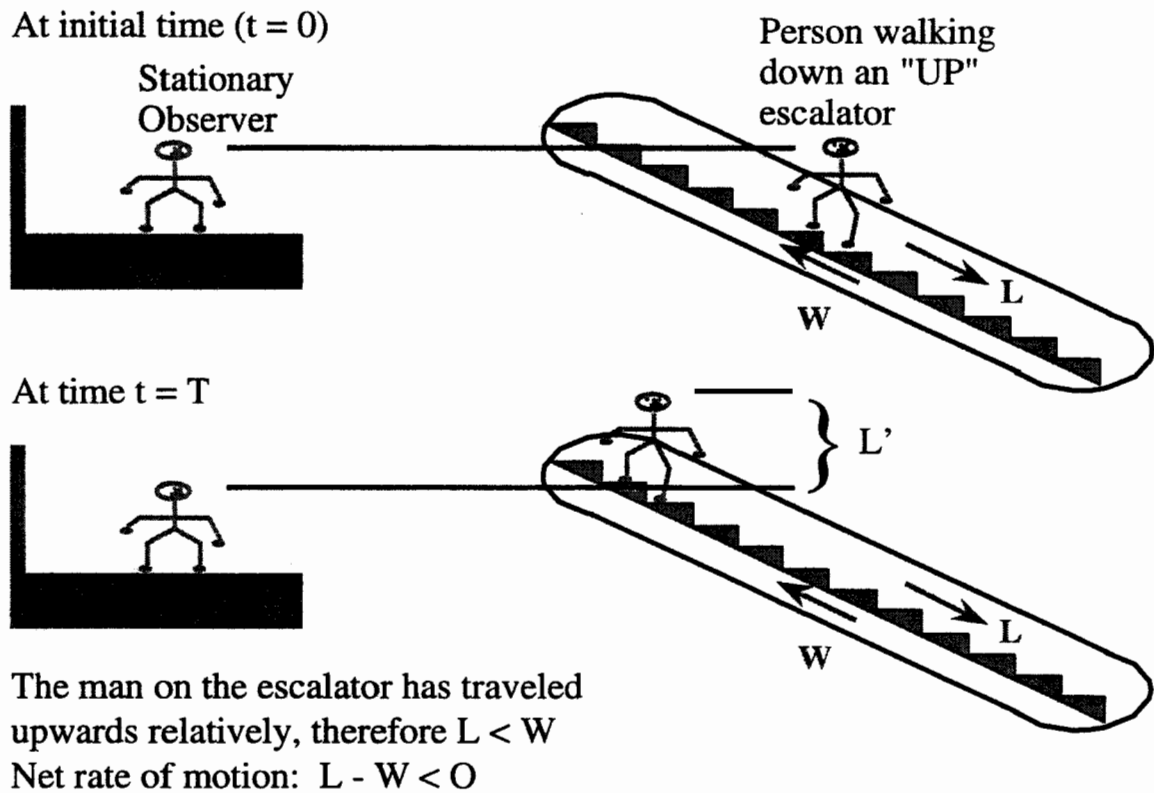


Figure II-14
Non-Selective Void Volume Circulation (Zero)

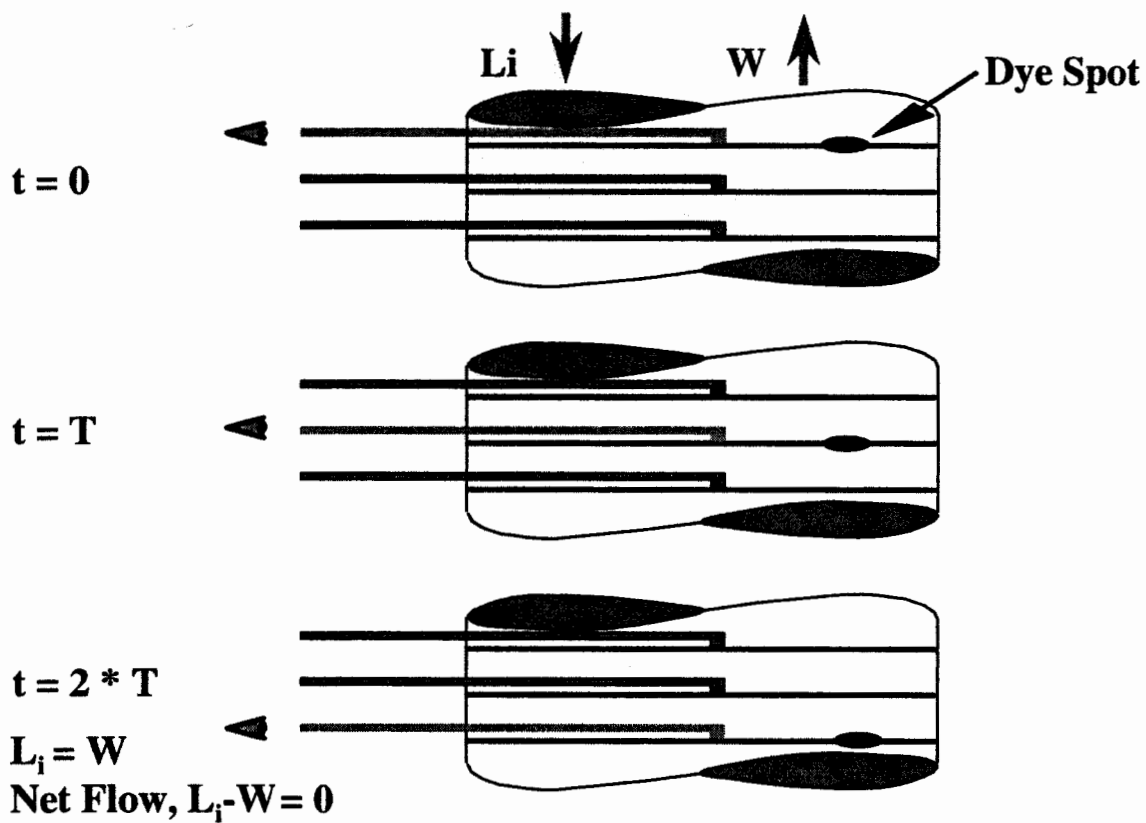


Figure II-15
Non-Selective Void Volume Circulation (Positive)
Zones I, II, III

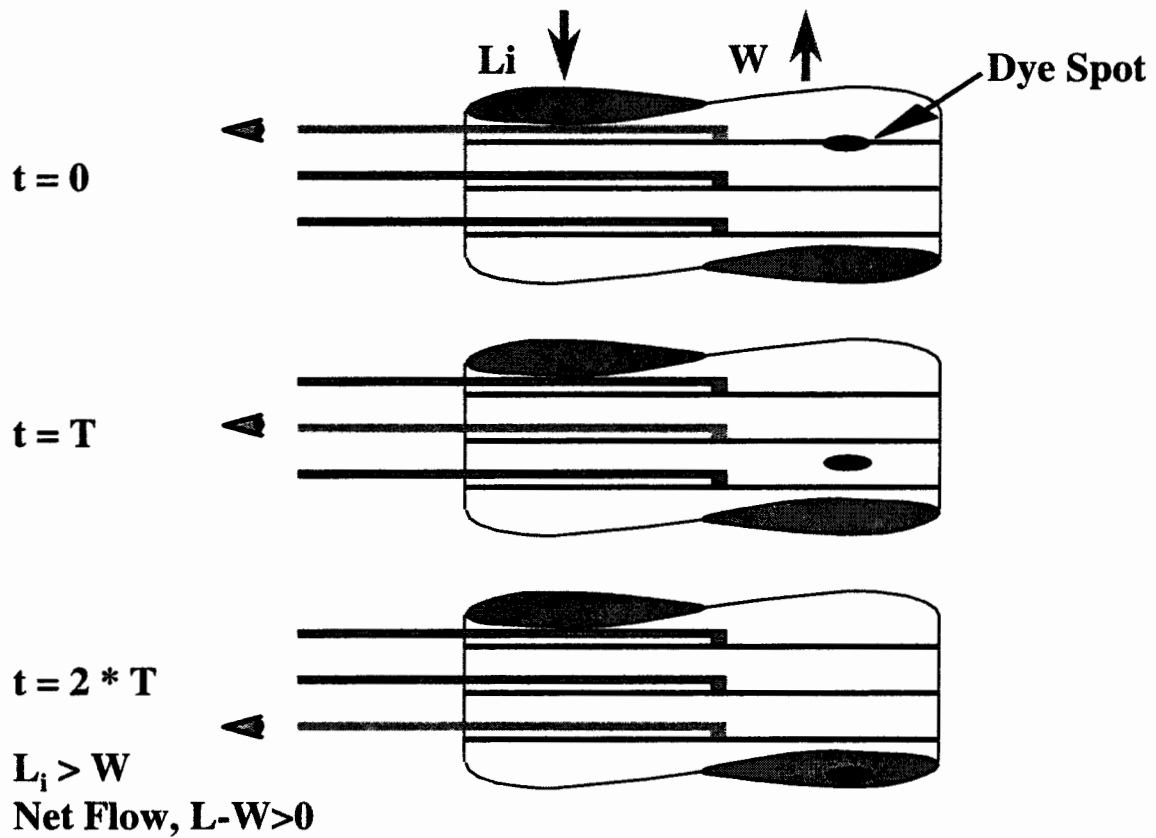


Figure II-16
Non-Selective Void Volume Circulation (Negative)
Zone 2B and IV

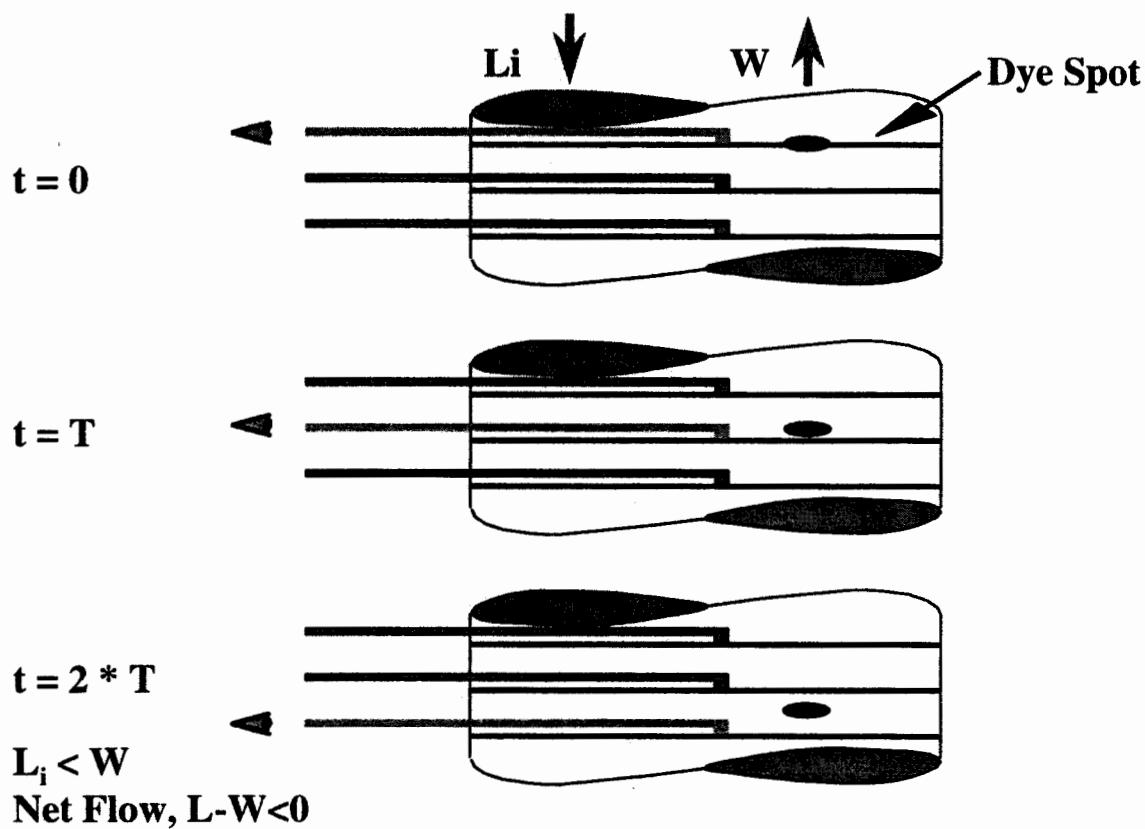
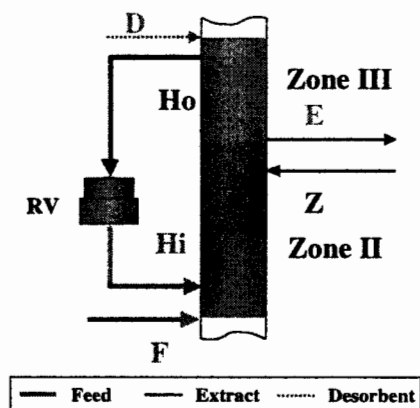
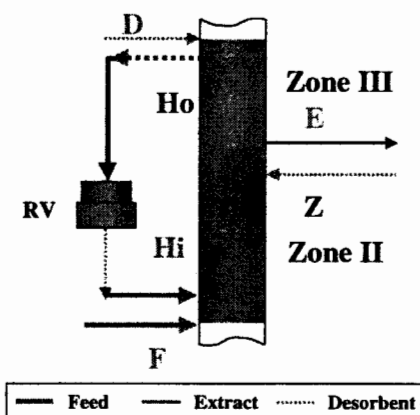


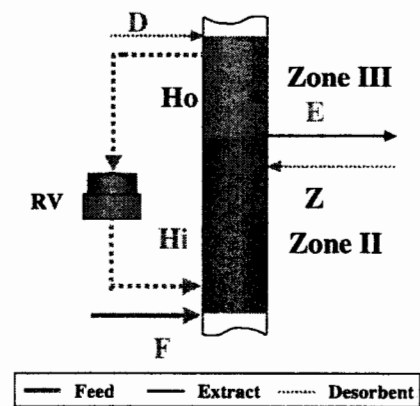
Figure II-17
Line Flushes



Composition at beginning of step.



Composition at middle of step.



Composition at end of step.







III. PROCESS VARIABLES

A. INTRODUCTION

This section will discuss the process variables used by the operations group to affect performance.

- | | |
|--------------------|-------------|
| 1. Temperature (T) | 5. L_2b/W |
| 2. Pressure (P) | 6. Dn/A |
| 3. A/Fn | 7. L_4/W |
| 4. L_2/W | |

Each of these variables is specified by the operations group and allows all the plant conditions to be calculated. After a specific set of "specified variables" is selected and the plant is lined out at the conditions, the performance is measured and evaluated. Depending on the performance, a decision must be made on which process variables to change to optimize the operation. This section will help the operators decide how to change the process variables to affect the performance in the proper direction and optimize the unit's operation. To begin this discussion, the variables used to measure performance will be presented and defined.

B. PERFORMANCE

The main variables used to measure performance in the UOP Molex Process are product purity and recovery. The product purity is defined as the weight percent n-paraffins in the Extract Column bottoms.

The recovery indicates the yield of n-paraffins. Recovery can be calculated in three different ways:

$$(1) \text{ Recovery} = \frac{\text{n - paraffins in the Extract Product}}{\text{n - paraffins in the Molex Feed}} = \frac{E(X_e)}{F(X_f)}$$

$$(2) \text{ Recovery} = \frac{\text{n-paraffins in the Extract Product}}{\text{n-paraffins in the Extract and Raffinate Product}} = \frac{E(X_e)}{E(X_e) + R(X_r)}$$

$$(3) \text{ Theoretical Recovery} = \frac{X_e(X_f - X_r)}{X_f(X_e - X_r)}$$

where

- F = Feed rate in weight per unit time
- E = Extract rate in weight per unit time
- R = Raffinate rate in weight per unit time
- X_i = concentration of n-paraffins in stream i

Derivation of Theoretical Recovery equation:

$$F = E + R \quad \text{Mass Balance} \quad (1)$$

$$F(X_f) = E(X_e) + R(X_r) \quad \text{Component Balance} \quad (2)$$

$$\text{Recovery} = \frac{E(X_e)}{F(X_f)} \quad \text{Definition} \quad (3)$$

where Recovery = fraction of n-paraffins entering process which leaves in the Extract.

Multiplying Equation 1 by X_r, and subtracting it from Equation 2:

$$F(X_f - X_r) = E(X_e - X_r) \quad (4)$$

$$E/F = \frac{X_f - X_e}{X_e - X_r} \quad (5)$$

Substituting Equation 5 into Equation 3:

$$\text{Recovery} = \frac{X_e(X_f - X_r)}{X_f(X_e - X_r)} \quad (6)$$

In this form, Equation 6 is the theoretical recovery term.

The first two methods of calculating recovery require both analytically determined concentrations and mass flow rates of the streams. The problem frequently arises that the flow meters are not calibrated and the mass balance does not close. This miscalculation of flow meters leads to recovery numbers that are incorrect. To avoid this problem with possible flow meter error, the theoretical recovery can be used which is only a function of analytically determined concentration. This is the method UOP recommends for the calculation of recovery.

The operator uses the n-paraffins purity and theoretical recovery to judge performance. The figures discussed in this section show how the process purity and recovery change with process variable changes. The next sections will discuss how process variable changes affect the purity and recovery of the UOP Molex Process.

C. ADSORBENT CHAMBER TEMPERATURE AND PRESSURE

The Molex is run at constant temperature (350°F; 177°C) and pressure (350 psig; 24.6 kg/cm²). These are secondary variables and are not changed during normal operations. Adsorbent Chamber pressure is specified in order to maintain a liquid full Adsorbent Chamber. The temperature is set as a compromise among the increase in mass transfer rates and the decrease in Rotary Valve Teflon sheet stability with increasing temperature.

D. PRIMARY VARIABLES

The following section will try to illustrate the different effects that occur when changes are made to the primary variables. It should be noted at this time, however, that the absolute values indicated in the enclosed figures represent general operations while each individual plant will be somewhat different from these illustrations. The graphs may be considered indicative of direction and relative strength of dependence between variables. The figures are used to determine the changes necessary to adjust performance in the desired direction.

1. L_2/W Effects

Zone II is the purification zone and therefore the zone ratio L_2/W is the critical control for maintaining product specification. If L_2/W is too low, impurities will be carried up through Zone II with the adsorbent and reduce the n-paraffin purity. If too high, a high purity will result but a loss of product recovery will occur.

Figure III-1 and Figure III-2 illustrate the relationship between L_2/W , purity and recovery. In this set of curves in order to obtain 99% n-paraffin purity, an L_2/W value of 1.12 is necessary, resulting in 98.6% extraction efficiency (recovery). If L_2/W is increased to 1.15, purity will increase slightly but recovery will drop. Therefore, at high purities a small increase (0.03) will have slight effect on purity but will change recovery considerably.

Going the other way, if L_2/W is decreased too far, a sharp purity breakpoint occurs while recovery has begun to stabilize and changes only slightly. This may be best seen in Figure III-2.

Changes in L_2/W of 0.02 are normally considered acceptable unless operations are well down on the performance curves.

2. L_2b/W Effects

Zone IIb is a buffer zone between the purification zone (Zone II) and desorption zone (Zone III). Iso-octane is used in the zone flush to prevent n-pentane from Zone III from entering Zone II and desorbing the purified normal paraffins and thus reducing the recovery. Para-xylenes are used to physically displace adsorbed aromatics and consequently flush them toward the Raffinate.

If L_2b/W is high, there will not be enough para-xylenes to displace the adsorbed aromatics and the extract product will contain a higher level of aromatics. Figure III-3a illustrates the relationship with pX. As L_2b/W increases above 0.97 the product aromatics content increases rapidly. Below 0.97 very little additional purity can be obtained. The breakpoint in purity may vary slightly with each individual plant and must be determined after startup.

As the ratio goes even higher (>1.05) the nC_5 from Zone III will displace the n -paraffins from the selective pores in Zones II and I. If Zone IIb is too low, an excessive amount of Zone Flush is needed which requires extra utilities and wastes money (see Figure III-3b).

3. Dn/A Effect

Zone III is the desorption zone in the Molex process. Although Zone III has little effect on product purity, it does have a pronounced effect on product recovery. See Figures III-4 and III-5.

If Dn/A is run higher than the specified operating range, it is seen that little or no increase in recovery results. However, the higher Dn/A requires a larger circulation of desorbent. This becomes very costly in terms of utilities and is not necessary.

If Dn/A is run too low, however, incomplete desorption occurs. The undesorbed n -paraffins then are carried up through Zone III and Zone IV to Zone I, where it competes with feed n -paraffins for space in the pores in Zone I, resulting in a higher concentration of n -paraffins in the raffinate, reducing the recovery.

Dn/A is not as sensitive to changes as L_2/W . It is also not an "everyday" variable. In other words, once the operating range is found through pumparound pump samples, Dn/A is rarely changed.

It should be noted that in Figure III-4, Dn/A is based on the nC_5 flow rate since only nC_5 (not iC_8) contributes to the desorption of the n -paraffins.

4. L_2/W Effects

Zone IV acts as a buffer zone between Zone I and Zone III. If not controlled correctly, raffinate material will migrate through Zone IV and into Zone III. From here it will travel down Zone III and exit with the extract contaminating the n -paraffin.

Figures III-6 and III-7 illustrate the effects of zone ratio L_4/W on the extract purity. It is seen that little or no non-normals break through Zone IV until L_4/W becomes greater than 1.

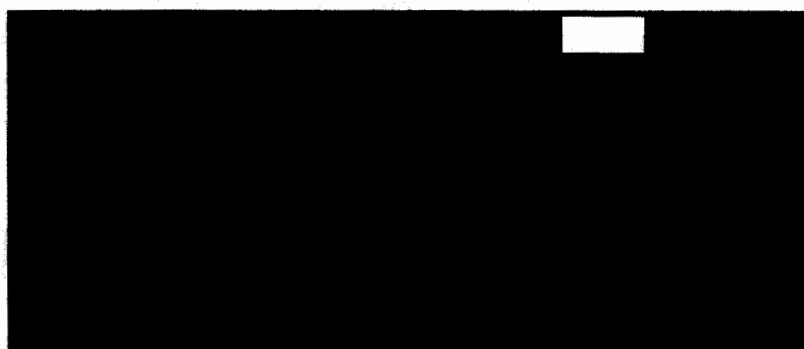
It is not desirable to run L_4/W any less than required since desorbent circulation, hence utilities go up as L_4/W goes down. This can be seen by taking a volume balance around Zone III and Zone IV as follows:

$$(L_4/W) \times W = L_4 \quad (1)$$

$$[A \times (Dn/A) / (\%nC_5)] + W = L_3 \quad (2)$$

$$L_4 + H_{out} = L_3a \quad (3)$$

$$D = L_3a - L_4 \quad (4)$$



If L_4/W is decreased, L_4 also decreases. (Equation 1) Since it is desirable to maintain a constant Dn/A ratio, it is necessary to keep the Zone III liquid rate (L_3) constant. From equation 3, if L_3 is kept constant and H_{out} is constant, then L_3a is constant. Since L_4 is lower and L_3a is constant, then the desorbent rate is increased as per equation 4.

5. A/Fn Effects

Figure III-8 illustrates the operating curve for the Molex unit. Also shown on the curve are the effects of A/Fn changes on the unit performance. Unlike the other three variables, A/Fn changes the position of the operating curve.

Another way to look at the A/Fn variable is to go back to the definition of A/Fn. A/Fn is the ratio of the selective pore rate (A) to the feed rate of normals (Fn). This means that A/Fn relates the amount of selective pores circulated past the feed

point to the feed rate. Intuitively it stands to reason that if you circulate more selective pores per unit of feed introduced that a greater recovery can be achieved. This phenomenon occurs until there is an excess of selective pores per unit of feed at which point there is no benefit derived from circulating extra selective pore volume. This means that at this point if A/F_n is increased, recovery will not increase. A procedure of increasing A/F_n can continue until the maximum required selective pore circulation per unit of feed is reached and no recovery improvement is seen with additional increases in A/F_n . At this point you have achieved the optimum A/F_n ratio and the outer most purity-recovery operating line. See Figure III-8.

This means that if A/F_n is increased, while n-paraffins purity is held constant, recovery will increase to some maximum level. A/F_n should not be increased past this maximum value that obtains maximum recovery because increased A/F_n values cause increased desorbent circulation which increases the utility costs. The performance and economic optimum is at a minimum A/F_n with maximum n-paraffin recovery (see Figure III-9).

While discussing A/F_n , it is a good time to describe the process significance of the cycle time (θ) (See Figure III-10). The cycle time (θ) is time required for the feed to step through complete cycle 24 beds. It is a calculated variable using the specified values of A/F_n and F_n . In the Molex system, the following variables; cycle time (θ), A/F_n , and F are all independent variables and by specifying any two, the third can be calculated. We have selected to specify the F and A/F_n and calculate cycle time (θ). The cycle time (θ) does have some operational limits that must be met by the design. In general, the cycle time for Molex should not be lower than 45 minutes. This limit exists because when the cycle time becomes too short, the molecular exchange between liquid and adsorbed phases becomes limited by diffusion rates. This means that if one wants to maximize the feed rate to the chamber section and has enough fractionation capacity, one would find the minimum A/F_n possible and then raise the feed rate until the cycle time (θ) reaches 45 minutes. At this point, further feed increases will push the cycle time (θ) to levels where the diffusion rates become limiting and the n-paraffin recovery will drop off to unacceptable levels.

Figure III-1
Effect of L2/W on Purity and Recovery

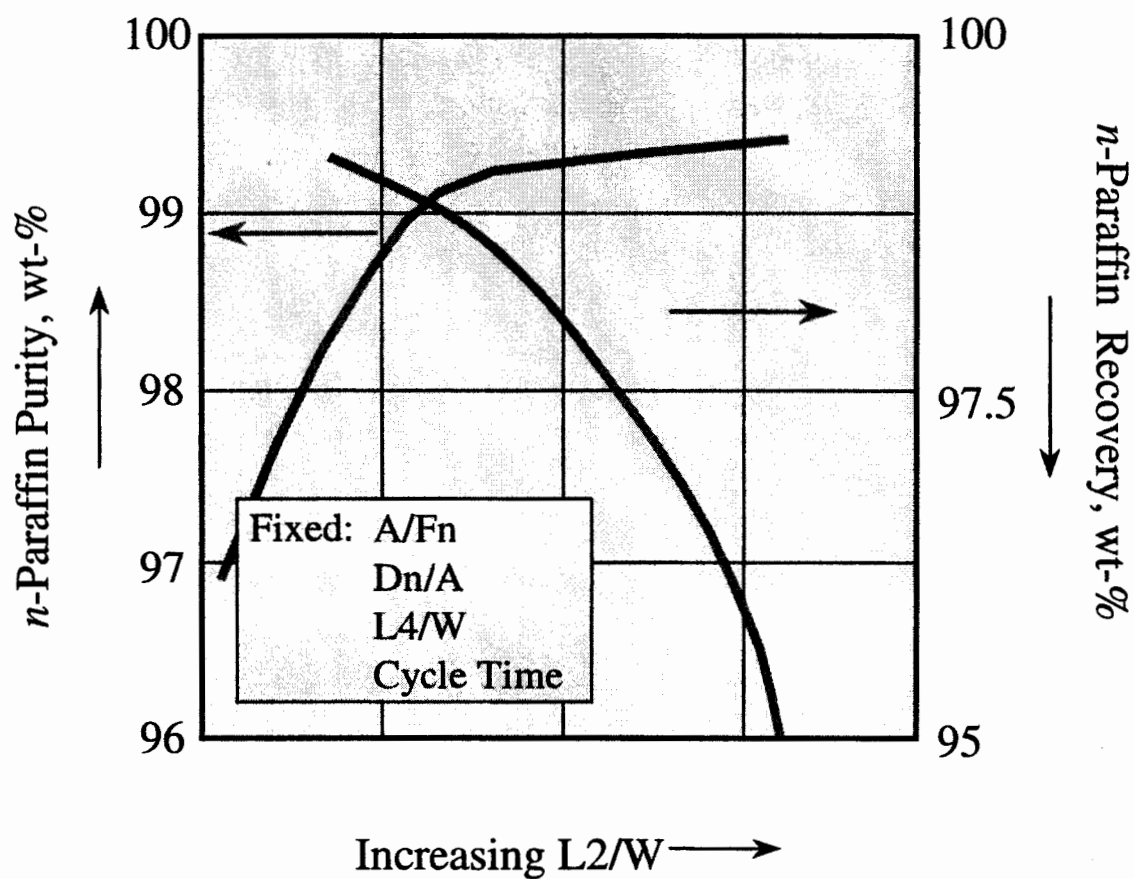


Figure III-2
Effect of L2/W on Purity and Recovery

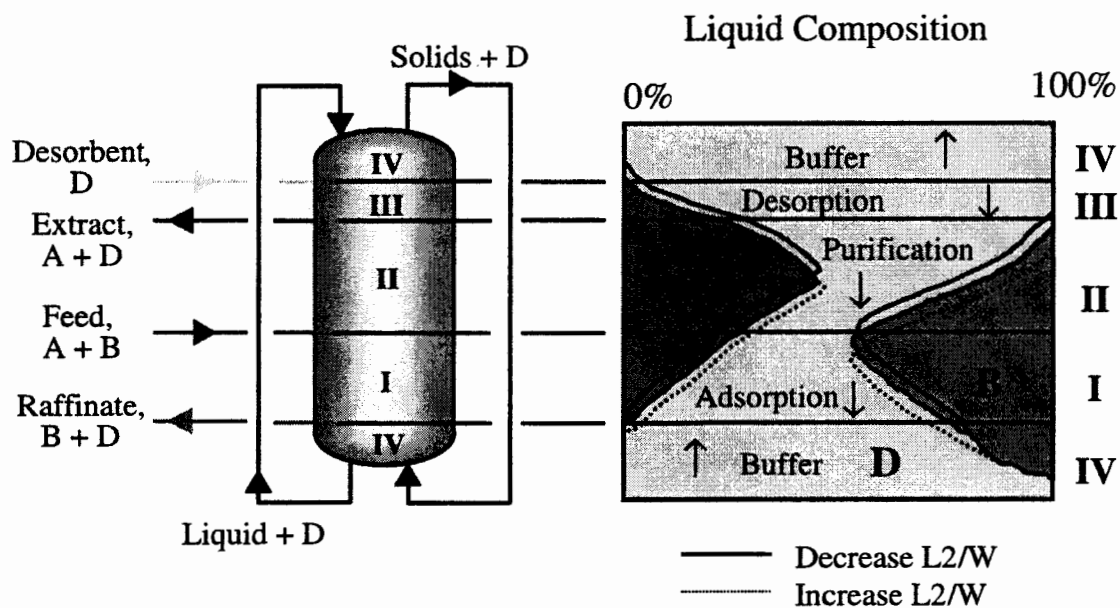


Figure III-3a
Aromatics in *n*-Paraffin Product vs. L2b/W
(with *pX*)

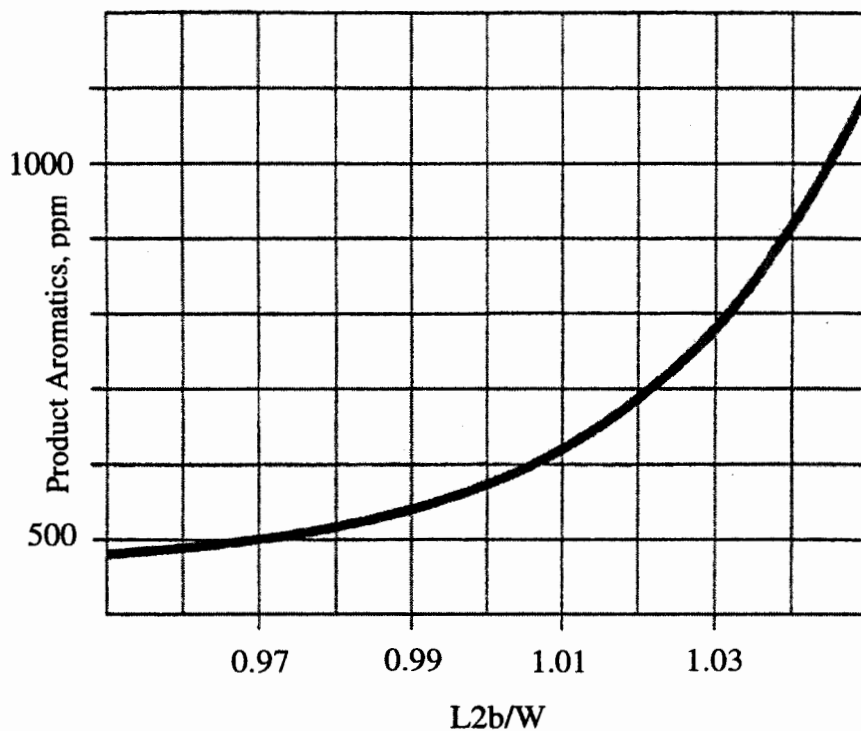


Figure III-3b
***n*-Paraffin Recovery vs. L2b/W**
(with or without *pX*)

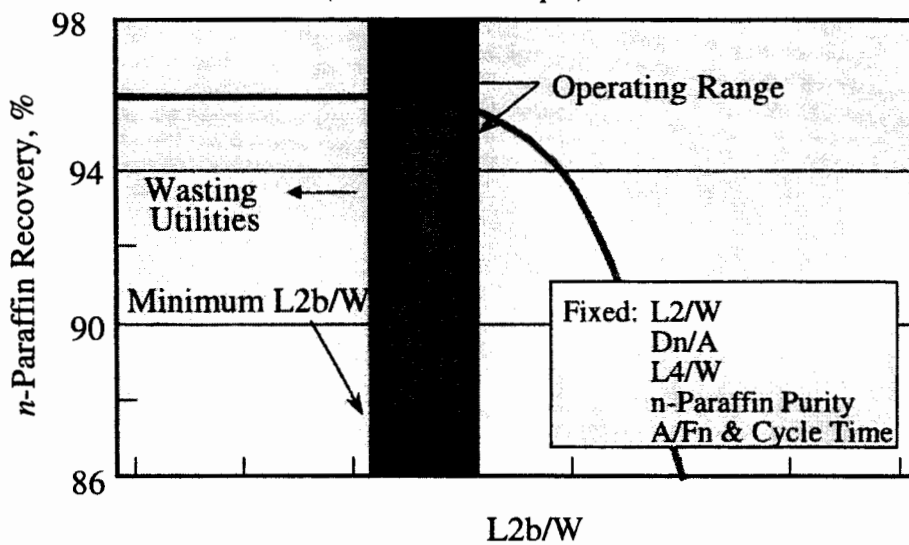


Figure III-4
Effect of Dn/A on n -Paraffin Recovery

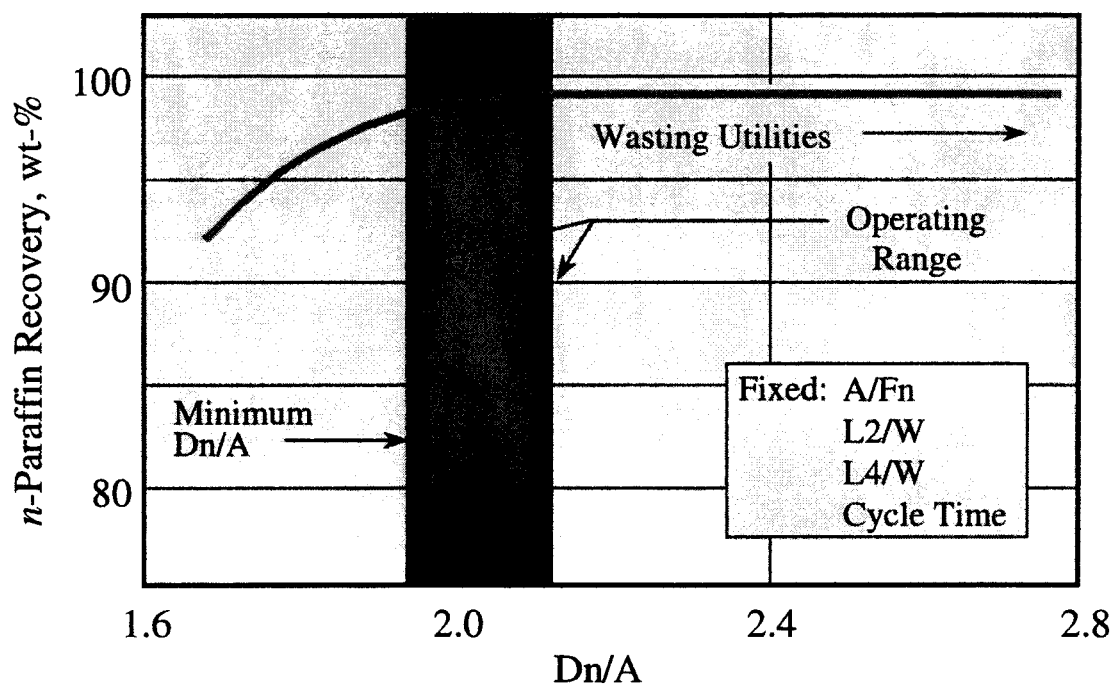


Figure III-5
Effect of Dn/A on n -Paraffin Recovery

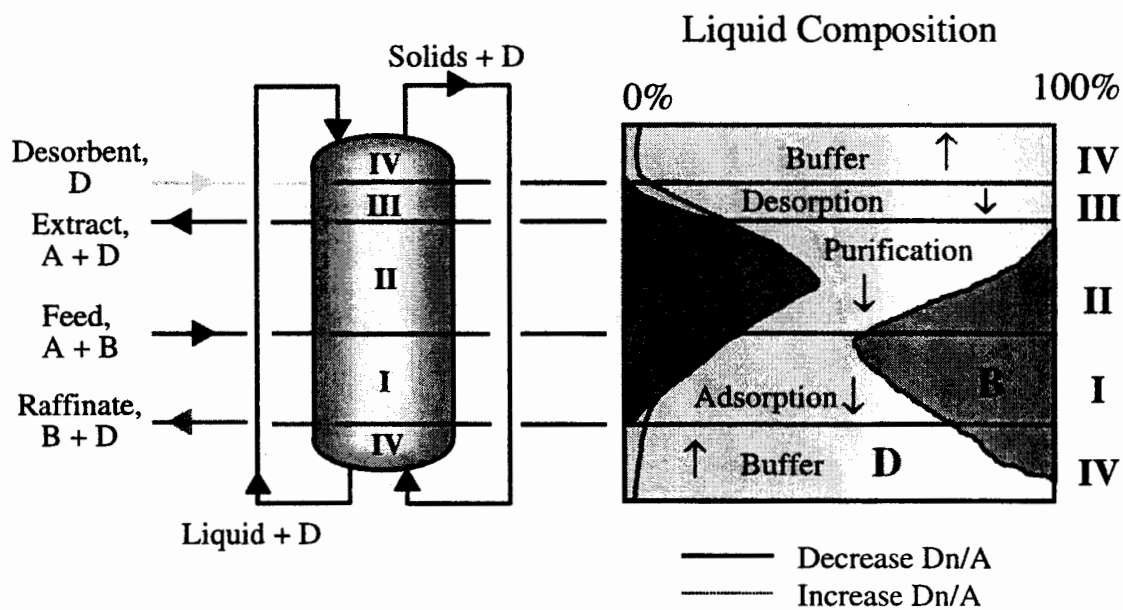


Figure III-6
Impurities from Zone IV in Extract vs. $L4/W$

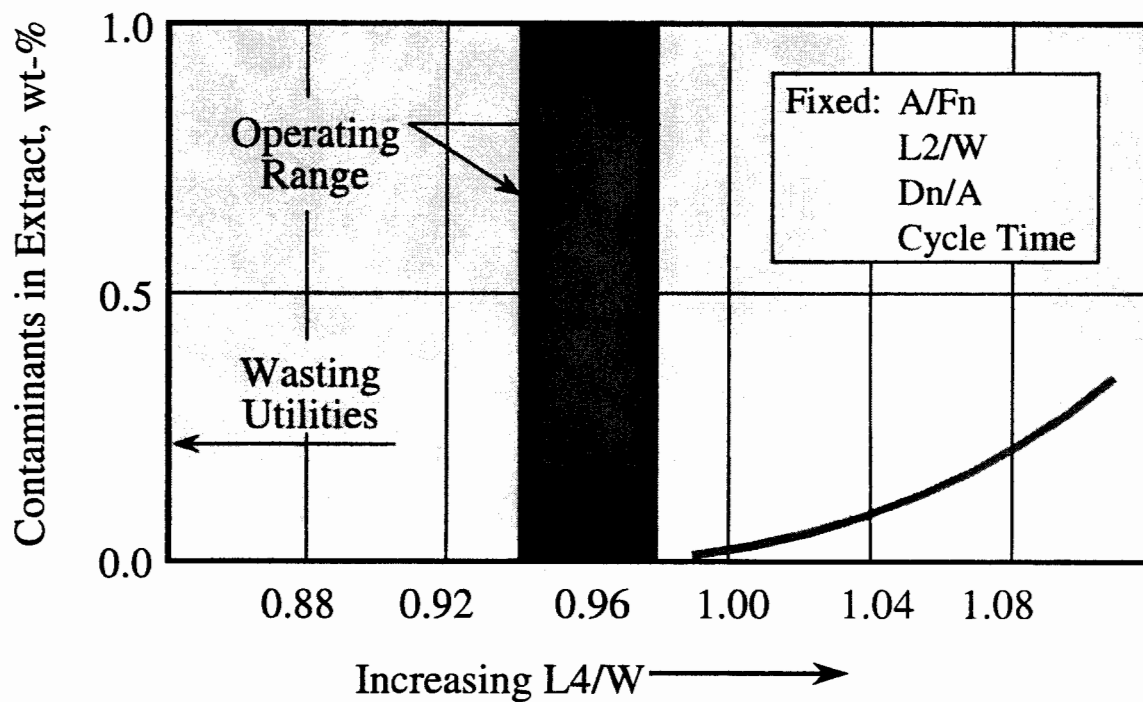


Figure III-7
Impurities from Zone IV in Extract vs. L_4/W

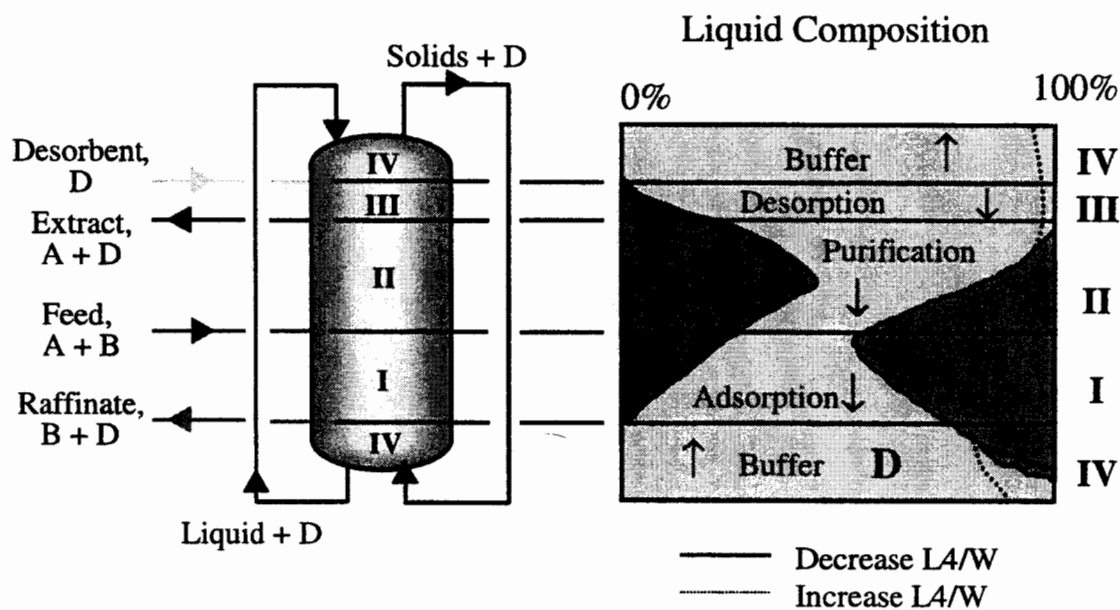


Figure III-8
***n*-Paraffin Purity vs. Recovery**

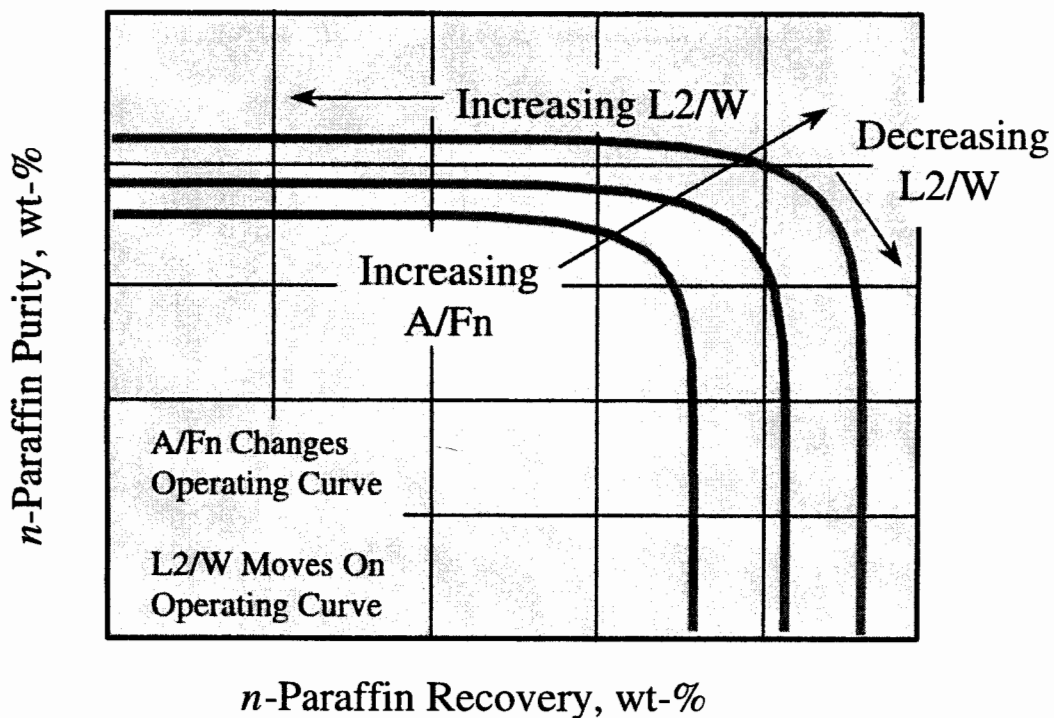


Figure III-9
***n*-Paraffin Recovery vs. A/Fn**

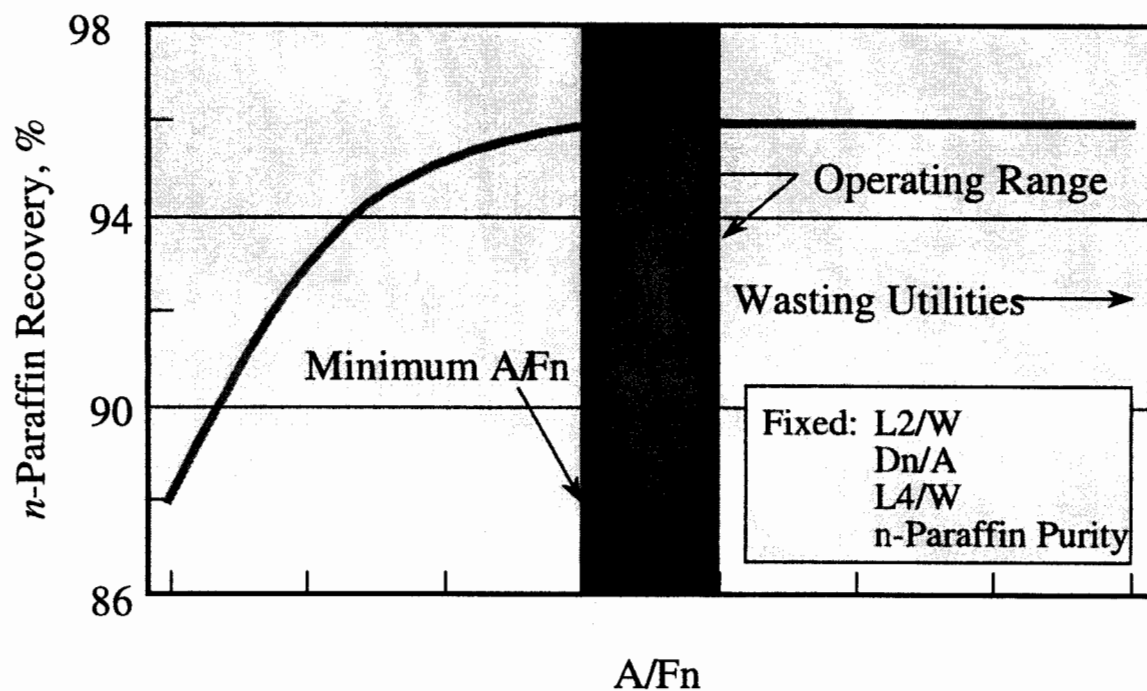
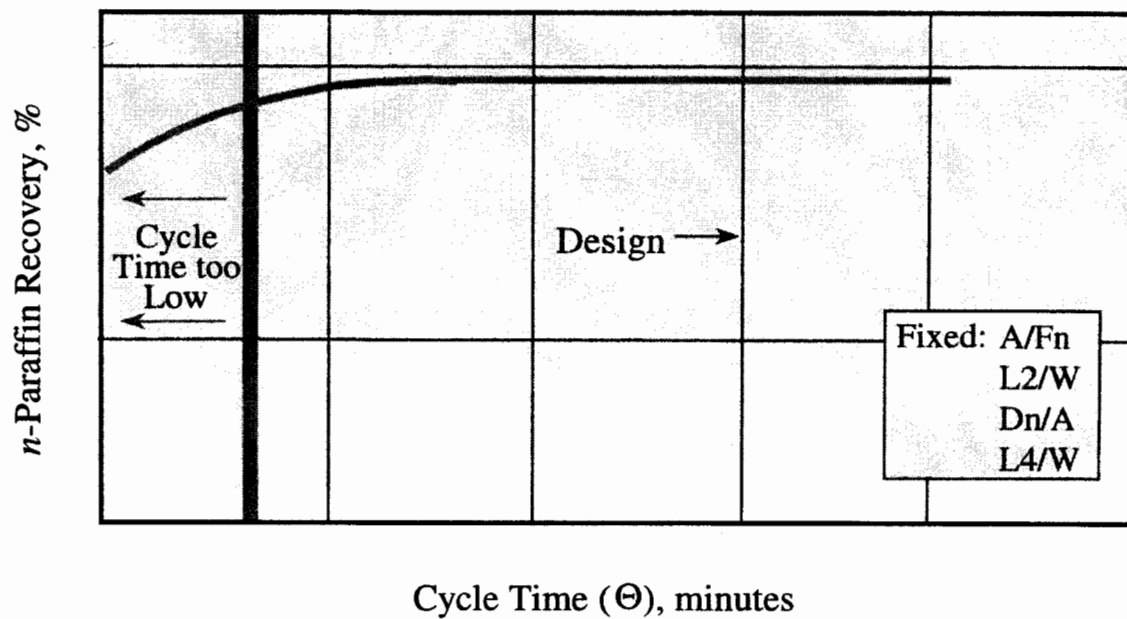


Figure III-10
***n*-Paraffin Recovery vs. Cycle Time**









IV. PROCESS FLOW AND CONTROL

A. PROCESS FLOW

The following is intended as a general description of the Molex unit process flows. Individual units may differ in such details as heating medium, cooling medium, and perhaps the fractionation of the n-paraffin product. Such items are, of course, dictated by the refiner's desired degree of flexibility, economics, and practices. A typical process flow diagram is included at the end of this section.

1. Feed Flow

Feed is normally pumped to the unit directly from the upstream hydrotreater stripper bottoms or from the Molex feed surge drum, through the feed filter as a precautionary measure against possible damage to the turbine meter or rotary valve, and directed to the rotary valve on flow control. The feed enters the chambers between Zones I and IIa. The temperature of the feed stream sent to the adsorbent chambers should be maintained at constant value. The hydrotreater stripper bottoms is temperature controlled by exchange with stripper feed so as to maintain the desired Molex feed temperature. In the event that the stripper bottoms stream has undergone additional exchange or cooling, then a feed heater is provided to raise the stream temperature to design value.

2. Adsorbent Chamber Flow

The adsorbent loading is equally divided into 24 beds contained in two chambers. At any one time, there are four streams entering and three streams leaving the chamber in the bed lines connected to the rotary valve. The streams entering the chambers are the feed, desorbent, zone flush and line flush in. Extract, raffinate and line flush out exit the chambers. A pumparound stream continually circulates from the pumparound pump to the top of and through Chamber No. 1, to the top of and through Chamber No. 2, and back to the pump. The pump around stream is

primarily used to control the rate of flow through discrete sections of the chambers referred to as zones. The chambers are divided into seven continuous circulating zones; their functions are described below.

Zone 1 The beds between the raffinate and feed inlets. In Zone I the normal paraffins in the feed are adsorbed by the adsorbent and the raffinate is rejected.

Zone IIa The bed between the feed and the line flush inlets.

Zone II The beds between the line flush and the zone flush inlets. Zone II acts as a non-normal desorption zone, or purification zone.

Zone IIb The bed between the zone flush and the extract inlets. Zone IIb flow is controlled to eliminate the flow of desorbent or n-paraffins into Zone II.

Zone III The beds between the extract and line flush out inlets. In Zone III the normal paraffins in the adsorbent are desorbed and exchanged with the desorbent. This is referred to as the desorption zone.

Zone IIIa The bed between the line flush out and desorbent inlets.

Zone IV The beds between the desorbent and raffinate feed inlets. Zone IV also acts as a buffer zone separating adsorption and desorption zones.

Since the zones are defined with respect to the active bed lines through which the streams are entering or leaving the chambers and at each valve step these bed lines are switched downward in sequence, it becomes apparent that each zone travels downward through the Chamber No. 1, out the bottom and via the pusharound circuit re-enters the top of the Chamber No. 2. The material leaving the bottom of the Chamber No. 2 is pumped via the pumparound pump back to the top of the Chamber No. 1.

The flow rate in the pump-around circuit is controlled by a flow recording controller. Since the flow rate is different in each zone, it is a requirement that the pumparound flow rate change as each zone passes through the pumparound circuit. This flow controller is thus reset by the Rotary Valve Control System. Facilities are provided for recording the differential pressure across each chamber.

With the exception of the raffinate stream, all flows entering and leaving the chambers are on flow control. The raffinate is pressure controlled and maintains the base of the Adsorbent Chamber No. 2 at constant pressure. It is a requirement that all six flows be accurately controlled and turbine type meters are used to achieve the required accuracy. For those streams that do not vary significantly in composition, like the feed and desorbent streams, flow to the rotary valve can be by straight orifice control. Turbine meters are still provided, as signals from those meters are fed to the rotary valve digital control system.

3. Extract

The extract, containing the recovered n-paraffins, desorbent and zone flush material leaves the chambers between Zones II and IIb, flows through the rotary valve, protective filter, turbine meter, flow control valve and on to the extract mixing drum. The mixing drum is designed to smooth out the concentration fluctuations in the column feed, which could upset column operations. Slight variations in the extract concentration can be expected.

The extract and line flush out streams merge and enter the extract column. The lighter desorbent and zone flush components are taken overhead, leaving the purified n-paraffin product to exit the column bottom, where it can be sent to storage or down-stream units. The nC_5 and iC_6 in the overhead are directed to the desorbent surge drum. A side-cut stream is withdrawn a few trays from the top. This stream is richer in iC_5 and is fed to the desorbent splitter.

4. Raffinate

The raffinate, containing the rejected non-normal paraffins, desorbent, and zone flush material leaves the chamber between Zones I and IV on pressure control, flowing through the rotary valve and on to the raffinate mixing drum. From the mixing drum, designed to smooth out concentration fluctuations, the raffinate is joined by the rotary valve dome sealant, heated in the feed/bottoms exchanger and enters the raffinate column.

The raffinate column sends the lighter desorbent and zone flush components overhead and drops the non-normals out the column bottom. The non-normals are cooled in the raffinate bottoms desorbent exchanger, a fin fan and trim cooler to recover as much heat as possible before sending them to storage. The nC_5 and iC_8 in the overhead flow are directed to the desorbent surge drum. Similar to the extract column, a side-cut stream is again withdrawn and serves as the major feed source to the desorbent splitter.

5. Desorbent and Zone Flush

A mixture of recyclable nC_5 and iC_8 is taken overhead from both the extract and raffinate columns and directed to the desorbent surge drum. The side-cut streams which are richer in iC_8 are sent to the desorbent splitter at rates required to meet the iC_8 demand of the system. The purpose of the desorbent splitter is to produce a high purity iC_8 stream for the zone, line and head flushes. The surge drum material, a mixture of nC_5 and iC_8 is used as desorbent.

The desorbent surge drum serves as surge capacity for the desorbent. If any makeup nC_5 or iC_8 is required, it can be sent to the raffinate column. The composition of the surge drum can be regulated by making up the required component or sending a slipstream from the desorbent splitter bottoms to remove iC_8 . Desorbent itself, consisting of approximately 60/40% (volume basis) of nC_5/iC_8 , can be removed in a controlled manner from the tops of the extract and raffinate columns.

Desorbent is pumped to the rotary valve on flow control, through the raffinate bottoms-desorbent exchanger, desorbent heater, filter, turbine meter, and control valve. The temperature of the desorbent is controlled by the flow of heating medium to the desorbent heater.

The desorbent splitter column sends the stripped nC_5 overhead vapor stream back to the raffinate column, while producing high purity iC_8 or iC_8/pX , in the bottoms. This material is recycled back to the adsorbent chambers as zone flush, line flush, head flush and dome sealant.

Purified flush material exits the desorbent splitter bottom, is heated across the flush heater, filtered and split into the various flush streams. The zone flush and line flush enter the rotary valve on an orifice meter measured flow control. Turbine meters are also provided that send signals to the digital controller.

Any nC_5 material contaminating these streams will reduce product recovery if it enters Zone II, where it will desorb the desirable n-paraffins. Any C_9^+ will contaminate the extract if it is coming from the raffinate column sidecut or reduce capacity if it is coming from the extract column sidecut. Adjust the sidecut TRC's to prevent accumulation of C_9^+ .

The temperature of the flush stream is regulated by a temperature controller that regulates the heating medium flow through the flush heater.

A nC_5/iC_8 system is typically referred to as a dual split desorbent system. Most Molex designs are based on this system. Certain units, however, employ a triple desorbent system comprising nC_5 , iC_8 and pX (para-xylene). The flow scheme remains identical except that the zone and head flush streams are now comprised of both iC_8 and pX. The desorbent stream directed to Zone III would still be an approximate 60/40 (vol-%) mix of nC_5/iC_8 . The desorbent splitter bottoms would contain approximately 70-80 vol-% iC_8 and 20-30 vol-% pX. The triple desorbent scheme is employed in those instances where very low aromatic contents are desired in the extracted normal paraffins product.

6. Line Flush

Since each bed line successively carries feed, zone flush, and extract, it is necessary to flush the residual feed material from the line before the zone flush is injected or extract is withdrawn. This is done because with only one bed between the zone flush and the extract withdrawal points, the extract product would be contaminated with feed components. This same principle applies to the line after being used for extract removal that is next used for desorbent. The line volume of extract would be displaced back into the chamber at the interface of Zone III and Zone IV, and some of the n-paraffin would be lost to the raffinate stream causing a loss of recovery. To eliminate these sources of contamination and product loss, two line flushes are provided. The line flush in stream, consisting of essentially pure iC_8 , is injected one bed after the feed. Flow control is similar to that of the flush out stream. The line flush out stream is withdrawn from the first line below the desorbent injection, flushing the line of extract to the extract column. In both cases sufficient liquid is pumped to leave the line clean. The line flush rates, which are on flow control using turbine meters, are calculated such that the longest bed line will be flushed with twice its own volume during one valve step.

7. Head Flushes

Head flushes are provided at the top and bottom of each adsorbent chamber to prevent contamination caused by the dead volumes of the elliptical heads. A small purge of flush material is withdrawn downstream of the flush filters and sent to the top and bottom heads of both chambers on flow control. These streams will then enter the main body of the chambers through the top and bottom grid snorkels. The head flush material slightly dilutes the circulation liquid with flush material and leaves with the raffinate stream. The smallest flow that will prevent contamination determines the head flush rate.

B. PROCESS CONTROL

1. Adsorbent Chambers

The two process variables for the chambers that need to be controlled are the temperature and pressure. The chamber temperature is controlled by the incoming feed and desorbent streams at approximately 177°C (350°F). The pressure is set at 24.6 kg/cm² (350 psig) which is high enough to prevent flashing of the hydrocarbon liquid.

Adsorbent chamber pressure is very important. Since the chamber is liquid full, a small change in liquid rate entering or leaving the chamber will have a drastic effect on chamber pressure, unless the pressure controller is in operation and functioning properly. The normal operating pressure of the chamber is sufficiently high to keep the hydrocarbons from boiling. If the pressure falls below the bubble point, the liquid inside the chamber will boil and vaporize. This must be avoided as the vaporization may damage the adsorbent structure. The chamber pressure is controlled by a pressure controller at the bottom of Adsorbent Chamber No. 2 that controls the flow of raffinate from the adsorbent chambers to the raffinate column.

There is an emergency system to protect the chambers from losing pressure. This consists of a pressure switch on the Chamber No. 2 pressure controller that closes the extract control valve if the chamber pressure falls below a predetermined point. This means that if there is a drop in the chamber pressure, the raffinate control valve will close with the PRC and the extract FRC flow will close with this emergency system and keep the chamber pressure above an acceptable level.

Pressure drop in the chamber should also be watched carefully. There may be several reasons for a sharp increase in pressure drop during operation, the most common of which is plugging of the beds or their support screens by either fines or dirt and scale. Any unexpected pressure drop should be promptly investigated. The maximum pressure drop allowed is 0.35 kg/cm² (5 psi) per individual bed.

2. Rotary Valve

a. Seating/Sealing of the Rotary Valve

The rotor plate of the rotary valve is seated against the bottom head of the rotary valve by applying pressure to the top of the rotor plate using desorbent. The desorbent to be used as rotary valve dome sealant is introduced to the rotary valve on flow control. Dome sealant is released from the rotary valve on pressure control located on the dome sealant return to the raffinate column. Figure IV-1.

Excessive pressure in the rotary valve dome could damage the cross over bellows of the rotor plate so a control scheme is provided to protect the cross over bellows of the rotary valve rotor plate. What will actually cause the damage is differential pressure from the inside to the outside of the cross over bellows. A low signal selector is used between the set point that the operator provides and a maximum set point calculated by the ACCS controller which is designed to prevent the differential between across the rotor plate from exceeded the design limit of the bellows. The controller uses the pressure of the bottom of the No. 2 adsorbent chamber for comparison. Sufficient margin is allowed in the setpoint of the controller to allow for the normal pressure differential between the inside of the crossovers of the rotor plate and the bottom pressure of the No. 2 adsorbent chamber.

A hand switch is provided such that the dome of the rotary valve can be connected to the raffinate line exiting the rotary valve via an on/off control valve. When this connection is open the dome of the rotary valve will be balanced with the pressure of the raffinate line.

b. Indexing the Rotary Valve

The rotary valve is stepped using the logic system of the ACCS. Switches are provided on the rotary valve that provide information to the ACCS on the location of the hydraulic cylinder and the shaft of the rotary valve.

When the ACCS determines that the rotary valve must step, the ACCS operates the flow directing solenoid of the hydraulic system of the rotary valve to rotate the rotary valve rotor plate with the hydraulic cylinder. The ACCS sets the flow directing solenoid of the hydraulic system to force the hydraulic cylinder forward until a switch on the rotary valve indicates that the hydraulic cylinder is extended to the correct location to have advanced the rotor plate of the rotary valve by 1 port. When this indication is received the ACCS redirects the hydraulic fluid to return the hydraulic cylinder to its rest location. When the hydraulic cylinder is retracted the ACCS closes the flow of hydraulic fluid to the hydraulic cylinder. For the system of two rotary valve on the same adsorbent train the rotary valves are stepped simultaneously and are synchronized so that the each net flow passing through the rotary valve are directed to the same bedline, e.g. the flow through the first rotary valve of feed would go to the same bedline from each rotary valve, when the first rotary valve is directing the feed to bedline 1 the second rotary valve is also directing the feed to bedline 1.

3. Extract and Raffinate Columns

The extract and raffinate columns flow and control schemes are shown in Figures IV-2 and IV-3. The primary purposes of both columns are to separate the recyclable desorbent and yield a purified bottoms product, n-paraffins from the extract and non-normals from the raffinate column, as well as to provide the feed source to the desorbent splitter column.

Bottoms product flow is regulated by the bottoms level controller and pumped out to storage after being cooled or for further processing. The reboiler heat is supplied by a heater or exchange with circulating hot oil on flow control. Heat input is assumed to be constant during normal operations at a level to achieve the required reflux for separation.

The overhead vapor from the column passes through the overhead condenser and drops into the overhead receiver. Pressure control is by a butterfly valve upstream from the condenser. A bypass line connects the overhead receiver with the

overhead vapor line upstream from the pressure control butterfly valve through a differential pressure control valve.

This type pressure control is very susceptible to non-condensable gases. These gases will not only reduce the efficiency of the condenser, but in addition, they will cause erratic pressure control. Vents are provided on both the overhead condenser and receiver to rid the system of non-condensable gases.

It is desirable to run the outlet of the overhead condenser at a temperature slightly below the point where all vapors will condense. If the condenser outlet liquid is too cold, heat will be unnecessarily wasted from the process, and if the liquid is too hot all material entering the receiver will not condense. Poor control of the condenser outlet temperature will result in erratic pressure control of the column. Hand control of air fan blade pitch will achieve the desired liquid outlet temperature.

The side-cut product from the raffinate column is pumped on flow control to the desorbent splitter. Suction to the side-cut pump is provided by the liquid collected in the weirs. The side-cut pump returns the major portion of the liquid inventory pumped from the weir back to the column to a tray just below the weir. This rate is on flow control reset by a TRC located a few trays below the weir. The reflux rate to the top of the column is on flow control reset by the weir's liquid level controller. The net overhead from the raffinate column is pumped out on flow control reset by the level in the overhead receiver to the desorbent surge drum. The reflux pump also acts as the net overhead pump.

The reflux to the extract column is pumped by the reflux pump which also pumps the net overhead liquid. The amount of reflux returned to the column is reset by the overhead receiver level controller. The net drawoff is sent out on flow control that is reset by the column temperature recording controller (TRC) and sent to the desorbent surge drum. The iC_8 rich side-cut is withdrawn and pumped on flow control to the desorbent splitter. This flow rate is reset by the desorbent splitter bottoms level.

It is important to maintain tight and accurate control of these columns since any upset could cause of the loss of n-paraffin purity or recovery, or the loss of desorbent. If the extract overhead becomes contaminated with n-paraffins, a subsequent loss in product recovery will result. If desorbent contaminates the bottoms, there will be a physical loss of desorbent as well as a reduction in n-paraffin product purity. In the raffinate column, a loss of non-normals overhead will result in a reduction in product purity as they eventually work their way through the desorbent splitter and into the adsorbent chambers with the zone flush.

If desorbent is lost in the raffinate bottoms, fresh desorbent will be needed to maintain a sufficient inventory for proper operation. On line chromatographs can be used to constantly monitor these streams for product purity and desorbent losses. These instruments can be very cost effective at the current costs of desorbent and energy.

4. Desorbent Stripper Column

This is typically a 20-30 tray vessel (See Figure IV-4). The object of the desorbent splitter is to produce a high purity stream of iC_8 for the zone flush, line flushes, head flushes and dome sealant. An nC_5 material contaminating this stream will reduce product recovery as it enters Zone II and desorbs the desirable n-paraffins. The extract and the raffinate side-cut streams merge and enter the splitter column on flow control. The side-cut from the extract column is additionally reset by the column bottoms level controller. Purified iC_8 exits the bottoms, is pumped through the desorbent splitter column bottoms heater (flush heater), to the filter, and on to the adsorbent chambers. The temperature of the zone flush leaving the exchanger is regulated by flow control of the heat medium, typically hot oil. The zone flush enters the rotary valve on flow control.

Desorbent splitter overhead, primarily stripped nC_5 material is returned to the raffinate column to a point just above the sidecut tray. The desorbent splitter thus operates at the pressure of the raffinate column.

Heat input to the desorbent splitter reboiler is provided by exchange with the raffinate column bottoms.

5. Turbine Meters

Turbine meters are used instead of orifice meters to measure the F, D, E, Hi, Ho, Hz, and pumparound flows because of the higher accuracy they afford.

The turbine meter element is a tubular housing flanged into a pipe of the same diameter and inside which a vaned rotor is made to spin on an axial shaft by movement of fluid through the pipe.

The rotation of the rotor is sensed by a magnetic pickup that transmits a pulse each time one of the rotor blades passes the pickup coil. The frequency of these pulses is proportional to the volumetric flow rate of fluid. The frequency is converted to a standard milliamp signal to the FRC.

The axial position of the turbine rotor on its shaft is maintained by hydrodynamic balance. If foreign material enters any of the passages that effect that balance, the turbine will quickly destroy itself, so filters or strainers are provided upstream of each turbine meter.

Figure IV-1
Rotary Valve Piping Configuration

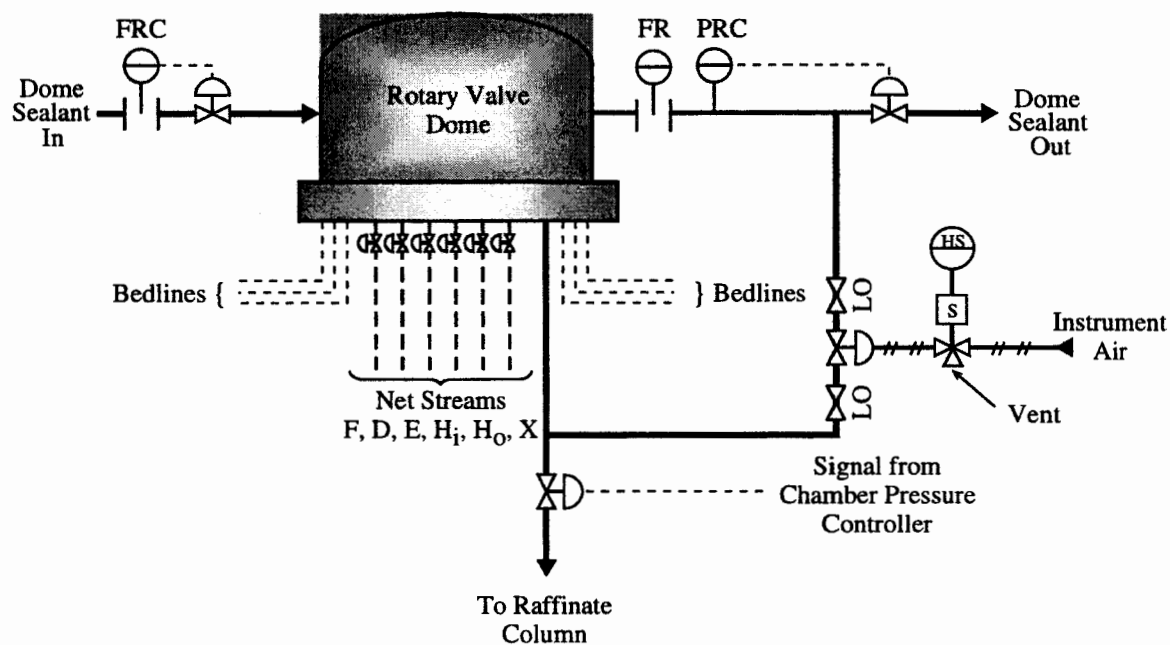


Figure IV-2
Extract Column

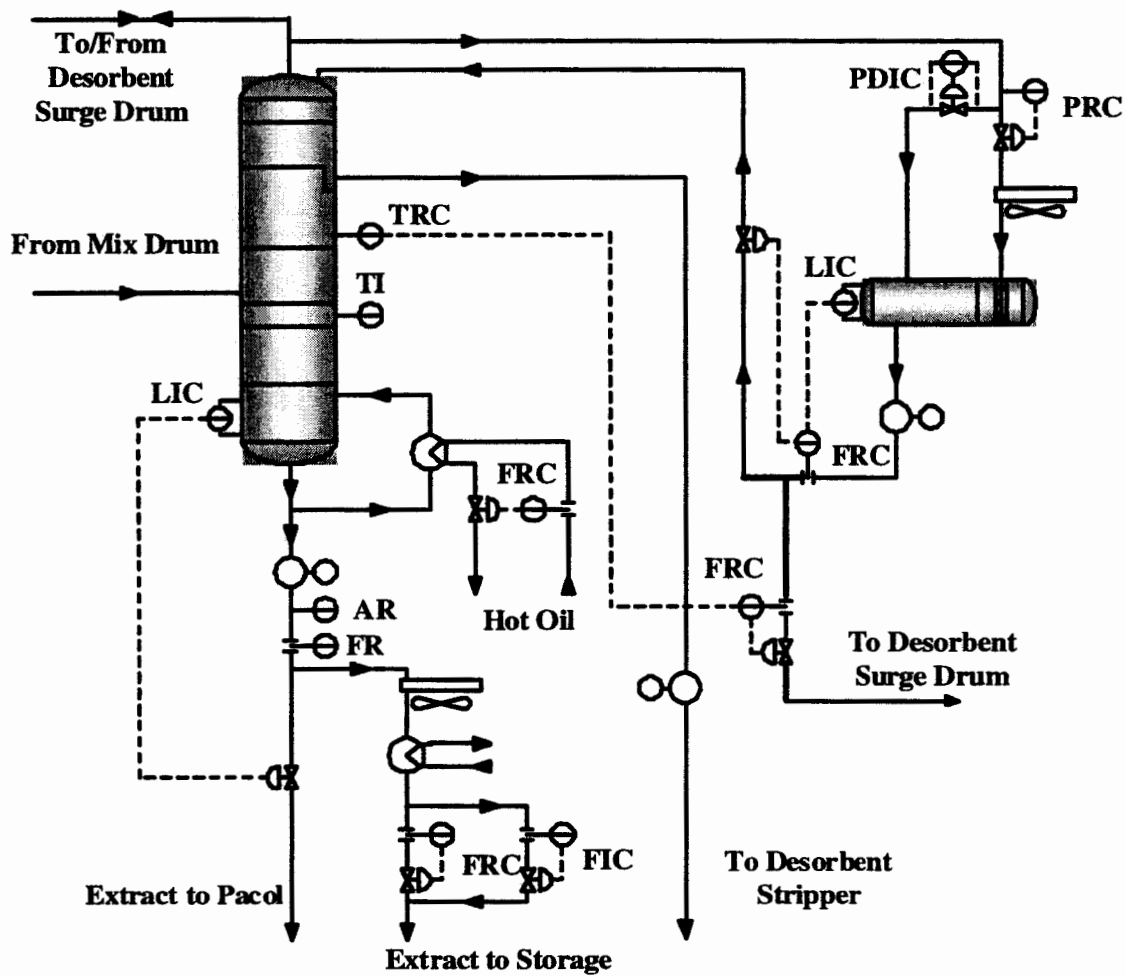


Figure IV-3
Raffinate Column

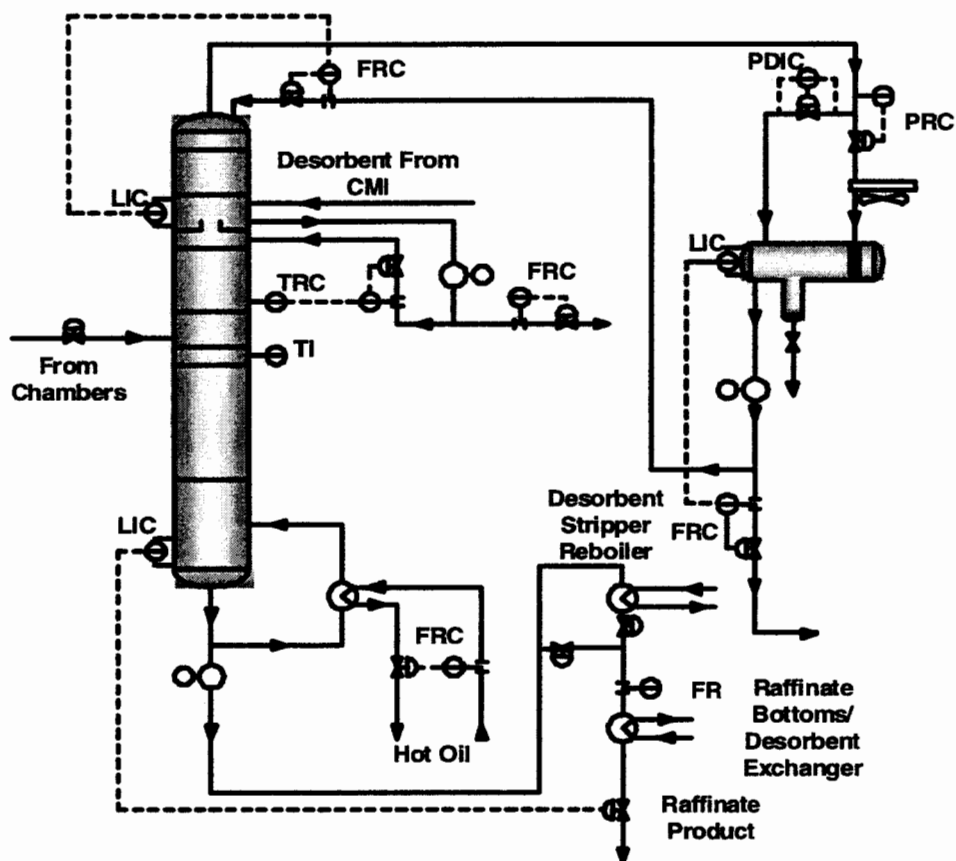
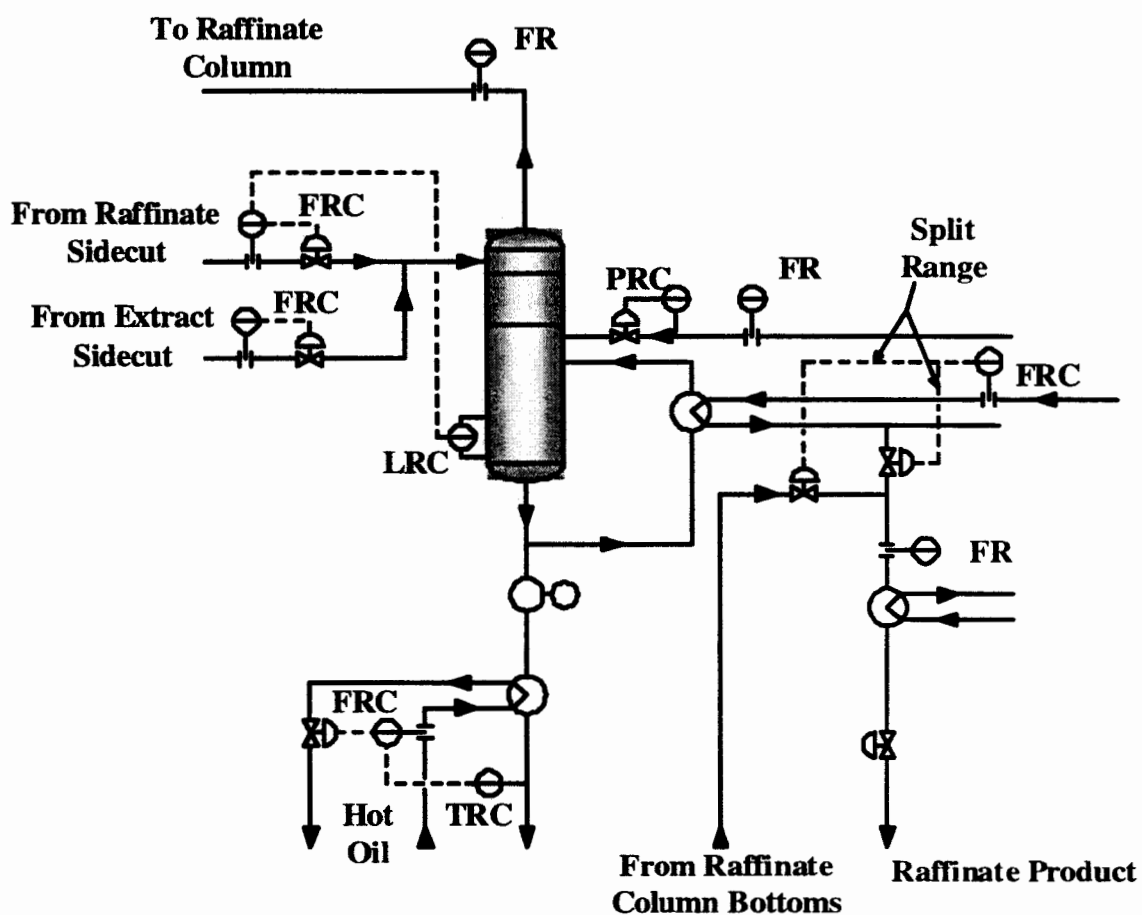


Figure IV-4
Desorbent Stripper







V. PROCESS EQUIPMENT

A. ROTARY VALVE

The rotary valve is a carbon steel multichannel, multiported valve. Seven process channels can be directed to the 24 ports of the rotary valve. The rotary valve consists of a bottom head which the 7 process lines and 24 bedlines of the adsorbent chamber connect to. A rotor plate inside the rotary valve provides a means of directing the flows. The top head or dome of the rotary valve provides an area that is used to seal the rotor plate using pressure and also supports the drive assembly and instrumentation for the rotary valve. Figure V-1.

The bottom head of the rotary valve consists of a thick forged plate which the nozzles for the 7 process lines and the 24 bedlines are welded to. In the center of the bottom head is a bushing that the drive shaft of the rotary valve rides in. Seven concentric channels are cut around the center of the shaft opening for the 7 process streams. The nozzle for each process stream connects to one of the concentric channels through the bottom head. A transition piece gradually changes in cross section from the circular pipe to the curved rectangular opening in the bottom of the channel. Outside the 7 concentric channels are 24 evenly spaced circular openings through the plate that the bedline nozzles are attached to. The upper most surface of the plate is flat and smooth to make the seal with the rotor plate.

The rotor plate, or more correctly, the rotor plate assembly is placed on top of the bottom head of the rotary valve. The rotor plate is a flat, circular, piece of steel with an elastomer sealant sheet attached to the bottom. Crossover pipes on the rotor plate direct flow from the process channels, or tracks, of the bottom head out to the ports at the perimeter of the bottom head. The crossover pipes start at the process tracks with a curved rectangular profile and make a transition to the circular cross section at the ports. In the larger crossover pipes an expansion bellow is included to help maintain the flexibility of the rotor plate. Attached to the bottom of the rotor

plate is an elastomer sheet that covers the bottom surface of the rotor plate except where there are openings for the process to pass through. The sealant sheet is held in place by retaining rings and washers that are attached to the rotor plate with screws. The retaining rings and washers support the sealant sheet in the open places of the bottom head. The sealant sheet contacts the top surface of the bottom head between the ports and tracks. The rings are above the tracks and the washers are above the ports. The sealant sheet is a special composite of various materials with Teflon that improves the wear of the sealant sheet. Figures V-2 and V-3.

The top head of the rotary valve seals against the bottom head of the rotary valve and can be pressurized by desorbent. The pressure in the top head will force the rotor plate against the bottom head to seal the process channels from each other and the ports from the process channels and each other. The top head also supports the drive mechanism and contains a mechanical seal to seal the shaft of the drive where it enters the top head of the rotary valve.

The position and zone indicator is a highly accurate and reliable form of position transducer used to determine the actual bed and zone position of the rotary valve. It consists of a resolver mounted on top of the rotary valve and a decoder mounted in the ACCS. The combination of the resolver and the decoder sends a signal to the logic system which indicates the position of the rotary valve, determines which zone is in the pumparound loop, and sets the correct pumparound flow setpoint.

The drive mechanism for the rotary valve is a simple ratchet and pawl assembly that is driven by a hydraulic cylinder. See Figure V-4. The drive assembly moves the rotor plate in a stepwise fashion through the 24 possible positions. The hydraulic cylinder forces a pawl into the ratchet which is attached to the shaft which passes through the seal on the top head of the rotary valve, through the rotor plate and is positioned by the bushing in the bottom head of the rotary valve. Each stroke of the hydraulic cylinder forces the ratchet to advance by one tooth.

The rotary valve directs the flows to the correct beds of the adsorbent chamber by aligning the crossover pipes of the rotor plate to the ports of the bottom head of the rotary valve. The alignment is set by adjusting the stroke of the hydraulic cylinder that adjusts the position of the rotor plate.

Due to deflections in the components of the drive assembly, the position of the end of the shaft nearest the drive is not an accurate indication of the position of the rotor plate. A sight port is provided such that the actual position of the rotor plate can be determined during normal operation. Two vertical wires are included in the sight assembly. By aligning the wires with a target attached to the rotor plate the position of the rotor plate can be determined. Figure V-5. The wires can be calibrated when no hydrocarbon is in the rotary valve by comparing the alignment of the rotor plate to the bottom head of the rotary valve by looking through one of the ports. Figure V-6.

A hydraulic system is provided to drive the rotary valve. A reservoir, hydraulic pumps, a cooler, hydraulic accumulators, and valves are provided to drive the rotary valve. Hydraulic pressure is developed by the hydraulic pumps by pumping the hydraulic oil from the reservoir to the hydraulic accumulators. The hydraulic pressure is used to drive the hydraulic cylinder by the operation of valves in the lines between the hydraulic accumulators and the hydraulic cylinder and the reservoir. Excess heat developed in the hydraulic oil is removed in the cooler.

For a more complete description of the rotary valve, see the documentation provided with that equipment.

RV Stepping in Molex Units

UOP recommends that the rotary valve not be stepped when the rotary valve is not seated. When the valve is not seated, the shaft wind up will be different than in the seated condition that will significantly change the alignment. Since the valve alignment is set when the valve is seated, stepping the rotary valve unseated will cause misalignment of the rotor plate to the track plate of the rotary valve. A second concern when the rotor plate is not seated the rotor plate may not be in complete

contact with the track plate of the rotary valve. If the valve is stepped in this condition, there is a higher probability that some debris could be trapped between the rotor plate and the track plate resulting in damage to the track plate.

B. ADSORBENT CHAMBERS

Two chambers are provided to hold the adsorbent loaded into the unit. The adsorbent in each chamber is divided into 12 beds by the chamber internals or "grids." See Figure V-7. The top grids act as a distributor and are seal welded in place to isolate the semi elliptical heads from the adsorbent. The intermediate grids between the top and bottom grids act as distributors, collectors, adsorbent supports, and baffles in the adsorbent bed. There is no seal between the intermediate beds. Each grid section is attached to the distributor that divides the bedline flow into the grids. Within each grid section is a conduit that has holes in it to act as the distributor for the flows that enter the adsorbent chambers via the bedlines. See Figure V-8. These same distributors act as collectors when a stream is withdrawn from the adsorbent chamber through the bedlines. Each set of grids supports the adsorbent above it to distribute the hydraulic load between the grids. The grids carry the load to the walls of the adsorbent chambers. Most of the area of the grids is covered by the internal baffle of the grids. Ceramic rope is packed between the grids and the vessel to force the liquid passing down the vessel walls out onto the grids. The internal distributor, baffles, and supports of the grids are sealed by screens on the top and bottom of the grids. The screens prevent migration of the adsorbent through the distributors. The bottom grids act as a collector at the outlet of the adsorbent chambers, support the lowest bed of adsorbent in each adsorbent chamber and seal the adsorbent beds from the semi elliptical heads of the adsorbent chambers. The bottom grids are seal welded in place.

Although the top and bottom grids are seal welded in place, they would be damaged if a large differential pressure could develop across the grids. In order to maintain a low differential pressure across the top and bottom grids, a vent is placed in each of the sealed grids. To prevent contamination of the process fluids in

the adsorbent by the material in the adsorbent chamber head, the vents are made in the form of a small channel. By purging clean desorbent through the vents and out to the raffinate column, contamination from the process cannot reach the large volume of the adsorbent chamber heads and the majority of the desorbent used for the flush does not need to pass into the adsorbent chambers where it would occupy adsorbent capacity.

C. FILTERS

Recently, UOP has received unprecedented reports of performance failures of feed and desorbent filters. Specifically, these reports have involved cases in which the filter elements were (1) not installed correctly or (2) improperly supplied.

1. To reduce the possibility of such failures, this advisory recommends that the filters be installed to operate in series, that is, in lead-lag operation (Figure V-9).

Even though the operation of the filters in series flow increases the complexity slightly, it minimizes the possibility of filter failure causing damage to the rotary valve or the adsorbent chamber internals.

The recommended setup calls for the filters to be in series and to have additional piping that allows the relative position of the filters to be interchanged.

Instrumentation should be provided to monitor the pressure drop of each filter when it is operating in either position. Normally, when the lead filter reaches the end of its useful life, as indicated by an increase in pressure drop, it is taken off-line, and the elements are changed. The clean filter is then returned to service in the lag position. Pressure drop in the lag filter indicates that the lead filter is not functioning properly (Figures V-10A-10D).

A clean filter has an approximate pressure drop of 0.14 kg/cm^2 (2 psi). UOP recommends changing the filter elements when the pressure drop reaches 1.05 kg/cm^2 (15 psi). The differential pressure indicator of the filter board should have a range of 0 to 1.5 kg/cm^2 (0 to 21 psi) and an alarm at 1.0 kg/cm^2 (14 psi).

UOP believes that the recommended two-filter, series-flow arrangement represents an enhanced and relatively inexpensive safeguard against the effects of filter failure. Additional protection can be achieved by using a third filter piped to allow its placement into the series-flow arrangement in the lag position before one of the two filters already in service is decommissioned. In such an arrangement, two filters would always be in series even when one is being changed.

This recommendation applies equally to both the desorbent and feed filters.

UOP also recommends that the filter elements be changed out every six months regardless of the pressure drop observed across the filters.

2. UOP has learned that in some cases, filter vendors are not supplying filter elements that meet UOP specifications. UOP recommends that the filter elements meet the following specifications:

- Fiber wound depth cartridge elements.
- Fiberglass as the filter media.
- Filter elements should have an efficiency of at least 99.8% per ASTM F 797 with Coarse test dust at one minute after start-up.
- Single piece (that is, no stacking, no joints, no glues, sealants or elastomers in the elements).
- Stainless steel metal core.
- Internal relieving systems that can bypass material around the filter elements are not acceptable.
- Specified to nominally exclude 10 micron particles for both desorbent and feed filters. Until the early 1990s, UOP had specified 100 micron for the

feed filters and 10 micron for the desorbent filters. Although this previous specification was found acceptable for many years, UOP believes this simplification of our specification meets the majority of requests received from customers to specify only one type of filter. This change can be simply handled when ordering replacement filter elements.

The licensee should verify that the filter elements supplied and installed meet all of these specifications.

Suggested Supplier:
Perry Equipment Corporation
Filtration Division
P.O. Box 640
Mineral Wells, Texas 76068
U.S.A.
Tel: (940) 325-2575
Fax: (940) 325-4622

D. LINE FLUSH PUMPS

The line flush pumps provide the hydraulic head to move line flush material from one bedline to another. The line flush pumps are single stage process pumps. The line flush pumps share a common spare pump.

E. CHAMBER CIRCULATION PUMPS

Chamber circulation pumps supply the necessary head to allow the proper circulation flow to pass from one adsorbent chamber to the other. The chamber circulation pumps are single stage process pumps. The chamber circulation pumps share a common spare pump.

F. MOLEX FEED SURGE DRUM

The hydrotreated stripper bottoms product (Molex feed) is typically directed to the adsorbent chambers from the feed surge drum (Figure V-11). This vessel serves two functions: a) during normal operations it provides the necessary surge capacity so as to enable maintaining a constant feed rate to the adsorbent chambers; and b) during startup and off-spec periods, both the extract and raffinate column bottoms streams are recirculated back to the surge drum (termed "short circulation"). The surge capacity provided will help dampen any composition variations during these operations. The surge drum has a pressure equalization line that ties into the raffinate column. A level recorder is provided for purposes of continuous monitoring of the liquid level in the drum.

G. EXTRACT AND RAFFINATE MIXING DRUMS

Mixing drums (Figure V-12) are located upstream of the extract and raffinate columns. The purpose of these vessels is to smooth out concentration fluctuations that could upset column operations. The inlet distributors inside of the drums contain several specially designed eductor nozzles that promote thorough mixing. The vessels must be liquid full for proper operation. Vent lines are provided on top of the drums for removal of any gas.

H. DESORBENT DRIER SYSTEM

The desorbent drier serves to remove from the desorbent the small amount of water that leaves the adsorbent chambers. The feed to the drier may be a flow controlled slipstream from the desorbent surge drum pump discharge through the cooler and drier and back to the surge drum; or alternatively, it may emanate as slipstreams from the extract and raffinate column net overhead liquid streams.

The drier is normally filled with 3A molecular sieves. When it becomes saturated with water, it must be regenerated. The regeneration is accomplished with desorbent vaporized in a hot oil heater. The excess liquid contained in the desorbent drier is displaced with vapor to the regeneration surge drum. The normal regeneration is then established.

The circulation pump takes suction from the coalescer and flow proceeds through the heater, downflow through the drier to the condenser and back to the coalescer.

The water separates from the condensed vapor and collects in the coalescer boot. When the regeneration is complete the coalescer is drained and refilled from the regeneration surge drum. Liquid is circulated through the drier to cool it to the normal operating temperature before it is returned to service. The desorbent drier is usually a package system supplied by others. The manufacturer should be consulted for specific operating instructions.

The drier will normally operate on a seven day drying cycle and then be regenerated for 24 hours. This will, however, depend on the quantities of water to be removed from the desorbent.

This system is not included in newer design.

I. TURBINE METERS

Turbine meters are used instead of orifice meters to measure the F, D, E, Hi, Ho, Hz, and pumparound flows because of the higher accuracy they afford.

The turbine meter element is a tubular housing flanged into a pipe of the same diameter and inside which a vaned rotor is made to spin on an axial shaft by movement of fluid through the pipe.

The rotation of the rotor is sensed by a magnetic pickup that transmits a pulse each time one of the rotor blades passes the pickup coil. The frequency of these pulses is proportional to the volumetric flow rate of fluid. The frequency is converted to a standard milliamp signal to the FRC.

The axial position of the turbine rotor on its shaft is maintained by hydrodynamic balance. If foreign material enters any of the passages that effect that balance, the turbine will quickly destroy itself, so filters or strainers are provided upstream of each turbine meter.

J. CHAMBER CIRCULATION TURBINE (OR VORTEX) METER

As implied above, the pumparound FRC is intended to keep each zone rate constant as the zone moves around the adsorption circuit. However, imprecision in the FRC settings, as well as erroneous turbine meter readings, can cause the zone rates to vary depending on the zone rate being controlled by the FRC.

In the circulation line from the bottom of Chamber 1 to the top of Chamber 2 is a flow meter. This meter is provided to permit an independent check of the constancy of zone rates not in the pumparound FRC; any variation in a zone rate implies an inconsistency between the zone rate set points and the net input or withdrawal flow rates. The vortex meter recording is similar to that of the pumparound FRC, except:

- a. It is not as accurate as a turbine meter.
- b. It is out of phase with the pumparound trace by 12 beds.

The inferior accuracy of the vortex meter does not matter; however, it is used only to look for step changes in flow rate within a zone, and for that it is quite sufficient. In fact, the zero and range of the vortex meter recorder may be adjusted to expand the trace to fill the chart for better precision.

K. DESORBENT TANKAGE SECTION

A typical desorbent tankage section consists of a separate tank for each desorbent component (nC_5 , iC_8 and pX) plus a tank to hold the desorbent blend. All of the tanks are nitrogen blanketed. Three pumps are provided. One is for pumping

desorbent components from drums into their respective storage tanks. A second is used to transfer components into the blend tank and also to circulate the blend tank. A third pump is provided to pump the desorbent blend, or individual components to the raffinate column. A diagram of the desorbent storage section is shown in Figure V-13.

Figure V-1
Rotary Valve Assembly

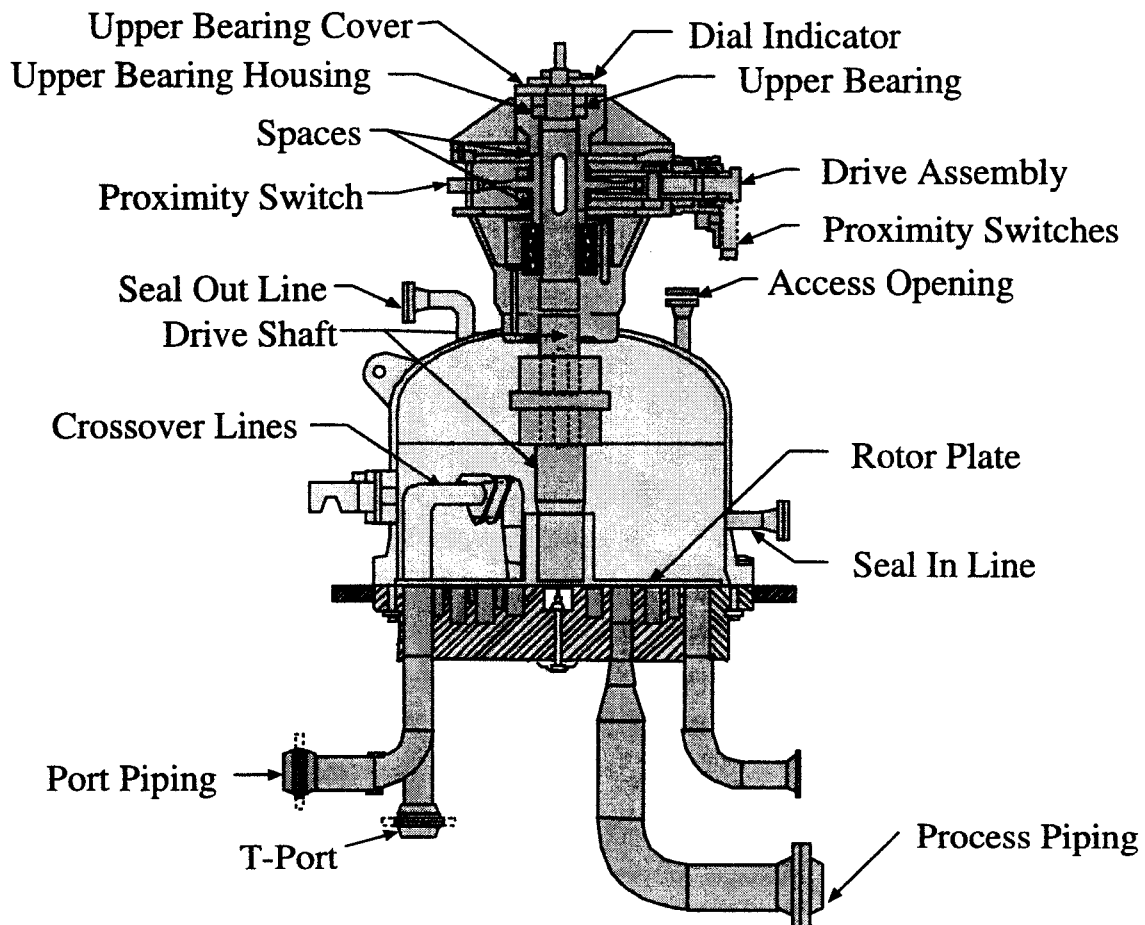


Figure V-2
Rotary Valve

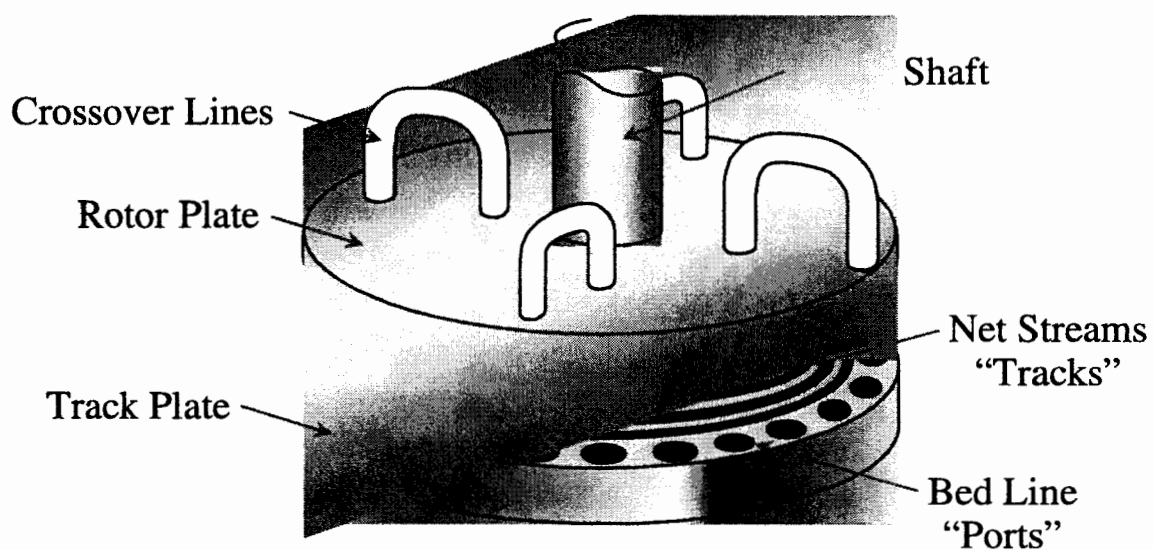


Figure V-3
Rotary Valve Assembly

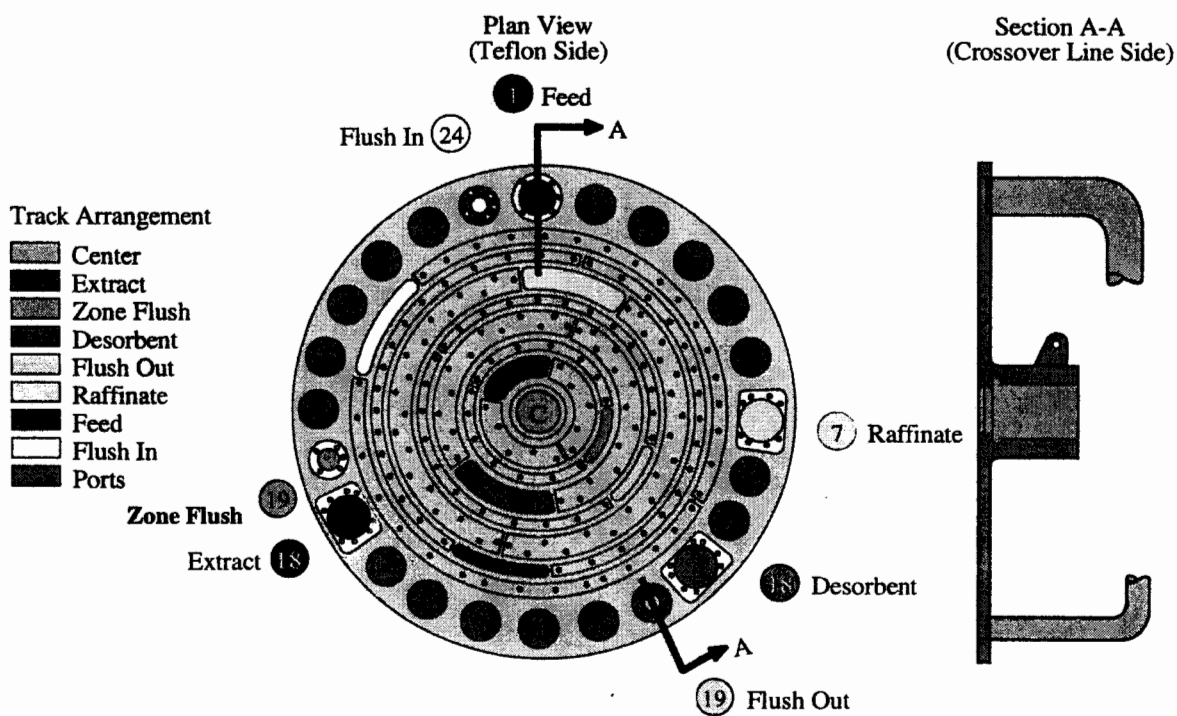
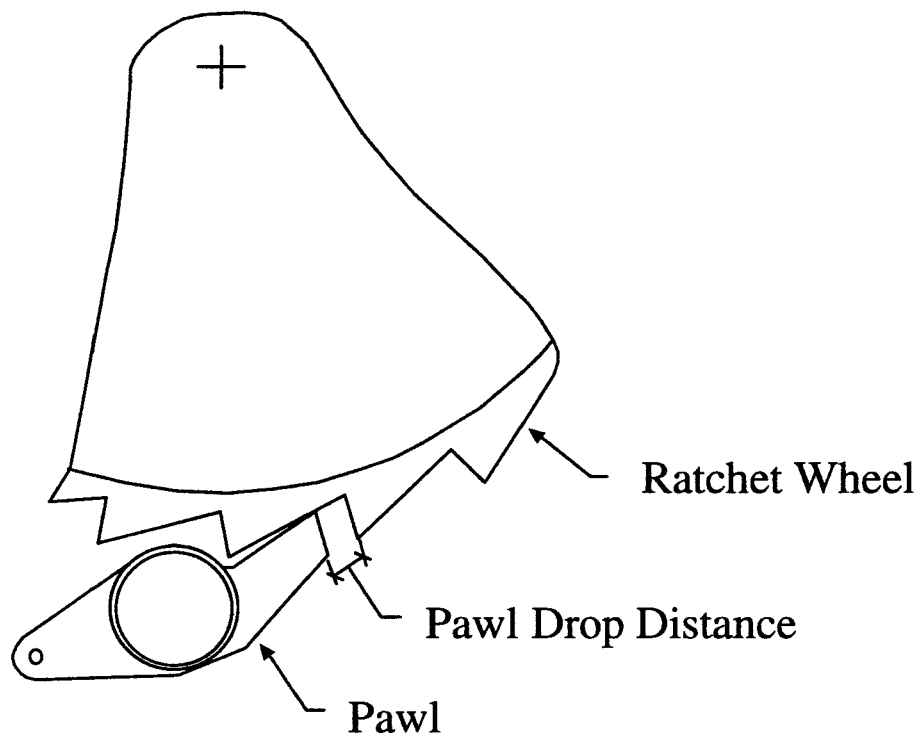
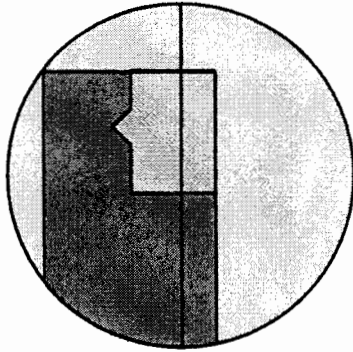


Figure V-4
Ratchet Wheel

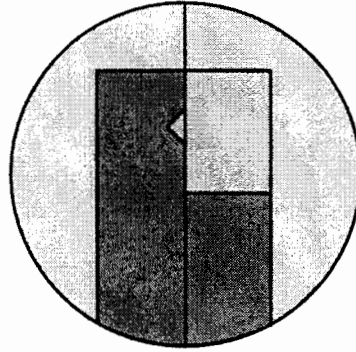


Top View of Ratchet Wheel

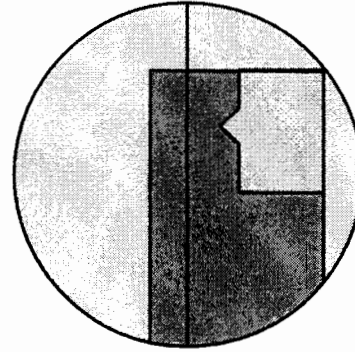
Figure V-5
Target Sight Assembly



**Retarded
Alignment**

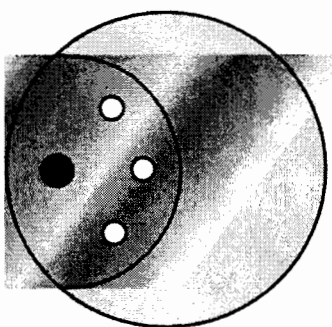


**Correct
Alignment**

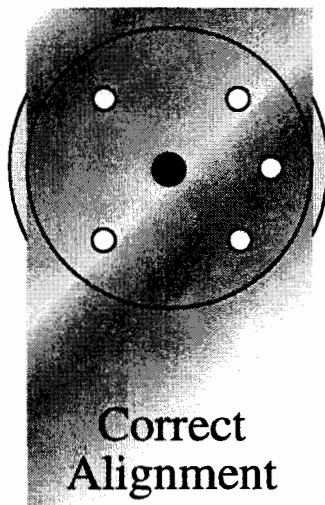


**Advanced
Alignment**

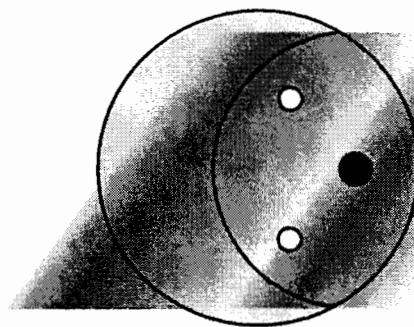
Figure V-6
T-Port Alignment



**Retarded
Alignment**



**Correct
Alignment**



**Advanced
Alignment**

Figure V-7
Adsorbent Chamber

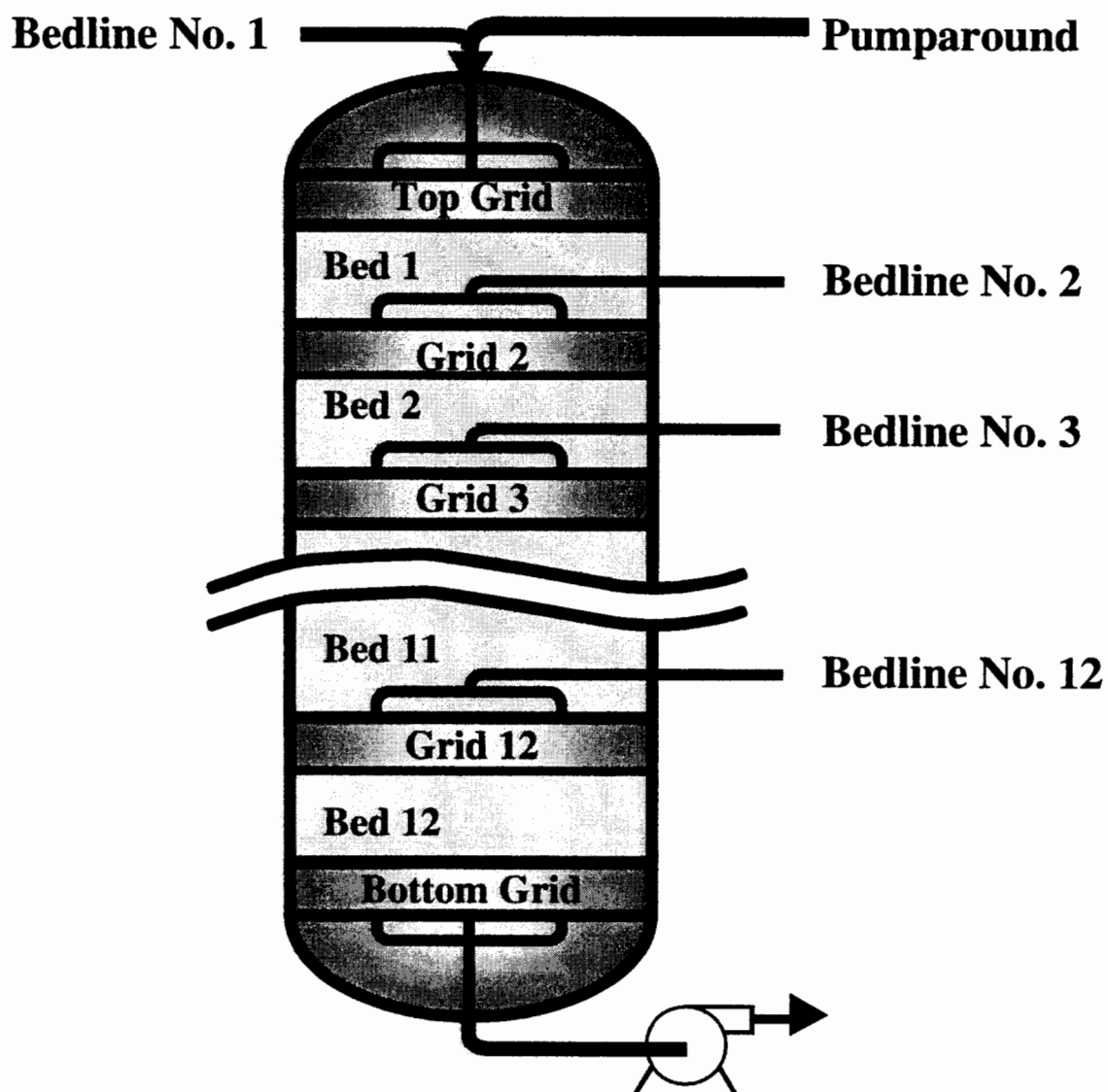


Figure V-8
Flow Through Grids

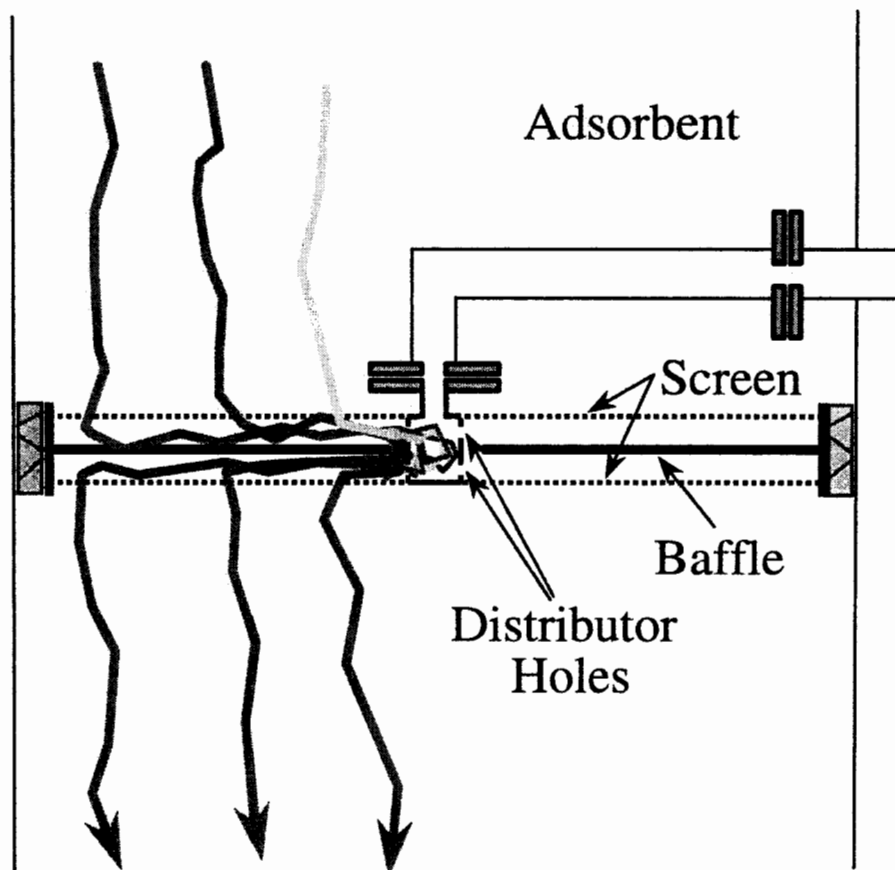
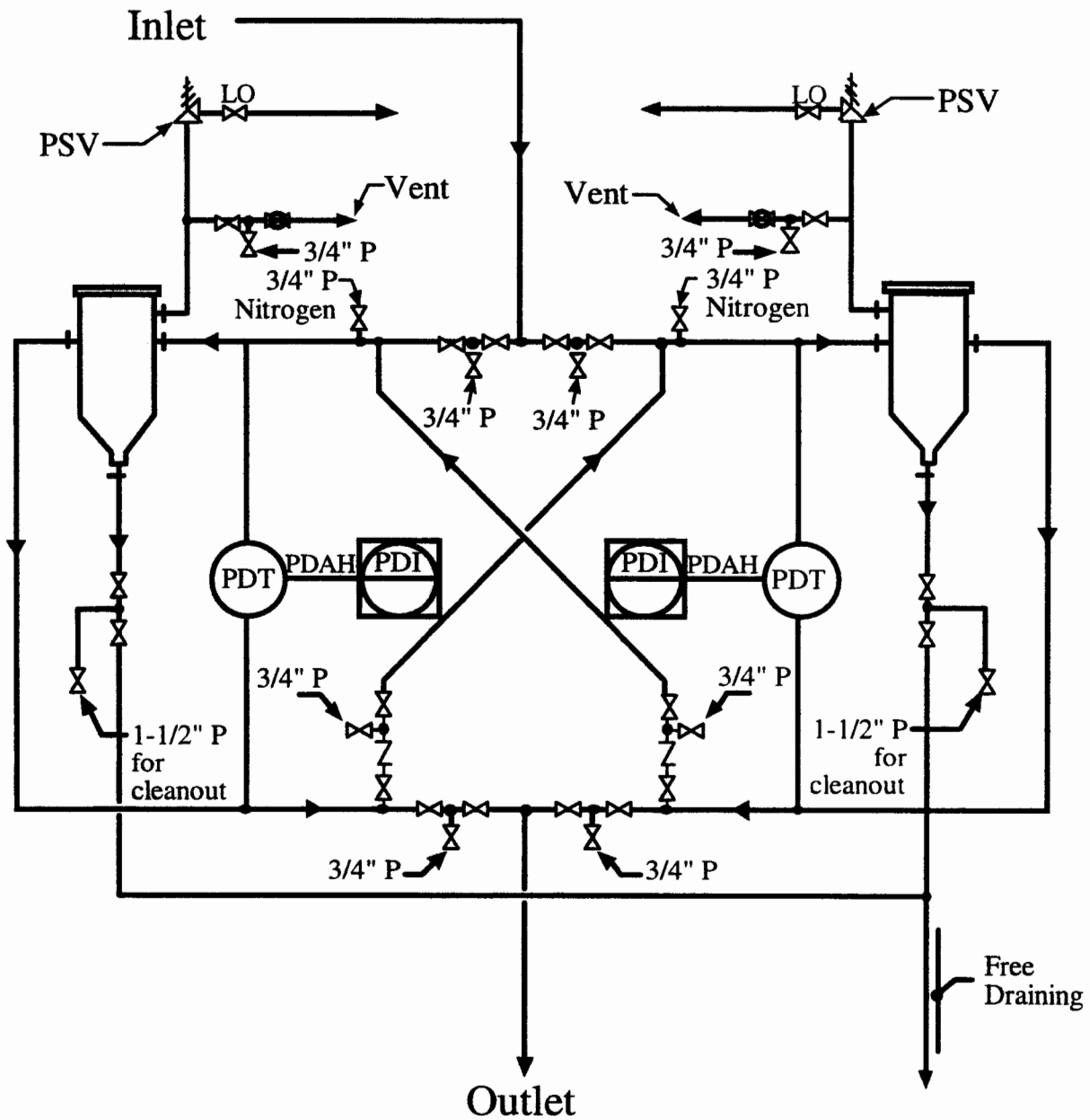
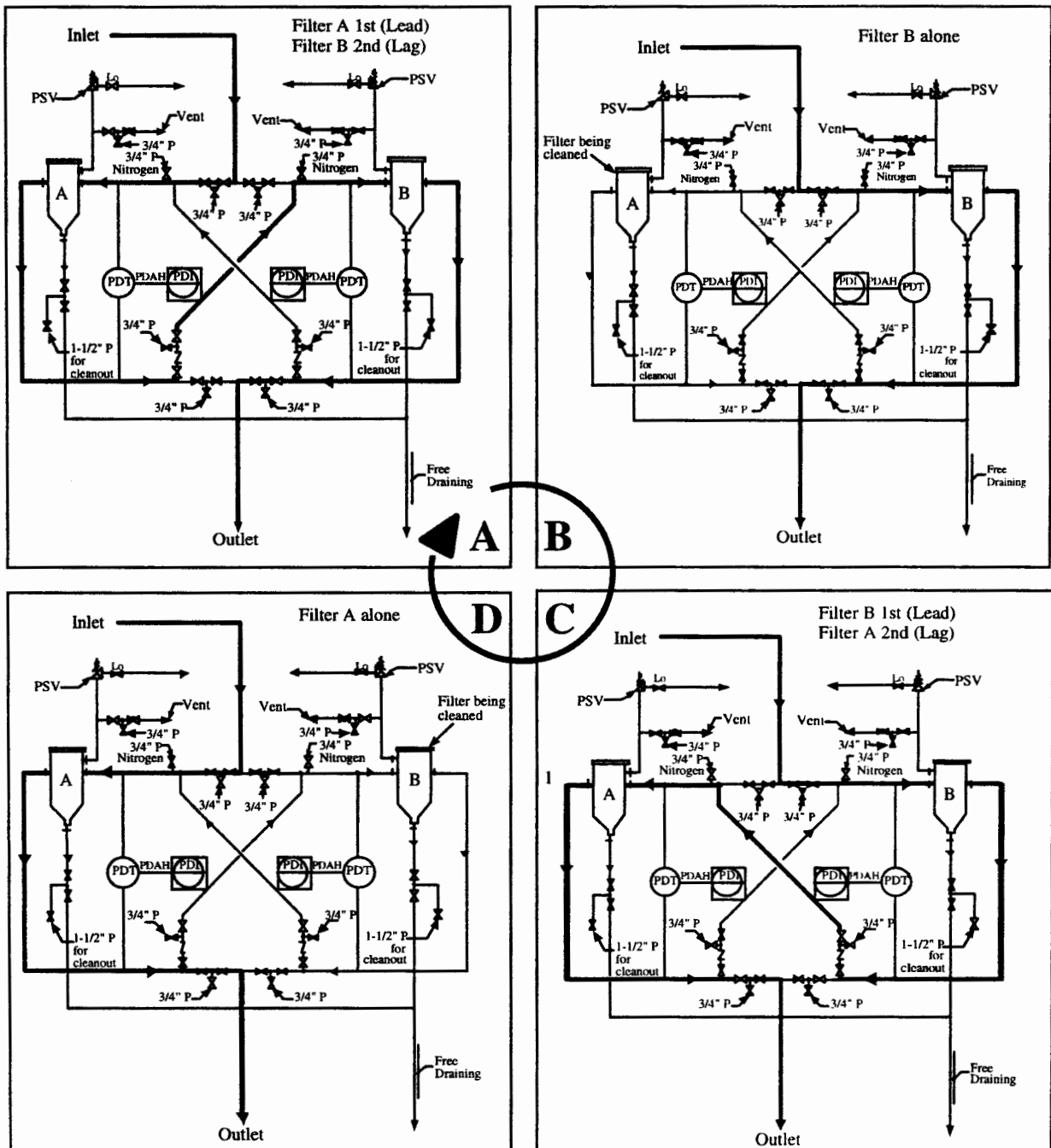


Figure V-9
Desorbent or Feed Filters



PRX-R00-111

Figure V-10
Filter Rotation



PRX-R00-112

Figure V-11
Feed Surge Drum

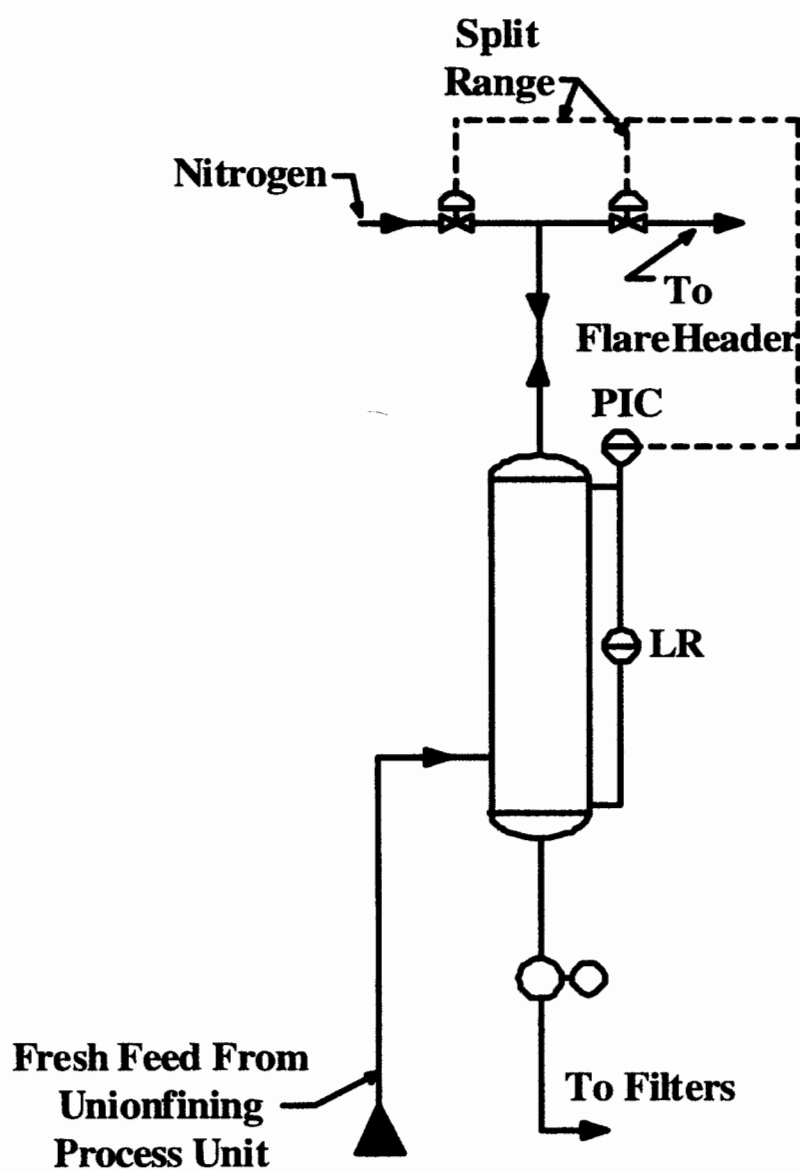


Figure V-12
Mixing Drums

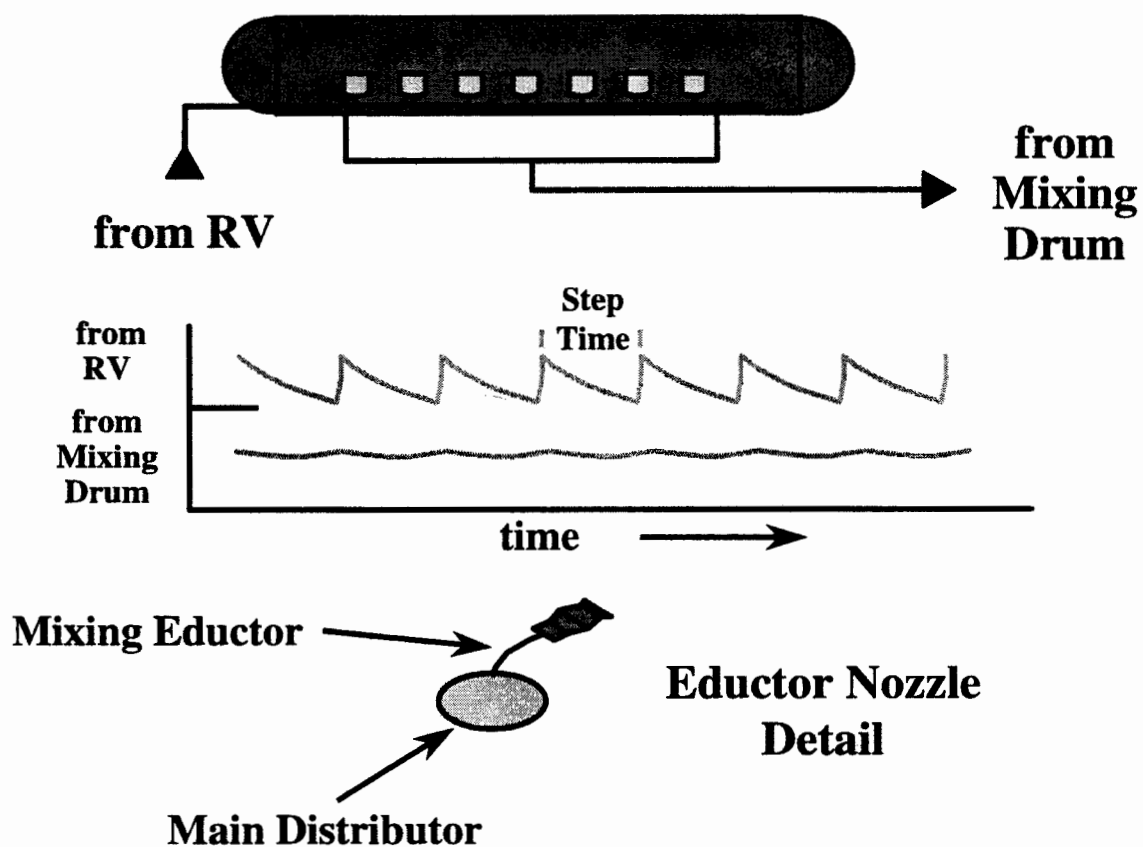
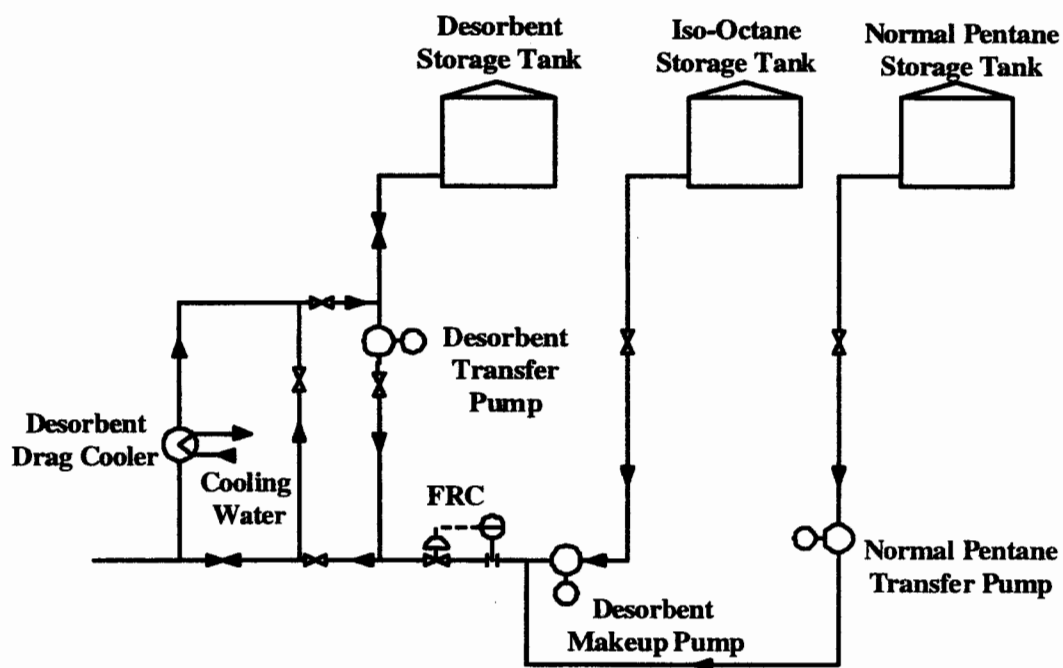


Figure V-13
Desorbent Storage







VI. COMMISSIONING

A. INTRODUCTION

When the unit is being commissioned for the first time, significant planning and work will be spent on the prestartup requirements and initial startup procedures. This includes preparing both the unit and adsorbent.

B. PRESTARTUP REQUIREMENTS

As the construction of the unit nears completion, the startup crew must begin to prepare it for operation. These precommissioning activities have three main purposes:

- (1) to ensure by thorough inspection and testing that the unit is safe, operable, and constructed as specified;
- (2) to prepare equipment for operation by washing, running in, etc., and
- (3) to acquaint the operators with the unit.

The importance of these activities cannot be over-emphasized. No matter how well designed a unit is, if the equipment provided is not what was called for, is not properly broken in, or is not understood by the operators, it will not perform as expected.

The material in this section gives general guidelines for preparing a unit for startup. Some sections need to be expanded to give specific directions (water washing procedure, for example); this can best be done by personnel on site.

1. Vessel Inspection

Vessel inspections can be made at an early stage of construction. The actual installation should be compared to the vessel drawings. Particular attention should be given to the following details:

- a. Vessel temperature and pressure rating
- b. Vessel metallurgy
- c. Vessel elevation
- d. Vessel lining
- e. Trays: number, type, spacing, levelness, liquid seal, weir height, valve number and operation, metallurgy, weep holes, supports
- f. Distributors: location, alignment, size and number of openings
- g. Nozzles
- h. Vortex breakers
- i. Demisters: size, materials, support
- j. Thermowells: location and length
- k. Level instruments
- l. Linings
- m. Fireproofing
- n. Inspection plates in place
- o. Cleanliness

CAUTION: Before entering a 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.**

2. Piping and Instrument Check

The unit should be checked to ensure that it conforms to the UOP Piping and Instrument Diagram. This can best be done by coloring every line and notation on the drawing with marking pencils as the check is done. In addition to the process flow, the inspection should include the following:

- a. Line sizes and materials
- b. Flanges and gaskets
- c. Bolting
- d. Drains and vents
- e. Pump ratings
- f. Relief valve settings
- g. Lines and equipment supports
- h. Equipment accessibility
- i. Expansion provisions
- j. Utility lines
- k. Tie-ins with other units/storage
- l. General safety requirements
- m. Heaters including burners and internals

3. Strength and Leak Testing

Any equipment – whether piping, vessels, or exchangers – which has been modified, repaired, or had connections made to it by welding during a downtime or construction period preceding a startup should be **STRENGTH TESTED**. This strength testing is normally done by hydrostatic pressure. When hydrostatic pressure testing is performed in the field, operating personnel assigned to witness and record the test should determine that blinds have been properly placed to isolate the equipment to be tested from all other equipment which has a lower design pressure.

All equipment newly constructed or opened during a downtime should be **LEAK TESTED** by refinery operating personnel before any hydrocarbon is admitted. Normally flanges and connections found to be tight under hydrostatic test do not need to be leak tested again provided these lines are not subsequently opened.

Distillation towers and certain associated piping can be leak tested under steam or N₂ pressure during the air freeing operation.

NOTE: Although leak testing as above gives reasonable assurance of startup without serious leaks, it must be borne in mind that leaks often develop after equipment has reached full operating temperature, when lines and vessels have expanded. Operators must be alert as startup progresses for development of leaks.

- a. Blind (or block and bleed) all safety valves, instruments, and pressure gauges that would be overpressured by the hydrostatic test pressure.
- b. Test Gauges must be placed at the low point of a large line or vessel so that the liquid head will be taken into account.
- c. High Point Vents must be provided on all lines and vessels to allow purging out of all air when pressuring with H₂O.

NOTE: Before draining any liquid from a vessel, a vent must be opened on top to avoid pulling a vacuum and collapsing the walls.

- d. Hydrostatic Test Pressure is normally set at 1.5 times the cold design pressure, local codes prevail however. In some cases, vessels that have been shop tested cannot be hydrostatically tested in the field and must be blinded from the process piping under test. These are vessels that are so designed and/or supported that they cannot safely be filled with water.

The refinery piping code ANSI B31.3 must always be followed when testing process piping.

- e. Fine Mesh Screens should be placed in the suction lines of all pumps before any liquid or gas is allowed to pass through them from newly constructed or dirty lines. If this is not done, scale, weld metal, slag, stones, etc., may freeze or damage the impellers of the pumps. The screens also act to trap such dirt in a place where it can be conveniently removed. Suction screens are normally placed in a flange or T-type strainer (for larger lines) between the suction valve and the pump. The screens should be made of fine screen retained by a heavy gauge, punched hole, cone-type strainer. These should

be placed in the lines before the unit is pressure tested so that the test liquid can be pumped through the lines for the purpose of washing them (See: 4. Flushing).

- f. An Air Pressure Test using plant air can be placed on the sections of the unit prior to the hydrostatic test so that any open lines or flanges may be discovered and taken care of before water is admitted.
- g. Water Temperature. In pressure testing equipment, particularly in cold weather, care should be taken that the testing of the vessels is not carried out at temperature levels so low that the metal becomes brittle. As metal temperatures decrease, the tendency for brittleness increases. Temperatures above 16°C (60°F) are considered minimum for testing to eliminate the possibility of cold fracturing of equipment. Such temperatures can be attained by warming of the testing medium with steam.
- h. Isolate Sections. It is not practical to test all of the equipment together. Thus the unit will be divided into sections as governed by the location of the various items of equipment and the test pressures to which each item will be subjected. Suitable blinds must be made up for insertion in nozzles and between flanges to isolate the various sections of equipment as required by different pressure ratings. Normally the exchangers, receivers, etc., for the various towers will be tested together with the piping.
- i. Exchangers. Special attention should be given to testing of heat exchangers and coolers using shell and tube type construction. If there is a possibility that materials such as heating fluid or water can enter the process side of such exchangers, the tube rolls should be given a very careful hydrostatic test. Any leaks detected, however small, must be repaired before the unit is considered ready for startup.

NOTE: Check exchanger vendor name plate test pressure regulations in cases where differential design is used on the tube bundles.

4. Flush Lines

All piping should be thoroughly cleaned before startup. This is a very important step since dirt, rust, scale, and other foreign objects, may plug the control valves, exchangers and pump strainers causing startup delays. Generally, lines designed to handle liquids are flushed with water and drained. Lines designed to handle gas can be cleaned by blowing with air or nitrogen or, if their supports are designed for liquid, flushed with water. Frequently a convenient source of water is that which has been used for hydrostatic testing. NOTE: If the adsorbent chamber piping or bed lines are rusted, an acidizing flush step may have to be performed. This decision should be made by the UOP Chief Operator, after inspection. In the adsorbent chambers piping section, foreign material is critical as it may plug internal adsorbent chamber grid screens and/or damage the Teflon seal in the rotary valve. Ammoniated citric acid is the recommended acidizing agent. A few preliminary steps, if acidizing is required, are listed below:

- a. Install blinds where necessary to prevent acid from entering and damaging non-acid resistant equipment.
- b. All turbine meters, rotameters, and orifice plates should be removed; provide spool pieces where necessary.
- c. Instrument lead lines should be disconnected to prevent the possibility of acid entering the instruments.
- d. Jumper lines may be temporarily installed to allow circulation of acid through the lines downstream of the filters on the feed, desorbent, and flush, and through the 24 bed lines entering the chambers, if necessary.
- e. It is not necessary to remove chrome steels during acidization as they are not attacked by citric acid.
- f. Brass equipment should not be exposed to high ammonia concentrations. It is not recommended to exceed a pH of 8 during the final ammoniation step.

- g. Upon completion of the acidization, continue with the water flushing procedures as usual.

Notes applicable to water flushing are:

- a. Orifice plates should not be installed prior to flushing.
- b. Control valves should be rolled out. Flush to both sides of the valves.
- c. Instrument air lines should be thoroughly blown with clean, dry air, then closed off or disconnected.
- d. Relief valves should be blinded. (Set pressure can be checked at this time in the shop.)
- e. Dirty lines should not be flushed through exchangers. Disconnect lines at exchanger inlet and outlet and flush through the open end of the lines to the exchanger.
- f. Pump suction spool pieces should be rolled out and basket screens installed after flushing the pump. Pump suction and discharge piping should be flushed while the pump is disconnected.
- g. When filling or draining vessels, open a vent.
- h. When draining vessels, care must be taken not to pull a vacuum. Adequate venting or nitrogen pressure must be provided and water should not be drained to rapidly.
- i. Lines should be flushed away from vessels. Do not flush debris into equipment.
- j. Where possible, flush down or horizontally.

- k. Flush headers from source to end, then flush lines away from header to end.
- l. Flush through open end lines. When possible, do not restrict flow.
- m. Flush through all drains and vents.
- n. In all cleaning, use maximum safe volume and velocity.
- o. Regulate flushing water from source.
- p. Water flushed lines which do not drain freely should be blown clean with air.
- q. Upon completion of line flushing for any system, carefully check that all temporary breaks are reconnected, control valves replaced, blinds removed, and pumps aligned.
- r. Turbine meters should be replaced by spool pieces.

5. Run-In Pumps and Drivers

Proper installation and operation of pumps and drivers is essential for trouble-free performance. All drivers should be run in for a minimum of four hours at no-load conditions. Following this, pumps and drivers should be coupled, properly aligned, and serviced for operation. This initial run-in is usually done with water and frequently can be included with the water flushing. However, when the fluid used for run-in is heavier than what the pump was designed for, precautions must be taken to avoid overloading the motor.

Operation notes are as follows:

- a. Check rotation of electric motors uncoupled from the pump. Run in for a minimum of four hours uncoupled, verify operability.

- b. Run in, uncoupled from pump, drivers other than electric. Check overspeed trip, bearings, control, etc.
- c. Prior to running in the pump, do the following:
 - (1) Review the manufacturer's operating instructions.
 - (2) Check that the overall installation is complete.
 - (3) Verify that the pump and driver are properly aligned.
 - (4) Verify that cooling water piping is connected and in service where required: pedestals, bearing jackets, stuffing boxes, etc.
 - (5) Verify that steam piping is connected where required.
 - (6) Check gland or seal piping. Conventionally packed pumps in hot service are generally furnished with gland oil. When a pump is furnished with mechanical seals, verify that all components of the flushing system, such as strainers, separator, restriction orifices, and coolers have been correctly installed and are clean. It is very important that the flush system be clean as the loss of flush or dirty flush can cause the loss of seals.
 - (7) Verify that packing on seals is installed.
 - (8) Verify that temporary strainers have been installed in the suction piping.
 - (9) Verify that bearings and shafts are clean and properly lubricated.
- d. When ready to start the pump, do the following:
 - (1) Rotate pump and driver by hand, verifying that they will roll freely.

- (2) Check that run-in water supply and delivery circuits are lined up.
- (3) Establish cooling water, where required.
- (4) Check that lubrication is satisfactory.
- (5) Check that pump vent and case drains are closed.
- (6) Open suction valves fully, venting air from piping and pump, completely filling with liquid.
- (7) Open pressure gauge tapping.
- (8) Make sure that electric power is available from the switch gear to the starter of the electric motor drivers. For steam turbine drivers, the turbine is warmed by slowly opening the exhaust valve. Back-in steam while draining water from the casing. The exhaust valve is always fully opened before starting. Where the exhaust system is under vacuum, the case must be warmed by gradually introducing the driving steam, after opening the exhaust valve. In a vacuum exhaust system, sealing steam to the turbine shaft labyrinths must be admitted to avoid loss of vacuum.
- (9) Barely open the discharge valve of a centrifugal pump and fully open the discharge valve of a positive displacement pump before starting.
- (10) Gradually warm up pumps in hot service to near operating temperature before placing in service. Uneven expansion of the internals and casing may cause seizure and damage when starting.
- (11) Start the pump and bring up to speed immediately, observing the discharge pressure. If the pressure does not build immediately, stop the pump and find the cause. Refill the pump and restart when the previous difficulty is corrected.

- (12) When the discharge pressure of a centrifugal pump has increased satisfactorily after starting, gradually open the discharge valve to obtain the desired flow rate.
 - (13) In the event of unusual noise, vibration, overheating or other abnormal conditions, shut the pump down immediately. Correct the cause before resuming operation. Continue checking for abnormal conditions as these may occur after prolonged operation.
 - (14) 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. A mechanical seal may show leakage on startup. After the pump has been started and stopped a few times, the leakage should stop. If the seal leakage persists, then steps are to be taken to correct the problem.
- e. The pumps are normally delivering a material of lighter weight than the water circulated during initial run-in. The pump driver is sized for the normal pumping fluid; consequently, while pumping water, the electric motors are easily overloaded.
- To avoid overloading the motor driving a centrifugal pump, the flow must be limited by throttling the pump discharge valve. If possible, check the amp usage against design. Avoid restricting the discharge to the extent that it will cause internal slippage and excessive heat generation.
- f. The discharge valve of an operating positive displacement pump, rotating or reciprocating, should never be closed as the case can be overpressured.
- g. During run-in, the strainers may cause some restriction on flow. As debris is collected, flow to the pump will fall off. Do not permit the pump to cavitate, that is, lose suction, which may cause damage to the pump.

- h. Inspect and clean screens as required.
- i. Recheck and realign, if required, after any disturbance of piping such as required for suction screen cleaning.
- j. Operate the pump directing flows through all suction and discharge piping.
- k. In shutting down a centrifugal pump, close the discharge valve first, thereby maintaining discharge pressure while the pump rolls to a stop. This also prevents rolling backward after shutdown if the discharge check valve leaks. Positive displacement pump discharge valves must not be closed until the pump is shut down and rolling stops.
- l. After all lines available to the pump have had suitable flushing, the temporary suction strainer may be removed only after showing no debris on two successive examinations. The permanent strainers may then be installed where required.
- m. Packing, in conventionally packed pumps normally not in water service, must be replaced upon completion of run-in screen removal.

6. Clean and Service Utility System

For ease of operation, the utility system (water, air nitrogen, steam, fire main) should be put into service as soon as the construction schedule allows. Fuel gas, fuel oil, and flare lines should be prepared for service but not commissioned until the unit is prepared to receive hydrocarbons. The various lines must be tested for leakage and washed free of debris. Steam lines can be conveniently cleaned by blowing them as long as necessary with steam. They should be warmed up slowly to minimize water hammer. The other utility lines can be cleaned by blowing with steam or air, or by water flushing if possible.

Commissioning of the steam system requires certain precautions. Hazards that should be avoided include thermal and mechanical shock, overpressuring of steam turbine casings, and introduction of condensate into other systems.

Before bringing steam into any system, all drains, vents, and bypasses on steam traps should be opened. Steam should be very carefully cracked into a system by slowly opening the block valve until steam is heard to just pass through the valve. Withdraw condensate through drains and warm up the system slowly in order to avoid water hammer and thermal shock. Keep drains and vents open until dry steam issues from each point, then throttle valves back slowly until the lines are at the desired pressure. Place all steam traps and condensate lines in service.

After commissioning the steam, block valves at vessels, snuffing steam valves, atomizing steam valves, and turbine steam supply valves should be kept blocked in.

All feed, chemical, and product storage tanks must be thoroughly washed, leak tested, dried and air free prior to bringing in hydrocarbons. Where possible, flush all lines to and from these storage tanks, and run a "pig" through these lines to assure that they are free of debris. Blow out the main volume of water with N_2 , then tape the flanges and check for leaks. When storage tanks are air free, leave them under a positive N_2 blanket.

7. Service and Calibrate Instruments

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

- a. Visual inspection of instruments to verify compliance with specifications
- b. Visual inspection of instrument installation to verify compliance with specifications and installation details in the instrument instruction manual
- c. Pressure testing of instrument piping

- d. Testing and calibration of instruments
- e. Testing of instrument air signal lines
- f. Testing of thermocouple wiring
- g. Loop checking
- h. Final preparation

When 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 or 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 for proper coverage and insulation. Orifice installations should be checked as soon as possible to ensure that straight run requirements are met, so that only piping change required can be made.

Instrument piping should be inspected visually for completeness and condition of welds and connections, 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 service (such as charge and product meters). Temperature transmitters should be checked for proper thermocouple burnout protection. Meter factor tags and valve position indication on controller output indicators should be installed at this time, and control actions should be set.

Instrument air signal lines should be checked for leakage, kinked tubing, and, for connections to the proper port of shutdown, solenoid valves and controllers. Thermocouple wiring should be inspected for proper polarity at the thermocouple head. Point by point verification of the switch identification at the temperature indicator on the operator's desk can be accomplished by selecting a point to be checked, opening the circuit at the thermocouple head, and checking the temperature indicator to see if it drives upscale or otherwise indicates an open circuit. The same applies for TRC instruments on the control board. Loop checking involves checking inputs and outputs to ensure that piping and wiring has been properly done. A signal should be simulated at the transmitter, and the control board instrument should be watched for response. The controller output should be adjusted manually, and the control valve watched for response. Special attention should be given to split range loops and complicated systems.

Final preparation includes checking instrument piping to verify that the valves are in the proper position, making preliminary control mode adjustments, filling seal pots and seal legs, 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.

In general, analyzers should be inspected, calibrated, and commissioned in accordance with procedures and instructions in the instruction manual for the analyzer. The specifications for the chromatographs include the services of a competent service engineer to review installation and supervise calibration and initial startup. Chromatographs should be inspected to see that all external piping and wiring is complete, and that no parts have been lost or damaged in shipment. No other action should be taken until the arrival of the service engineer.

8. Tubular Heater Exchangers

A tubular heat exchanger consists of a tube bundle housed inside a cylindrical shell. Flow through the tube bundle is isolated from the flow in the shell. By the use of internal baffling, flow through the tube bundle can be redirected to make several passes through the exchanger. Allowance for relative thermal expansion of the tube bundle and the shell during operation is included in the tubular exchanger design. Tubular exchangers are used for a wide range of applications including condensing, cooling, heating and vaporization. Attention should be given to the following details during inspection:

- a. Metallurgy and temperature/pressure ratings of shell and tube.
- b. Internal condition of tubes.

- c. External condition of tubes.
- d. Condition of tube sheet, baffles, tube supports, etc.
- e. Sufficient number and proper location of vents and drains.
- f. Proper piping arrangement in/out of tubes and shell.
- g. Installation of exchanger bypasses where specified.
- h. Installation and proper location of instrumentation and pressure relief valves where necessary.
- i. Inspection of tube to tube sheet strength welds. Strength welds are used to reduce the risk of cross contamination.

9. Fin-Fan Heat Exchangers

A fin-fan exchanger consists of a motor driven fan with one or more finned tube sections fixed between header boxes and mounted on a frame. The exchangers should be installed and commissioned prior to startup according to the manufacturer's recommendations. They are generally run in for a minimum of four hours with the blade pitch adjusted (when variable) to pull the maximum amperage on the driver. During checkout, attention should be given to the following details:

- a. Metallurgy and temperature/pressure ratings
- b. Condition of fins: free of debris
- c. Headers boxes: one anchored and the other free to permit thermal expansion
- d. Grounding wire connected
- e. Sufficient vents and drains
- f. Louvers: present, if specified, and operable from grade
- g. Alignment and coupling of motor and fan
- h. Bearings: clean and properly lubricated
- i. Free rotation of fan and motor

- j. Direction of rotation of fan
- k. Belt tension of fan drive pulleys
- l. Vibration switches set properly
- m. Fan pitch and speed: if variable, verify operation
- n. Guards: fans must be safely enclosed

10. Fired Heaters Dry Out (if applicable)

Before startup circulation is established, it is necessary to slowly expel the excess moisture from the insulating concrete of the fired heaters by gradually raising the temperature. During this initial period, it is preferable that no material be flowing through the tubes.

- a. For UOP-type heaters, the following dryout procedure and precautions apply. (Other types of heaters may require changes in the procedure that are to be made according to the manufacturer's recommendations.)
 - (1) Make a temporary installation of thermocouples for temperature measurement in the hip sections of the heater. The tips of these thermocouples should extend 15-20 cm beyond the inside face of the insulating concrete.
 - (2) Before starting to light any burners, all individual burner block valves are to be closed and the fire box steamed out to remove any gas accumulation. Open all burners.
 - (3) Light pilots and one or more burners, as required, in each section of the heater and fire slowly so that the temperatures in the fire box, as indicated by the thermocouples in the hips, are increased at a rate of about 15°C per hour until a level of 110-120°C is reached.
 - (4) Maintain this temperature for at least 48 hours, during which period the burner operation is to be rotated frequently in order to distribute the heat as evenly as possible throughout the length of the setting.

- (5) Light additional burners as required after the 48-hour period to increase the temperature at a rate of about 40°C per hour to a level of 500°C. Hold this temperature for ten hours.
 - (6) All of the burners should now be shut off and the heater allowed to cool slowly by keeping the air inlet doors and stack dampers closed.
 - (7) After the heater has been dried out, the temperatures can be subsequently raised or lowered at any desired rate unless the heater has been shut down for a prolonged period of time.
- b. The following is a general procedure for firing a heater on fuel gas.
- (1) When the unit is shut down, always blind off the fuel gas supply line because gas may leak through the block valves at the heaters and fill a furnace.
 - (2) Before lighting any burners, see that all individual burner block valves are closed and steam out firebox to remove gas accumulation. Make sure that the dampers are opened.
 - (3) Light each burner individually with a torch, opening the gas valve to each burner after the torch is inserted in front of the burner.
 - (4) Burners should be fired to produce a blue flame with a yellow tip, obtained by regulating the primary and secondary air supply. The furnaces should be checked over frequently for dirty burners that might give either too long, too short, or misdirected flame. There must be some excess of air to the burners so that an increase in fuel gas flow will have sufficient air to produce complete combustion. Proper spud size is important so that the burners will have the correct fuel gas pressure for operation. A hydrogen rich fuel usually requires burner pressures in the range of 1.25 to 1.50 Kg/cm² for best performance on most types of burners.

- (5) If, for any reason, the fires in a furnace go out, then:
- (a) Shut off gas supply immediately. This can be done by closing the main block valve in the fuel gas supply to the furnace. This will take care of any bypass and pilot lines that might be open around the control valves.
 - (b) Put snuffing steam in the fire box.
 - (c) Close individual burner valves and pilots.
- (6) As in all heaters, care must be taken that no flame impingement on the tubes is permitted.

11. Air Freeing

After completion of the water flushing and prior to admitting hydrocarbon, the plant must be purged to remove the air and all free water. It is assumed that all hydrotesting and tie-ins have been completed.

12. Chamber Evacuation and Nitrogen Purge

To prepare the adsorbent chambers for introduction of hydrocarbon material, all oxygen must be removed from the adsorbent chambers, rotary valve and connected piping. The air freeing procedure uses a steam ejector to evacuate the associated lines and equipment and a nitrogen purge line to pressure up and purge the system until the oxygen content is less than 0.3 mole-%.

- a. Prepare for air freeing by removing all blinds from the 24 bedlines.
- b. Close the low point drain and high point vent valves on each bedline. The rotary valve dome sealant inlet, dome sealant outlet, and seal vent lines should already be blocked closed.

- c. All process lines to the rotary valve: feed, desorbent, extract and raffinate, line flush in, line flush out, and secondary flush should be closed.
- d. Block in the suction and discharge headers of the chamber circulation pumps to isolate the pumps.
- e. The rotary valve dome equalization line to the raffinate line should be closed.
- f. The dome sealant lines to and from the rotary valve should be closed.

NOTE: It may be necessary to blind the suction of the chamber circulating spare pump before the ejector can be used if this pump cannot be isolated from the vacuum source.

- g. The chamber circulation lines to the top of the chambers are opened with the control valves in manual.
- h. Confirm that all adsorbent chamber head flush lines and head vents are closed.
- i. Isolate the instrumentation from the rotary valve, adsorbent chambers, and adsorbent chamber circulation lines to prevent damage by the vacuum.
- j. All turbine meters (feed, desorbent, extract, secondary flush, line flush, and pumparound) should not be installed, and spool pieces should be inserted in their place.

The turbine meters could be damaged by overspinning and overheating during the air freeing procedures as large volumes of gas will be used and gas has insufficient capacity to cool the bearings of the meters. It is also desirable to flush liquid through the unit before the turbine meters are commissioned since debris dislodged by initial circulations may damage the turbine meters. It is recommended that the unit be operating normally for a few days before the turbine meters are installed.

- k. Any other equipment that could be damaged by vacuum should also be isolated prior to starting the evacuation procedure.
- l. The evacuation/filling header at the bottom of the adsorbent chambers should be set up to the steam ejector but line to the ejector should remain closed until the ejector is in operation.

For the best results, the evacuation procedure can be performed on one adsorbent chamber at a time.

- m. Open the bedlines from the first chamber to be evacuated to the Rotary Valve.

Since the adsorbent can be damaged by free water it is recommended that the steam ejector be operating properly before connecting the adsorbent chambers to the ejector and that the chambers be isolated prior to closing the steam flow to the ejector. This should reduce the risk of steam being accidentally injected to the adsorbent chambers in the case of a malfunction of the steam ejector.

- n. Start steam flow to the steam ejector. When the ejector is functioning normally, open the evacuation line to the adsorbent chamber(s) and pull a 635 mm Hg (25 in. Hg) vacuum on the system. A pressure gauge at the ejector should be used for measuring the vacuum.
- o. Completely isolate the system. Using a second vacuum gauge installed at a convenient location, conduct a 20-minute vacuum test to verify tightness of the system.
- p. Pressurize the system to 0.5 kg/cm²g (7 psig) by introducing nitrogen into the circulation line upstream of the chamber being evacuated first.

- q. Repeat steps n. through p. at least 3 times until the system contains less than 0.3 mole-% oxygen. The vacuum leak test can be omitted after it is successfully completed once.
- r. Check the gas at the bottom of both adsorbent chambers to verify that the entire system is free of air.
- s. Pressurize the chamber up to 3.5 kg/cm²g (50 psig) with nitrogen after the system has been purged of air and block it in.
- t. Isolate the rotary valve from the chamber by closing the bedline ball valves on the 24 bedlines.
- u. Perform steps m. through t. for the second chamber.

The easiest way to assure that all piping is air-free is to divide the unit into sections (off-sites, chambers, columns, etc.) and purge each part separately. Normally, mechanical flow diagrams will be posted in the control room so that all piping can be inked out as the lines are purged and sampled for O₂. A final unit leak test can now be performed as all hydrotesting blinds should have been removed.

Steaming or evacuation is the most common method to air free a system. When a vacuum cannot be pulled on a vessel, the following procedure should be used.

- a. Nitrogen is brought into the vessels and lines and the system pressure brought to 7 kg/cm² (100 psig) (or available nitrogen pressure). While the system is under full nitrogen pressure, soap test all flanges and screwed connections for leaks.

NOTE: Always follow design pressure limitations when air freeing vessels. Most storage tanks are designed for a maximum of 3" (76 mm) of H₂O pressure and most fractionation columns are designed for 3.5 kg/cm² (50 psig).

- b. Depressure the vessels and lines to a low positive pressure 0.7 kg/cm² (10 psig). It is important that the system be depressured on the opposite end from which it is filled in order to be sure that a maximum amount of oxygen is removed in the depressuring process. Vent at each available low and high point vent in a downstream pattern.
- c. Perform steps a and b two times.
- d. 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. Theoretically, after the second displacement, the oxygen content should be down to near 0.5 mol%. If the analysis shows the oxygen content to be below, the nitrogen displacement step is complete. If the oxygen content is greater than 0.5 mol%, then repeat the nitrogen displacement procedure.
- e. Upon completion of the air freeing operation, leave all vessels and lines under a positive pressure of 0.3 kg/cm² (5 psig). Storage tanks are left at 25 mm (1 in.) of H₂O pressure.

All lines to and from offsite storage tanks and the unit battery limits must be air-freed. This includes the feed lines, chemical makeup lines, product rundown lines, and off test lines. By using nitrogen or steam, the lines can also be blown dry prior to admitting hydrocarbon.

13. Rotary Valve Control System

The rotary valve and the Adsorbent Section Control System (ACCS) should be prepared for operation in accordance the technical information provided by UOP in accordance with the warranties of the equipment. After the equipment has been prepared the operators should familiarize themselves with the operation of this equipment.

- a. The various graphic pages of the ACCS should be reviewed so that the operators learn the inputs required to operate the unit.
- b. The purpose of manual switches must be clearly understood.
- c. The location of the field station and its use must be understood.
- d. The alarms and their meaning should be reviewed.

Refer to the documentation supplied with the rotary valve and the rotary valve control system for full details on how to prepare and commission the rotary valve.

C. DESORBENT PREPARATION

1. Introduction

The desorbent components, normal pentane and iso-octane must be oxygen stripped and rerun to remove impurities that would poison the adsorbent if not removed. The raw nC_5 and iC_8 (see Table 1) are stripped on the raffinate column and rerun in the extract column before they are sent to their respective clean, gas-blanketed storage tanks. In some units, due to problems observed with alkylation unit quality control, the iC_8 is routed through a desorbent drier as a safety precaution to protect the adsorbent from any residual acid (used in the manufacture of the iC_8). However, the current design is able to omit this due to increased quality control in the production of iC_8 . If Para-xylene is going to be used as a desorbent component it must also be stripped and rerun.

NOTE: Since this is the first time that hydrocarbons will be processed, special care should be taken to assure that the plant is ready for operation.

- a. Check that all utilities such as steam, instrument air, cooling water, and electricity are available. Be sure the proper fire fighting equipment is in place.

- b. Check that all instruments are calibrated and ready for operation.
- c. If not already done, free the extract and raffinate columns of oxygen by steaming out. Connect steam to the steamout connection at the bottom of each column, opening all vents, drains and associated lines. When the steam vapors leaving the vents are at 100°C (212°F), steamout can be considered complete. Block in all vents and drains and pressure the columns to 1.0-1.4 kg/cm² (15-20 psig) with steam, effectively superheating the vapors in the column. Shut off the steam quickly, open the top vent and purge the columns with nitrogen. This serves to dry the columns. Close the top vent and leave a slight positive pressure on the columns to reduce the risk of oxygen leaking back into the unit.

2. Process Flow

A schematic of the desorbent preparation flow is shown in Figure VI-1. To minimize losses, the volatile nC₅ should be stored under pressure; the nC₅ storage tank and the desorbent surge drums are designed for this purpose. The iC₈ and desorbent storage tanks are atmospheric tanks, blanketed with N₂ to keep oxygen out.

3. Normal Pentane (nC₅) Preparation

Line up the plant for nC₅ preparation as follows:

- a. Send raw nC₅ from the N₂ blanketed desorbent surge drum to the raffinate column feed via the rotary valve bypass, using the desorbent pump.
- b. Oxygen stripped nC₅ through raffinate bottoms pump to extract feed column.
- c. Rerun nC₅ overhead from extract column; send bottoms to slop.
- d. Route nC₅ from extract overhead receiver through the desorbent cooler, bypassing the desorbent drier section. The cooled nC₅ is then sent to the clean N₂ blanketed nC₅ storage tank.

- e. Be sure all necessary blinds have been installed prior to stripping and rerunning.

The details of nC₅ preparation are as follows:

- (1) Bring in the nC₅ from the desorbent surge drum and build a working level in the raffinate column reboiler. The surge drum should be N₂ blanketed via the equalization line from the extract column overhead receiver.
- (2) Put the raffinate column reboiler into operation slowly, in order to prevent pressure surges. Set the overhead pressure controller at 2.5 kg/cm² (36 psig). Commission the overhead condenser fin fans. Set the overhead receiver PDIC controller at 0.5 kg/cm² (7 psig).
- (3) As soon as sufficient level is seen in the overhead receiver, put the column on total reflux by commissioning the overhead pumps and the side-cut pumps and begin sending net bottoms liquid to the extract column, bypassing the raffinate bottoms cooler.
- (4) Adjust the reflux, if necessary, to obtain an oxygen-free product. The receiver water boot should be checked for water accumulation and drained as water is collected.
- (5) The raffinate column side-cut accumulation tray level controller resets the reflux flow, while the raffinate bottoms level controller resets the net stripped product flow. The sidecut pumparound flow, back to the tower is reset by the TRC a few trays below the accumulator tray.
- (6) With the raffinate bottoms product, establish a working level in the extract column reboiler.
- (7) Slowly bring the extract column reboiler heater into operation to avoid pressure surges. Set the overhead pressure controller at approximately 2.5 kg/cm² (36 psig). Commission the overhead

condenser fin fans. Set the overhead receiver PDIC controller at 0.5 kg/cm² (7 psig).

- (8) As soon as a working fluid level is built in the overhead receiver, establish reflux to the column and withdraw the rerun nC₅ from the overhead receiver.
- (9) As necessary, withdraw a stream of heavy material from the extract column bottoms through the cooler to slop.
- (10) Adjust the reflux to eliminate any heavy ends from the nC₅. The extract overhead level controller resets the net nC₅ product flow.
- (11) Send the stripped and rerun nC₅ product through the desorbent cooler to the nC₅ tank, bypassing the desorbent drier section.
- (12) Continue this operation until the required clean nC₅ inventory is filled and the desorbent surge drum level is low. At this time, close off the desorbent pump spillback and pumpout as much nC₅ as possible to the raffinate column. Minimize the inventory of nC₅ in the raffinate and extract columns by exporting as much nC₅ as possible.

4. Iso-octane (iC₈) Preparation

Line up the plant for iC₈ preparation. The process scheme is identical to nC₅ preparation with the exceptions of:

- a. The raw iC₈ can be stored in either the desorbent storage tank or the desorbent surge drum. If the desorbent storage tank is used, the raw iC₈ can be sent directly to the raffinate column feed line using the desorbent filling pump, circumventing the need to use the desorbent pumps and routing flow through the rotary valve bypass.

- b. The iC_8 must pass through the desorbent drier section that includes a feed/effluent heat exchanger, heater, and a drying bed.
- c. Stripped, rerun and dried iC_8 is finally sent to the blanketed iC_8 storage tank.

The details of iC_8 preparation are as follows:

- (1) When as much nC_5 as possible has been removed from the surge drum and columns, immediately begin charging raw iC_8 to the raffinate column to the raffinate column feed.
- (2) Slowly decrease the raffinate and extract column overhead pressure controller to approximately 1.0 kg/cm² (15 psig).
- (3) Continue operations as outlined in the nC_5 preparation.
- (4) When the iC_8 shows up in the extract column overhead, redirect the flow through the desorbent drier section. The iC_8 should now pass through the regeneration feed/effluent exchanger, heater, drying chamber, back to the feed/ effluent exchanger and on to the desorbent cooler and iC_8 storage. The heater temperature should be slowly brought up to the point where the heater outlet stream is at 177°C (350°F), then set on automatic temperature control.
- (5) Continue until the required inventory of clean iC_8 is filled. All of the raw iC_8 must either be stripped, rerun and dried, or removed from the plant, to avoid any risk of contamination.

5. Para-xylene Preparation

Para-xylene will be utilized as a flush component and it should be prepared in a similar manner to the nC_5 . The para-xylene does not have to pass through the desorbent drier if one is provided.

The details of Para-xylene Preparation are as follows:

- a. When as much iC_8 as possible has been removed from the surge drum and columns, immediately begin charging raw para-xylene to the raffinate column.
- b. Continue operations as outlined in the nC_5 preparation. The column overhead pressures should be maintained at approximately 1.0 kg/cm² (14 psig).
- c. Stripped and rerun para-xylene is finally sent to the para-xylene storage tank.

D. ADSORBENT CHAMBER FILLING (Figure VI-2)

1. Establish Desorbent Inventory

The objective of this procedure is to fill the adsorbent chambers and saturate the sieve with a 60:40 mixture of stripped and rerun nC_5 and iC_8 . If para-xylene is going to be used, the desorbent component concentrations should be adjusted accordingly. The raffinate and extract columns perform the stripping and rerunning operation as in the desorbent preparation procedure. The adsorbent chambers are filled slowly to allow the sieve to adsorb as much nC_5 as possible. The desorbent should be kept as cool as possible and the chamber pressure kept at 7 kg/cm² (100 psig) to reduce the risk of flashing nC_5 ; the heat of adsorption of nC_5 is quite high.

- a. Bring in an appropriate quantity of nC_5 and iC_8 from storage to the desorbent storage and mixing tank in order to make a 60:40 mixture.
- b. Pump the 60:40 desorbent mixture to the raffinate column. As desorbent fills the raffinate column, start up the column on the total reflux with the reflux on the trap tray level control. Commission the overhead condenser, column reboiler, and the overhead pressure controller (typically) at 2.5 kg/cm² (36 psig). When a level is established in the overhead receiver, start the reflux pump. The reflux rate should be brought up slowly on manual control until the overhead receiver level is stable; then it can be placed on automatic control. The side-cut pump around should also be placed in service at this time. The hot oil rate to the reboiler should be increased slowly until the desired amount of reflux is obtained.
- c. As soon as the raffinate column is inventoried, start the raffinate bottoms pump and send the raffinate bottoms to the extract column bypassing the bottoms cooler.
- d. Import sufficient desorbent to permit extract column operations. Bring the column up slowly just like the raffinate column. Set the reflux at a sufficient rate, commission the overhead receiver level controller, and begin sending overhead material to the desorbent surge drum.
- e. Establish a good level in the desorbent surge drum and begin the following circulation.
 - i. Raffinate column feed to raffinate column.
 - ii. Raffinate column bottoms to extract column feed.
 - iii. Extract column feed to extract column.
 - iv. Extract column overhead to the desorbent surge drum.
 - v. Desorbent surge drum to the rotary valve bypass.
 - vi. Rotary valve bypass to raffinate column feed.
 - vii. Raffinate column feed to raffinate column.

- f. Be sure that the desorbent pump spillback is open for good pump operation. Commission the chamber pressure controller and open it 50% on manual control to allow desorbent to flow back to the raffinate column.

2. Flushing

The following desorbent flushing procedure is necessary to ensure that the process lines are free of moisture and scale. Any residual water in the circulation loop will show up in the raffinate overhead condenser water boot, where it can be drained.

When all the lines have been thoroughly flushed clean, as shown by a steady decline of water accumulation in the raffinate overhead boot, proceed with the chamber filling.

3. Filling Adsorbent Chamber No. 1

- a. One adsorbent chamber will be filled at a time. The adsorbent chamber pressure must be at 7 kg/cm² (100 psig) before filling can be started. Install a temporary 0-15 kg/cm² pressure gauge on each chamber bottom head, or use temporary impulse lines. The gauge should be located such that it is visible from the bottom head's seal angle vent and snorkel sample probe. Install a temporary 0-15 kg/cm² pressure gauge at the top of each chamber. Ensure that the bottom head flush control valve is blocked in. The orifice plate on the bottom head flush flow meter should be removed. The restriction orifices on the head flush and seal angle vent lines should not be removed. These are sized to allow the heads to be filled in 45 minutes. Obtain two small sight glasses and attach these to the low point drain on bed line No. 2 (No. 14 when filling chamber No. 2) and the bottom head snorkel sample probe. The connection should be made with suitable high pressure tubing. The outlet of the sight glasses should be directed to a closed drainage system. Station operators at the top and bottom heads of the adsorbent chamber. All bed lines except No. 2 (No. 14 when filling chamber No. 2) should be blocked in to the rotary valve. Open the communication line at the chamber top between the pumparound line and the top head.

- b. Desorbent circulation should have already been established in the raffinate and extract columns as per above. During the initial filling of the adsorbent chamber, "cold" desorbent at approximately 37°C (100°F) is used to wet the sieve. The temperature of the desorbent to the chamber is controlled by adjusting the extract column overhead condenser. Begin filling the bottom head of the adsorbent chamber with the "cold" desorbent through the head flush control valve bypass. The restriction orifice on the head flush line will limit the flow to the bottom head at roughly 11.5 m³/hr. As the bottom head begins to fill, the operator at the bottom of the chamber should fully open the snorkel sample probe and vent off nitrogen. The pressure in the chamber should be controlled at approximately 7 kg/cm² (100 psig) during the entire liquid filling operation. If the pressure builds up with the sample probe line fully open, then the seal angle vent on the bottom head should also be opened so that the pressure can be controlled. In the event that the chamber pressure continues to rise with both the sample probe and seal angle vent fully open, the operator at the top of the chamber should control the chamber pressure by venting through the sight glass to the raffinate column.

NOTE: The communication line and the vent to the raffinate feed line should be unblinded.

- c. The operator at the bottom of the chamber should continuously monitor the sight glass on the sample probe line for liquid. Once liquid is observed, the flow into the head through the flush line should be stopped before the vents on the bottom heads are closed. Once the desorbent flow to the bottom head is stopped, block in all vents from the chamber in order to hold 7 kg/cm² (100 psig).
- d. Prepare to fill the main shell of the adsorbent chamber with desorbent as per the normal procedure. The main points to observe are:
- i. Fill the bottom bed to the top bed in 12 hours.

- ii. During filling, monitor the three adsorbent bed TI's – they will show a temperature rise as the benzene adsorption wave progresses up through the chamber.
 - iii. Open up the low point drain on bed line No. 2 (No. 14 when filling chamber No. 2).
 - iv. Once a temperature rise is observed in the top bed or when liquid is seen at the low point drain on the open bed line No. 2 (No. 14 when filling chamber No. 2), immediately slow down the filling rate to 11.5 m³/hr. The top head must be filled at this low rate to prevent top grid damage.
 - v. Use the vent on top of the chamber to maintain 7 kg/cm² (100 psig) pressure.
 - vi. The columns and the desorbent surge drum should be at maximum stable levels.
- e. As soon as the liquid filling of the main shell of the adsorbent chamber begins, the seal angle vent line on the bottom head should be opened and all the gas in the bottom head vented off until the sight glass is liquid full.
- f. Open the jumper line from the raffinate line to the suction header of the chamber circulating pumps. Unblock to the bottom of the chamber from the circulation pumps' suction header. Set the raffinate PRC on automatic at 14 kg/cm² (200 psig). Ensure the proper chamber PRC is controlling the raffinate control valve. Vent N₂ from the top of the chamber to the raffinate column in order to keep 7 kg/cm² (100 psig) at the chamber top during the filling operations.
- g. As soon as the main chamber filling is started, desorbent from the desorbent storage tank is fed to the raffinate column to make up the difference between the desorbent accumulation in the unit and the volume of desorbent required to fill the chamber.

- h. When bed No. 1 (or bed No. 13 for chamber No. 2) TI shows a temperature rise or if liquid is observed at the low point drain on bed line No. 2 (bed line No. 14 for chamber No. 2), slow the filling rate to 11.5 m³/hr to prevent top grid damage. Continue filling until the top head and pumparound line are liquid full. This is determined by the sight flow located on the chamber high point vent line.
- i. Slowly close the vent from the top of the chamber. While doing this the chamber pressure will slowly increase to 14 kg/cm² (200 psig) and the pressure control valve on the raffinate line will open automatically and divert the desorbent flow from the chamber to the raffinate column. The chamber is now floating on the PRC at 14 kg/cm² (200 psig).
- j. Purge liquid from the top of the chamber via the vent line (Figure VI-3) to the raffinate column until the concentration of the stream is a 60:40 mixture of nC₅ and iC₈. This will assure that nC₅ has totally filled the selective pore volume (only nC₅ is adsorbed by the sieve).
- k. Lower the raffinate pressure control valve to 7 kg/cm² (100 psig) and block in the filling line jumper from the raffinate line.

NOTE: The heat of adsorption of the nC₅ can cause the bed temperatures to rise over 100°C (212°F). These temperatures must be carefully monitored as sieve damage may result if the nC₅ flashes in the sieve. The iC₈ is not adsorbed and acts as a heat sink.

4. Filling Chamber No. 2

Fill identically as Chamber No. 1 with the exception of:

- a. Rerouting desorbent flow to the bottom of Chamber No. 2.
- b. The pressure connection for the raffinate pressure transmitter must be reconnected at the bottom of Chamber No. 2 rather than that of Chamber No. 1.

E. BED LINE FLUSHING (Figure VI-4)

The 24 bed lines must be flushed free of debris and moisture to avoid plugging the grid screens or allowing water to come in contact and damage the adsorbent. The flow of flush material must originate from the chamber and be directed to the rotary valve housing, from where it can be sent to the raffinate column for stripping.

1. Line up the following flows: raffinate bottoms to extract column feed, net extract overhead to desorbent surge drum, to the desorbent pump (with spillback open) through the desorbent FIC on the pump discharge to the pumparound piping into the tops of adsorbent chambers No. 1 and No. 2, through the beds, out a bedline to the rotary valve housing, out the raffinate port and back to the raffinate column. The desorbent FIC will be used to regulate the flow to the chambers and the chamber bottoms PRC will control the chamber pressure by passing flow out of the raffinate line.
2. To flush bedlines Nos. 1-12, set the chamber No. 1 pressure controller at 14.4 kg/cm² (205 psig), open the jumper line between the bottom of Chamber No. 1 and the raffinate line, and start the desorbent pump, controlling the desorbent flow with the desorbent FRC. Slowly bring up the desorbent rate, making sure that the chamber pressure controller is working properly. Raise the desorbent flow to ½ of the design rate.
3. Slowly crack open the valve on bedline No. 1 and close the chamber outlet valve effectively redirecting the desorbent flow through the bedline rather than downflow through the chamber. Flush the bedline for one hour.
4. When finished with flushing bedline No. 1, slowly open the valve on bedline No. 2 and close the one on bedline No. 1, continuing flushing. Proceed in this manner until bedline No. 12 is flushed clean.
5. Stop the desorbent flow and block in the chamber. N₂ should be added if necessary to maintain pressure (minimum 7.0 kg/cm² (100 psig)) on the chambers.
6. While bedline flushing, flush the dome sealant into the rotary valve at design rate with desorbent using the jump-over line used in bottom head filling.

7. Connect the chamber pressure transmitter so that it senses the pressure at the bottom of Chamber No. 2. Open the jumper line between the bottom of Chamber No. 2 and the raffinate line, and start the desorbent pump, controlling the desorbent flow with the desorbent FIC. Slowly bring up the desorbent rate, making sure the chamber pressure controlling is operating properly. Raise the desorbent flow to ½ of the design rate.
8. To flush bedlines #13-24, repeat instructions 3, 4, and 5.

For ease of operations, the filling of Chamber No. 1 and bed line flushing of beds #1-12 may be done consecutively and then filling of Chamber No. 2 and bed line flushing for beds #13-24.

F. ROTARY VALVE ROTOR PLATE INSTALLATION

After the bed lines have been flushed the rotary valve must be isolated from the plant by closing all the block valves on the bed lines, dome sealant in/out, the seal flush and the dome equalization line. The hydrocarbon in the rotary valve must be purged out using nitrogen and flexible line from the nitrogen header to empty all the lines and the rotary valve. The hydrocarbon should be drained from all low point drains including the six track lines. When the rotary valve is hydrocarbon free, it will be opened and the rotor plate installed. (See Rotary Valve Manual.)

Following the rotor plate installation, the rotary valve and connecting lines should be air freed by purging with nitrogen and then refilled with liquid by slowly opening the bed lines to the rotary valve.

The gas should be vented from the top of the rotary valve using the high point vent line until the dome is liquid full. Pressure on the system (adsorbent Chamber No. 1 and No. 2 and rotary valve), should be maintained at 7 kg/cm² (100 psig) using nitrogen as necessary. The dome equalization line must now be opened to prevent seating pressure on the rotor plate when the rotary valve is not in service. The equalization valve is used for this purpose.

Table 1
Chemical Specifications for Desorbent Components

Normal Pentane

Provide a hydrogenated, normal pentane-rich stream, that satisfies the following specification:

Composition, wt-%

Normal Pentane	95 min.
Iso-pentane, Cyclopentane	Balance
Butanes and lighter	0.5 max.
Hexanes and heavier	0.5 max.

Total Sulfur, wt-ppm	1 max.
Total Nitrogen, wt-ppm	0.5 max.
Chloride, wt-ppm	0.1 max.
Fluoride, wt-ppm	0.1 max.
Carbonyl, mg/L	2 max.
Bromine Index	150 max.
Aromatic Content, wt-ppm	100 max.

Iso-Octane

Provide a hydrogenated, octane-rich stream, preferably derived from a sulfuric acid Alkylation unit, that satisfies the following specification:

Composition, wt-%

2,2,4, Trimethylpentane (Iso-octane)	60 min.
Other Octanes	Balance
Heptane & lighter	10 max.
Nonanes and Heavier	0.5 max.
N-Paraffins	0.5 max.
Distillation (Typical)	

<u>Percent Over</u>	<u>°C</u>
IBP	91
5%	96
10%	98
50%	100
90%	102
95%	104
EP	116

Table 1 (continued)
Chemical Specifications for Desorbent Components

Total Sulfur, wt-ppm	1 max.
Total Nitrogen, wt-ppm	0.5 max.
Carbonyl, wt-ppm	2 max.
Chloride, wt-ppm	0.1 max.
Fluoride, wt-ppm	0.1 max.
Bromine Index	150 max.
Aromatic Content, wt-ppm	1000 max.

In addition, before discharging the "Iso-octane" to storage, it shall pass over a bed of molecular sieves at 177°C (350°F), in the liquid phase, and at a rate not to exceed 2 liquid hourly space velocity.

Figure VI-1
Desorbent Preparation

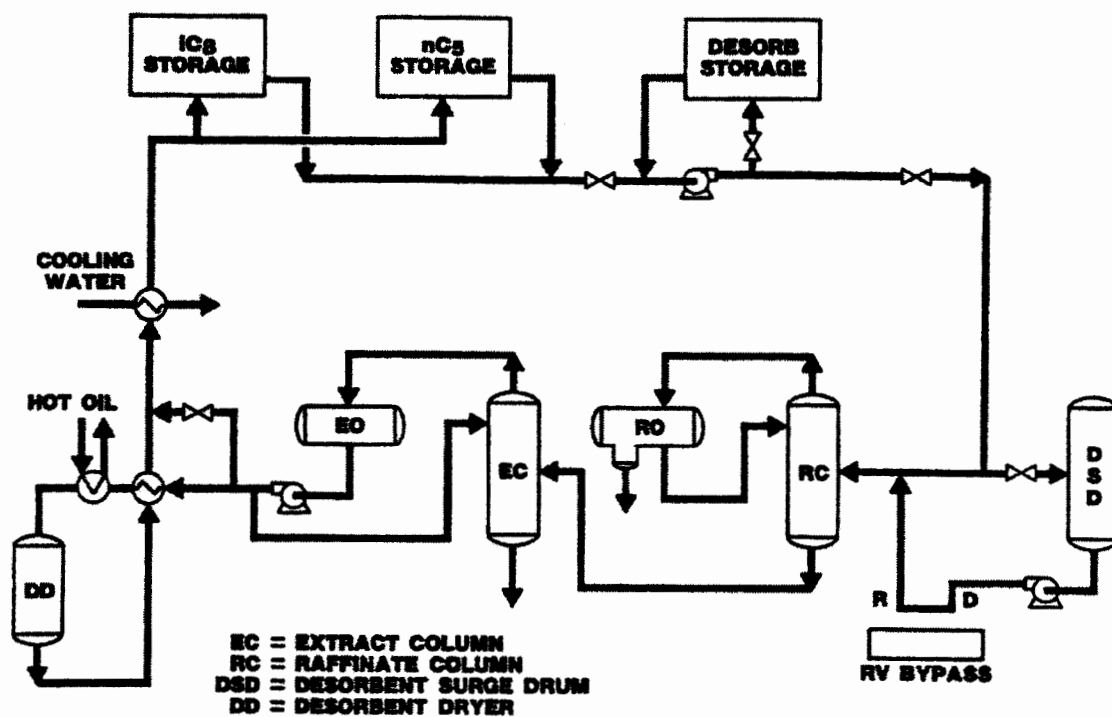


Figure VI-2
Chamber Filling

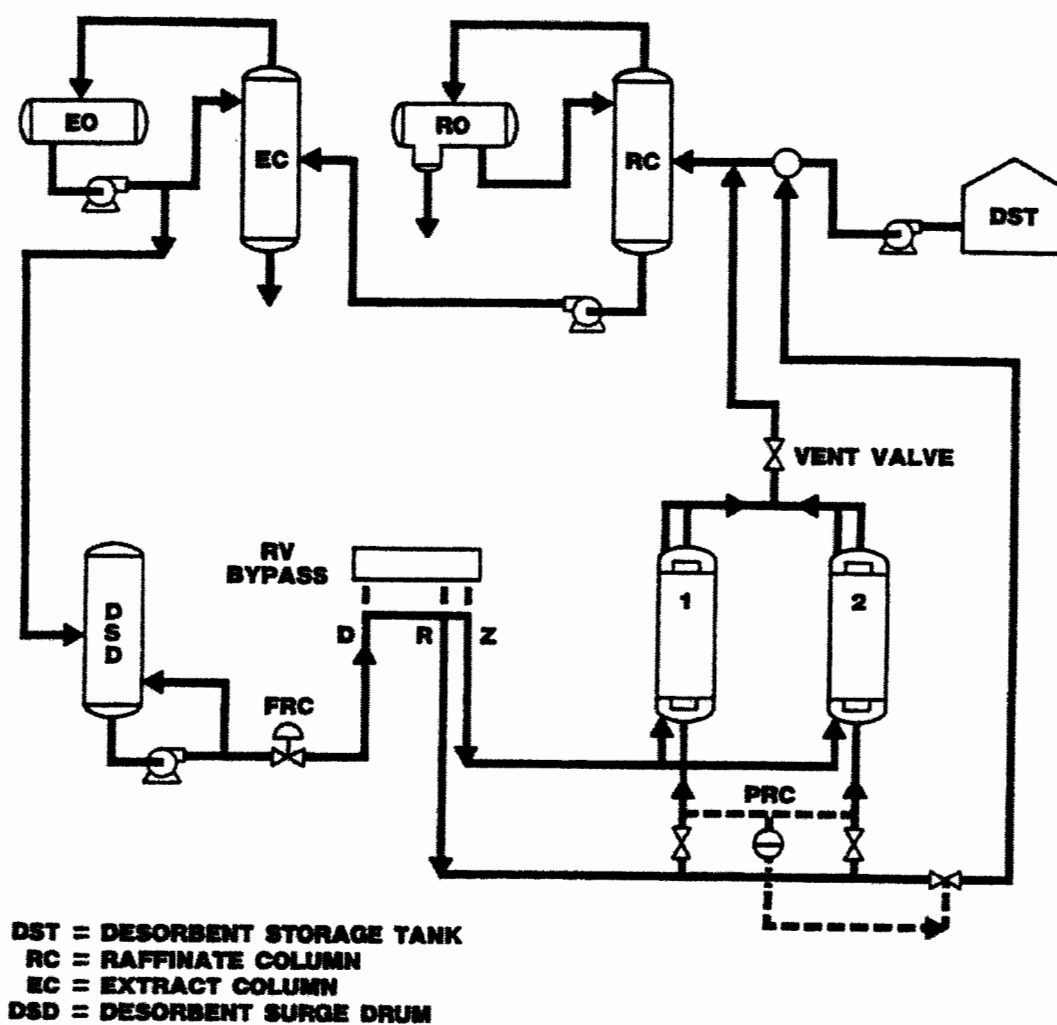


Figure VI-3
Venting of Adsorbent Chamber

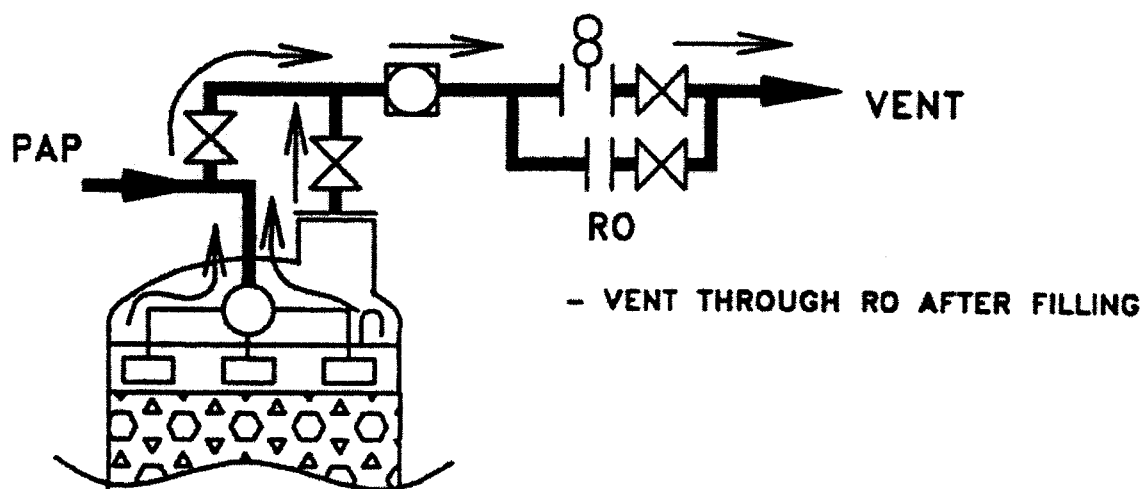
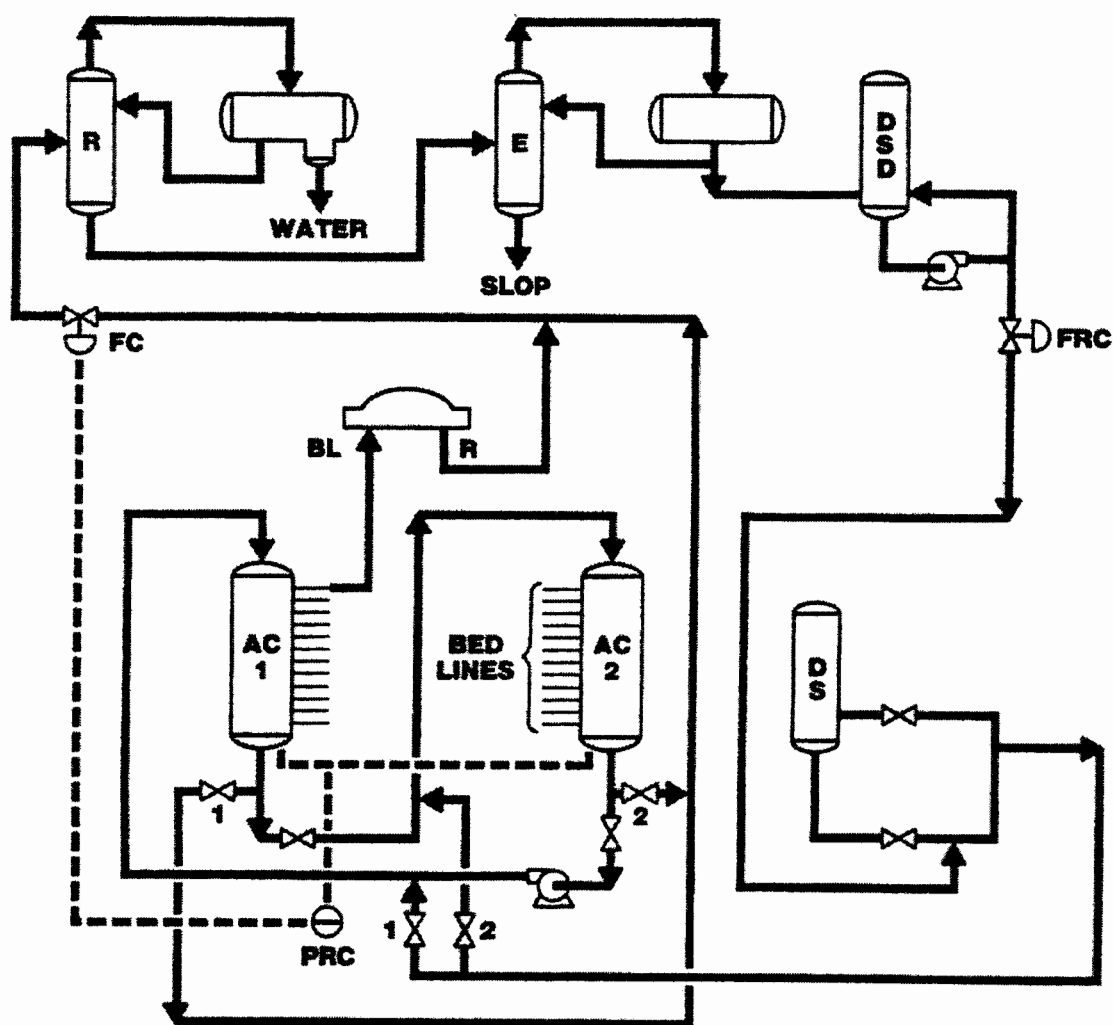


Figure VI-4
Bed Line Flushing



✕ 1 VALVES OPEN DURING FLUSHING OF BEDLINES 1-12 (IN CHAMBER 1)

✕ 2 VALVES OPEN DURING FLUSHING OF BEDLINES 13-24 (IN CHAMBER 2)







VII. NORMAL STARTUP

A. ASSUMED CONDITIONS

This procedure assumes that the adsorbent chambers are full of desorbent, the rotary valve rotor plate is installed, and that a supply of desorbent has been prepared. This assumption also implies that air has been purged from the unit and that all lines and equipment are free of water. If these assumptions are not correct, refer to the appropriate part of the preceding sections before proceeding further.

The normal startup can be divided into the following steps:

1. Establish levels in the raffinate, extract, and desorbent stripper columns and start circulation of desorbent and feed through the rotary valve bypass manifold.
2. Start desorbent, zone flush, line flush, and feed flow to adsorbent chamber. Adjust inventories as needed.
3. Start up fractionation section and line out.
4. Warm up the adsorbent chambers.
5. Start the chamber circulation pump and put rotary valve into operation.
6. Establish the proper zone flow rates in the adsorbent chambers, and the proper conditions in the extract, raffinate and desorbent stripper columns to make specification products and minimize desorbent loss.
7. Bring unit to desired charge rate and line out.

If the startup follows desorbent preparation, some of these steps may already have been done, or partly done. In this case, appropriate parts of the procedure may be omitted.

B. GENERAL STARTUP FLOW SCHEME

The unit should be lined up for normal operations. Route the extract and raffinate column sidecuts to the desorbent stripper. The overhead products should be lined up to the desorbent surge drum. The desorbent stripper bottoms should be lined up through the flush pumps to the head flush, line flush and zone flush lines. Blind off all desorbent preparation piping if this has not already been done.

C. EXTRACT, RAFFINATE, AND DESORBENT SPLITTER COLUMN STARTUP

1. Bring in the 60:40 nC₅/iC₆ desorbent mixture from storage and establish levels in the extract and raffinate columns. Commission the overhead condensers, column reboilers and overhead pressure controllers. Start heat to the columns slowly and establish operation on total reflux. The heat input rate to the reboilers should be increased slowly until the desired amount of reflux is obtained. When a level is established in the overhead receiver, start the reflux pump. Start sidecut pumparound reflux on the raffinate column. The reflux rate should be brought up slowly on manual control until the overhead level is stable; then it can be placed on automatic.
2. As soon as the extract and raffinate columns are inventoried, begin sending the column overheads to the desorbent surge drum. Begin sending sidecut streams to the desorbent stripper.
3. Once a good working level is obtained in the desorbent stripper establish raffinate column bottoms flow through the reboiler. The heat input rate to the reboiler should be brought up slowly.
4. Circulate desorbent from the desorbent surge drum with the desorbent pumps (make sure the spillback is open for good operation), through the desorbent/raffinate exchanger, desorbent heater, filter, FRC, rotary valve bypass and out the extract and raffinate lines to the columns.

5. Circulate desorbent stripper bottoms (mostly flush material, iC_8) with the bottoms pumps, through the flush heater, flush filter, to the zone flush and line flush lines, through the FRC's, the rotary valve bypass and back to the extract and raffinate columns.
6. When the hydrobon splitter bottoms is on specification, start feed through the feed filters, flow controller, rotary valve bypass, and on to the extract and raffinate columns.
7. Line out operation of the extract, raffinate and desorbent splitter columns so that the proper split is made between the nC_5 , iC_8 and feed components. Send extract and raffinate column bottoms back to the Molex feed surge drum.

D. ADSORPTION SECTION STARTUP

The adsorbent chambers and pumparound pump piping should be full of the 60:40 desorbent mixture and pressurized at 7 kg/cm² (100 psig) at this point.

1. Open all the bedlines to the rotary valve.
2. Start all head flushes at normal operating rates while watching the chamber pressure and the vents. Vent off all gases, if any, from the tops of the chamber. The pressure will build rapidly once the chamber becomes liquid full. Once the pressure starts to build, slowly open the raffinate line to the rotary valve from the rotary valve bypass and set the chamber pressure controller on automatic at 24.6 kg/cm² (350 psig). If there is no pressure increase within a short period of time, a vapor pocket may be present and the chambers may need to be vented.
3. Rotary Valve Startup
 - i. Check that all 24 block valves on the chamber lines are open to the rotary valve.

- ii. Start sealant flow as specified in the project specifications through the flow controller to the rotary valve dome. Make certain that the dome pressure control valve and equalization line are open.
- iii. Vent the dome through the sight flow indicator for several minutes to assure that no gas is present.
- iv. Begin valve rotation, setting the step time as determined from the zone calculation sheet.

4. Adsorbent Chamber Warmup

The adsorbent chambers must be warmed up slowly to prevent excessive piping stress due to thermal expansion. The temperature differential between the cool beds and the warmer streams entering the chambers should not exceed 28°C (50°F). Therefore, it is necessary to cool the circulating desorbent and flush streams from the columns as much as possible. The temperature of the feed, once it has left the hydrobon stripper bottoms should be controlled, using the flow of feed to the rotary valve to stay within the 28°C (50°F) differential temperature requirement. Initially, the differential temperature may exceed the 28°C (50°F), so the flow should be minimized until the chamber temperatures come up.

The flow of liquid through the sieve creates a pressure drop. During normal operation at 177°C (350°F), this pressure drop is small and causes no problems. If, however, the pressure drop is high, serious damage may result. The sieve bed supports (grids) are designed for a maximum pressure drop of 0.34 kg/cm² (5 psig) per bed, this value must never be exceeded. The pressure drop normally encountered during operation is a result of both the flow rate and viscosity of the material passing through the bed. The viscosity of cold liquid hydrocarbons is several times greater than hot liquid hydrocarbons. This effect becomes more pronounced as the molecular weight increases; the desorbent is less viscous than the feed at a given temperature. High pressure drop may, therefore, result from high

concentrations of cold hydrocarbons, such as the feed, in the chambers as a result of starting the pumparound. During startup, the chamber circulation should be started slowly and the chamber pressure drops carefully observed. As the temperatures inside the chamber rise, the viscosity of the streams decrease and the flow rates through the chambers may be increased.

It is assumed that the extract, raffinate and desorbent stripper columns are operating, sending their products through the rotary valve bypass, the chambers have been liquid filled and are on pressure control, and the rotary valve is stepping. With the above points in mind:

- i. Slowly cut in cool desorbent and feed to the rotary valve, while leaving their rotary valve bypass lines open. Use the chamber temperature/stream differential temperature as a guide to regulating the feed and desorbent flows. Continue increasing the flow rates of the streams to the rotary valve by slowly closing the rotary valve bypass valves, then regulate the flows to the rotary valve with the desorbent and feed flow controllers. The line flushes and zone flush may be isolated during this time. One at a time, slowly open each line to the rotary valve, then slowly close off on each rotary valve bypass. Each stream should be flowing directly to or from the rotary valve and all the bypasses closed except for the one on the raffinate line, which may be closed slowly at this time. While raising the individual flow rates, make sure all the flows to and from the rotary valve are balanced (feed + desorbent + line flush in + zone flush = extract + raffinate + line flush out). The raffinate flow is controlled by the chamber pressure controller; however the remainder of the flow rates are controlled by an FRC. Avoid depressuring the chambers by allowing the extract and line flush out rates to exceed the total incoming flows.

- ii. After closing the rotary valve bypasses, close the dome equalization line, and at the same time, raise the dome pressure controller to a level 1.5 kg/cm^2 (21 psig) above the average chamber pressure, and set it on automatic. If the rotary valve does not seat (indicated by dome sealant leakage into the bed lines), open the dome equalization line, then close again.

NOTE: As feed enters the chamber, it displaces the desorbent initially present. Some desorbent may need to be exported via the desorbent pumpout if the desorbent surge drum cannot handle the additional volume.

- iii. As the adsorbent bed/stream differential temperature decreases, the desorbent and flush line heaters may have to be commissioned. Slowly bring in the hot oil and gradually raise the stream temperatures, staying within the 28°C (50°F) limits.
- iv. When the average bed temperature reaches 140°C (284°F), the pumparound pump can be turned on. Start the pumparound pump and control the circulation rate so as to limit the pressure drop across each chamber to 2 kg/cm^2 (28 psi). Keep the pumparound controller on manual and establish Zone II flow.
- v. Continue heating and circulating until the average chamber bed temperature has reached 177°C (350°F).

5. Line Out to Final Operations

- i. Line out the inlet and outlet streams, and the individual zone rates as per the zone calculation sheet, then place on automatic control.

- ii. Line out the average adsorbent chamber temperature to 177°C (350°F) by regulating the hot oil to the desorbent and flush line heaters, then place the hot oil flow controller on automatic. The feed TRC must also be commissioned and lined out at 177°C (350°F).
- iii. Check the rotary valve alignment.
- iv. Bleed the chambers and the extract and raffinate feed mixing drums periodically to assure removal of gas pockets.
- v. Check for leaks in the rotary valve bypass line. Drain the line and isolate any block valve leaks.
- vi. Regulate the flow of extract and raffinate sidecuts to the desorbent stripper and desorbent surge drum in order to produce the desired amount of iC_8 . Adjust the desorbent stripper column as necessary to obtain the desired nC_5/iC_8 split. The stripper bottoms should be pure iC_8 . Adjust desorbent inventory as needed.
- vii. When the extract and raffinate bottoms material are on-spec, begin routing the extract and raffinate products to storage.







VIII. NORMAL OPERATIONS

Normal on-line operation implies that the Molex unit is lined out at the desired capacity and is producing specification product at the guaranteed quality with the normal temperatures, pressures, and flows established. It is possible, however, to optimize both the adsorbent chamber section and fractionation section so that utility consumption is reduced. This is accomplished by lowering reflux ratios in both sections while maintaining maximum recovery and the desired product purities.

The principal duty of the operators is to achieve a steady-state condition and optimize further whenever possible. Abnormal conditions or emergencies should be detected quickly, interpreted accurately, and acted on promptly and correctly. This requires that each operator have an intimate knowledge of the unit and its component parts, and a thorough understanding of the process variables.

For good plant operation, the following things should be done:

1. Weight balance the plant.
 - a. Unit should balance $100 \pm 2\%$.
 - b. Periodically, component balance the plant. This will help check the overall balance and the laboratory analysis.
2. Watch the pumparound dielectric analyzer for abnormalities in Zones II and IV.
3. Take a pumparound survey periodically.
4. Maintain desorbent inventory control, try to balance the desorbent inventory monthly.

5. Check the rotary valve position and alignment.

6. Analyze the composition of the top heads regularly.
7. Check the gas blanketing systems.
8. Maintain good records.
 - a. Have operators log data in a convenient form.
 - b. Plot data to watch long term trends.
 - c. Fill out and mail to UOP the UOP computer input form.
9. Optimize the unit. Continue to trim these variables after the initial optimization because the sieve action and feed composition may change with time.
 - a. Maximize purity and recovery.
 - b. Minimize utility consumption.
 - (1) Minimize A/F_n and D_n/A
 - (2) Maximize L_4/W
 - (3) Minimize R/F on columns

A. INTRODUCTION

This section of the manual will probably be referred to more than any other section. It presents the actual calculations that are performed to operate the UOP Molex Unit. It will be divided into three main sections that are:

- (1) plant constants,
- (2) specified variables, and
- (3) calculated variables.

The plant constants are a function of the design. The specified variables are the independent parameters that one specifies to operate the plant and affect plant performance. The calculated variables are those variables which are calculated from the plant constants and specified variables, and the calculated variables specify all operating parameters which have to be dialed in to run the Molex unit.

Once these calculated variables are implemented and the plant is lined out, the plant performance is measured by purity and recovery. Performance of the unit will dictate what changes to the specified variables are necessary. The specified variables are changed and new calculated variables are dialed into the plant and a new performance is evaluated. This continues until the performance is optimized. The changes made to the specified variables to affect performance were discussed in the Process Variable section of this manual.

B. PLANT CONSTANTS

The plant constants for the plant calculations are the adsorbent loading volumes (V_s , V_a , V_w), and the longest bed line volume (V_{bl}).

The adsorbent volume is specified by the adsorbent chamber size. From the loaded adsorbent volume (V_s), the selective pore volume (V_a), and the non-selective void volume (V_w) are calculated from the loading data. These two values are considered constants for that specific plant and are used in the calculations. The volume of the longest bed line is calculated for a given plant and used to calculate the line flush rates. These are the only numbers that will be permanently entered on the calculation sheets.

The calculation of the selective pores and non-selective voids from the loading data is very important and warrants a detailed discussion at this point. This discussion should begin with the difference between the selective pore volume and non-selective void volume.

Consider a vessel of 1 m³ capacity filled with coarse sand. A mixture of normal and non-normal paraffins are added to the vessel until it overflows. A tap is then opened on the bottom of the vessel and the liquid is drained and measured. This volume, divided by 1 m³, is the void fraction of the sand. The sand is then replaced with adsorbent and the experiment repeated. The liquid drained from the adsorbent will be about 0.55 m³. The vessel is then heated and the vapor driven off is condensed and measured; its volume will be 0.11 m³. Whereas 0.55 m³ of liquid could be drained off freely, the 0.11 m³ was bound to the adsorbent and could be removed only by breaking the adsorption bond with heat. Furthermore, whereas the liquid drained from the sand had the same composition as that added to it, the 0.11 m³ of liquid boiled off the adsorbent was richer in n-paraffin and the drained liquid correspondingly leaner in n-paraffin than the original.

The Molex adsorbent, unlike sand, is a synthetic crystalline porous solid composed of many units held together by a binder to form a single particle. An adsorbent particle consists of two major parts: the molecular sieve particle and the binder. The sieve particle consists of selective micro pores and non-selective macro pores. In addition, there is a large volume of non-selective volume in the interstitial volume between adsorbent particles. The total non-selective volume is about five times the selective volume.

Figure VIII-1 shows a simplified sketch of the adsorbent. It can be seen where the selective and non-selective volumes exist. The selective volume is the pores that exist in each molecular sieve particle. These pores are sized so that only n-paraffins can pass through, leaving the other feed components outside. This where the separation takes place. The non-selective volume can be divided into three separate categories. The first is the interstitial void volume that exists between adsorbent particles. The second is the non-selective macro pores that exist in the clay binder. The third is volume of the grids and the pumparound piping.

This must be considered as non-selective void volume and taken into account when calculations are made.

When loaded to the Lab ABD (Apparent Bulk Density) the total selective volume of the adsorbent is about 11% of the bulk volume of adsorbent. About 54% of the bulk volume consists of non-selective macro pores and interstitial voids between individual particles. The remaining 35% is the actual bulk of the adsorbent.

After the concept of selective and non-selective voids is understood, the calculation of the selective pores and non-selective voids from loading data can be performed. The following example is presented to illustrate the calculation of V_a and V_w :

Necessary data:

*Weight adsorbent loaded:	<u>75000</u> kg
+Water on adsorbent:	<u>5.5% LOI 900°C</u>
*Calculated Chamber Volume:	<u>100</u> m ³ after subtracting out distributor pipe, grid and beam volumes
+Percent selective pores:	<u>11.1%</u> when loaded to lab ABD
+Percent non-selective voids:	<u>53.9%</u> when loaded to lab ABD
+Percentage solids:	<u>35.0%</u> when loaded to lab ABD
*Pumparound + grid volumes:	<u>5.0</u> m ³ (calculated for given plant)
+Lab loaded ABD:	<u>740</u> kg/m ³ , dry 900°C

NOTE: All values for purpose of example only.

The calculation procedure is:

- (1) Correct Adsorbent weight to dry basis:

$$W_s = (1.0 - 0.055)(75000) = 70875 \text{ kg, dry } 900^\circ\text{C}$$

- (2) Actual loaded ABD = $70875/100 = 708.75 \text{ kg/m}^3$, dry 900°C

- (3) Loading efficiency, LEF = $708.75/740 = 0.9578$
- (4) To calculate the selective pores and solid volume loaded, the lab ABD, 740 kg/m³ dry 900°C is used with the adsorbent loaded volatile free weight of 70875 kg. This is done because the volume of selective pores and solid is independent of the loading efficiency.

$$V_a = \text{Selective pores} = \frac{70875 \text{ kg} \times 0.111}{740 \text{ kg/m}^3} = 10.63 \text{ m}^3$$

$$\text{Solids} = \frac{70875 \text{ kg} \times 0.35}{740 \text{ kg/m}^3} = 33.52 \text{ m}^3$$

$$\text{Total of Selective pores and solids} = 44.15 \text{ m}^3$$

- (5) The total adsorbent bed voids must be the total bed volume minus the sum of the selective pore volume and the solid volume:

$$V_{ns} = 100 \text{ m}^3 - 44.15 \text{ m}^3 = 55.85 \text{ m}^3$$

The total system voids is the sum of the total adsorbent bed voids and pumparound + grid volumes:

$$V_w = 55.85 \text{ m}^3 + 5.0 \text{ m}^3 = 60.85 \text{ m}^3$$

C. SPECIFIED VARIABLES

After the chamber loading is completed, V_a and V_w can be calculated. With these numbers and the longest bed line volume (V_{bl}), the operator is ready to proceed with the process calculations. The operator must now select a set of values for the specified variables that are: temperature, pressure, feed rate, feed composition, A/Fn , L_2/W , L_2b/W , Dn/A , and L_4/W .

The chamber temperature and pressure are considered secondary variables and are specified in the design and not normally changed. The chamber temperature is controlled by the incoming feed and desorbent streams at 177°C (350°F). The chamber pressure is controlled by the raffinate outlet stream and the pusharound PRC and is set at 26.4 kg/cm² (350 psig). The temperature is selected to provide sufficient kinetic activity for good mass transfer and the pressure is selected to prevent vaporization at that temperature. The feed rate is selected depending on feedstock availability and product demand within the constraints of the design of the plant. The feed composition is controlled by the operation of the upstream fractionation unit and the quality of fresh feed. A sample of the Molex feed should be taken and used to specify the feed composition. The percentage of normals in the Molex feed is required because we base our adsorbent circulation on the normals feed rate (F_n) only and not total Molex feed (F). The final specified variables (A/F_n , L_2/W , L_2b/W , Dn/A , and L_4/W) are the **primary control variables** for the UOP Molex Process.

These primary control variables are ratios of gross liquid flow rates (L_2 , L_2b , L_3 , and L_4) in the chamber, the non-selective pore rate (W), and the selective pore rate (A). The exact definition of these variables will be covered in the final section on calculated variables. The value for these ratios have been developed by experimental work and commercial operation and for the initial startup, the process design numbers should be used as a starting point. An example calculation will be presented in the next section and typical values for the ratios will be used.

D. CALCULATED VARIABLES

With the given plant constants and the specified variables, the plant calculations can now be performed. This section can be divided into the following sections:

- (1) Cycle time calculation
- (2) Zone rates
- (3) Volume balances

After these topics are discussed in detail, the entire calculation will be put together in a sample presented in the next section.

1. Cycle Time Calculation

Cycle time (θ) is defined as the time required for the feed to make a complete cycle starting at one feed location and stepping through all the beds and returning to the original location. The Greek letter theta " θ " is used to symbolize the cycle time. To calculate the cycle time, the following equation is used:

$$\theta \text{ (minutes)} = \frac{V_a \text{ (volume)}}{A \text{ (vol/min)}}$$

Where A is the rate of simulated movement of selective void volume expected in units of volume per unit time.

The selective pore volume V_a is one of the plant constants but A must be calculated. In the Molex process, the quantity of sieve required to do the separation is a ratio of the feed normals rate (F_n , Feed rate $\times$ vol fraction of normals). If the feed rate is increased, A must be increased to adsorb the additional normals. Thus the desired rate of adsorptive volume, A, is calculated:

$$A = F_n \times (A/F_n) \text{ (volume per unit time)}$$

This means that with a given feed rate and an assumed A/F_n , we calculate A and then use A and V_a to calculate the cycle time (θ).

After the cycle time (θ) is known, the valve step time can be calculated as follows:

$$T = \theta \times (60 \text{ seconds/minute}) / \text{number of beds (24)}$$

This valve step time is the time the valve will stay in each location.

When you simulate the movement of the selective volume V_s , non-selective volume V_w is also moved. To purge out this interstitial volume, an additional flow W is required as;

$$W = V_w$$

$$V_w = \text{non-selective void volume}$$

$$\theta = \text{Cycle time}$$

$$W = \text{Non-selective void volume rate volume per unit time}$$

"W" is the flow rate necessary to compensate for the simulated circulation of adsorbent. For example, each 1 m³ of adsorbent carries with it not only 0.11 m³ of absorbed material but also 0.55 m³ of interstitial liquid. If this were not compensated, the two phases would continue to move together and the desired separation would not be possible. Therefore, each 1 m³ of simulated adsorbent circulation requires 0.55 m³ of interstitial liquid pumped in the opposite direction.

To illustrate this concept of "W", see Figure VIII-2. It shows several beds in which a small amount of dye is injected at time $t=0$ and the movement of the dye in the flowing liquid is compared to the movement of the chamber bed lines. When the dye is keeping pace with the bed lines, then the flow rate in the chamber is said to be equal to W .

2. Zone Rates

The gross flow rate, L_g , represents the actual flow rate within the adsorbent chamber. For example, if L_g is 200 m³/hr, then the flow rate for Zone I in the pumparound would be set for the 200 m³/hr. The gross zone rates are conventionally expressed in terms of the following equation:

$$L_1 = (L/W) \times W$$

gross zone flow	=	(L/W) zone reflux ratio	×	W non-sel. void rate
-----------------------	---	----------------------------------	---	-------------------------------

To calculate the gross zone rate, it is convenient to express things in terms of the zone reflux ratio, L/W . The net flow rate, $L_1 - W$ is either positive or negative, as required to do the separation (see Figure VIII-2). As stated earlier, W represents the flow rate required to keep the liquid and the sieve in the same relative position. Net flow is the flow greater than or less than W to effect a flow greater or less than the sieve. If L_1 is greater than W ($L/W > 1$), the liquid moves faster relative to the sieve. If it is less than W ($L/W < 1$), it moves slower relative to the sieve.

It is convenient to express it in terms of a ratio L/W , the zone reflux ratio. This relates the net flow to the sieve flow. The reflux ratio is thus somewhat constant at various feed rates and A/Fn ratios.

The desired L_2/W , L_{2b}/W and L_4/W are chosen according to experimental work and operating experience.

$$\begin{aligned} L_2 &= L_2/W \times W \\ L_{2b} &= L_{2b}/W \times W \\ L_4 &= L_4/W \times W \end{aligned}$$

In Zone III, the nC_5 in the desorbent must displace the n -paraffin in the pores of the adsorbent. In contrast to the other zones, the effectiveness of the zone depends not on the flushing of the interstitial volume, but rather upon the displacement of material from the pore volume. Thus, the rate of interest is Dn/A , where Dn is the net flow of normal pentane to the zone, and A is the selective pore rate. This ratio is used to calculate the Zone III flow rate.

$$L_3 = [(Dn/A) \times A / \%nC_5] + W$$

gross flow	=	net zone	×	% nC_5 in Desorbent	+	W non-selective void flow
---------------	---	-------------	---	--------------------------	---	---------------------------------

reflux

Typically, the desorbent is a mixture of 60% nC₅ and 40% iC₈. The resulting value in the brackets is the net flow of desorbent passing through Zone III only, not the total flow of desorbent entering the chambers. Some of the incoming desorbent stream flows into Zone IV.

The LINE FLUSH rates (H_i and H_o) are generally twice the longest bed line volume divided by the step time.

$$H_i = H_o = 2.00 \times V_{bl} \times (60/T), \text{ m}^3/\text{hr}$$

3. Volume Balances

Volumetric balances may now be made through the chamber to calculate the remaining flow rates. (See Figure VIII-3)

At the line flush withdrawal point:

$$L_3a = L_3 + H_{out}$$

At the extract withdrawal point:

$$E = L_3 - L_2b$$

At the Zone Flush point:

$$L_2 = L_2b + H_z$$

At the line flush addition point:

$$L_2a = L_2 + H_{in}$$

At the feed point:

$$L_1 = L_2a + F$$

At the raffinate withdrawal point:

$$R = L_1 - L_4$$

At the desorbent point:

$$D = L_3a - L_4$$

The raffinate rate, R , is on pressure control and therefore must balance both the net input to the adsorbent chamber,

$$R = F + D - E + H_z \quad (H_{in} = H_{out})$$

and the difference between L_1 and L_4 ($R = L_1 - L_4$). This calculation serves as a check on the others.

E. SAMPLE CALCULATION

1. Terminology

F	Feed Rate
F _n	n-Paraffin Feed Rate
D	Desorbent Rate
D _n	Desorbent's normal pentane rate
E	Extract Rate
R	Raffinate Rate
H _z	Zone Flush Rate
H	Line Flush Rate (H _i or H _{in} is line flush in and H _o or H _{out} is line flush out)
V _s	Total Volume of Sieve Loaded (Not Chamber Volumes)
V _a	Adsorptive (selective) volume or pore volume (11.1% of sieve vol.)
V _w	Non-selective Void Volume [(53.9% of Sieve volume)+(Grid Vol. - Dead Space) + Pumparound Vol.]
V _{tbl}	Longest Bed Line Volume
A	Adsorptive (selective) volume rate or pore circulation rate

A/Fn	Adsorptive volume rate/normals feed rate Note: Ratio used in connection with the Molex adsorbent because only normal species can be adsorbed
W	Non-selective void volume rate
L _i /W	Zone Flow Ratio ("Reflux" ratio) for Zone i
L _i	Gross Zone Rate (L _i ' + W) for Zone i
K	Gross Zone II rate (Computer Register)
θ	Valve Cycle Time (complete cycle to circulate through the total number of beds)
T	Valve Step Time (θ/24 beds)

2. Plant Constants

Assume a dimensionless number for volume so that either English or metric units can be used. (1 volume unit = vol.)

$$F = \text{design feed rate} = 10000 \text{ vols/day @ } 15.5^{\circ}\text{C}$$

$$V_s = \text{total volume of sieve loaded} = 1000 \text{ vols.}$$

$$V_a = (1000 \text{ vols}) (0.11) = 110 \text{ vols.}$$

$$V_w = (1000 \text{ vols.}) (0.539) + (1000) (.05) = 589 \text{ vols.}$$

(assume grid vol. + PA piping is 5% × V_s)

$$V_{bi} = (1000) (0.1\%) = 1 \text{ vol. (assumed)}$$

See page V-3, for an example of a detailed calculation of the selective pores (V_a) and non-selective voids (V_w) from loading data.

3. Calculation

NOTE: All flows (except raffinate) are measured by turbine meters at the operating temperature of 177°C (350°F).

(a) Normal Feed Rate (assume 20 vol% normals)

$$F = 10000 \text{ vols/day} \times \text{EXP. Factor}$$

$$F = 10000 \times 1.18 \frac{(0.78 \text{ sp. gr. @ } 15.5^{\circ}\text{C})}{(0.66 \text{ sp. gr. @ } 177^{\circ}\text{C})} = \text{Expansion Factor}$$

$$F = 11800 \text{ vols/day} \times \frac{\text{day}}{24 \text{ hrs}} = 492 \text{ vols/hr}$$

$$F_n = 492 \text{ vols/hr} (0.20) = 98 \text{ vols/hr}$$

(b) Specified Variables (Based on Process Design)

$$A/F_n = 1.20$$

$$L_2/W = 1.12$$

$$L_2b/W = 0.97$$

$$D_n/A = 2.20$$

$$L_4/W = 0.95$$

(c) Valve Step Time

Adsorbent Volume Rate:

$$A = F_n \times A/F_n = 98 \times 1.20 = \underline{118 \text{ vols/hr}}$$

$$\text{Cycle Time: } \theta = V_d/A = \frac{110}{118} = \underline{0.932 \text{ hr (55.93 min)}}$$

$$\text{Step Time: } T = \theta/24 = \underline{55.93 \text{ min}/24} = 2.33 \text{ min} = \underline{139.8 \text{ sec}}$$

(Process Timer)

(d) Zone Rates (Pumparound Flow Rates)

Non-selective Volume Rate:

$$W = V_w/\theta = \frac{589 \text{ vols}}{0.932 \text{ hr}} = \underline{632 \text{ vols/hr}}$$

(Note that this is approximately 6 times the adsorptive volume rate.)

$$\begin{aligned} L_2 &= K = (L_2/W) \times W = 1.12 \times 632 = 708 \text{ vols/hr} \\ L_{2b} &= L_{2b}/W \times W = 0.97 \times 632 = 613 \text{ vols/hr} \\ L_4 &= L_4/W \times W = 0.95 \times 632 = 600 \text{ vols/hr} \\ L_3 &= [A \times (Dn/A) / \%nC_s] + W \\ &= (118 \times 2.2) / 0.60 + 632 = 1065 \text{ vols/hr} \end{aligned}$$

(e) Line Flush

Line Flush is set at 200% of the longest line (V_{tbl}).

Therefore,

$$\begin{aligned} H &= (\% \text{ Line Flush}) \times V_{tbl} / T \\ &= \frac{(2.0) \times 1 \text{ vol}}{139.8 \text{ sec}} \times \frac{3600 \text{ sec}}{\text{hr}} = 52 \text{ vols/hr} \end{aligned}$$

NOTE: $H_{in} = H_{out}$

(f) Remaining Flow Rates (Calculated from Volume Balances)

				vols/hr
L_{2a}	$= L_2 + H_{in}$	$= 708 + 52$	$=$	760
H_z	$= L_2 - L_{2b}$	$= 708 - 613$	$=$	95
E	$= L_3 - L_{2b}$	$= 1065 - 613$	$=$	452
L_{3a}	$= L_3 + H_{out}$	$= 1065 + 52$	$=$	1117
D	$= L_{3a} - L_4$	$= 1117 - 600$	$=$	517
L_1	$= L_{2a} + F$	$= 760 + 492$	$=$	1252
R	$= L_1 - L_4$	$= 1252 - 600$	$=$	652

Check Calculations

$$R = F + H_{in} - H_{out} + H_z - E + D$$

$$R = 492 + 52 - 52 + 95 - 452 + 517 = \underline{652}$$

NOTE: Operators must perform these calculations in order to set the plant flow rates whenever an operating change is made. This applies whether or not the computer or manual set point stations are used.

F. OPTIMIZATION

Product purity and recovery are the primary index of a good operation. After the unit is lined out at the desired operating conditions, samples should be taken to determine product purity and recovery. In the optimization, there are four major variables which are adjusted, normally only one at a time, A/F_n , L_2/W , D_n/A , and L_4/W .

The most sensitive control of the product purity is the Zone II flow rate. A comparatively small change, say of 0.02, in the reflux ratio (L_2/W) will produce marked changes in purity and recovery and during optimization of the unit. Moves of this order should be made and the unit stabilized.

A/F_n is set to give complete adsorption of n-paraffins in Zone I. Increasing this ratio markedly will be of no benefit and will cause excessive utility usage.

Once the Zone IV rate is set such that there is no significant contamination of the n-paraffins product across Zone IV (this will be checked by reference to the pumparound chromatograph), no further changes should be required under stable operating conditions.

Desorbent flow should be sufficient to complete desorption in Zone III. If it is suspected that desorption is not complete, a small change in Zone III rate (say 0.02-0.05 in reflux ratio, Dn/A) should be made with the corresponding change in desorbent flow and the effect noted. Excessive desorbent flows will consume utilities.

Figure VIII-1
Adsorbent Volumes

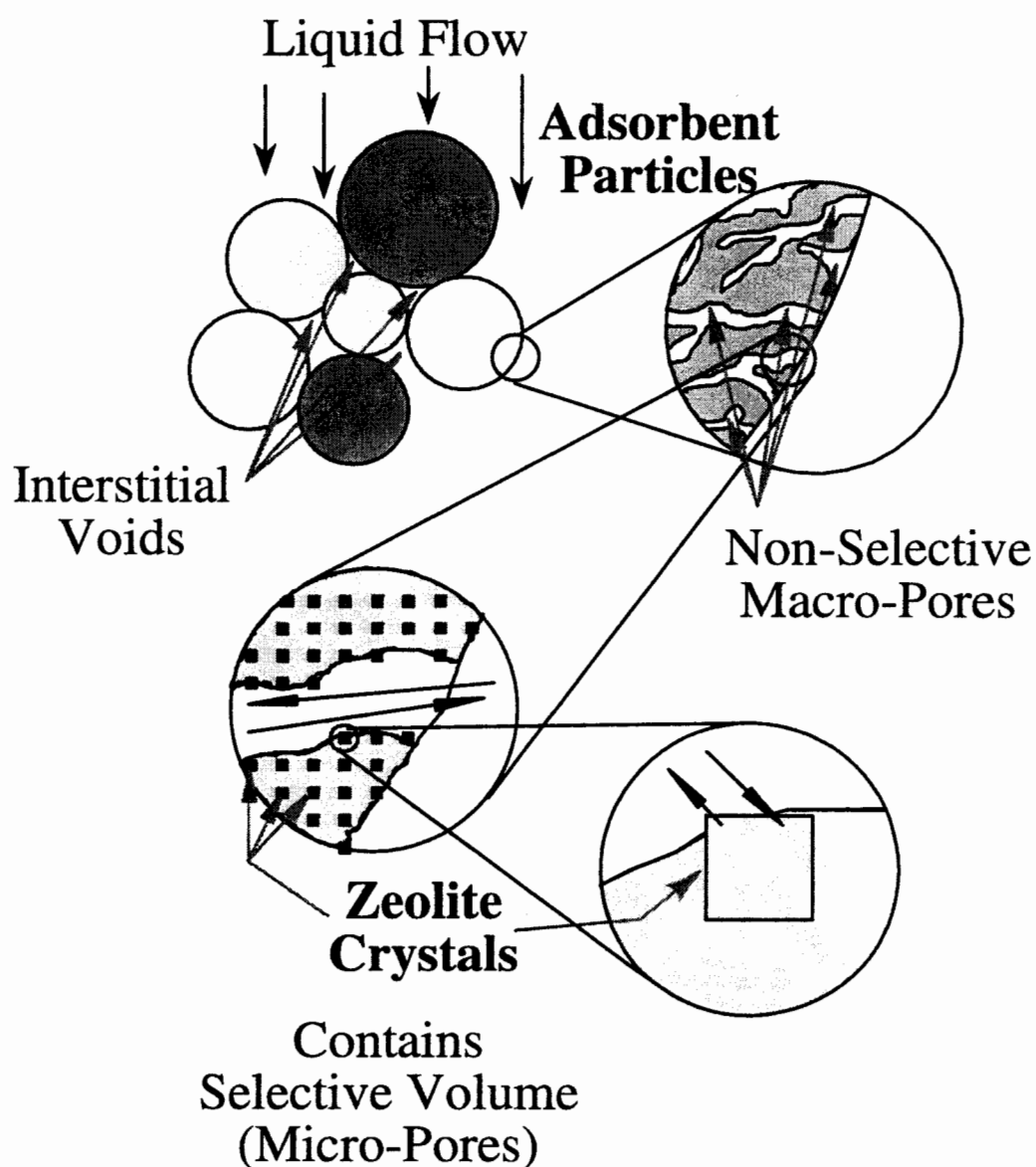


Figure VIII-2
Illustration of the "W" Concept

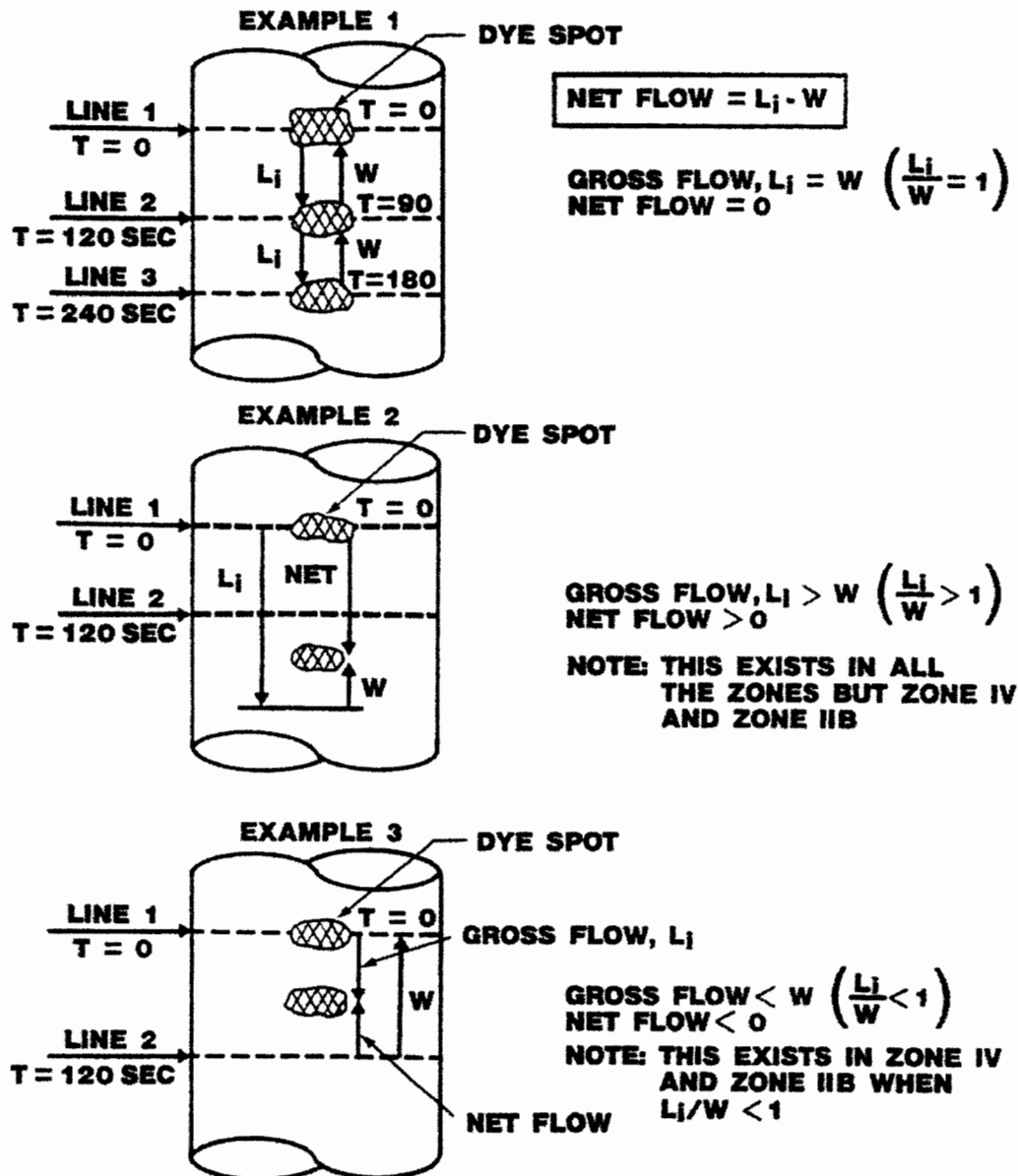
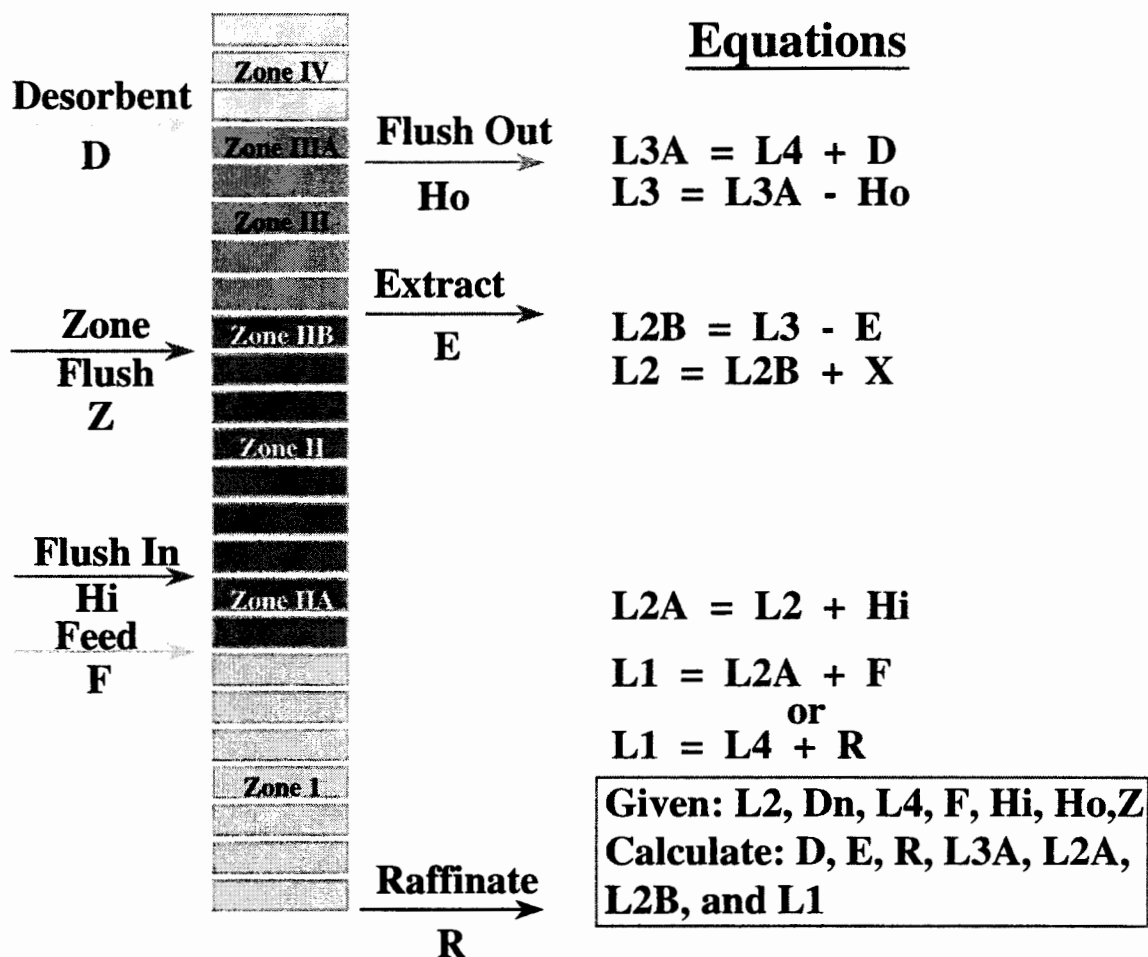
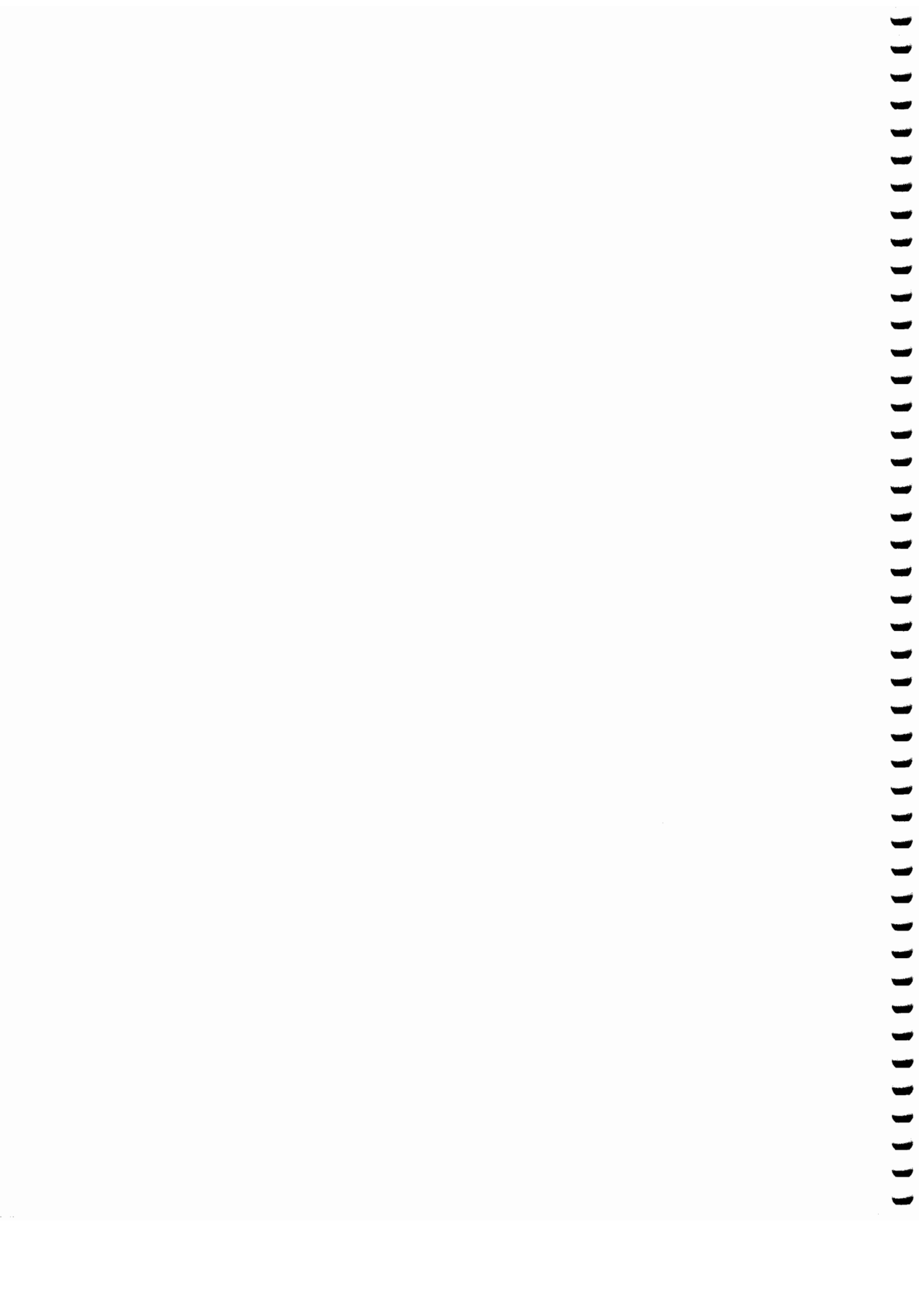


Figure VIII-3
Calculation of Zone Flows from Volume Balances









IX. ANALYTICAL INFORMATION

A. SAMPLING

Sampling process streams normally requires some care in order to limit personnel exposure and prevent the release of potentially dangerous compounds into the environment. All streams should be sampled carefully. Even if the stream is thought to contain non-hazardous and non-toxic chemicals, good sampling techniques and care should be employed.

For liquids, the sample containers can be made of glass or metal. Metal cans have the advantage of being unbreakable while 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 normally occurs because the level in the container can be observed while filling.

UOP recommends the use of glass bottles where excessive breakage does not occur. It is recommended that the bottles be permanently labeled and used repeatedly for the same samples.

A typical sample connection for a liquid stream 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 through the $\frac{3}{4}$ inch pipe would be in the opposite direction of that shown, and the positions of the gate and globe valves would be reversed.

For hot liquid streams, a 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 one foot. This limits the amount of liquid that must be drained in order to flush the line prior to sampling.

The sample circulation loop should be put into operation during the initial unit startup. The following is the procedure used to put the loop into operation:

1. Close the $\frac{1}{4}$ inch globe 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 unit 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 from the cooler.

To obtain a sample, the $\frac{1}{4}$ inch valve is opened to produce a steady flow of liquid to the drain. The sample container should be filled about 25% full two or three times and rinsed out with the liquid flowing from the sample connection. Finally, the sample container should be filled about 75% full. It should not be completely filled because a small vapor space is desirable to adjust for changes in temperature.

Gas samples are normally taken using a closed sample connection like that shown in Figure IX-2. At the bottom of the figure a detailed procedure for using the sample connection is outlined. However, it should be emphasized that the sample cylinder (bomb) should be nitrogen purged in the laboratory and pressured to 20-30 psig with nitrogen prior to taking a sample. The sample cylinder should then be well purged with the process gas so that a good representative sample of the gas stream is obtained.

B. LABORATORY TEST SCHEDULE

In order to fully evaluate the performance of any process unit it is necessary to maintain detailed analytical as well as operating records. All streams entering and exiting the Molex process unit should be routinely analyzed. The analyses conducted should be detailed and should include stream compositions, specific gravities, and other properties that are important from a process and catalyst protection standpoint. The stream compositions should indicate not only the major components but also certain minor components or contaminants. This is very important because even small concentrations of certain impurities can have an adverse impact on the overall process performance.

The laboratory staff and personnel should be thoroughly familiar with the various analytical test methods prescribed for the Molex process unit. A detailed schedule listing the various streams to be analyzed, analysis type, prescribed method, and the frequency of sampling has been prepared and is attached to this section. This is a fairly exhaustive schedule and may be modified as operating experience is gained. However, the UOP Chief Technical Advisor on site for unit startup may require more sample analyses to line out the unit and check its performance in the shortest period of time.

C. SAMPLE SHIPPING TO UOP

Listed below are general procedures to use when sending samples(s) for analyses to UOP. By following these directions, delays in sample analyses will be minimized and aid in a quicker turnaround, once the sample has been received.

General Letter for Domestic Samples (from within the USA)

Revised February 2, 2002

Following are the general procedures to be used when sending sample(s) to UOP for analysis, with the exception of SPA Catalyst samples, which will continue to be handled as before.

Information:

1. Please complete the attached Form and Fax it to UOP in Des Plaines, IL at (847) 627-8225 when the samples are shipped. Please note that completing this form will minimize delays in sample analysis.

Samples:

1. Label samples clearly with refinery name, location, sample description, and date collected.

2. Ship samples to:

UOP LLC
Sample Receiving
200 E. Algonquin Road
Des Plaines, Illinois 60017-5016

Attn: Technical Service Sample Coordinator

3. We recommend that samples be shipped by Federal Express or Emery. US Mail may be used for non-hazardous samples when expedited service is not required. Samples must be classified for "IATA" or "DOT" regulations and MUST be accompanied by the appropriate Material Safety Data Sheet (MSDS). The MSDS should include CAS numbers for identification per TSCA (Toxic Substances Control Act).
4. Include a copy of the attached Form with the samples.
5. Hand carrying of samples to UOP without proper documentation is illegal. Therefore, UOP employees and customers are to refrain from transporting any samples by this method, from any refinery to any UOP laboratory or office.

DOMESTIC
Analytical Requisition Form - UOP Technical Service

TO: UOP Technical Service Sample Coordinator Phone 847-391-2620 FAX: 847-627-8225

UOP Technical Service Contact _____ Phone 847-391- Fax 847-391-2253

Customer: Refinery _____
Address _____
Contact Name _____ Phone _____ Fax _____
e-mail address _____

Liquid & Gas Sample(s)

<u>Description</u>	<u>Analysis Required:</u>	<u>Check if MSDS is Included</u>	<u>Rush or Standard</u>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Catalyst & Adsorbent Sample(s):

<u>Catalyst Type</u>	<u>Regenerated or Coked</u>	<u>Analysis Required</u>	<u>Check if MSDS is Included</u>	<u>Rush or Standard</u>
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Billing Information:

Address: _____

Attention: _____

Sample Shipping Information:

Shipped Via: _____
Phone Number of Carrier: _____
Airwaybill No.: _____

SAMPLE CONTAMINATION - CONTAINER PROBLEMS

There have been found to be problems associated with the shipment of liquid samples shipped in standard laboratory vials with conical polyethylene caps. It has been found, through controlled tests, that samples permeate out of the containers in question, and can actually cross permeate between samples.

Tests were performed with the type of vials generally available in the laboratory using caps with liners of various materials. In a typical test, a vial containing only benzene and a vial containing only orthoxylene were placed together in a sealed jar for a period of time. The jars were heated to 40°C (a very possible shipping temperature) to speed the permeation. Tests were done both with the vials in an upright position and with the vials lying on their side so that the liquid would contact the caps. The common conical polyethylene lined caps cross-permeated from 0.50 to 4.0% under these conditions. Paper lined caps produced terrible results. Corks and Teflon linings worked well until they were tipped over, at which point these containers also produced terrible results. The best caps found thus far are the foil lined caps. The aluminum foil lined caps cross-permeate only slightly and the tin foil lined caps do not cross-permeate at all.

The following recommendations are made: If possible, keep the samples cool. Temperature does affect the permeation rate through some materials. Use tin foil lined caps when available and keep the samples as far apart as possible so they will have less tendency to cross-permeate.

SHIPMENT OF OVERSEAS SAMPLES

(current date)

Company Name
Address
State, City, Zip
Country

Dear Sir or Madam:

**SUBJECT: SAMPLE SHIPPING PROCEDURE:
To Be Used For All Samples Except SPA Catalyst**

In order to comply with U.S. regulatory laws outlined by the Environmental Protection Agency (EPA) and United States Customs, UOP LLC must assure that all chemical substances imported into the United States adhere to the following procedures.

IMPORTANT: The use of a freight forwarder (e.g. Burlington Air Express (BAX) or Emery Worldwide) or commercial cargo airline is preferred. Please note that UOP, as importer, must provide the TSCA (Toxic Substances Control Act) Certification. For samples sent by a freight forwarder or a commercial cargo airline, UOP will provide the TSCA Certification directly to our Customs Broker.

Overnight couriers (e.g. DHL, FedEx, UPS) may be used only if the written TSCA certification, authorized, signed and dated by a UOP LLC employee, is obtained prior to shipment, and physically included with the documentation shipped with the package.

All samples should be routed through Chicago O'Hare International Airport.

All imports must have the following documentation:

1. Bill of Lading (also known as a Master Airwaybill) which includes vessel/flight information. If using BAX Global, the Bill of Lading description field **must** include the words **"TSCA Certified Chemical Sample." "Customs Clearance Through BAX Global, O'Hare International Airport, Chicago, IL Attn: Frances Willis"**. If **not** using BAX Global, the Bill of Lading description field **must** include the words **"TSCA Certified Chemical Sample." "Customs Clearance Through Expeditors International, 1300 Arlington Heights Rd, Itasca, IL Attn: Bob Taussig"**.
2. Pro forma invoice (See attachments 2a and 2b: a blank form and example) including the following information:
 - Shipper's name, address, contact name, phone number and fax number.
 - Importer's name, address, and contact name, (this must include the UOP contact person within the U.S.). UOP has provided this information, except for Contact Person.

- Consignee/Delivery name, address, contact name, phone number and fax number (this must include the actual delivery location). UOP has provided this information.
 - Approximate market value (for Customs purposes), for each item, in U.S. Dollars.
 - Sample descriptions in English.
 - Packing details – the number and types of containers.
 - Net weights and Gross weights for each item, in kilograms.
 - Country of origin. This is the country where the sample was taken.
3. TSCA Certification (See attachment 3) including the following information:
- Sample description in English, UOP has provided this information.
 - Date the sample is to be shipped, in English.
 - Signature by UOP LLC employee (this is included, please do not change).
4. Analytical Requisition Form (Attachment 4) including the following information:
- Shipper's name, address, contact name, phone number, fax number, and e-mail address.
 - Sample shipping information: Carrier and flight information, phone number of the carrier, airwaybill number.
 - Fill in all pertinent sample and analytical request information as required.
5. Material Safety Data Sheet (MSDS) if the sample material is regulated as hazardous by IATA, IMO or DOT.

All samples must be prepared according to the hazardous materials shipping regulations of the International Air Transportation Association (IATA), if shipped by air, or International Maritime Organization (IMO) if shipped by sea.

Prior to shipping, the UOP Technical Services Sample Coordinator must be notified of all imports. **Please fax a copy of the Pro Forma Invoice, the Bill of Lading, TSCA Certificate Form, and the Analytical Requisition Form to the UOP Technical Services Sample Coordinator at 847-391-2253. In addition, include a copy of all the documents inside the package, with the samples.** UOP will then ensure proper Customs and TSCA clearance.

Failure to send samples with the proper documentation and information will result in a delay of Customs clearance or refusal of the sample.

Sincerely,
UOP LLC

(name of person signing letter)
Technical Services Department

Attachment 2a

Pro-Forma Invoice

International Shipper/Exporter (Name & Address)

Contact Name: _____
Contact Phone No. _____ Fax No. _____

Invoice No: _____
Invoice Date: _____

Terms: Pro-Forma Invoice
Reference No: _____

DESCRIPTION	AMOUNT		
*** NO CHARGE INVOICE *** QUANTITY: _____ DESCRIPTION OF SAMPLE(S): _____ PACKED IN _____ BOX(ES) GROSS WEIGHT: _____ KGS. COUNTRY OF ORIGIN (where sample was taken) _____ MARKS: AS ADDRESSED <table border="0"><tr><td>IMPORTER OF RECORD UOP LLC 25 EAST ALGONQUIN RD. DES PLAINES, IL 60017 ATTN: _____ UOP Contact Name</td><td>CONSIGNEE / DELIVERY ADDRESS: UOP LLC SAMPLE RECEIVING 200 EAST ALGONQUIN RD. DES PLAINES, ILLINOIS 60017-5016 ATTN: UOP Tech Service Sample Coordinator</td></tr></table> _____ Refinery Representative Signature _____ Refinery Representative Name: please print I HEREBY CERTIFY THAT THIS INVOICE IS TRUE AND CORRECT.	IMPORTER OF RECORD UOP LLC 25 EAST ALGONQUIN RD. DES PLAINES, IL 60017 ATTN: _____ UOP Contact Name	CONSIGNEE / DELIVERY ADDRESS: UOP LLC SAMPLE RECEIVING 200 EAST ALGONQUIN RD. DES PLAINES, ILLINOIS 60017-5016 ATTN: UOP Tech Service Sample Coordinator	US DOLLARS \$ _____ MARKET VALUE -- DECLARED FOR CUSTOMS CLEARANCE PURPOSES ONLY
IMPORTER OF RECORD UOP LLC 25 EAST ALGONQUIN RD. DES PLAINES, IL 60017 ATTN: _____ UOP Contact Name	CONSIGNEE / DELIVERY ADDRESS: UOP LLC SAMPLE RECEIVING 200 EAST ALGONQUIN RD. DES PLAINES, ILLINOIS 60017-5016 ATTN: UOP Tech Service Sample Coordinator		
ORIGINAL INVOICE			

Attachment 2b Example Form

Pro-Forma Invoice

International Shipper/Exporter (Name & Address)

Mountain View Refining
1000 Mountain View Dr.
Boulder City
Refining Country
Contact Name: Joe R. Engineer
Contact Phone No. (12) 3 456 -7890 Fax No. (12) 3-098-7654

Invoice No:
Invoice Date:

Terms: Pro-Forma Invoice
Reference No:

DESCRIPTION	AMOUNT
*** NO CHARGE INVOICE ***	US DOLLARS
QUANTITY: <u>Three 1 liter catalyst samples</u>	\$ <u>30.00</u>
DESCRIPTION OF SAMPLE(S): R-134 CCR Platforming Catalyst <u>Two regenerated samples and one coked sample</u>	MARKET VALUE -- DECLARED FOR CUSTOMS CLEARANCE PURPOSES ONLY
PACKED IN <u>1</u> BOX(ES)	
GROSS WEIGHT: <u>3.5</u> KGS.	
COUNTRY OF ORIGIN (where sample was taken) <u>Refining Country</u>	
MARKS: AS ADDRESSED	
IMPORTER OF RECORD UOP LLC 25 EAST ALGONQUIN RD. DES PLAINES, IL 60017 ATTN: <u>Robert S. UOP</u> UOP Contact Name CONSIGNEE / DELIVERY ADDRESS: UOP LLC SAMPLE RECEIVING 200 EAST ALGONQUIN RD. DES PLAINES, ILLINOIS 60017-5016 ATTN: UOP Tech Service Sample Coordinator	
<u>Joe R. Engineer</u> Refinery Representative Signature	Joseph R. Engineer Refinery Representative Name: <u>please print</u>
I HEREBY CERTIFY THAT THIS INVOICE IS TRUE AND CORRECT.	
ORIGINAL INVOICE	

Attachment 3
TSCA Certification Form

To: Area Director of Customs

Date: _____

Description of Sample: _____

(customer name and location)

(X) TSCA Positive Certification

I certify that all chemical substances in this shipment comply with all applicable rules or orders under TSCA and that I am not offering a chemical substance for entry in violation of TSCA or any applicable rule or order under TSCA.

Signature (Authorized UOP LLC employee)

Printed Name (Authorized UOP LLC employee)

Attachment 4**International Analytical Requisition Form - UOP Technical Service****TO: UOP Technical Services Sample Coordinator Phone 847-391-2620 FAX: 847-627-8225**UOP Technical Service Contact _____ Phone 847-391- Fax 847-391-2253

Customer: Refinery _____
Address _____
Contact Name _____ Phone _____ Fax _____
E-mail _____

Liquid & Gas Sample(s)

<u>Description</u>	<u>Analysis Required:</u>	<u>Check if MSDS is Included</u>	<u>Rush or Standard</u>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Catalyst & Adsorbent Sample(s):

<u>Catalyst Type</u>	<u>Regenerated or Coked</u>	<u>Analysis Required</u>	<u>Check if MSDS is Included</u>	<u>Rush or Standard</u>
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Billing Information:

Address: _____

Attention: _____

Sample Shipping Information:

Shipped Via Carrier: _____
Phone Number of Carrier: _____
Airwaybill Number: _____

SAMPLE CONTAMINATION - CONTAINER PROBLEMS

There have been found to be problems associated with the shipment of liquid samples shipped in standard laboratory vials with conical polyethylene caps. It has been found, through controlled tests, that samples permeate out of the containers in question, and can actually cross permeate between samples.

Tests were performed with the type of vials generally available in the laboratory using caps with liners of various materials. In a typical test, a vial containing only benzene and a vial containing only orthoxylene were placed together in a sealed jar for a period of time. The jars were heated to 40°C (a very possible shipping temperature) to speed the permeation. Tests were done both with the vials in an upright position and with the vials lying on their side so that the liquid would contact the caps. The common conical polyethylene lined caps cross-permeated from 0.50 to 4.0% under these conditions. Paper lined caps produced terrible results. Corks and Teflon linings worked well until they were tipped over, at which point these containers also produced terrible results. The best caps found thus far are the foil lined caps. The aluminum foil lined caps cross-permeate only slightly and the tin foil lined caps do not cross-permeate at all.

The following recommendations are made: If possible, keep the samples cool. Temperature does affect the permeation rate through some materials. Use tin foil lined caps when available and keep the samples as far apart as possible so they will have less tendency to cross-permeate.

D. MINIMUM SAMPLE SIZE

Analyses	Preferred Minimum (cm ³)
Density	100
Distillation	210 ⁽¹⁾
Composition	50
Sulfur, Microcoulometric	10
Nickel Reduction	100
Nitrogen, Kjeldahl	800
Microcoulometric	10
Chloride	100
Arsenic	400
Lead	100
Product	
Density	100
Distillation	210 ⁽¹⁾
Composition by GC	150
Adsorbent	
All Common Analyses	2 liters

Note: (1) May also be used for density

The operation of the Molex Unit requires not only analysis of major components in the circulating stream, but of certain minor components that have an effect on the process. Analysis of the major components is fairly straight-forward and can be obtained directly from the Test Method write-ups. The attached Tables are organized by stream and tabulate the Methods and frequencies for the important streams. A complete laboratory test schedule is attached.

E. PHYSICAL PROPERTIES

Some physical property information about Molex feed and Desorbent components is enclosed.

LABORATORY TEST SCHEDULE

STREAM	TEST METHOD	FREQUENCY
--------	-------------	-----------

MOLEX FEED

Relative Density	ASTM D-4052 or D-1298	1/DN 3/DS
Distillation	ASTM D-86	1/WN 1/DS
Saybolt Color	ASTM D-156	1/WN 1/DS
Normal Paraffins Composition	UOP 411	1/DN 3/DS
Aromatics Content	UOP 495	1/DN 1/DS
Bromine Index	UOP 304	1/DN 1/DS
Water Content	UOP 481	2/WN 1/DS
Nitrogen	ASTM D-4629 or UOP 384	1/DN 3/DS
Sulfur	ASTM D-4045	1/DN 3/DS
Total Chloride	UOP 395	Occas 1/DS
Fluoride	UOP 619	Occas 1/DS
Peroxide No.	ASTM E-299	1/WN 1/DS
Carbonyl Number	UOP 624	1/WN 1/DS
Phenol	UOP 262	1/WN 1/DS

EXTRACT COLUMN BOTTOMS

Relative Density	ASTM D-4052 or D-1298	1/DN 3/DS
Distillation	ASTM D-86	1/WN 1/DS
Saybolt Color	ASTM D-156	1/WN 1/DS
Normal Paraffins Composition	UOP 411	1/DN 3/DS
Aromatics Content	UOP 495	1/DN 1/DS
Bromine Index	UOP 304	1/DN 1/DS
Water Content	UOP 481	2/WN 1/DS
Nitrogen	ASTM D-4629 or UOP 384	1/DN 3/DS
Sulfur	ASTM D-4045	1/DN 3/DS
Peroxide No.	ASTM E-299	1/WN 1/DS
Desorbent	UOP 621	1/DN 1/DS

STREAM	TEST METHOD	FREQUENCY	
--------	-------------	-----------	--

RAFFINATE COLUMN BOTTOMS

Relative Density	ASTM D-4052 or D-1298	1/DN	3/DS
Distillation	ASTM D-86	1/WN	1/DS
Saybolt Color	ASTM D-156	1/WN	1/DS
Normal Paraffins Composition	UOP 411	1/DN	3/DS
Aromatics Content	UOP 495	1/DN	1/DS
Bromine Index	UOP 304	1/DN	1/DS
Water Content	UOP 481	2/WN	1/DS
Nitrogen	ASTM D-4629 or UOP 384	1/DN	3/DS
Sulfur	ASTM D-4045	1/DN	3/DS
Peroxide No.	ASTM E-299	1/WN	1/DS
Carbonyl Number	UOP 624	1/WN	1/DS
Phenol	UOP 262	1/WN	1/DS
Desorbent	UOP 621	1/DN	1/DS

DESORBENT

Relative Density	ASTM D-4052 or D-1298	1/DN	3/DS
Distillation	ASTM D-86	1/WN	1/DS
Component Distribution	UOP 621	1/DN	1/DS
Bromine Index	UOP 304	1/DN	1/DS
Water Content	UOP 481	2/WN	1/DS

FLUSH

Relative Density	ASTM D-4052 or D-1298	1/DN	3/DS
Distillation	ASTM D-86	1/WN	1/DS
Component Distribution	UOP 621	1/DN	1/DS
Bromine Index	UOP 304	2/WN	1/DS
Water Content	UOP 481	2/WN	1/DS

EXTRACT COLUMN OVERHEAD

Relative Density	ASTM D-4052 or D-1298	1/WN	1/DS
Distillation	ASTM D-86	1/WN	1/DS
Component Distribution	UOP 621	1/DN	1/DS
Bromine Index	UOP 304	1/WN	1/DS
Water Content	UOP 481	1/WN	1/DS

STREAM	TEST METHOD	FREQUENCY
--------	-------------	-----------

EXTRACT COLUMN SIDECUT

Relative Density	ASTM D-4052 or D-1298	1/WN 1/DS
Distillation	ASTM D-86	1/WN 1/DS
Component Distribution	UOP 621	1/DN 1/DS
Bromine Index	UOP 304	1/WN 1/DS
Water Content	UOP 481	1/WN 1/DS

RAFFINATE COLUMN OVERHEAD

Relative Density	ASTM D-4052 or D-1298	1/WN 1/DS
Distillation	ASTM D-86	1/WN 1/DS
Component Distribution	UOP 621	1/DN 1/DS
Bromine Index	UOP 304	1/WN 1/DS
Water Content	UOP 481	1/WN 1/DS

RAFFINATE COLUMN SIDECUT

Relative Density	ASTM D-4052 or D-1298	1/WN 1/DS
Distillation	ASTM D-86	1/WN 1/DS
Component Distribution	UOP 621	1/DN 1/DS
Bromine Index	UOP 304	1/WN 1/DS
Water Content	UOP 481	1/WN 1/DS

RAFFINATE AND EXTRACT FROM RV

Normal Paraffins Composition	UOP 411	Occas.
Component Distribution	UOP 621	Occas.

PUMPAROUND SURVEY

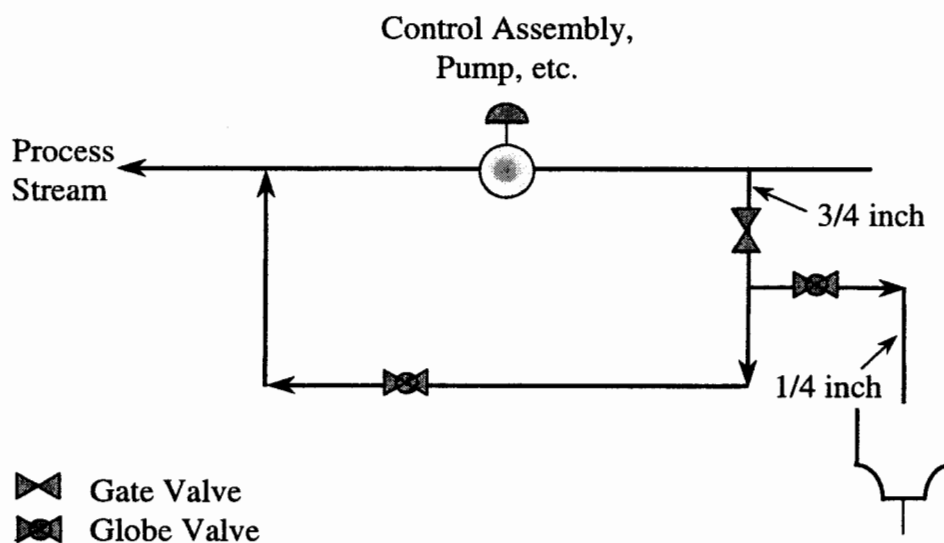
ON REQUEST. TYPICALLY 24 SAMPLES MUST BE SCHEDULED FOR PROMPT ANALYSES.

Normal Paraffins Composition	UOP 411	Occas.
Component Distribution	UOP 621	Occas.

1/WN: Once per week, normal operation
1/DN: Once per day, normal operation
3/DN: Three times per day, normal operation
1/DS: Once per day, startup
1/WS: Once per week, startup
3/DS: Three times per day, startup
3/WS: Three times per week, startup
Occas: Occasionally

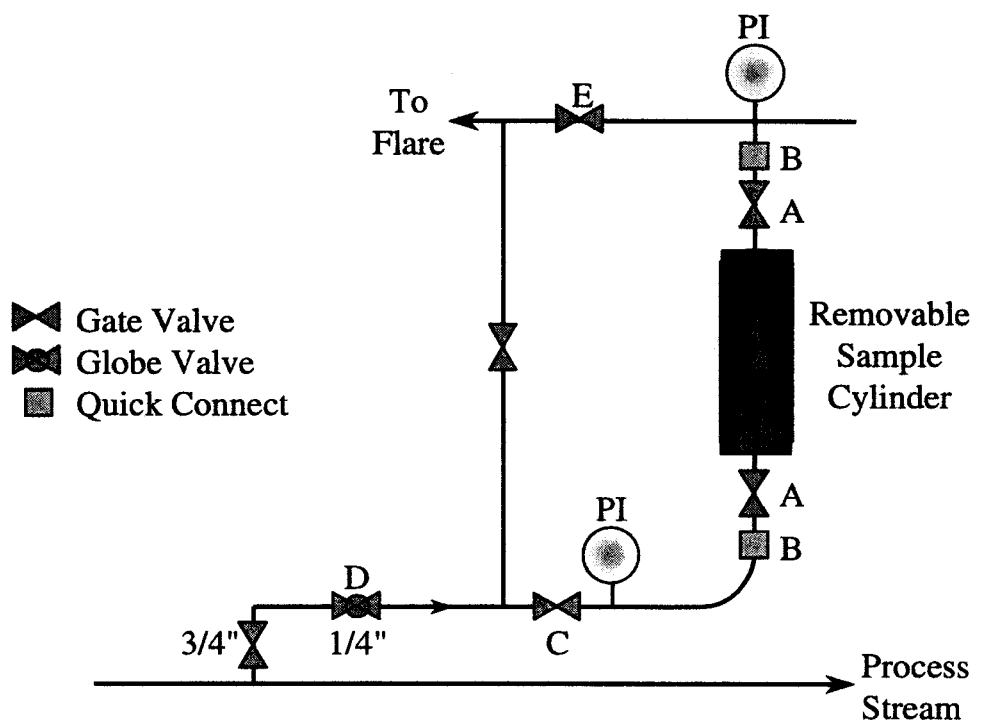
Frequency of required tests for startup operation will be determined by UOP chief technical advisor on site as he dictates the necessity during the startup.

Figure IX-1
Liquid Sample Connection



PAC-R00-71

Figure IX-2
Closed Flow-Gas Sample Connection



PAC-R00-72





X. TROUBLESHOOTING

Troubleshooting should be done on a continual basis. The operations group should always be on the lookout for potential problems developing. A general troubleshooting guide is presented in Figures X-1a and X-1b. Experience has shown that problems in the Molex unit have generally resulted from the following:

1. Flow Problems

a. Turbine Meters

Turbine meters are used instead of orifice meters to measure the F, D, E, Hi, Ho, Hz, and pumparound flows because of the higher accuracy they afford.

The turbine meter element is a tubular housing flanged into a pipe of the same diameter and inside of which a vaned rotor is made to spin on an axial shaft by movement of fluid through the pipe. The rotation of the rotor is sensed by a magnetic pickup that transmits a pulse each time one of the rotor blades passes the pickup coil. The frequency of these pulses is proportional to the volumetric flow rate of fluid. The frequency is converted to a standard milliamp signal to the FRC.

The axial position of the turbine rotor on its shaft is maintained by hydraulic balance. If foreign material enters any of the passages that affect that balance, the turbine will quickly destroy itself. For this reason, cartridge type filters or strainers are provided upstream of each turbine meter. Since the turbine meter can only handle flow in one direction, make sure to isolate the meter and open the bypass line if reverse flow is anticipated (i.e., during startup or dryout).

b. Backup Orifice Plates

All turbine meter streams have a backup orifice meter that is constantly recording that flow on a separate FR. In the case of a turbine meter failure, the operator must first switch from automatic digital control to the manual setpoint stations. The FRC

stream with the turbine meter failure is then switched over to the orifice plate flow signal and the flow adjusted to line up with the previously recorded value. Operation of any stream (including the pumparound) with an orifice plate flow meter results in a less than optimum plant operation. Normally, the plant can still produce on-spec product but at a reduced recovery.

c. Improper Flow Setpoints

If there is a problem with product quality and the zone rates are thought to be adequate to produce purity, the first step should be to recalculate all the settings as shown in Section VIII. If the recalculation shows that the flow settings are correct, a volumetric balance should be made across the chambers to ensure that all flow meter readings are correct. If these also are consistent, the next step should be to check that the flush rate is adequate. Normally this should be sufficient to flush twice the volume of the longest line per valve step. It is simple to make an increase in this and see if there is an appropriate response.

d. Chamber Circulation Vortex Meter ("Pusharound" FR)

(1) Discussion

The chamber circulation vortex meter is used on the Molex unit and other two chamber units using a digital pumparound controller. This system, if properly applied, can be used to balance and calibrate itself. For this discussion, the Molex described has 24 beds. The principles are the same for 20 bed Molex, but the bed numbers are different.

The pumparound flow is the heart of the rotary valve unit and is defined as the chamber circulation from Bed 24 to Bed 1. The pumparound controller is intended to keep each zone rate constant as the zone moves around the adsorption circuit. Proper zone equations are based on the rotary valve position at any given time (defined as the feed location).

In the circulation line from the bottom of Chamber No. 1 (Bed 12) to the top of Chamber No. 2 (Bed 13) is a vortex flow meter. This very important meter is provided to permit an independent check of the constancy of zone rates not in the pumparound flow. Any variation in a zone rate implies an inconsistency between the zone flow rates. This backside chamber circulation is on PRC control and is called the "pusharound" flow. The vortex meter recording is similar to that of the pumparound FRC, except it is less accurate but equally precise and it is out of phase with the pumparound trace by 12 beds. This is illustrated in Figure X-2.

The inferior accuracy of the vortex meter does not matter, however, as it is used only to look for step changes in flow rate within a zone, and for that it is quite sufficient. In fact, the zero and range of this elbow meter recorder may be adjusted to expand the trace to fill the chart for better precision.

The chart speeds for the pumparound and pusharound flow meters are normally double the speed for the rest of the charts.

(2) Extra Zone Effects

The pumparound FRC set point calculated by the digital controller (or by the operator when the manual set point stations are used) will be incorrect if there is an error in the flow measured by one or more of the turbine meters involved in the calculation. If this occurs, the flow pattern at the elbow meter will show shifts in the flow rate in between the normal seven zone changes per cycle of the rotary valve. These "extra zone effects" can be used to identify and correct the problem by observing where in the cycle they occur and whether the shift involves an increase or a decrease in the flow rate.

In all cases below, the pusharound zone remains the same, as the rotary valve steps from one position to the next. The pumparound zone changes, and thus the resulting equation used to calculate the pusharound flow changes by just one variable. If the rotary valve steps from Bed 7 to Bed 8, and turbine meter E is accurately measuring the extract flow rate, there will be no change in the Zone I flow rate and no extra zone effect. If, however,

turbine meter E is indicating a flow rate higher than the actual flow, and the pumparound turbine meter is correct, the resulting pusharound flow will be too large. Similar analysis can be used to determine errors caused by other turbine meters.

<u>Feed to Bed</u>	<u>Pusharound Zone</u>	<u>Pumparound Zone</u>	<u>Pusharound* Equation</u>	<u>Meter Tested</u>
1	III	IIa	$IIa + F - R + D - H_o$	H_{in}
2	III	II	$II + F - R + D - H_o + H_i$	
7	I	IIb	$IIb - H_z + H_i + F$	E
8	I	III	$III - H_z + H_i - F - E$	
15	II	IIIa	$IIIa - H_o - E + H_z$	D(E,F)
16	II	IV	$IV - H_o - E + H_z + D$	
24	III	I	$I - R + D - H_o$	F
1	III	IIa	$IIa - R + D - H_o + F$	

**See pumparound digital controller discussion.*

The erroneous flow rate can be corrected by adjusting the appropriate time gate or scaling factor until the actual, correct value for the turbine meter is reached and the extra zone disappears. The actual meter factor must then be determined by back calculating from the ratio of the two readings.

In the case of the change in valve position from Bed 15 to Bed 16, three turbine meters are involved. To determine that the D meter is at fault, it is necessary first to ensure that the E and F meters are correct by separate procedures above.

Also, in this case, it is necessary to eliminate one more variable before concluding that a turbine meter is in error. That variable is the flow rate of flush material to the adsorbent chamber heads. Since all other streams into and out of the adsorbent chambers are measured and controlled, the only exit available for the head flush is in the raffinate stream. Normally, the head flush flow rates are small enough to be

neglected. If, however, the head flush flow rates are excessive, an extra zone can appear in the pusharound flow.

When the valve position changes from Bed 15 to Bed 16, the raffinate changes from Bed 24 to Bed 1. When the raffinate is at Bed 24, the head flushes from Chamber No. 1 must pass through the pusharound and add to the Zone II flow in order to reach the raffinate. When the raffinate is at Bed 1, the head flushes from Chamber No. 2 must, in effect, pass backwards through the pusharound to reach the raffinate, thus subtracting from the Zone II flow rate in the pusharound. The magnitude of the extra zone effect is therefore directly a function of the magnitude of the head flush flow rates. If an extra zone appears in the pusharound when the valve moves from Bed 15 to Bed 16, the head flush flow rates should be checked first to ensure that they are not excessive and then the turbine meter can be checked.

Example 1 - Desorbent Turbine Meter Error

Basis: The pumparound rates calculated by the digital controller agree with the calculations done by hand.

Shift occurs when Zone IV is in pumparound.

$$\text{Zone IV rate} = "K" - H_z + E + H - D$$

When the feed switches from bed 15 to 16, the pusharound FR which is recording Zone II and this rate will shift if there is a desorbent turbine meter error. This is illustrated in Figure X-3.

The flow pattern is an indication that the turbine meter signal for the desorbent flow is in error (reading higher than actual flow). When Zone IV is in the pumparound, the desorbent and raffinate flows are in different chambers. If the actual flow of desorbent is low relative to the calculated Zone IV rate, Chamber No. 1 would tend to decrease in pressure (when the desorbent is in Chamber No. 1) resulting in a decrease in the "pusharound" flow. If the raffinate and desorbent flows are in the same chamber, the flow of raffinate (on pressure control) would automatically

compensate for the low desorbent flow as seen in the top half of the Zone II pattern above. If the erroneous Zone IV was in the "pusharound" flow, the difference between this flow and the calculated value would be extremely difficult to notice.

To balance the two-chamber system for this example, the desorbent time gate or scaling factor is decreased until the extra zone disappears from the "pusharound" FR (elbow meter). The time gate setting is the length of time the digital controller reads the turbine meter pulses. After this change, the controller is receiving a smaller signal for the desorbent flow, and the resulting Zone IV flow rate is higher than before (see Zone IV equation). To allow for a consistent material balance in the zone calculations, a new Zone IV reflux ratio must be "back calculated" from the new Zone IV flow rate. The desorbent turbine meter flow recorder will have a new pen factor so that the range corresponds to the smaller desorbent flow.

Similar zone shifts (extra zone effects) can be caused by the other 5 turbine meter inputs to the digital controller (F, H_i, H_o, H_z, E). However, because the line flush streams (H_i, H_o) are small and the "pusharound" zone patterns are too short (one bed) to see a shift, a zone imbalance caused by the line flushes cannot be identified. An imbalance caused by the other streams can be seen as they pass through the pumparound loop:

	<u>Pumparound (FRC)</u>	<u>Pusharound (FR)</u>
F	Zone I to Zone IIa	Zone III
E	Zone IIb to Zone III	Zone I
H _i	Zone IIa to Zone II	Zone III

To correct any zone shift caused by the turbine meters, the proper scaling factor should be adjusted until the imbalance disappears.

Example 2 - Excessive head flushes to the chambers

Basis: Controller calculations are correct, as before Pumparound flow switches from Zone IV to Zone I

All four head flushes entering the adsorbent chambers must be pressured out via the raffinate (PRC controlled) line, as all other flows are FRC controlled and do not account for these small flows. When these flushes are excessive, the extra zone is seen in the "pusharound" zone pattern as the raffinate switches from Bed 24 to Bed 1 (Chamber No. 1).

When the raffinate is in Chamber No. 2, the "pusharound" flow includes the total head flush from Chamber No. 1. When the raffinate switches over to Chamber No. 1, the Chamber No. 1 head flush flows exit through the raffinate directly, but the Chamber No. 2 head flushes will tend to increase the Chamber No. 2 pressure. This higher pressure in No. 2 opens the raffinate PRCV that lowers the Chamber No. 1 pressure. The net effect is that the lower Chamber No. 1 pressure decreases the "pusharound" flow (Chamber No. 1 to Chamber No. 2) by an amount equal to the sum of all head flushes.

The solution in this case is to decrease the head flushes until the extra zone disappears in the "pusharound" FR. This effect will occur in every system, but normally will be very small and impossible to notice.

2. Pumparound Pump Survey

a. General

The pumparound pump (PAP) survey is a series of samples taken at the PAP with the feed in each bed. Analysis of these samples shows the composition of each zone as it passes through the PAP. These results can be plotted and used as a tool to analyze the plant performance.

The sample system should have short lines from the PAP discharge through the sample cooler back to the PAP pump suction. A continuous flow should go through the sample lines. Special sample bombs are required because of the high volatility of the nC_5 . The samples should be analyzed using a

modification of UOP Method 411. A drawing of a typical sample bomb and a copy of the laboratory method are enclosed.

The sampling procedure is very important because of the changing composition at the PAP stream; not only because of the difference in zone composition but also because of the step wise advancement of the beds. The composition of the PAP streams at the start of a rotary step is different from the composition of the PAP stream at the end of the step. This is especially true at beds adjacent to process streams. Therefore, each bed should be sampled at exactly the same point in time during a given step for consistency. A good approach is to take each sample in the middle of each step.

For a full PAP survey:

- (1) Prepare 24 sample bombs that are clean, dry, and labeled.
- (2) Make sure the valves of the sample system are wide open and there is a good flow through the sample cooler.
- (3) Communication should be established so that the person at the sample cooler knows exactly when the valves step and what bed is in the PAP system.
- (4) (0.5T-20) seconds after the valve has stepped, the sample tap is opened to flush line for ten seconds; adjust the rate to about 10 cc/sec. "T" is defined as the step time in seconds. During the 20 sec. from $t = 0.5T - 10$ to $t = 0.5T + 10$ seconds the sample bottle is filled at a constant rate.

b. PAP Survey Results

Plot the percentages of the important components versus each bed sampled on semi-log paper. See the enclosed example Figure X-4. The following

areas should be analyzed and any necessary zone adjustments can be made.

- (1) How well is the extract component adsorbed in Zone I. This curve should show a fast decline of that component from the feed point down in Zone I. A convex curve would indicate insufficient adsorption and an increase in A/F_n may be considered.
- (2) In Zone II, we can see how well the undesired components are refluxed down.
- (3) In Zone III, we want to see that if at the end of Zone III the desorption is complete and nothing is breaking through into Zone IV.
- (4) In Zone IV, we can see if any raffinate material is breaking into Zone III.

3. Column Operation

- a. Although not considered a variable as such, column operation has a profound effect on plant performance. If the extract and raffinate columns have excessive heat input, the Molex feed components will go overhead contaminating the desorbent. In the case of the raffinate column, these impurities will lower the product purity. One may unthinkingly increase L_2/W , resulting in decreased recovery of n-paraffins.
- b. Poor operation of the extract column can cause n-paraffins to be present in the desorbent, thereby causing a loss in recovery.
- c. The desorbent splitter column must be operated correctly to produce a relatively pure iC_8 , 99%+, stream for the flushes. If nC_5 is allowed to drop into the column bottoms, a decline in recovery will result as the nC_5 strips the n-paraffins off the adsorbent in Zone II.

4. Mechanical Leakage

Mechanical leakage can result from contamination of the adsorbent chamber heads and leakage of the contaminated material into the pumparound system.

If this occurs, the unit should be checked for possible leakage by taking chamber head samples to see if the head liquid contains any feed components. If this is found to be so, then the flush to the heads should be increased. If this is found to be insufficient or if excessive loss of recovery results, then a shutdown will have to be scheduled to inspect the top heads.

UOP's design of chamber internals is such that if installation is properly carried out, the risk of contamination is minimal, provided flush is adequate.

5. Rotary Valve Problems

Problems caused by the rotary valve have been found to be comparatively rare.

Internal leakage in the rotary valve can affect either product purity or recovery, or both. The product purity is most sensitive to leakage and valve trouble may show as reduced purity. If internal leakage is suspected, an increase in dome pressure may be made.

A careful check should first be made on the rotary valve dome flush rates. If the rates into and out of the dome do not balance, leakage is possible. Any increase in dome pressure should be made with care since excessive dome pressure will lead to increased wear of the Teflon seal within the rotary valve. After the dome pressure is increased, the product purity and/or recovery should be improved if valve leakage is the cause of the performance debit. It is always desirable to operate the valve with as low a dome pressure as possible without excessive leakage. The likelihood of leakage increases as the rotary valve and chamber systems hydraulic loads are increased. A rotary valve track pressure survey may help diagnose hydraulic limitations in the rotary valve.

6. Contaminants

If a slow reduction in recovery is experienced during normal operations, or a sharp reduction after a feedstock upset, it is possible that adsorbent poisoning is occurring. This is most serious and the cause should be corrected at once. It is much better to stop feeding the Molex unit than risk adsorbent poisoning.

As one would suspect, there are many feed contaminants that are poisonous to the adsorbent and reduce its ability to selectively adsorb n-paraffins. Among the more common poisons are sulfur compounds, nitrogen compounds, oxygenated compounds including (water and alcohols), olefins, and mineral acids. Some cause serious loss of capacity, others may cause irreparable damage.

Olefins polymerize on the adsorbent and seriously reduces the adsorption capacity. These polymers are not easily removed from the adsorbent. Excessive water (due to water or steam exchanger failure) can cause rapid sieve deactivation, although in many cases no permanent damage is done. The first indication of water contamination would be very low recovery.

The Molex feed is first pre-treated to reduce the contaminants to a level that will give a reasonable adsorbent life. The following maximum limits have been established for this unit. It should be emphasized that these limits are maximum limits, and that contamination should, in all cases, be as limited as possible.

Water	5 wt-ppm
Total Oxygen	1 wt-ppm max., no standard method currently available
Sulfur	1 wt-ppm max., preferably less than 1 ppm
Nitrogen	0.5 wt-ppm max., preferably less than 0.1 ppm
Chlorides	0.1 wt-ppm max.
Fluoride	0.1 wt-ppm max.
Bromine Index	150 max.
Carbonyl	2 mg/L max.

Although there is no refinery method for determining the total oxygen content, the nitrogen specification is low enough that the oxygen level should be on spec.

It should be remembered that most corrosion inhibitors especially the filming amine type, are serious poisons. Such materials should not be used anywhere in the Molex complex. Also to be included in a list of possible accidental contaminants are pipe compounds, cutting oils, and drying oils.

7. Chamber Pressure Drop/Chamber Pressure

Pressure drop in the chamber should be watched carefully. There may be several reasons for a sharp increase in pressure drop during operation, the most common of which is plugging of the beds and their support screens by either fines or dirt and scale. Any unexpected pressure drop should be promptly investigated.

Adsorbent chamber pressure is also important. Since the chamber is liquid-full, a small change in liquid rate entering or leaving the chamber will have a drastic effect on chamber pressure, unless the pressure controller is in operation and functioning properly.

The normal operating pressure of the chamber is sufficiently high to keep the hydrocarbons from boiling. If the pressure falls below the bubble point, the liquid inside the chamber will boil and form vapor. This is undesirable and should be avoided.

A pressure switch is provided on the Chamber No. 2 pressure controller so that if the chamber pressure falls below a predetermined point, the extract control valve will close. A time delay is also provided so that a few seconds elapse before the control valve closes. This time delay reduces the chance that a pressure decrease of very short duration would close the extract control valve and trigger a series of pressure fluctuations leading to a major upset.

When starting flows to the chamber, the initial rate should be very small. If there is no pressure increase within a short time, a vapor pocket may be present. Keep initial rates low until a pressure buildup is noticed and then place the pressure controller in service. Do not make large increases in flows until the pressure controller functions satisfactorily.

8. Molex Adsorbent Chamber and Rotary Valve Pressure Surveys

The following are UOP's recommendations on how the pressure survey around the adsorbent chambers and rotary valve should be performed. These data will provide important information on the hydraulics of the Molex unit. This information may allow UOP to better determine the ultimate capacity of the rotary valve and adsorbent chambers or possibly indicate a problem internal to the adsorbent chambers.

- a. The unit should be in steady operation at the highest possible throughput. Document all the net stream flows (F, E, D, R, Z, H_{in} , H_{out}) and the pumparound flow rate for each zone in the attached tables.
- b. Check the individual bed pressure drops using a movable differential pressure indicator. The differential indicator should have a range of approximately 0-2 kg/cm².

The instrument should be located below the elevation of the grids being checked so that gas is not trapped in the impulse legs and the effect of static head is reduced.

Make temporary piping connections from the low point drains on each pair of adjacent bed lines to the indicator. Measure the pressure drop across the corresponding bed while Zone I flow is passing through the bed while there is no flow in the bedlines the indicator is attached to. For example to measure the pressure drop across bed 3 the high point of the indicator is connected to bedline 2 and the low point of the indicator is connected to bedline three. Readings are to be taken when the instrument indication is stable when feed is in bedlines 21, 22, 23, 24, or 1.

For checking the pressure drops across beds 1, 13, 12, and 24 the adsorbent chamber head sample points should be used to avoid including the pressure drop of the distributor piping in the measurement.

Record the data in Table 1. After checking the pressure drop for each bed, the indicator may be left connected to the bed with the highest pressure drop so that it can be easily monitored.

- c. Check the process track pressures at the rotary valve. Calibrate a test pressure gauge with a range of 0-40 kg/cm²g. Check the pressure of one of the net streams flowing to or from the rotary valve at a low point drain or other connection which is on the rotary valve side of that stream's control valve. Measure the stream's pressure for each rotary valve position (FIP) and record it in the attached table. After measuring the pressure of a stream for the entire rotary valve cycle, use the **same** pressure gauge to measure the pressures of the other streams in a similar manner. Record the data in Table 2.
- d. In evaluating a unit for a future increase in throughput the pressure drop through the rotary valve may also be helpful. The same differential pressure indicator that was used in "b." may be used. The pressure drop through the rotary valve can be measured by connecting the differential pressure indicator from a low point drain on a bed line to the hose connection at the pressure indicator located between the rotary valve bypass manifold and the rotary valve. The differential instrument should be located below the connections so that gas will not be trapped in the instrument. The pressure drop is measured when the process flow is in the bedline that the differential instrument is connected to.

Typically the feed, extract, desorbent and the raffinate flows should be documented as these are the flows most likely to be found limiting the throughput of the rotary valve.

Table 1

UOP**Molex Adsorbent Chamber Pressure Survey**

Customer Name _____

Date _____

Time _____

Zone Ratios A/Fn= , L2/W= , L2B/W= , Dn/W= , L4/W= , Cycle time, min.=

Bed		Differential Pressures kg/cm ² in Zone I
Dist Pipe	1/TH	
1	TH/2	
2	2/3	
3	3/4	
4	4/5	
5	5/6	
6	6/7	
7	7/8	
8	8/9	
9	9/10	
10	10/11	
11	11/12	
12	12/BH	
Dist Pipe	13/TH	
13	TH/14	
14	14/15	
15	15/16	
16	16/17	
17	17/18	
18	18/19	
19	19/20	
20	20/21	
21	21/22	
22	22/23	
23	23/24	
24	24/BH	

Track Flows, m ³ /h @ 177°C						
F	Z	R	D	E	H _{in}	H _{out}

Pumparound Flows, m ³ /h @ 177°C						
I	IIA	II	IIB	III	IIIA	IV

Chamber A bott pressure, kg/cm²g _____Chamber B bott pressure, kg/cm²g _____RV dome pressure, kg/cm²g _____Chamber A/B Overall Delta Pressure, kg/cm² _____ / _____

TH = top head, BH = bottom head

Table 2

UOP**Molex Rotary Valve Track Pressure Survey**

Customer Name _____

Date _____

Time _____

Zone Ratios A/Fn= _____, L2/W= _____, L2b/W= _____, Dn/A= _____, L4/W= _____, Cycle time, min.= _____

Track Pressures, kg/cm ² g								Delta p, kg/cm ² g	
FIP	Feed	Zone Flush	Raffinate	Flush In	Desorbent	Flush Out	Extract	Chamber A	Chamber B
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
Track Flows, m ³ /h @ 177°C									
Pumparound Flows, m ³ /h @ 177°C									
I	IIA	II	IIB	III	IIIA	IV			

Chamber A bott pressure, kg/cm²g _____Chamber B bott pressure, kg/cm²g _____RV dome pressure, kg/cm²g _____

9. Hydraulic Pump or Hydraulic Cylinder Failure

If a hydraulic pump fails, a valve malfunction alarm will be seen when the valve is supposed to step. The valve advance light will be one bed ahead of the valve position light and the manual valve advance button will not advance the valve. The spare pump should be started immediately. If a cylinder fails and cannot be immediately repaired, the Molex unit must be shut down.

10. Absorbent Sampling

UOP occasionally requests that a sample of the adsorbent be taken and sent to UOP for analysis. One bed in the adsorbent chambers (usually Bed 24) will contain connections for adsorbent sampling. A special apparatus and procedure are required to ensure that a representative sample is taken and that it does not become contaminated. The apparatus and procedure are described on Figure X-5. After the adsorbent sample is taken it may be transferred into a specialized container for easier transport. UOP will provide detailed instructions for adsorbent sampling if there is a need for sampling.

11. General Operations

When changes in operating conditions such as flow rate, temperature, pressure, instrument control settings, etc., are necessary, such changes should be made slowly and in small increments. Allow sufficient time between each change for the operation to adjust itself to new conditions, for instruments and control valves to adjust themselves to new positions, and for the process to line out once more. If possible, change only one variable at a time and observe the effect of this one change before making another.

The majority of variables in this section are under automatic control. The operator must make sure, however, that automatic controllers are functioning properly and must be prepared to switch from automatic to manual control or to use the control valve bypass valves if the automatic control system fails.

It is the operator's responsibility to make sure that all moving equipment is kept lubricated with the proper type of lubricant. Oil levels and lubricators and operation of pressure type lubricators should be checked periodically. The manufacturer's instruction manuals should be consulted to determine all points requiring lubricants.

Moving equipment such as pumps and drivers and control valves should be checked periodically and any abnormality such as high temperatures, vibrations, leaking shaft seals, unusual noises, etc., reported immediately to the proper authorities. In case of necessity or emergency, the equipment should be shut down, bypassed, switched to spare equipment, or whatever steps are required.

The process area should be kept neat and orderly. All tools and equipment should be kept in its proper place and trash should be disposed of promptly. Spills should be cleaned up as quickly as possible.

All fire fighting and safety equipment should be kept in its proper place and checked periodically to assure its readiness for immediate use.

Figure X-1a
Molex Troubleshooting Guide

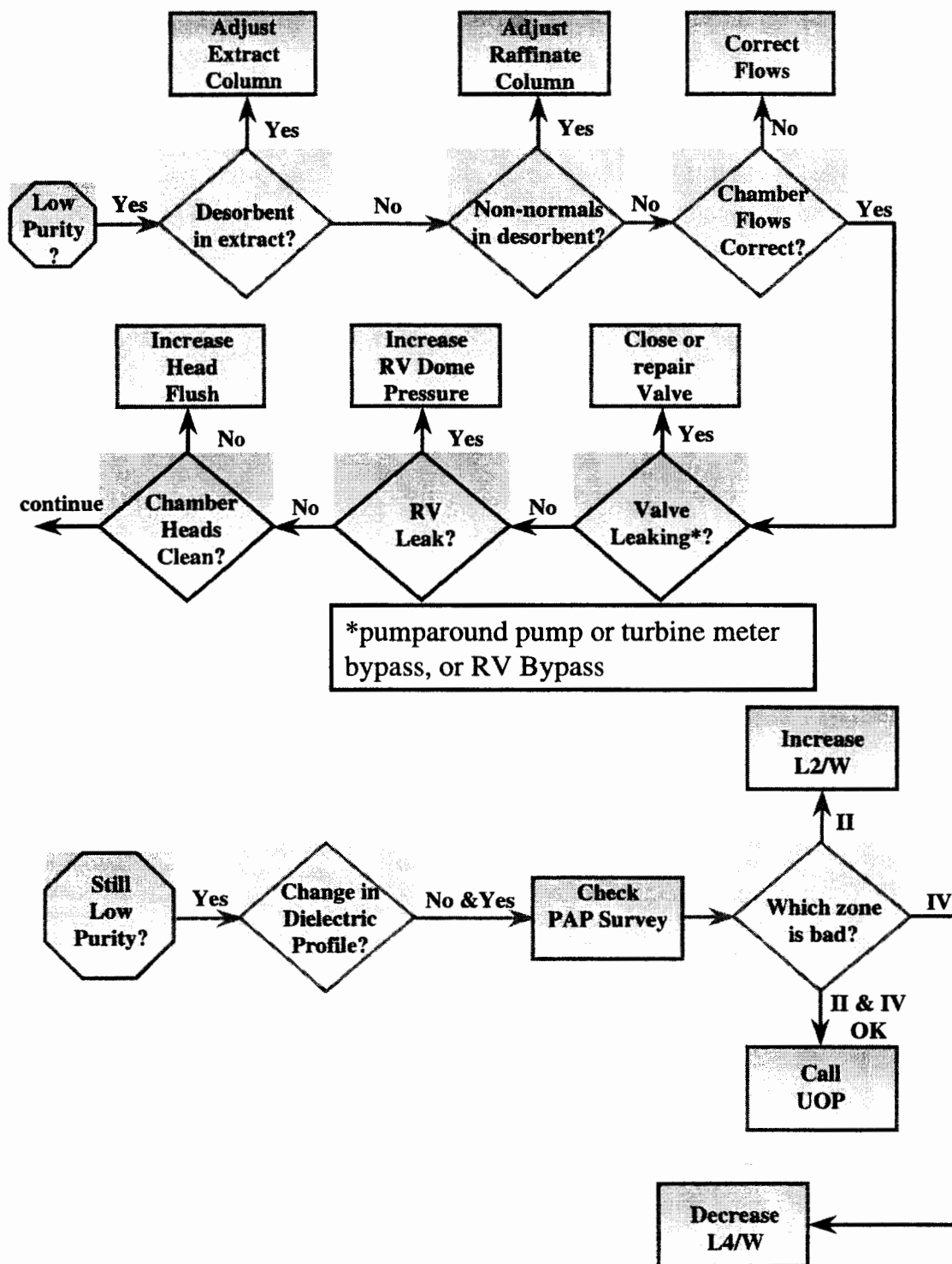


Figure X-1b
Molex Troubleshooting Guide

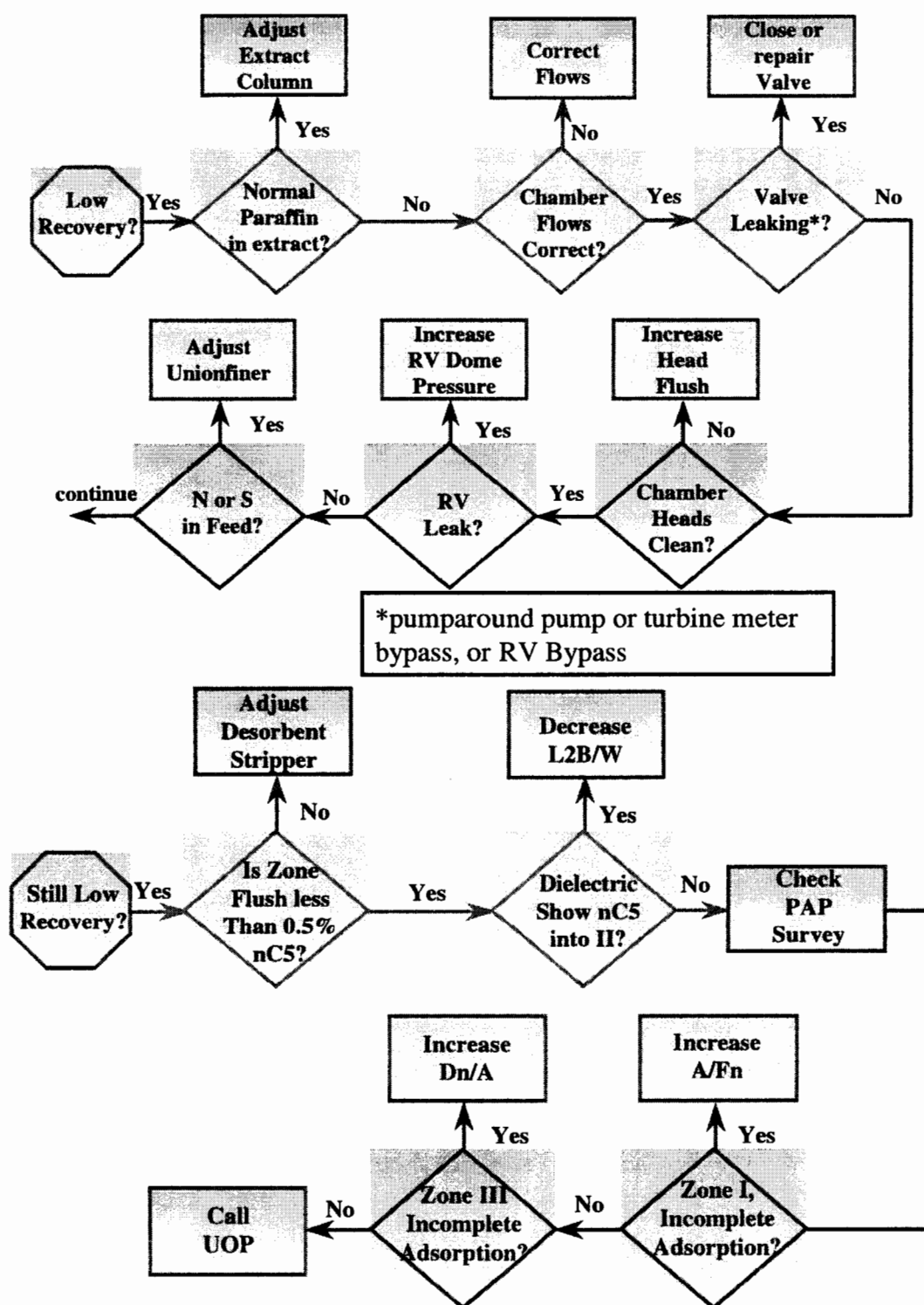
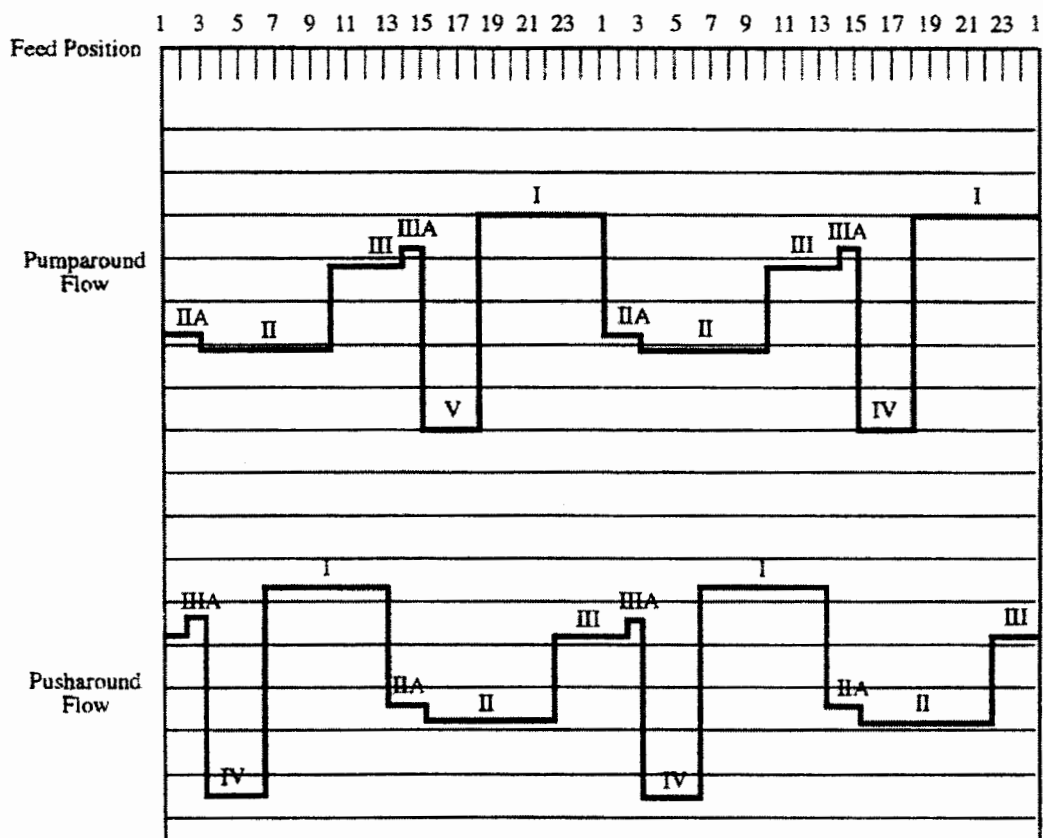


Figure X-2
Pumparound and Pusharound Flows
With Respect to Feed Position



PRX-R01-06

Figure X-3
Extra Zones

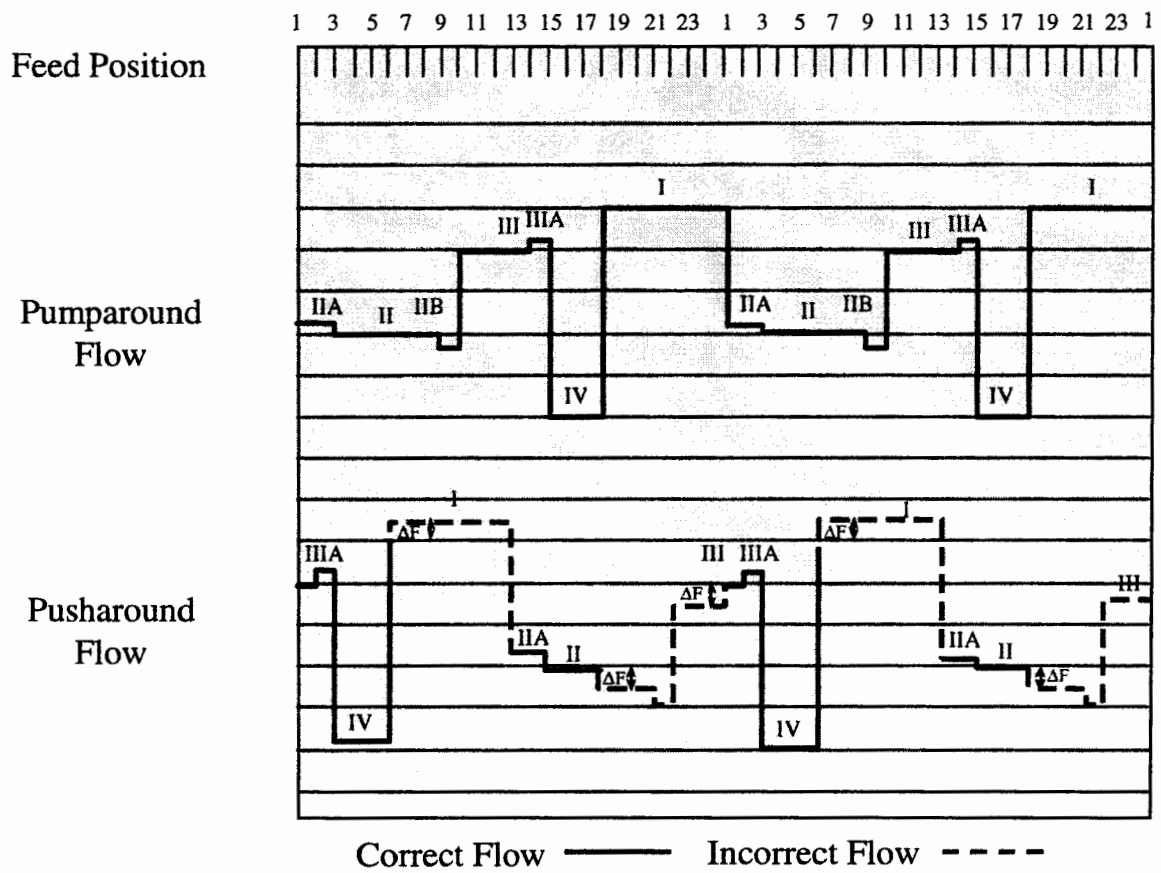


Figure X-4
Pumparound Survey

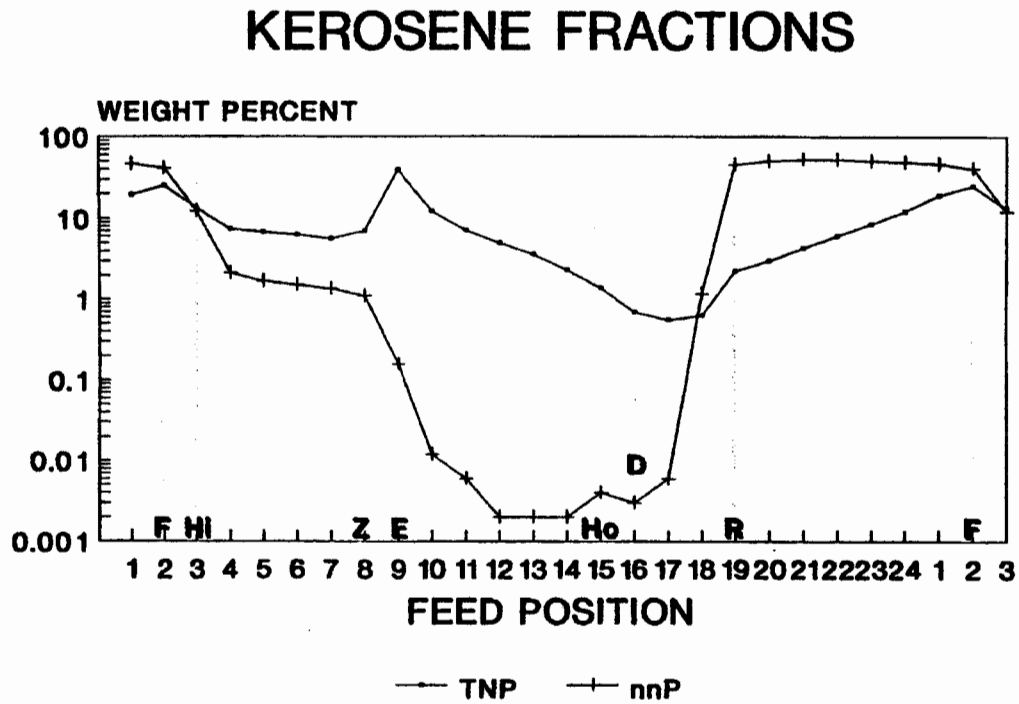
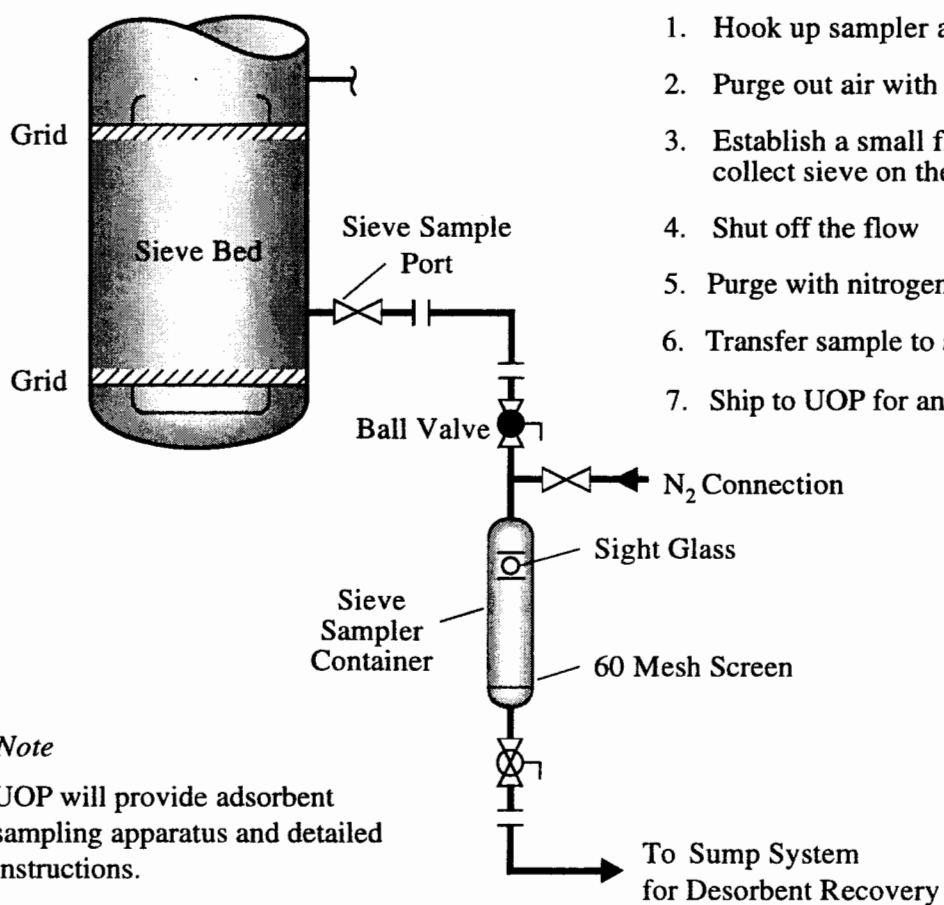


Figure X-5
Adsorbent Sampling Apparatus









XI. NORMAL SHUTDOWN

This section contains 2 distinct procedures. The first is for placing the Molex unit on circulation. This is used when the unit is upset and time is required to line out the unit. The Hydrobon unit product is sent to storage and the Pacol unit is fed from storage while the Molex unit is on circulation.

The second procedure is for a complete shutdown of the unit. The procedure contains details of a chamber cooldown and removal of hydrocarbons from the adsorbent chambers. The duration and reasons for the shutdowns will determine if these steps are necessary.

1. Placing the Molex Unit on Circulation

- a. Line up the extract and raffinate column bottoms back to the Molex feed surge drum. The hydrobon stripper bottoms should be sent to storage. The Pacol unit feed must come from storage.
- b. Recycle desorbent flow continues via the normal circuit.
- c. It is important to monitor and control the feed temperature when the unit is operating on circulation. The chamber temperature is 177°C (or 350°F). The temperatures of the extract and raffinate streams returning to the feed surge drum must be controlled so that the feed temperature to the rotary valve is kept at 177°C (or 350°F).

Normally the extract stream going to the Pacol unit is at 260°C (500°F). If the Pacol unit is not operating the extract is sent through an air cooler to storage. If necessary, this cooler can be used to cool the extract before it is sent back to the feed surge drum. The correct procedure would be to have the cold extract on flow control while the hot portion remained on level control. Since this stream is small relative to the raffinate flow rate, it is not required to cool the extract unless the desorbent heater is limited.

The connection for sending the raffinate back to the feed surge drum is downstream of the raffinate column bottoms cooler and upstream of the normal level control valve. The design temperature at this point is about 54°C (129°F). The raffinate temperature must be increased when the Molex is on circulation. This is done by using the bypass around the raffinate/desorbent exchanger and the raffinate column bottoms cooler. Use the upstream block valve on this bypass to control the quantity of raffinate going through the exchangers. As a rough approximation, 50% of the raffinate must go through the exchangers. The raffinate must be switched to the new level control valve on the short circulation line.

The lower flow of raffinate through the desorbent/raffinate exchanger means that there will be less heat transfer. The desorbent heater is used to provide the extra heat that will be required in this situation.

The raffinate and the extract temperatures can be controlled such that the combined temperature at the feed surge drum is about 177°C (or 350°C).

2. Complete Unit Shutdown

a. Shutdown of the Adsorption Section

- (1) Line up the extract and raffinate column bottoms back to the Molex feed surge drum.
- (2) Put the pumparound pump controller on manual. Use the manual setpoint stations to control the pumparound flow. Adjust all zone flow setpoints equal to the Zone II flow.
- (3) Unseat the rotary valve by opening the dome equalization line.
- (4) Decrease the feedrate stepwise to zero, block in.

- (5) Decrease the desorbent, flush and extract flows. Be certain to keep the chamber pressure at 24.6 kg/cm² (350 psig). Gradually decrease the extract flow to zero and block it in. Place the extract column on total reflux, blocking in the overhead and bottom lines; adjust the reboiler heat accordingly.
- (6) Feed is displaced from the adsorbent beds by the desorbent circulation after the feed is cut out. All the feed can be considered displaced when the raffinate net bottoms production falls essentially zero. Bring in desorbent as needed to the surge drum.
- (7) If the unit will be down more than a few hours the chambers should be cooled. The chambers can be cooled by decreasing the desorbent heater outlet temperature. This temperature must be decreased slowly to prevent excessive thermal stress in the adsorbent chambers and rotary valve. The difference between the desorbent temperature and the average chamber temperature should never exceed 28°C.
- (8) Shut down the pumparound pump.
- (9) Block in the extract and raffinate column sidecut flows. Shut down and block in the flush flow. The raffinate overhead material will be directed solely to the desorbent surge drum now.
- (10) Open the raffinate line at the rotary valve bypass. Then open the desorbent line at the rotary valve bypass.
- (11) Shut down the rotary valve system. The following steps should be done as rapidly as practical:
 - (a) Block flows to the rotary valve except that of the raffinate. (The only remaining flow to the rotary valve at this point should be the desorbent.)

- (b) Stop rotary valve at midcount to prevent an extend overload at the time. The best way to stop the rotary valve is to switch the field mounted local-remote switch to the local position. This keeps the UOP Adsorbent Chamber Control System working but disconnects the logic signals to 4-way solenoid valve. **NOTE:** When switching back the local-remote switch, the rotary valve will step immediately, so care must be taken.
 - (c) Shut down the hydraulic pump and block it in.
 - (d) Block in the raffinate at the rotary valve. desorbent and raffinate should still be flowing through the rotary valve bypass, however.
 - (e) Blanket both chambers with N₂ from taps in the pumparound lines and maintain a pressure of 7.0 kg/cm² (100 psig) to prevent vaporization of chamber liquids. Vaporization in the chambers could cause adsorbent damage.
- (12) Stop raffinate flow.
- (a) Shut down desorbent feed pump and block it in.
 - (b) Block in the rotary valve bypass manifold.
 - (c) Place raffinate column on total reflux.

b. Shut Down the Desorbent Stripper Column

This column is effectively shutdown when the feed to the column is terminated. The extract column sidecut pump is shut down. The raffinate column sidecut pump should not be stopped until the raffinate column is shutdown.

Stop heat to the column bypassing the reboiler. As the column cools off, the liquid in the bottoms is pumped to the desorbent surge drum and on to desorbent storage.

c. Shut Down the Extract Column

It is assumed that the column is already on total reflux.

- (1) Boil all the desorbent overhead and pump from the overhead receiver to the desorbent surge drum and on to the desorbent tankage.
- (2) Shut down the extract column reboiler and when the column cools down, stop reflux.
- (3) Put sweet fuel gas or nitrogen in the column to maintain a positive pressure on the column as it cools down.
- (4) As the column cools, pump liquid from the bottom to off- specification storage. Shut down the bottoms pump and drain the remaining liquid to slop.

d. Free the Adsorption Chamber of Hydrocarbon

It is assumed that the adsorption section has been shut down and freed of feed as directed in Part 2. The reasons for the shutdown will determine if this step is necessary. If not, the chambers should be isolated and the pressure should be maintained at 7 kg/cm² (100 psig) with nitrogen. The details of the hydrocarbon removal procedure are as follows.

- (1) Introduce nitrogen to the top of one chamber and displace desorbent to the raffinate column from the bottom of the chamber through the cross-over to the raffinate column feed line and into the raffinate column. Maintain the pressure in the adsorption chambers above 7.0 kg/cm² (100 psig) until all the desorbent is displaced. Boil the desorbent overhead in the raffinate column and send it to the desorbent surge drum and out to storage.

- (2) Depressure the column slowly to flare.
- (3) Displace the desorbent from the second chamber in the same manner.
- (4) If the adsorbent is to be discarded, the chambers may be steamed into the raffinate column.
- (5) If the adsorbent is to be unloaded for repair of the internals, the chambers should be evacuated and purged with nitrogen. Contact the UOP District Man for specific details.
- (6) Once the chambers have been freed of liquid, the raffinate column can be shut down.

e. Shut Down the Raffinate Column

- (1) Adjust heat in the raffinate column to maintain it on total reflux. Reduce the level in the overhead receiver by sending desorbent to the surge drum and out to tankage.
- (2) Shut down the raffinate column reboiler. When the top of the column begins to cool, shut down the reflux pump and the sidecut pumparound pump. Pump the remaining liquid in the overhead receiver out to the desorbent tankage. Put nitrogen or sweet fuel gas in the tower as it cools to maintain a positive pressure.
- (3) When the tower has cooled, pump bottoms to off-specification storage. Drain the remaining liquid to slop.



XII. EMERGENCY PROCEDURES

This section is meant to acquaint you with safe procedures to be used when problems occur in the Molex adsorption section, or in other areas that will ultimately affect the adsorption section. These procedures will normally get the shutdown as far as circulation, and then depending on the exact nature of the problem, a complete shutdown as outlined in A. Normal Shutdown may be warranted.

In case of an emergency, there are three very important things to protect in the adsorption section. The first to protect is the rotary valve rotor plate Teflon sheet, and this is done by unseating the rotary valve (open dome pressure equalization line). The second is the adsorbent, by keeping the chamber liquid full by maintaining 24.6 kg/cm² (350 psig) chamber bottoms pressure. A depressuring of the chambers at a high temperature can cause vaporization inside the adsorbent pore structure rupturing the adsorbent. The third is to prevent off-spec feed from entering the chamber. If there is a question of the feed quality the adsorption section should be put on circulation and hydrobon stripper bottoms sent to storage until laboratory analysis confirms that the Molex feed is on spec.

1. Loss of Feed

a. Loss of Molex Feed Pumps

- (1) Restart the feed pump, if possible, and bring charge rate back to normal quickly. Make certain the discharge of feed pump is sufficiently pinched closed when restarting to avoid a pressure surge on the system. Put the feed control valve in manual closed before starting the pump.
- (2) If you are unable to restart the charge pump do the following:
 - (a) Unseat the rotary valve by opening the equalizing valve.

- (b) Open all flows into and out of the rotary valve bypass manifold except the feed. Block in the feed line to the rotary valve. Reduce all flows to 50%.
- (3) Block in all flows, except the raffinate to and from the rotary valve to maintain pressure control on the adsorbent chambers. Switch to analog control of the pumparound flow.
- (4) The raffinate and extract column bottoms control valves should be closed to maintain bottoms inventory. Adjust the reboilers accordingly. Desorbent circulation can continue via the normal route.
- (5) Stop adsorbent chamber circulation pump. Isolate the adsorbent section as necessary.
- (6) Continue desorbent flow to the rotary valve bypass manifold, splitting the flow as required to the extract and raffinate columns.

b. Loss of the Hydrobon Section

- (1) Send the raffinate and the extract back to the feed surge drum to establish circulation as described in Section XI-1.
- (2) Maintain Molex unit in this circulation mode until feed from the hydrobon section is available.

2. Loss of Desorbent Flow

- a. Restart the desorbent pump as quickly as possible, making certain the pump discharge is sufficiently pinched closed to avoid a pressure surge when restarting. Put the desorbent flow control valve in manual closed before starting the pump.

b. If it is not possible to establish desorbent flow quickly, do the following:

- (1) Unseat the rotary valve by opening the equalizing line. Block in the desorbent flow to the rotary valve.
- (2) Open all flows into and out of the rotary valve bypass except the desorbent.
- (3) Block in all flows into and out of the rotary valve except the raffinate line to maintain chamber pressure control.
- (4) Establish circulation in the unit by rerouting the extract column net bottoms and the raffinate column net bottoms back to the Molex feed surge drum. Send hydrobon stripper bottoms to storage.
- (5) Continue Molex feed flow to the rotary valve bypass, splitting the flows as required to the extract and raffinate columns.
- (6) Desorbent circulation will stop. The raffinate and extract column net overhead to the desorbent surge drum should be blocked in as necessary to maintain overhead receiver levels. Place the columns on total reflux.
- (7) Continue flush circulation.
- (8) The extent of further shutdown procedures will be determined by the expected time necessary to re-start the desorbent circulation pump.

3. Loss of Flush Pumps

a. Restart flush flow as quickly as possible.

- b. If it is not possible to start the flush flow quickly:
- (1) Unseat the rotary valve by opening the dome equalization line.
 - (2) Open all flows into and out of the rotary valve bypass except the flush. Block in the flush to the heads and dome.
 - (3) Block in all flows into and out of the rotary valve except the raffinate line (to maintain pressure control).
 - (4) Establish circulation, as indicated in Section XI-1 that includes switching the Hydrobon stripper bottoms to storage. Continue desorbent circulation, but send all the extract and raffinate overheads to the desorbent surge drum.
 - (5) The expected time for restarting the flush pump will determine how much further the unit should be shut down.

4. Loss of Chamber Circulation Pumps

- a. Restart circulation flow as quickly as possible.
- b. If you cannot establish circulation flow:
- (1) Unseat the rotary valve by opening the equalizing valve.
 - (2) Open all flows into and out of the rotary valve bypass.
 - (3) Block in all flows, except the raffinate, to and from the rotary valve.
 - (4) Establish circulation as indicated in Section XI-1.

5. Loss of Desorbent Stripper Column

Follow the same instructions as in Part 3, Loss of Flush Pumps, except shut down pump between steps 3 and 4.

6. Loss of the Extract Column

- a. Cut out feed to the extract column.
- b. Cut back on feed and desorbent to the adsorbent chambers to maintain the same raffinate rate as before stopping the extract flow.
- c. If the extract column is to be down for a long period of time, the adsorbent section should be shut down according to the normal shutdown procedure.

7. Loss of the Raffinate Column

- a. Cut the feed, flush and desorbent flows to the rotary valve.
- b. Cut extract flow from the rotary valve and place the extract column on total reflux.
- c. The raffinate valve should close but care should be taken to maintain pressure in the adsorbent chambers. It may require the addition of nitrogen to the top of both chambers to prevent vaporization.
- d. If the raffinate column is to be down for a long period of time, the adsorption section should be shut down according to the normal shutdown procedure.

8. Loss of the Rotary Valve Hydraulic System

- a. Unseat the rotary valve by opening the equalizing valve.
- b. Open all flows into and out of the rotary valve bypass.

- c. Block in all valves to the rotary valve except the raffinate to maintain chamber pressure.
- d. Establish circulation.

9. Rotary Valve Stepping Error

If the rotary valve fails to step, an alarm will sound. The operator must take quick action to resolve the problem or the product will go off spec. The following procedure should be used to isolate the problem.

- a. Try to step the valve with the rotary valve advance button on the control panel. If it advances, the problem is in the stepping logic.
- b. If the valve does not step, investigate the hydraulic system. If the hydraulic pump is not running, try to start the spare pump. If the pumps are running, attempt to step the valve manually. Observe the pressure gauges. If the gauges read full pressure and the valve steps, the problem is in the automatic valves or the signal to the automatic valves. If the rotary valve fails to step and hydraulic cylinder moves, the problem is in the drive mechanism. If the rotary valve fails to step, the hydraulic system does not move and the hydraulic oil pressure is normal, then the rotary valve is stuck. Unseat the rotor plate and shut down the unit to inspect.

On rare occasions, the rotary valve has been known to double step. That is, take two steps instead of one. This may be due to a stray electrical signal or due to a malfunction in the stepping logic. If this occurs, the unit should be watched closely. The flows to the column will be slightly upset, and the product purity will decrease slightly but it should not be necessary to discontinue sending product to storage.

10. Loss of Chamber Pressure

- a. As mentioned previously, loss of the chamber pressure can result in damage to the adsorbent, rotary valve Teflon sheet and rotary valve expansion joint bellows. Two safety systems exist which should help prevent damage if a loss in chamber pressure should occur. The instrumentation systems to prevent adsorbent and/or equipment damage are described in sections IV.b.1-2 of this manual.
- b. Manual action should also be taken if the chambers begins to depressure. The actual situation will determine which steps should be taken.
 - (1) Block in the flows out of the chambers: Flush out, extract and raffinate.
 - (2) Unseat the rotary valve by opening the equalizing valve.
 - (3) Block in the flows to the chambers if they have stopped: feed, desorbent, zone flush and line flush.
 - (4) Stop chamber circulation.
- c. Repressure the chambers slowly. Be certain that the chamber PRC and raffinate control valve are back in service.

11. Electric Power Failure

Since the unit is primarily electric driven, a power failure means a complete shutdown. The electric motors may have a time delay in their starters that will restart the motors after a very short duration power failure. A power failure of more than a few seconds will stop the unit.

In any shutdown, either normal or emergency, special care should be given to safely shutting down the fired heaters. In case of an instrument air failure, the fuel gas and fuel oil control valves should close. The operator should also close a block valve in the main fuel gas header as soon as possible, and put snuffing steam into the firebox.

A power failure will sharply reduce the cooling capacity of the air coolers in the unit due to the loss of the fans. Watch for excessive pressures in the extract, raffinate, and desorbent stripper towers. Block in the extract and raffinate bottom products to storage line. Be sure that all condenser fin fans are restarted when power is available.

The feed pump may be in another process area and might not be affected by a power failure in the Molex area. If it is not, it should be shut down and blocked in.

When the extract, raffinate, and desorbent stripper towers start to cool, connect nitrogen or contaminate-free fuel gas to the overhead receivers and maintain a slight positive pressure on the towers.

After the power to the unit has been resumed, the unit may be restarted in the normal fashion. Of course, any other utilities that may have failed as a result of a power failure should be restored before attempting to restart the unit.

12. Instrument Power and/or Instrument Air Failure

Either instrument power or instrument air failure will force the unit to shut down if the failure is of more than a few seconds duration.

Be sure to block in the hot oil heater fuel gas line. Continue to pump through the hot oil heater for a short time, if possible. When the heater has cooled, shut down and block in the hot oil circulating pumps.

All control valves will go to their failure position if instrument air is lost. The control valves that fail open are listed below. All other control valves fail closed.

Control valves which fail open:

- a. Raffinate Column Reflux
- b. Raffinate Column Sidecut Pumparound
- c. Raffinate Column Pressure Control
- d. Extract Column Bottoms Pump Spillback
- e. Extract Column Reflux
- f. Extract Column Pressure Control
- g. Normal Paraffin Transfer Pump Spillback

Block in the product to storage lines and shut down the extract raffinate and desorbent splitter bottoms pumps. Continue refluxing the towers until they start to cool and then shut down the reflux pumps. Connect nitrogen or contaminate free fuel gas to the towers to maintain a positive pressure on them.

Shut down and block in the feed pump and the desorbent pump. Block in the zone flush, line flush (in and out) and extract lines at the rotary valve. The raffinate control valve will close on air failure. Block in the control valve and use the bypass if necessary to prevent a pressure buildup in adsorbent chamber.

The chamber circulation control valve will also close on air failure. Shut down the chamber circulation pump. Note the position of the rotary valve. If it has moved for any reason, it should be returned to its original position prior to restarting the unit.

13. Water Failure

The process coolers are mainly air cooled and are not affected by a water failure. There are several items of equipment, such as pump seals and bearing boxes, which have water cooling, and if overheated, may cause damage. Such equipment should be shut down where overheating is suspected.

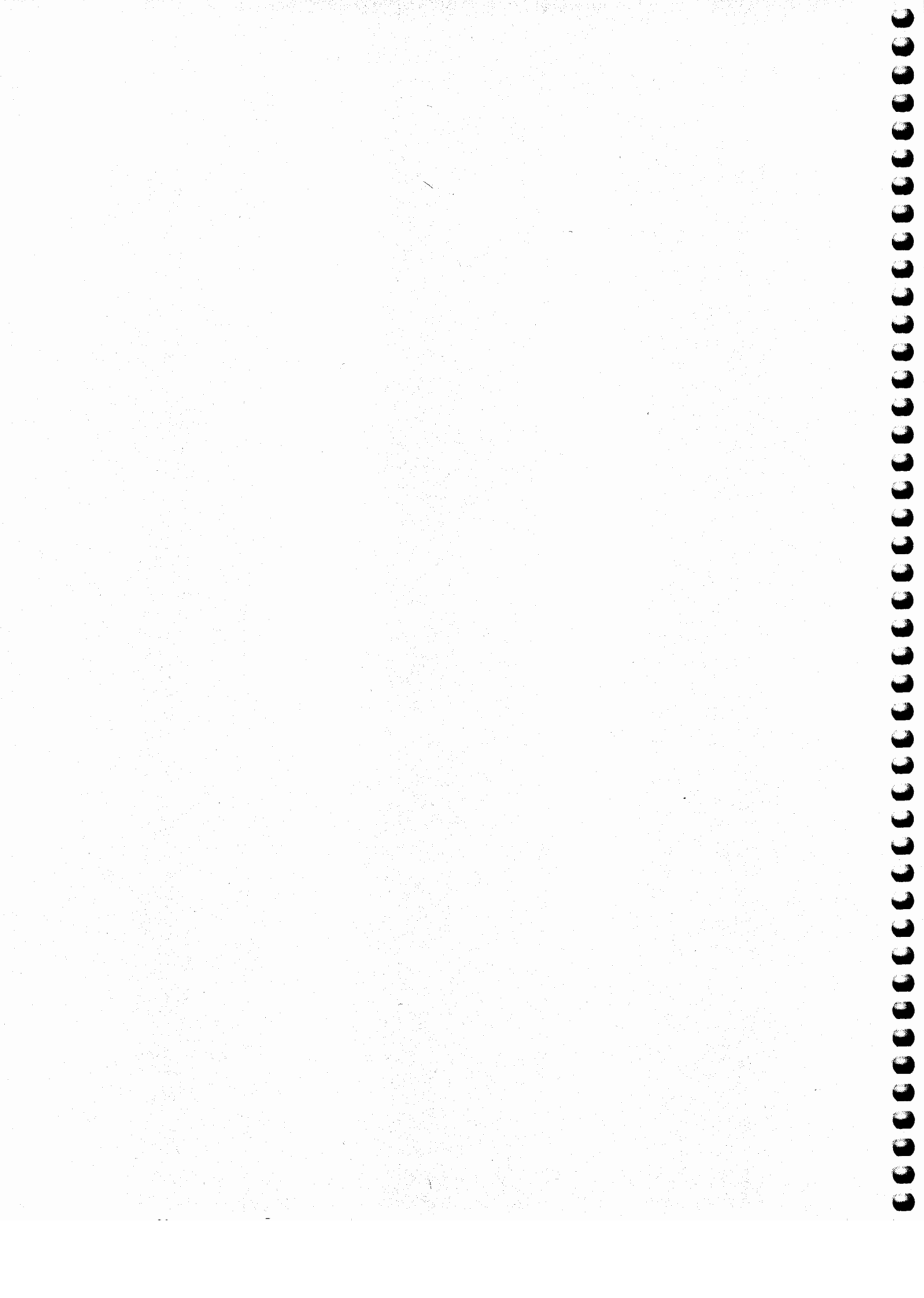
14. Steam Failure

Because steam is used solely for the ejector, a steam failure will not shut down the Molex unit. A steam failure may, however, affect other plants or utilities and force a shutdown of the Molex unit.

15. Fire, Explosion, or Major Leak

The first concern of the operators should be for their personal safety and the safety of other personnel in the area and adjacent areas. When this is accomplished, they should direct their attention to minimizing the damage to the equipment. Shut off the main fuel gas line and shut down and block in all pumps, if possible. Depressure the unit to the flare and isolate equipment as circumstances permit or require.





XIII. SPECIAL PROCEDURES

A. ADSORBENT CHAMBER LOADING

This section serves as a guideline for adsorbent loading. The UOP Chief Advisor will work with the Licensee or Contractor to develop a more detailed procedure. The detailed procedure will take into account the local conditions and the specifics of adsorbent chambers and internals.

A good adsorbent loading operation is a critical step in ensuring the Molex Unit will perform as guaranteed. Loading efficiency is defined as the actual loaded density divided by the maximum expected density measured by UOP. UOP targets a loading efficiency of 97% and has developed the loading procedure to ensure the loaded bed has a uniform density.

Adsorbent chamber internals are known as segmental grids. The connections between the grid internal distributors are achieved by distributor piping. Figures XIII-1 and XIII-2. With the segmental grids, beams are used to support the grid sections. Figures XIII-3 and XIII-4.

1. Planning and Preparation Work

a. Storing of Adsorbent Chamber Internals and Adsorbents

Adsorbent is shipped to the job site in 210 liter (55 gallon) drums or other sealed containers. Since the adsorbent is extremely hygroscopic and can be permanently damaged by free water, it is necessary to keep the adsorbent protected. In particular, adsorbent in drums or other containers must not be allowed to stand in water or be exposed to rain or snow. The adsorbent must be stored in a clean, dry, enclosed warehouse. Drums should be stacked on pallets no more than 3 drums high. If alternate packaging is used, the packages must not be stacked in such a way as they may become damaged. The adsorbent should be kept sealed until it is used for loading into the adsorbent chambers. Careful handling will prevent the adsorbent from being damaged or the packaging from being accidentally opened.

For the adsorbent chamber loading, provide temporary weatherproof shelter close to the adsorbent chambers for protection of the adsorbent and chamber internals.

Moisture ingress into the crates will result in corrosion of the carbon steel adsorbent chamber internals. The corrosion will cause critical profile wire dimensions to no longer meet UOP's strict tolerances. Manual or chemical cleaning will then be required to remove the corrosion and restore the material to UOP's strict tolerances, resulting in additional costs and project delays.

The following notes apply to the adsorbent chamber internals:

1. The crates must not be opened until just before installation on site. If customs inspection is required, it is best that the inspection be performed in the field just before installation. Customs inspection has caused some difficulties. It is important that if any customs inspection is performed that the packaging of the UOP adsorbent and grids be properly protected and resealed.
2. Storage of the crates must be indoors. Storage outdoors under a shed, tarp or other non-six-sided enclosure is unacceptable.
3. Storage of crates must be elevated above the floor. This to prevent damage from a leaking roof or floor spill of liquids.
4. Storage of the crates must be in a temperature-controlled environment with the minimum temperature being 32°F and the maximum temperature being 90°F.
5. Storage of the crates must be in a humidity-controlled environment with the maximum relative humidity being 75%.
6. Inspection of the crates must be made every three (3) months. NOTE: The inspection must be an EXTERNAL - VISUAL inspection, looking for signs of

crate damage by impact or moisture ingress. The inner seal of the crates must NOT be opened during the three (3) month routine inspections.

7. In the instance where the routine external visual inspection of crates indicates damage, the client is advised to contact UOP - KME immediately for advice and corrective action. UOP - KME can be reached at fax 1-847-391-2592 or e-mail at KME @ UOP.com.

b. Scheduling

Good organization and preparation are key to a smooth, fast adsorbent loading. All concerned groups should meet to define and assign areas of responsibility well in advance of the loading operations. It is recommended that a UOP Technical Service Advisor should be on site prior to this operation to advise the customer on specialized techniques required for inspection, grid installation, and loading. UOP also offers specialized training for adsorbent loading which is highly recommended a few months before adsorbent loading begins.

The method used successfully on most Molex loadings uses two specialized crews per shift. One crew consists of riggers who also double as the adsorbent loading crew. The second crew does internal pipe fitting, welding, ceramic rope packing and final cleanup. These crews alternate chambers to maximize the use of the crane(s), or overhead hoist and trolley, which can be a limiting factor.

One crew loads adsorbent and then installs the next set of internals in Adsorbent Chamber No. 1. At the same time, in Adsorbent Chamber No. 2, the other crew is completing the internal piping and clean up. The timing can be coordinated so that each crew finishes its task at about the same time. The time required for each bed is very dependent on the coordination of personnel, materials, and equipment – particularly the crane. Typically, two to three beds can be loaded and tested in a 24-hour shift. An additional three to four days must be allowed for each bottom and top grid assembly since considerable fit-up and welding is required. A more detailed breakdown of the timing is listed in Table XIII-1.

Alternatively, a single crew can work in each chamber covering both the functions of loading adsorbent and placing the adsorbent chamber internals. This is typically less efficient. Making the welds on the bedlines is one of the most skilled activities in the adsorbent chambers, and these workers can alternate between chambers to fulfill that function.

A thorough understanding of the work required and of the available personnel enables the best guidance that can be given in determining how to staff the loading.

The customer must allow sufficient time so that the loading can be finished before the unit's mechanical completion date. No maximum has been established for the length of time the chambers may be loaded before initial operation.

After completion of the loading it is imperative to positively isolate the chamber and maintain a positive pressure with either nitrogen or dry instrument air.

TABLE XIII-1
TIME REQUIREMENTS FOR A TYPICAL LOADING CYCLE

Typical Grid Installation Schedule

Work Description	Time (hours)
Grid Placement and BIA Installation	3.0
Grid Finalization	
Grid Orientation and Tie Down	3.0
Internal Piping, Welding and Ceramic Fiber Rope Packing	3.0
Cleanup	<u>0.5</u>
TOTAL	9.5

Typical Adsorbent Loading Schedule

Work Description	Time (hours)
Final Inspection and Preparation	1.0
Sieve Loading	8.0
Pressure Up for Integrity Test	<u>1.0</u>
TOTAL	10.0

Time to pressure up chamber to 0.35 kg/cm²g for integrity test depends on dry air supply that is available.

This is a general schedule and depending on each particular site, chamber size, and equipment available, the distribution of time will vary. Top and bottom grid installation usually takes 2-5 days.

c. Manpower

The customer must decide on the exact number of people required for the specific situation. Table XIII-2 contains an estimate of the personnel necessary for loading. A guideline to the skills required for loading is the following:

- (1) Rigging Personnel for handling and installation of adsorbent chamber internals inside and outside of the adsorbent chambers, loading adsorbent material, and handling the adsorbent drums.
- (2) Loading Crew with off-site drum handlers and sock operators.
- (3) Installation Crew including personnel for bolt up, rigging, pipe fitters, and welders.
- (4) Inspectors to inspect the grids before, during, and after installation. Also, inspect adsorbent loading.
- (5) Operators for specialized equipment used for the loading, e.g. truck drivers, crane and forklift operators.

If the correct people are available, the rigging and loading crew can be the same individuals, and the installation and inspection can be handled by the other crew. The UOP Technical Advisor(s) assist in all areas to coordinate and advise in the operations. Inspection of grids is extremely important, and the UOP Technical Advisor(s) will be closely involved in this activity. A detailed breakdown of manpower requirements is listed in Table XIII-2. The manpower listed is exclusive of UOP Advisors present.

As expected, the time required for loading is dependent on the number of people available to accomplish the work and their familiarity with their work scope. For the loading to progress on a reasonable schedule it is important for all workers to quickly learn the procedure and complete their tasks on schedule and be prepared. It is expected that after 3-4 beds have been loaded the pace of work will meet the

time requirements provided in Table XIII-1. For small units with adsorbent chambers less than 3 m (9 ft) in diameter, the number of people that can effectively work inside the adsorbent chamber is limited. As the adsorbent chamber diameter approaches 5 m (15 ft), the most efficient number of workers increases. The standard specification for adsorbent handling indicates that the number of loading socks suggested is dependent on the diameter of the vessel.

**TABLE XIII-2
MANPOWER REQUIREMENTS PER SHIFT**

Work Description No. of People

1.	Sieve Loading	
a.	Sieve loaders - inside of chamber for sock handling and adsorbent leveling per sock	3
b.	Guiding loading hopper and crane signaling also controlling sieve flow to hoppers - on top of chamber	2
c.	Movement of drums on loading platform and filling loading hoppers	3
d.	Crane operator	1
e.	Drum handling and transport from storage area, 2 drum handlers and 1 fork lift operator	3
f.	Supervisor	<u>1</u>
	Total	13
2.	Grid Installation	
a.	Grid placement, bolt up, and ceramic fiber rope packing 1 experienced rigger, 4 general workers	5
b.	Welding bed lines, intermediate grids (welder)	1
c.	Grid handling, crane signaling - top of chambers	2
d.	Crane operator	1
e.	Grid handling - at crane hook-up point	2
f.	Grid transport from storage area, 1 truck driver, 1 small crane operator, 2 riggers	<u>4</u>
	Total	15
3.	Supervision: 1 supervisor inside chamber, 1 supervisor at grade	2
	Total Manpower Requirements	30

The above manpower requirements are representative, assuming that the sieve loading and grid installation operations are completely separate. Actually, these operations are seldom, if ever separate. Therefore, through proper personnel scheduling, the manpower requirement can be reduced to approximately 23 people (i.e. the people at the top of the chamber can be switched from sieve to grid loading, drum handlers switched to grid handlers, etc.). These requirements are for the normal loading sequence. It is obvious that the top and bottom grid installations will require a shifting of personnel. The top and bottom grid installations require 2 welders per chamber minimum. Up to 4 welders may be useful

as the chamber diameter approaches 5 m (15 ft) where the larger vessel provides sufficient space for more welders.

d. Materials and Equipment

In terms of what materials and equipment are required for adsorbent loading, the loading procedure is broken down into four major categories.

- i. Preparation, Grid Loading, and Inspection
- ii. Grid Installation and Repair
- iii. Adsorbent Loading
- iv. Grid Screen Integrity Test

NOTE: Some items listed are supplied with adsorbent chamber internals. The information provided with the adsorbent chamber internals should supply the details.

- i. Preparation, Grid Loading, and Inspection
 - (1) A protective wood, rubber, or plastic ring to cover the adsorbent chamber top manway flange.
 - (2) Adequate lighting inside each chamber. An average chamber requires a minimum of five low voltage flood lamps, and three flashlights.
 - (3) A sectional or extendible ladder to permit safe internal access from the top to the bottom of each chamber. Rope or cable ladders are not considered acceptable. Fall protection should also be provided. Platforms inside the chamber should be constructed of grating to prevent adsorbent accumulation thereby reducing the slipping hazard.
 - (4) Radios or telephones for communication from ground level to the top of the chambers and communication inside the chambers.

- (5) Sawhorses, or scaffolding, to support all pieces of at least one complete grid level for visual inspection. The platform should be approximately 1 meter (3 feet) high. Sufficient space should be provided to allow access to all sides of each grid section.
- (6) Four to six air-powered eductors to ventilate the adsorbent chambers. If possible, the eductors should be flanged for easy attachment to and removal from the adsorbent chambers. Air eductors are very noisy and may hinder communication inside the chamber during loading. If available, fan-type air movers with flexible ducts have also been found acceptable. Depending on local regulations it may not be permitted to exhaust the dust from the loading directly to the air outside of the adsorbent chamber. Some form of dust collection may be required; see the material safety data sheet for the molecular sieve.
- (7) A portable air compressor, or instrument air supply, to supply air free of oil and water for the integrity test of each bed and also to the air ejectors if they are used.
- (8) A lifting harness. The harness should be kept close at all times to permit rescuing an injured person from the chamber.
- (9) Adsorbent chamber internals.
- (10) Water proof canvas and plastic covers for the chamber top structure, grids, and adsorbent drums in case of rain.
- (11) Four magnifying glasses with 10 x magnification, 25 (1 inch) diameter, 1 kg (2 lb) of chalk or soap stone marking sticks, and six yellow or white carpenter crayons.

- (12) 6 mm (1/4 inch) plywood and cardboard covers for grid sections. Sufficient covers are required for four intermediate grid levels and two bottom grid levels.

NOTE: The covers must cover the entire grid screen area. The covers must be fabricated so that they may be removed after the grids and other internals are installed in the adsorbent chambers. The covers must also have some method to prevent them from falling and injuring personnel or damaging the grids in the event that they become detached from the grids during lifting and placement in the adsorbent chambers.

- (13) Rigging equipment for lifting grid sections and other internals including shackles (U-type fastener), chokers (Nylon slings and metal cables), and comealongs (portable hand operated hoist). See Figure XIII-5.
- (14) Machinist marking stamps – both numbers and letters.

ii. Grid Installation and Repair

- (1) Metal wedges of various sizes to move grids and get equal spacing around the outside perimeter.
- (2) Metal spacers to help maintain uniform grid spacing.
- (3) Six 20 liter (5 gallon) buckets.
- (4) Welding brackets (three per weld on the top and bottom grid piping (Figure XIII-6).
- (5) Both inert gas and electrical welding machines and supplies.

- (6) Four crowbars; 2 approximately 2 meter long (5-6 feet), 2 approximately 0.5 meter long (18-24 inches), four 1 kg (2 lb) hammers, and two copper, lead, or rubber mallets.
- (7) Two ceramic blankets large enough to cover a complete grid level to protect grids from weld spatter.
- (8) Typically braided ceramic rope is supplied. The braided rope may be unwound to allow it to be packed into the smaller areas. The ceramic rope is used to fill the space between the grids and the vessel wall. As the vessel may have variance in the roundness, the gaps between the grids and the vessel may vary. Typically 4 to 5 courses of ceramic rope are required for each grid level. Sufficient 19 mm rope should be provided for the complete installation, with typically 30-50% spare for contingency. The smaller diameter rope is required to pack the space between the vessel and the unloading nozzles and the thermowells. Alternatively ceramic rope of varying sizes may also be used, 19 mm (3/4") and 2 mm (1/16") diameter.
- (9) Four sets of ceramic rope packing tools (Figure XIII-7).
- (10) Box and open-end wrenches for grid piping flanges.
- (11) Strong plastic adhesive tape.
- (12) Hand brooms and a portable industrial vacuum cleaner.
- (13) Distributor piping gaskets, asbestos-free flat composite type suitable for the process fluid.
- (14) Thermowells with gaskets.
- (15) Adsorbent sampling valves as per UOP drawings.

- (16) Grid repair equipment:
- (a) Fine tip acetylene torch: Victor No. 0323-0059-000T4 (0.027 inch (0.686 mm) opening).
 - (b) Gas cylinders for silver brazing.
 - (c) Portable grinder.
 - (d) Wire brushes.
 - (e) Cold chisels.
 - (f) 15 kg. (35 lb.) of high temperature silver brazing alloy (44 to 57% Ag. See Table XIII-3 for additional detail on the silver solder composition.
 - (g) Flux for soldering with silver on carbon steel or stainless steel. (Older screens used stainless mesh, newer units utilize carbon steel screens. Confirm type required.)
 - (h) Electrodes (No. 309) for welding stainless steel to carbon steel. (Only required for units that utilize grids with stainless steel mesh screen.)
- (17) Rubber soled safety shoes for all personnel working inside the chambers. The rubber-soled shoes shall provide adequate traction for safe walking in the area and inside the adsorbent chambers. It is a good idea to check the soles of the shoes worn in the adsorbent chamber upon entry to make sure they have not picked up foreign debris that could damage the grids.
- (18) Various thickness, 0.5-6.0 mm (0.02-0.25 inch) of 50 mm x 50 mm (2 inch x 2 inch) carbon steel shims. Shims are used to level and stabilize grid section placement.
- (19) Water level (Figure XIII-8) or optical level.

TABLE XIII-3
SILVER SOLDERS AND BRAZING TORCH TIP

UOP Specification Listed in the Project Specification 330

	BSg-1 wt-%	BAG-1 wt-%
Ag	44-46%	55-57
Cu	14-16	21-23
Sn	0	4.5-5.5
Zn	14-18	15-19
Cd	23-25	
Brazing Temp. Range	1145-1400°F	1205-1400°F

Fine-Tip Acetylene Torch: Victor #0323-0059-000T4

Opening Size = 0.027 inches (0.686 mm)

iii. Adsorbent Loading

- (1) Protective clothing for workers handling adsorbent both inside and outside the chamber. Although the adsorbent is not immediately toxic, it will generate heat when it contacts moisture; therefore if the adsorbent comes into contact with skin, it may cause burns. The dust formed during loading can cause respiratory disorders if inhaled over an extended period of time. Personnel working inside the chambers must wear dust masks at all times. When loading the adsorbent, personnel should also wear:
 - (a) Tight fitting overalls. TYVEK disposable overalls with hoods work well.
 - (b) Long rubber or leather gloves.
 - (c) Full face dust masks. Provide one for each worker with at least 10 spare filters. Do not use filter canisters designed for toxic gases. Respiratory protection should comply with the requirements provided in the MSDS for the adsorbent being loaded.
- (2) Bolt cutter for opening drums.
- (3) Two loading hoppers and two manway mounted loading hoppers. See UOP Standard Drawing No. 9-111-0.
- (4) Rubberized, or Dacron, 100-150 mm (4-6 inch) diameter canvas socks with grounding wire. The best type of sock has a wire mesh grounding wire sewn into the sock. Provide as many socks per chamber as the number of outlets on the manway mounted loading hopper with at least one spare for each adsorbent chamber.

- (5) Adsorbent leveling boards. Boards with a cross section of 12 mm x 100 mm (1/2" x 4") and a length that can reach between two beams is a useful size. Several boards should be prepared to allow for loss and breakage.
- (6) Scaffolding for making work platforms inside the chamber.
- (7) Adsorbent loading shoes, two sets of shoes for each loading person. Figure XIII-9.
- (8) Protective cover for all flanges and open pipe of the grids and distributor piping to prevent adsorbent from getting inside. Adsorbent inside the grid or piping could result in a failed integrity test.

iv. Grid Screen Integrity Test

- (1) Blind flanges, or blind plates, for all nozzles on the adsorbent chambers.
- (2) Several 1/4 turn ball valves of the same size as the adsorbent chamber bed lines. These valves are used for releasing air pressure when checking the integrity of the grid screens.
- (3) Test baskets for collecting any debris removed from the grids during the integrity test (Figure XIII-10).
- (4) Special equipment for testing the integrity of the bottom grids. Typically a custom-made reducing section is used to connect the ball valve mentioned above to the bottom grid distributor piping that exits the chamber.

2. Chamber Preparation and Material Handling

a. Chamber Preparation

- (1) The adsorbent chambers must be prepared for safe entry. At a minimum:
 - i. The vessel must be positively isolated.
 - ii. Safe access to the vessel must be provided.
 - iii. Personnel entering the vessel must be provided with safety harness and proper fall protection.
 - iv. Two people must be available outside the vessel to assist personnel inside the vessel.
 - v. The safety of the atmosphere must be confirmed by checks for toxic gases and oxygen content and for explosive gases.
 - vi. A nearby supply of fresh air must be available for those who will rescue personnel that encounter difficulty.
 - vii. A vessel entry permit must be obtained and registered with the responsible persons.
- (2) Have a protective cover installed on the top manway flange made from either metal, wood, rubber, or plastic. This will hopefully avoid the necessity of resurfacing this flange later when loading is complete.
- (3) Although the process specifications require the adsorbent chambers to be cleaned and sealed at the point of fabrication, immediately before adsorbent loading it may be necessary to sandblast the inside of each chamber to remove rust if the vessel was not adequately protected. Brush out all dust, working from top to bottom, and remove any oil or grease from the flange faces. Care should be taken to avoid damaging the leveling bolts in the support lugs if they are used.

- (4) Install air eductors or movers on several chamber nozzles to provide adequate ventilation.
- (5) Since the adsorbent chambers are opened and closed frequently for integrity tests during the loading, construction of a temporary handling platform on the top grid supports should be considered. Such a platform would allow a short run of ladder to be moved into and out of the adsorbent chamber as needed to open and close the vessel.

b. Beam installation

When moving beams into and out of the adsorbent chamber it is important that the beams be securely held by the rigging and that the work be done very carefully. UOP suggests a small lug be temporarily welded to the beams such that the beam cannot slide out of the rigging. Figure XIII-10. Newer sets of beams have a hole provided to secure the beams for lifting. Figure XIII-11.

- (1) Install all grid support beams for the bottom grid.
- (2) Insert leveling bolts into the leveling plate at the bottom of the support lugs for all of the beams at this elevation.
- (3) The level of the beams are determined by the use of a water level or optical level. The water level should consist of a large water reservoir, a smaller water reservoir with a scale for determining the height of the liquid level, and a plastic tube connecting the bottoms of both water reservoirs (Figure XIII-8). Clear tubing should be used for the leveling instrument such that bubbles in the tubing can be removed as they can lead to inconsistent readings and require the measurements to be performed again.

- (4) Find the elevations of the support ring by using the leveling device. If using a water level place the large reservoir on the ring and gauging the height around the ring. Although the ring should be installed and the vessel should be installed so that the support ring conforms to the ± 3 mm ($\pm 1/8$ inch) specification, it is not possible to adjust the level of the support ring without extensive work. Level the beams with the highest point of the support ring. Shims may be used at a later stage if the grids are not stable after placement. Figure XIII-12.
- (5) Level all beams to ± 3 mm ($\pm 1/8$ in) to each other and the support ring utilizing the beam leveling bolts in the support lugs. Ensure the high point previously identified on the ring is still the highest point of both the ring and beams. Record all measurements. Poor leveling can create later difficulties in grid installation it is very important to obtain good levelness.
- (6) After all beams are level, locate and punch the beam web for drilling the beam. Locate the holes on the vertical centerline in the hole or slot in the support lug. Figure XIII-13.

NOTE: If the hole is drilled in the beam at the exact center of the hole or slot in the support lug, when the beam is installed the top of the beam will be lower than when it was leveled because of the clearance between the bolt and the slot and the bolt and the hole in the beam.

As an example let's say a 30 mm (1 1/4 inch) bolt is to be used in a 32 mm (1 3/8 inch) hole. If the beam is marked directly in the center of the hole or slot in the lugs and drilled to 32 mm, when the beam is installed and the leveling bolts are removed the beam will rest below the location it was leveled by 2 mm. The easiest solution is to punch the beam 2 mm lower than the horizontal centerline of each hole. For the example above, a

rod of steel can be prepared with a point that is 28 mm in diameter. This can be held against the bottom of the hole or the slot in the support lug to mark the center of the hole for drilling. When this adjustment is made, the beam, when installed, should be at the same elevation as it was when it was leveled.

Another method is to punch the beams in the center. It is then necessary to adjust the center punched mark to compensate for the lug slot diameter and bolt diameter and drill to the specified hole diameter. As mentioned before, if an adjustment is not made, the tolerances of the hole in the beam and lug add together and drop the beam below the desired level.

- (7) Match-mark the beams and lugs using machinist stamps such that the beams will be placed back in the identical location. It is customary to label the grid levels with the same number as the bedline that connects to them. The top and bottom grids are usually referred to as top and bottom.
- (8) Remove beams and drill proper sized holes. Several techniques are available such that the holes may be drilled without removing the beams from the adsorbent chamber. If this can be done, it is not necessary to remove the beams for drilling again.
- (9) Reinsert beams. Select the correct number of hold down lugs (top grids only), as shown on the grid drawings, and slip over the beam from one end. Check that all lugs will slide along the beam. Install the support beam bolts, but do not tighten them, and then remove all leveling bolts from that elevation. Figure XIII-14.

- (10) Recheck levels, make a record, and if they are satisfactory, start the procedure again at the next elevation.
- (11) The elevation between the top of the support beams for the next and each successive level must be within ± 6 mm (1/2 inch) of the distance required in the Project Specifications from the top of beam at the previous level. The exception is the top grid level where this specification may be relaxed such that the beams can be leveled with the top grid support ring.
- (12) The top grid beams may require an offset from the level of the support ring. Check the Project Specifications.
- (13) Check the orientation of the thermocouple nozzles to ensure that there is enough clearance for thermocouple installation. The thermocouples should not come closer than 25 mm to any beam. If necessary, a shorter thermocouple than called for in the Project Specifications may be used.
- (14) The beams can be a real hindrance in loading as they interfere with movement of the loading socks. This may slow the progress of the loading. One approach to minimize this is to remove any beams from the adsorbent chamber that are unnecessary for rigging of ladders or work platforms. Typically with this approach, the beams for the top grids are left in place and the outer-most beams are also left in place.

Another technique is to remove the central beam(s) at each bed level, rotate the beams 90 degrees around a vertical axis, and place the beams on adjacent beams so as to produce an opening from the top of the chamber to the bottom. Such an arrangement may make distributing the adsorbents using the loading socks difficult.

c. Material Handling

- (1) Erect a waist-high pipe bridge, or saw horses, for inspecting grid sections in a paved area near the drop area of the chambers which is accessible to the crane or overhead hoists. Sufficient area is desired such that two grid levels can be inspected. The area should be set up so that there is enough space so that all areas of the grids to be inspected can be easily reached.
- (2) Construct platforms and ramps so adsorbent drums can be unloaded from a truck and moved to the lifting area without machinery, if possible. See Figure XIII-15.
- (3) Arrange a route for removing empty adsorbent drums from the loading area to a suitable storage area.

d. Grid Inspection

- (1) The screens of the grid sections are inspected prior to loading the grid sections into the adsorbent chamber as it is easier to make repairs with the grid sections out of the adsorbent chamber. Platforms are fabricated to facilitate grid inspections. See Figure XIII-16.
- (2) Inspect each grid for:
 - i. screen damage or abnormally large slots openings
 - ii. pinholes in the seal weld
 - iii. deformities in the seal weld
 - iv. deformities in the distributor tie-in weld and collar
 - v. any other possible source of sieve leakage into the grid or distributor piping
 - vi. Screen flatness and note any movement of screens.

- (3) The grid sections have been inspected once at the fabricator shop, so repairs at this point are intended to correct damage incurred via shipping or storage. Two types of damage may occur: rusting or mechanical damage to the screens.
- (4) Should the grids have become unacceptably rusted, it may be recommended that the grids be cleaned chemically. If this is necessary, an experienced chemical cleaning contractor should be consulted for the work. UOP Standard Specification 1-13 addresses chemical cleaning. A brief discussion of the general guidelines of chemical cleaning are discussed under a separate heading in this section.
- (5) Damage to wires of the screens. If damage during transport causes the wires of the screens of the grids to become weakened or causes the gaps between the wires to become too large then the screens must be patched or repaired.

Grids utilize screens formed from parallel wire welded to support rods. The shape of the wires is a triangle. The screen is welded directly to the frames of the grid. Minor damage to this type of screen can be corrected by either filling the oversized gaps between the wires of the screen with silver solder or the wires can be adjusted carefully with a chisel and hammer. In the latter method, a sharp chisel is placed on the top of the wire to be moved and tapped with a hammer. Some skill must be developed to correctly carry out this technique, but once mastered, it is more efficient than filling in minor defects with silver solder. More severe damages to the screen may be repaired by silver solder, Table XIII-3. Small holes and scrapes are repaired by covering the damage with silver solder. Adequate flux is necessary to get the solder to stick to the screen. Alternatively the vendor representative may employ

other weld techniques for screen repair which require more specialized skill.

Occasionally slots are found to be out of tolerance as being too wide without indications of physical damage to the grids. Most of the time this is the result of improper use of thickness gauges in taking the measurement. A thickness gauge is not a yes/no go instrument. A thickness gauge is meant to be used to determine the actual clearance between two parts. As such, they are expected to be used with a certain amount of skill. When using a thickness gauge to determine a gap, the gauge should be able to slide very easily between the two parts with an almost imperceptible pull or “feel”. This pull or “feel” as the gauge slides between the parts is one reason that thickness gauges are frequently called feeler gauges. Since the wires of the screen are not perfectly parallel, it is preferred that a thickness gauge with a width reduced to about 3-5 mm (0.1-0.2 inches) be used.

For example, it is possible to push a 0.20 mm (0.0079 inch) between two wires that are actually only 0.19 mm (0.0075 inch) apart with a fairly small amount of pressure. This is especially true when a gauge has been used for a long time and the tip of the gauge has been worn. Since over 99.5% of the adsorbent is typically larger than 0.3 mm (0.012 inches) making a “repair” at such a point would either be a waste of time or cause a reduction of open screen which would be considered detrimental due to the unnecessary loss of open area.

- (6) For the bottom grids, a seal plate is welded to the flange of the grids. This is the plate that supports most of the angle plate used to seal the grids to the vessel wall. The junction between this plate and the plate installed at the ends of the T-bars frequently leaks when the seal weld of the bottom grid is

checked. The plate attached to the grid flanges is typically a shop weld. The plate installed between the seal plate on the grid is a field weld. Steps should be taken such that the field weld will not have a defect where it meets the shop weld. Some contractors have ground the shop weld back where the field weld connects as a preparation prior to installing the grid sections into the adsorbent chambers. Figure XIII-17.

3. Bottom Grid Installation

The following procedure is given to minimize the forces on the seal welds of the bottom grids. Poor installation could lead to a mechanical failure.

- a. One eductor should be installed on the unloading nozzles above the bottom grid for ventilation, and, if possible, another eductor should be connected to the bottom manway. The fan type air mover with flexible duct can also be used to ventilate the bottom head.
- b. The bottom distributor piping needs to be thoroughly cleaned and then arranged in the bottom head of the chamber. It is difficult or even impossible to get the piping in after the grids are in place.
- c. After the bottom grid sections have been inspected and repaired and the protective cardboard and plywood have been secured over the fine screen, the grids can be installed in the chamber. Great care must be taken in lowering the grids so as to protect the screen. Figure XIII-18. The grid sections should be placed so that the grid vent (snorkel nozzle) is as shown on the drawings contained in the Project Specifications.
- d. The grids need to be placed correctly and their spacing adjusted to fit the T-bars. To ensure that the grids are placed correctly, it is helpful to mark the supports with chalk before placing the grids. The gap between the vessel wall and the grid should be adjusted also. Spacer bars may assist in arranging the grids. Figures XIII-19 and XIII-20.

- e. After there is proper ventilation in the bottom head, the distributor piping needs to be arranged and the grids should be adjusted to accommodate any prefabrication of the piping. The pre-fabricated piping is supplied with over-length to allow flexibility in installation. However it is necessary to verify the approximate fit-up before beginning t-bar welding. Since some distortion of the grids may occur during the welding of the T-bars a final fit of the pipes should not be made at this time.
- f. After the grid spacing is acceptable, fix the grids to the support by tack welding or using temporary holddown lugs (see Figure XIII-21) to minimize the grid movement while welding the T-bars.
- g. Ensure that the T-bars will still fit in between the adjacent grid sections and that any prefabrication of the piping still fits.
- h. The T-bars can be cut to size and laid between the grid sections. Always have one beveled edge at each T-bar intersection or joint to allow proper welding as shown in Figure XIII-22. The ends of the T-bars should be notched to fit up with the outer seal plate that fits below the seal angle. It is not acceptable to cut back the angle plate used to seal the grid to accommodate T-bars that are too long.
- i. The T-bars can be tack welded into place.
- j. The seal welding of the T-bars should begin in the center of each bar. Use ceramic blankets to prevent slag from damaging the fine screen by falling between the grid frame and plywood. To prevent warping by localized overheating, use an alternating welding pattern, with each weld being 100-150 mm (4"-6") long. Welders should be asked to avoid welding on sections of the grid that are still hot from welding. The idea is to have the grids welded in as they sit in the cold position.

If possible, Tungsten Inert Gas (TIG) weld the T-bars to avoid slag splatter. Less heat is generated via TIG and less repairs are usually required later as TIG is less prone to leak at the starting and stopping points.

After the seal plates are installed at the ends of the T-bars, inspection can be made at the connection of the plates at the ends of the T-bars and the plates on the grids. At this time there will be access; after the bent angle (seal angle) is installed, this inspection will be more difficult.

- k. The temporary restraint welds holding the grids to the supports should be removed after completing the T-bar welding. Any gaps between the grids and the supports should be shimmed with metal plates which must be tack welded in place to the beam or support ring. At least two tack welds should be made per shim between the shims and the supports. Either SMAW or TIG welding may be used for the tack welds.

NOTE: The gaps below the bottom grid can be made up with shims and little structural problems will exist, but a similar condition may occur during installation of the top grids where this remedial action is not possible. The installation of the bottom grid can be a guide as to the adequacy of the skip weld technique carried out by the welders. If excessive warping occurs, the welding technique should be improved for the top grids.

- l. Verify fit-up of the seal angle. In some cases it may be necessary to modify the seal angle if the vessel was not fabricated to specification.
- m. To get good piping alignment and ensure a full penetration weld, the use of welding brackets or welding dogs (see Figure XIII-6) is advised. In order to reduce distortion, it is also useful to tack weld the pipes together at three points 120 degrees apart prior to the first full-pass of root weld. All root (initial) passes must be welded with inert gas TIG (GTAW) welding to ensure a clean, slag-free weld. When all these root welds are completed, they must be checked for cracks and pinholes. A dye-penetrant check is a fast and easy way to do this.

The basic steps of the dye penetrant test are: apply the dye over the weld, allow the dye to penetrate any defects, wipe excess dye from the surface,

apply developer, allow the dye in the defects time to mark the developer. Any defects indicated by staining of the developer with dye should be ground back and welded over using the TIG technique.

- n. After the root passes on the distributor piping have been checked, the pipe to pipe welding can be completed with normal arc welding.
- o. Place and fit the seal angles into position. Tack weld the seal angles in place. It is best to start the tack welds at the center of the seal angle pieces. Seal weld the seal angles - TIG welding is preferred in order to avoid slag formation. Use an alternating welding pattern, with each weld being 100-150 mm (4"-6") long. Complete the seal welding. Figure XIII-23. The complete installation is shown in Figure XIII-24.
- p. To check the integrity of the T-bar and seal angle welds, place a blind in the grid vent (snorkel) flange and seal off the bottom head. See Figure XIII-25.
- q. The bottom head should be pressured up to 380 mm (15 inches) H₂O pressure (maximum) with clean, dry air. A water manometer is a good way to check the pressure, and it can also act as a safety device to prevent over-pressurization if the water manometer tube is larger in diameter than the air supply line. The manometer must be connected to the bottom head with no block valves in between the manometer and the bottom head.

NOTE: On more than one occasion inattention to this step of the procedure or incorrect setup has caused the seal welds of the bottom grid to rupture requiring rework.

It is recommended that no personnel be standing on the bottom grids when the bottom head is being pressurized for the leak test. Take extreme care that the bottom head is not over-pressurized. See Figures XIII-26 and XIII-27 for the recommended air supply piping arrangement and the water manometer details.

- r. Disconnect the air source then enter the chambers for the leak test. Test all the welds with a soap and water solution. This includes the welds of the continuous ring to the vessel wall. Any leaks have to be repaired after the bottom head has been depressured. Repeat this procedure until no leaks are found. The soap and water solution should be wiped off of the grids.
- s. During the bottom grid weld integrity test, the adsorbent unloading nozzle and thermocouple can be installed and packed with ceramic rope as per Figures XIII-28 and XIII-29. Also, the adsorbent sampling valves need to be put in place at this time where they are specified. The sample valves should not be packed with ceramic rope.
- t. The beams for the next level should be installed NOTE: The support lugs should not be tight against the beam. Should the support lugs be pressed against the beams by the bolts this may restrict movement of the beams. If one nut is used on a bolt it should be pulled hand tight to the support lug and tack welded in place. If two nuts are provided the first is pulled hand tight to the support lug and the second and first nut are tightened to each other not the support lug. There must be clearance between the support lugs and the beams and clearance between the end of the beams and the vessel wall.
- u. After all the grid repairs are finished, the bottom head must be cleaned up, the grid vent (snorkel) blind removed and the correct size grid vent (snorkel) installed. The remaining pipework must be completed and inspected prior to closing the bottom head manway.

4. Final Inspection

- a. Remove the plywood and cardboard. It is a good idea to check the soles of the shoes worn in the adsorbent chamber again to make sure they have not picked up foreign debris or welding slag that could damage the grids.

- b. Thoroughly clean the screen's surface with an industrial vacuum cleaner. The cleanliness of the grids can have a significant impact on the time required for inspection. Work above the grids being inspected and moving of equipment into or out of the adsorbent chamber should be minimized to avoid knocking debris onto the grids during inspection.
- c. Using a wrench, check all bolting. Confirm that the correct gaskets are in place (intermediate beds). Use an appropriate checklist to ensure that no items get missed. See example at the end of this section.
- d. Inspect the adsorbent unloading nozzle to make sure that the ceramic rope has been properly installed.
- e. Also check that the peripheral ceramic rope has been properly installed, packed tightly and is flush with the grid (intermediate and top grids only).
- f. Check that the thermowells and adsorbent sampling valves have been installed, if required. The thermowells are packed with ceramic rope. Do not pack the adsorbent sampling valves with ceramic rope.
- g. Measure the average bed depth and diameter and record these numbers for use in the loading calculations (Figure XIII-30).

5. Adsorbent Loading

a. Adsorbent Loading System

A large platform at ground level for dumping the adsorbent drums into the loading hopper may be necessary. The means of transporting the adsorbent from storage to the adsorbent chambers will determine the type of platform required (for possible methods, see Figure XIII-15). Dumping drums by hand is no longer acceptable in some locations due to the risk of injury while handling the drums. Many types of equipment are now available for dumping drums of material, and the type chosen will determine the platform required

for the lifting hopper. All equipment should be verified by the UOP Loading Advisor for suitability prior to use.

As the drums are loaded into the lifting hopper, one person should record the lot and drum number, and net weight of each drum loaded, and also a composite adsorbent sample should be taken. This person should also check to make sure no damaged drums of adsorbent are loaded. The personnel handling the adsorbent should wear dust masks and leather or plastic gloves.

It is very important not to overfill the lifting hopper. Adsorbent can be lost to the wind if the hopper is overfilled. The adsorbent has a high value and so it should be handled carefully and not lost. Lost adsorbent will make calculating the loading efficiencies difficult or erroneous. It is very important to have the unit loaded properly, and without a correct loading efficiency, it will be difficult to be sure this is done. Typically a cover is secured over the lifting hopper and the bottom outlet is positively sealed to prevent adsorbent losses.

The use of two lifting hoppers allows one hopper to be filled while the other hopper is filling the adsorbent chamber. A large hopper at the top of the adsorbent chamber saves a substantial amount of time by keeping the crane or overhead hoist working. The lifting hopper dumps the adsorbent into the hopper at the top of the chamber that acts as a reservoir of adsorbent while the next full lifting hopper is being delivered. The adsorbent is then distributed in the adsorbent chamber through the loading socks attached to the hopper at the top of the adsorbent chamber. The socks should be equipped with grounding wires. (See Figure XIII-31.)

b. Adsorbent Loading Procedure

- (1) Before the adsorbent loading begins, make sure that sufficient ventilation is provided. Either equipment to remove and contain the dust from the loading (as required by local regulations) or at least 2 air

eductors are connected to the lowest possible unloading nozzles above the bed to be loaded. Confirm that this equipment is functioning properly. Also make sure that all the materials needed inside the adsorbent chamber are available. All personnel in the adsorbent chamber should be properly outfitted with the correct safety equipment (dust masks, gloves, etc. refer to the MSDS for the adsorbent).

- (2) The coarse adsorbent is loaded first in each bed as a support layer. The amount of coarse adsorbent to be used is found in UOP Specification 105. This material is typically divided evenly among the beds to be loaded such that all of the material is used during the loading. Take care to spread the adsorbent evenly, covering all of the screen with a 25-50 mm (1" to 2") layer.

The loading personnel should avoid standing on the adsorbent or the distributor piping. A temporary platform can be made one or two levels above the bed being loaded. Such a platform should be supported well, and proper fall protection should be provided. The material used to make the platform should not allow the accumulation of adsorbent that could cause personnel to slip. Steel and plastic grating materials have been used in the past.

After the coarse adsorbent is loaded, the fine adsorbent should be spread slowly in a fine, wide spray so that the coarse layer is not disturbed. This can be done by pinching the end of the sock while spreading until the fine adsorbent layer is 100-150 mm (4" to 6") deep.

- (3) After the initial fine adsorbent layer of 100-150 mm (4" to 6") deep is loaded, the loading personnel equipped with "adsorbent loading shoes" (Figure XIII-9) walk across the bed to prevent mounding and level the bed. This will help to ensure the bed has been loaded uniformly.

- (4) The loading sock should be cut so that the adsorbent falls at least 1 to 2 meters (4' to 8'). To get the maximum loaded density, move the end of the sock back and forth and lay down the adsorbent in thin layers (75 mm (3") max.). Mounding of the adsorbent due to inadequate movement of the sock will cause a lower average bulk density (ABD) per bed that reduces the capacity of the unit and can ultimately reduce the performance.

The adsorbent loading rate should not exceed 20 drums/hour/100 mm sock to ensure good loading efficiency. (The UOP Standard Drawing 9-111 will specify the number of socks.) As each bed level rises, the sock will have to be cut several times to maintain the 1 to 2 meter (4' to 8') falling height.

The unit has support beams and holddown plates for the top grid, must be placed at their correct positions prior to beginning the loading since the adsorbent will interfere in their installation.

The adsorbent should be loaded until the bed is even with the top of the support beams. Level the bed using the leveling boards suggested in the material and equipment section. Take care not to overfill the bed because removing adsorbent is difficult whereas adding adsorbent is relatively easy.

- (5) Record the total number of drums (and fractions of drums) loaded. The bed density should be calculated based on the weight of adsorbent loaded, and the calculated bed volume less the volume of the piping and beams. Calculate the loading efficiency and compare it with the expected number supplied by the UOP Technical Advisor. If the loading efficiency is not near the 97% target, then adjustments in the loading technique must be made. Typically, values at 97% +/- 1.5% are considered acceptable. This must be done as loading progresses not at the end of the loading.

6. Grid Screen Integrity Test

After loading each bed of adsorbent, a test must be performed to ensure that the screen has no leaks before continuing with the loading.

- a. Close up the adsorbent chamber and pressurize it to 0.35 to 0.5 kg/cm²g (5 to 7 psig) with clean, dry air (see Figure XIII-32). Pressurizing the adsorbent chambers can be the most time consuming part of the integrity test.

If a clean, idle vessel can be found to provide surge capacity for the air system, this part of the procedure can be greatly accelerated. One good example used an idle fractionation column and large plastic hoses to pressure up the adsorbent chamber with air. Typically the dry air source is sized to allow the empty chamber to be pressurized in 15-30 minutes.

- b. Install a block valve on the bed line connected to the grid that has just been covered with adsorbent (or on the adsorbent chamber circulation line for the bottom grid).
- c. After the vessel has been pressurized, open the block valve for about 30 seconds to blow out any adsorbent or debris in the grid and piping.
- d. Attach the integrity test basket (Figure XIII-33) to the block valve. The integrity test basket is fabricated such that the flow through the basket shall be maximized and no parts of the basket will trap adsorbent caught during the test.
- e. Open the valve and depressure the chamber through the basket for at least 3 minutes. The basket is carefully removed and dumped out onto a dark surface. If any adsorbent is found in the basket, repeat the test until no adsorbent is found and there is no doubt that the screen does not leak. If it cannot be determined there is no leak, the adsorbent must be removed and the grid must be inspected, cleaned, and repaired as necessary.

Rarely will a unit fail the integrity test if the installation instructions have been strictly followed. It is more economical to spend some extra time in ensuring the grid installation is done correctly than taking the time to unload, reinspect and repair the grid level. Very seldom has this occurred, but the failed integrity tests that have been recorded could have been avoided by installation technique.

7. Grid Loading and Installation

- a. Recheck the level of the adsorbent bed using the correct leveling board.
- b. Brush the grid supports free of adsorbent.
- c. Lower the grid sections into place using the crane and/or hoist and comealongs.
- d. Grating may be used for the installation personnel to stand on inside the chambers.

NOTE: The grids should be numbered in the correct installation sequence to avoid excessive handling of the grids inside the chamber. The grids must be placed directly in their final position on the supports. Sliding the grids on the supports to get them into position is not acceptable. Sliding the grids can trap mounds of adsorbent under the grid's bottom screen (Figure XIII-34). This adsorbent can damage or plug the bottom screen, and can also prevent the grid from sitting correctly on the supports.

As each grid is put in place, check each grid carefully by standing on the grid and shifting your weight to ensure that the grid is supported well. If the grid shifts under your weight, it should be lifted, and the support and grid must be checked. Shims may be required, and these should be tack welded into place onto the support. See Figure XIII-35.

- e. After all the grids are in place, they need to be moved using crowbars to achieve equal spacing. Adjustments may be necessary to account for irregularities in the shape of the vessel. Spacers to get the correct gaps between grid sections and between the grid sections and the vessel can be prepared. The spacers can be made from bar stock and a metal rod welded across the top to prevent them from falling into the sieve bed (Figures XIII-19 and XIII-20).
- f. Wedges can be placed around the grids to prevent the grids from shifting.
- g. After the grids have been secured in place, the pipe fitting can be started. Align each pipe so that there is no stress placed on it. All first-pass welds must be done with inert gas to prevent slag from entering the piping and the rotary valve where the elastomer sealant sheet can be damaged. Check the first passes for cracks and pinholes before they are welded over. Use welding dogs (brackets) wherever possible. The additional filler weld passes can be done by normal arc welding. The alignment of the piping should be checked during the welding to make sure that the welding is not significantly misaligning the pipe work.
- h. For installation of Dur-o-lok couplings, use the procedure provided in Appendix 1: Fit the piping in place and check the alignment. If the alignment is correct, complete the piping installation. Note to obtain proper pipe to grid alignment, the grids may have to be shifted.
- i. When the piping is complete, pack the ceramic rope around the outer circumference of the grids until it is at least flush with the top of the grid or slightly above it. Depending on the accuracy of the fabrication of the adsorbent chamber it may be necessary to use various sizes of ceramic rope or make additional rounds of rope to fill the gap adequately to make up for differences between the vessel shape and the round cross section of the grids. Spare rope is provided for this purpose. See Figure XIII-35.

Care should be taken that the position of the grids is not changed by the packing of the ceramic rope. If it is found that the packing of the ceramic rope alters the position of the grids, it is permitted to install the ceramic rope prior to the pipework. Small adjustments of the position of the grids can be obtained by adjusting the position of the grids using a large crowbar and adjusting the ceramic rope packing.

- j. After the pipes have been welded on, the pipe can be reinstalled and bolted into position.
- k. Perform a final inspection of the grids and distributor piping after installing the ceramic rope packing.

8. Top Grid Installation

Before loading the last two beds in each chamber, check to see whether the top grids can be installed in the chamber without disturbing the top adsorbent bed. If there will be difficulty, it may be necessary to bring the top grid sections into the chamber and to install them and hang them out of the way before loading the top two beds. Lugs are provided on the top head of the chamber for this purpose.

After the last bed of adsorbent is loaded and leveled, the top grids can be lowered into place. A section of grating can be used to avoid disturbing the adsorbent bed while lowering the grid sections. It is helpful to mark the position of each grid with chalk on the supports.

The finishing of the top grid is similar to the procedure used for bottom grid.

- a. The grids should be adjusted to accommodate any prefabrication of the piping. Since some distortion of the grids may occur during the welding of the T-bars, a final fit of the pipes should not be made at this time.

- b. After the grid spacing is acceptable, fix the grids to the support by using holddown lugs similar to what was used for the bottom grid (see Figure XIII-36) to minimize the grid movement while welding the T-bars.

The holddown plate for the top grids should be pulled toward the vessel head and tack welded into place. This can be done by attaching a loop of rope between the top head and the plate and twisting the rope to put tension on the plate. It is recommended to tap the plate with a hammer to make sure there is not adsorbent caught between the plate and the beam. Figure XIII-37.

- c. Ensure that the T-bars will still fit in between the adjacent grid sections and that any prefabrication of the piping still fits.
- d. The T-bars can be cut to size and laid between the grid sections. Always have one beveled edge at each T-bar intersection or joint to allow proper welding. The ends of the T-bars should be notched to fit up with the outer seal plate that fits below the seal angle. It is not acceptable to cut back the angle plate used to seal the grid to accommodate T-bars that are too long.
- e. The T-bars can be tack welded into place.
- f. The seal welding of the T-bars should begin in the center of each bar. To prevent warping by localized over-heating, use an alternating welding pattern, with each weld being 100-150 mm (4"-6") long. Welders should be asked to avoid welding on sections of the grid that are still hot from welding. The idea is to have the grids welded in as they sit in the cold position.

If possible, TIG (GTAW) weld the T-bars to avoid slag splatter. Less heat is generated via TIG and less repairs are usually required later as TIG is less prone to leak at the starting and stopping points.

- g. The temporary restraint welds holding the grids to the supports should be removed after completing the T-bar welding.

- h. Install the ceramic rope around the top grids.
- i. To get good piping alignment and ensure a full penetration weld, the use of welding brackets or welding dogs is advised. In order to reduce distortion, it is also useful to tack weld the pipes together at three points 120 degrees apart prior to the first full pass of root weld. All root (initial) passes must be welded with inert gas TIG (GTAW) welding to ensure a clean, slag-free weld. When all these root welds are completed, they must be checked for cracks and pinholes. A dye-penetrant check is a fast and easy way to do this.
- j. After the root passes on the distributor piping have been checked, the pipe to pipe welding can be completed with normal arc welding.
- k. Place and fit the seal angles into position. Tack weld the seal angles in place. It is best to start the tack welds at the center of the seal angle pieces. Seal weld the seal angles – TIG welding is preferred in order to avoid slag formation. Use an alternating welding pattern, with each weld being 100-150 mm (4"-6") long. Complete the seal welding. The completed top grid installation is shown in Figure XIII-38.
- l. To check the integrity of the T-bar and seal angle welds, place a blind in the grid vent (snorkel) flange and seal off the bottom head. See Figure XIII-39.
- m. The adsorbent chamber should be pressured up to 380 mm (15 inches) H₂O pressure (maximum) with clean, dry air. Figures XIII-26 and XIII-27 show the recommended piping configuration for the leak test.. A water manometer is a good way to check the pressure, and it can also act as a safety device to prevent over-pressurization if the water manometer tube is larger in diameter than the air supply line. The manometer must be connected to the adsorbent chamber with no block valves in between the manometer and the adsorbent chamber head.

No incidents of rupture of the top grid seal welds have been reported, which may be a result of either lessons learned while installing the bottom head or the larger capacity of the adsorbent chamber as compared to the bottom head.

It is recommended that no personnel be standing on the top grids when the adsorbent chamber is being pressurized for the leak test. Take extreme care that the top grid is not over pressurized.

- n. Disconnect the air source and proceed with the leak test. Test all the welds with a soap and water solution. Any leaks have to be repaired after the adsorbent chamber has been depressured. Repeat this procedure until no leaks are found. The soap and water solution should be wiped off of the grids.
- o. Remove the grid vent (snorkel) blind and install the correct size grid vent (snorkel). Install the remaining piping.
- p. Clean the top grid and close the top manway. Care should be taken that the top manway is aligned properly with the attached piping.

9. Protection

After the adsorbent chambers have been loaded, take extreme care to prevent the accidental introduction of water or other contaminants into the chambers. The adsorbent chambers should be blinded off and pressurized to at least 0.35 kg/cm²g (5 psig) with nitrogen or dry air.

B. ADSORBENT CHAMBER UNLOADING

GENERAL OUTLINE

1. Prepare Adsorbent Chambers for Unloading

- a. Desorbent displacement
- b. Adsorbent saturation with water
- c. Hydrocarbon purge with nitrogen
- d. Steamout for hydrocarbon removal
- e. Evacuation and nitrogen cooldown

2. Removal of Chamber Internals

- a. Grid and residual adsorbent removal
- b. Intermediate grid cleaning and inspection
- c. Bottom grid inspection and top grid reconditioning

Prior to the shutdown of the unit, materials and equipment must be prepared. Since once the unit is unloaded it is normal to reload the unit, all of the materials required for loading must be prepared with additional material listed here:

- 1) It is important to secure a dependable, large volume vacuum truck. Two is preferred if available and budget allows. Delays will occur if the vacuum for removing the adsorbent breaks down. One solution is to use a cyclone separator before the vacuum truck which can be dumped into a separate vessel for removal from site. The available solids handling equipment for each site should be reviewed. NOTE: Moving dry adsorbent can cause static discharge and protective grounding of all adsorbent handling equipment is required.
- 2) Equipment for cutting/removal of top grids and distributor pipes.

- 3) T-bar and bent seal angle to replace the material destroyed during removal of top grids.
- 4) New holddown plates for the top grid will be required
- 5) Additional bolts for the pipe flanges and beam to cover loses and damage during removal.
- 6) Replacement screen and spare parts package for grid repairs.
- 7) Ceramic rope

If free water enters the adsorbent chambers during normal operation, not only will it destroy the ability of the adsorbent to function but it may also cause the adsorbent to agglomerate. If this is suspected, the availability of air-driven hammers may also be required.

Prior to the shutdown of the unit, special equipment should be prepared for use during the shutdown procedure.

The lines used during initial startup circulations will need to be prepared to accommodate the circulations used in the unloading shutdown procedures. Typically these are the lines used during initial filling of the adsorbent chamber and flushing of the bedlines.

A water injection piping, pump, and flow meter must be arranged for use during the water saturation step of the procedure. Typically condensate is used for this purpose.

Large quantities of steam are used to purge hydrocarbon from the adsorption chamber. The steam must be provided at a temperature low enough not to exceed the allowable temperature of the adsorbent chambers during the steamout. Depending on steam availability, this may require the use of a steam desuperheater. While the maximum temperature of the steam is important, it is also

important that condensate not enter the adsorbent chamber during the steamout procedure to avoid agglomeration of adsorbent. See comment earlier about air-driven hammers. Some form of condensate trap located a minimum distance to the adsorbent chambers is recommended.

Although the normal nitrogen connections may be used to remove the hydrocarbon from the system during draining of the adsorbent chambers, they may not be sufficient for the purge and cooldown step after steamout of the adsorbent chambers. After the adsorbent is steamed to remove hydrocarbon, the adsorbent chambers must be cooled for vessel entry. Introduction of large volumes of nitrogen to the adsorbent chambers can be used to speed cooling of the adsorbent chambers.

During steamout, large quantities of steam with undetermined quantities of hydrocarbon must be removed from the adsorbent chamber. Temporary piping from the adsorbent chamber circulating pumps suction manifold to a cooler or condenser should be arranged along with facility of separation of hydrocarbon from the condensate produced. Typically, one of the air-cooled condensers of the unit is used for this service.

a. Desorbent Displacement

Existing units may have different startup lines and procedures which must be followed. The startup lines are required for this procedure so blinds and spools must be put into startup position. Once the flow of desorbent is stopped to the adsorbent chambers, the desorbent contained in the chambers is quickly replaced by feed. During the desorbent displacement procedure, the limiting operation may be the removal of desorbent from the heads of the adsorbent chambers. To facilitate removal of desorbent from the top heads, the vent with the restriction orifice may be opened to the raffinate column. Clearing the bottom heads is not as easy. The drains on the bottoms of both adsorbent chambers could be used to route material from the bottom heads through the sump to storage. Desorbent is exported via the desorbent removal system and the Desorbent Pumpout Cooler.

Desorbent from the Desorbent Surge Drum and Desorbent Stripper Bottoms Pump are exported via the Raffinate column Overhead Pumps.

1. Short circulation is established by normal shutdown procedures.
2. Shut down and block in the line flushes and zone flush.
3. Reduce the desorbent flow to the rotary valve. Begin exporting desorbent to storage. The faster all desorbent is removed from the unit, the faster this step will be completed.
4. Import feed to the unit as needed to replace displaced desorbent. Raffinate Column bottoms may be sent to the Extract column feed line for further fractionation if necessary. The columns bottoms is then circulated back to the feed line.
5. When the desorbent flow to the rotary valve has been reduced to zero block in the desorbent line to the rotary valve.
6. Put the pumparound FRC controller in analog control and set all the zone setpoints equal to the normal Zone II flow. Rotary Valve step time may be increased to 5 minutes to maximize flushing.
7. Direct feed from the outlet of the feed filter through a temporary jumper line to the outlet of the flush filter in order to displace desorbent from the chamber heads. Maximize the rate of feed flushing to the chamber heads.
8. Continue sending feed through the rotary valve to the chambers for three complete rotary valve cycles. The pressure at the bottom of chamber No. 2 will still be controlled using the raffinate control valve. This procedure will flush the desorbent from the adsorbent chamber bed lines and the adsorbent beds. Continue until desorbent recovery decreases to an acceptable level.

9. One option which allows isolation of the Rotary Valve in future steps is to install a temporary jumper line from the feed filter outlet to the bedline flushing startup line at the outlet of the desorbent filter. This allows feed to be introduced into the chamber circulation pump discharge line during water saturation without going through the rotary valve. After stopping the feed flow, unseat the rotary valve and block in all bed lines to the rotary valve. Stop the rotary valve's hydraulic system. Drain down the rotary valve and all piping low points. Purge the rotary valve with nitrogen. Install blinds in all bed lines, dome sealant lines, and process lines to the valve. Install these blinds at the rotary valve. Inspect the rotary valve at this time if required.
10. Shut down the pumparound and pusharound pumps as per standard procedures. Block in the pumparound pump but leave the pusharound pump's block valves open. The pumparound and pusharound control valves should be in manual and fully open.
11. The adsorbent chambers must be kept hot during the desorbent displacement. Use the heat exchange as required to heat the feed sent to the chambers up to normal operating temperatures.

b. Adsorbent Saturation with Water

1. Control the pumparound flow rate at the normal Zone II rate. The feed circulation through the chambers and the columns is the same for the water saturation procedure as it was for the desorbent displacement procedure. Continue heating the feed sent to the chamber to normal operating temperature. Filtered Feed is normally introduced into the chamber circulation discharge line and raffinate is withdrawn via the startup line from the chamber circulation pump suction line allowing chamber pressure control. Establish the feed and raffinate flows before starting the circulation pump to minimize chamber pressure fluctuations.
2. Begin water injection (steam condensate) to the top of chamber No. 1, via a direct connection to the pumparound line, at 2% of the PAP flow. Water

injection can be to both chambers to reduce time, each injection rate can be 2% of the PAP flow. A condensate pump and flowmeter or flowmeters will be needed for this purpose.

3. Drain water from the raffinate column overhead receiver's water boot as needed. Check this location frequently.
4. Take samples of the raffinate column feed on a regular basis (every 30 minutes). If second injection point is used, both chamber outlets must be checked. Once the liquid becomes cloudy (water saturated), continue water injection for two more hours and then proceed to the hydrocarbon purge step. It may not be possible to continue for these two hours if the circulation pumps begin to cavitate.
5. While injecting water to the chambers, monitor the adsorbent temperatures. If the temperatures begin to rise, reduce the water injection rate to stabilize the temperatures. The water injection rate can be increased as long as temperature rises do not occur. Do not exceed 2% of the PAP flow.
6. Also monitor the adsorbent chamber pressures and pressure drops during the water saturation. Decrease the pumparound circulation rate and/or the water injection rate if either chamber pressure drop exceeds 30 psi (2.0 kg/cm²). Control the chamber pressures at design pressure, it is acceptable to lower chamber pressure if required.
7. It will be necessary to stop all the paraffin material in the column after the water saturation step.

c. Hydrocarbon Purge with Nitrogen

1. Stop the water injection.
2. Stop the circulation pumps as per standard procedures. Block in the pumps.

3. Slowly reduce the feed circulation to zero. Keep the raffinate column operating on total reflux. Shut down the extract column.
4. Open the common circulation pump suction valve between the bottom of chamber No. 1 and the line to the raffinate column. Slowly reduce the adsorbent chamber pressure to 85 psig (6 kg/cm²g) through the raffinate control valve.
5. As the chamber pressure drops, begin adding nitrogen to the top of each chamber via the pumparound lines. Check the nitrogen header pressure to make sure it is adequate.
6. Use the nitrogen to displace the feed in the chambers to the raffinate column. Keep the pressure at the top of the chambers at 85 psig (6 kg/cm²g).
7. Export the displaced paraffins from the unit to slop. Water should be stripped from the xylenes and will collect in the overhead receiver. Drain the receiver's water boot as necessary.
8. Once the chambers are empty of liquid, drain all low points (chamber circulation pumps, bottom heads, top heads, dead end lines, etc.) of hydrocarbons. It will take several hours to complete. Once partial vapor is detected in the raffinate line, proceed to the next step as it is very difficult to purge hydrocarbon from the adsorbent with nitrogen.
9. Once the low points are drained, block in the nitrogen and slowly depressure the chambers to zero via the raffinate column.
10. Repeat step 8. Continue to check low points as steaming is started until steam is present
11. Shut down the raffinate column, as per standard procedures.

d. Steamout for Hydrocarbon Removal

1. Make separate steam connections to the top of each adsorbent chamber. The connections can be made by disconnecting bed lines Nos. 1 and 13 at the pumparound lines and using these flanges. Blind bed lines Nos. 1 and 13 at the rotary valve.

NOTE: The rotary valve should not be steamed out or exposed to water at any time.

2. The steam chosen for use (low pressure, medium pressure, etc.) must not be capable of exceeding the design temperature and pressure of the adsorbent chambers. Also, the temperature of the steam must be capable of vaporizing xylenes at ~30 psig (2 kg/cm²g) (i.e. >375°F or 190°C). Three or four inch steam lines are suggested to allow sufficient flow of steam to the chambers.
3. A column, overhead condenser, or flare system may be used as the destination the steam effluent from the chamber. Open the valves in the line from the bottom of the chambers to the column overhead condenser or flare line. Start the condenser. (Typically the raffinate column or the raffinate column overhead condenser will be used.)
4. Slowly open steam to the top of each chamber. The steam will flow down through the chambers and out to the raffinate column/overhead condenser. The condensed hydrocarbons and steam will collect in the raffinate column or overhead receiver. Drain water and hydrocarbons as required. Set the steam flowrates as high as possible, but not so high as to exceed the design differential pressure between the top and bottom of each chamber.
5. As steam-out continues, open up all low point drains and high point vents on chamber piping, top and bottom heads, dead end lines, pumps, bed line drains, etc. Do not steam out the rotary valve. This step is critical in order to

prevent residual hydrocarbon from remaining in the unit and the accumulation of condensate in the lines.

6. While the chambers are being steamed out, the columns (raffinate, extract, rerun, finishing), and all other unit piping can be steamed out in preparation for the turnaround work.
7. Once steam is present at the bottom of the chambers, and at all low point drains and high point vents on the chambers and associated piping, condense and sample the steam for hydrocarbons. Once hydrocarbons are not detected (visually or analytically) continue to steam out for another 12 hours, and then proceed to the nitrogen purging step. Actually, once hydrocarbon is condensed, hydrocarbon in steam stabilizes at a low level, e.g. 10-50 ppm or so.
8. After steamout is complete drain all water and hydrocarbon from the column and any system exposed to steam. Ensure nitrogen blanketing is maintained on the equipment to prevent corrosion. Free water in a column could result in tray damage so it is important to ensure the removal of water is complete after the steamout step.

Nitrogen vs. Air Purging

Nitrogen is recommended for purging the chambers for safety reasons. If all low points and dead ends are adequately steamed clean, the use of air may be considered. One accident occurred as a result of incorrectly isolating the adsorbent chambers on a small unit. In place of blinding during hydrocarbon removal, the operator relied on the bedline ball valve for isolation. It is guessed that the evacuation drew some hydrocarbon past one of the bedline ball valves. When the operator introduced air after the first evacuation, the adsorbent was still hot enough that the hydrocarbon ignited and burned. Three grid levels were melted and the vessel was damaged.

Although nitrogen is recommended for safety reasons, some customers have used air successfully. Extra care was taken in vessel isolation and all dead ends in the piping were purged well during the steamout operation. There was a much lower cost in using air.

e. Evacuation and Nitrogen Cooldown

(see comments on nitrogen vs. air for purging)

1. Twelve hours after hydrocarbons are no longer detected or reach a stable low level, stop steam flow to the chambers and disconnect the steam lines.
2. Block in all low point drains and high point vents on the chambers.
3. Pressurize the chambers to 5 psig (0.5 kg/cm²g) with nitrogen. Drain all liquid from low points. Connect the ejector to the bottom of the chambers. It may be best to arrange temporary piping at the adsorbent chamber circulation pump manifold for a temporary ejector. Use a larger than originally installed ejector, if available, in order to save time on this step. It is also possible to use a vacuum pump capable of handling liquids to further speed up this step.
4. Block in all instruments, pressure gauges, etc. on the chamber.
5. Commission the ejector and pull a vacuum of 300-500 mm Hg. Continue to check for the presence of hydrocarbons and condensate in the chambers and associated piping.
6. Repeat steps 3 and 5 two more times. The evacuation step is important to remove all water vapor from the chamber before cooldown. Water vapors may condense on the adsorbent and cause agglomeration of the adsorbent making it more difficult to unload. This step also helps to cool the chambers and reduces the quantity of nitrogen required for cooldown.
7. Remove the ejector as it will restrict nitrogen flow.

8. Begin maximum nitrogen purging flow down through the chambers and out of the ejector. Use a direct connection to the nitrogen header for this purpose or separate high volume source such as a nitrogen truck.
9. Monitor adsorbent bed temperatures and keep chamber temperatures within 30°C range.
10. Reopen the low point drains and high point vents on the chambers and the associated piping.
11. Once the adsorbent bed temperatures drop below 100°F (40°C), adsorbent removal can begin.
12. Block in the nitrogen to the chambers.

3. Grid Removal and Inspection

a. Grid and Residual Adsorbent Removal

1. Test the nitrogen atmosphere in the chambers for hydrocarbon. Depressure the chambers completely.
2. Be sure the chambers are properly blinded and isolated. Blind off the nitrogen supply.
3. Connect air lines to the chambers and pressure to 5 psig (0.35 kg/cm²g).
4. Depressure the chambers.
5. Repeat steps 3 and 4 two more times. Test the chambers' internal atmosphere for oxygen content.

6. If the O₂ level in the chambers are sufficient, open the top and bottom manways on each chamber. If not sufficient, continue with steps 3 and 4 until it is.
7. Remove all remaining unloading nozzles. We recommend vacuuming of adsorbent begin at this point.
8. Install air movers.
9. Cut out and remove the top grid distributor piping. Make a minimum number of cuts if the top distributor piping is to be reused. Carefully label each pipe section and record its location before it is removed so that the sections are returned to their original locations. The cuts should be as thin as possible. Any gaps will have to be filled with pipe spool pieces, necessitating extra welding. Spool pieces should be added as time allows so that one weld for each cut is removed from the critical path during reinstallation. All piping removed should be prepared for reinstallation as time permits.
10. Grind out or air arc out all top grid sections. Air arc is recognized to be the fastest method and is recommended. Some contractors have applied small diamond bladed rotary saws with success. The T-bars and seal angles must be cut carefully to avoid damaging the grids. Study the grid drawings to determine the best place to cut. Instruct the people who will be doing the work that they must be careful. Carefully remove and store the top snorkel sample probe. Label and record the position of each grid section before it is removed from the chamber. Also mark the chamber wall for grid location. The grid sections must be reinstalled in their original positions.
11. Some used adsorbent will still be in the top bed. This material should be vacuumed out. People working in the chambers must be instructed to move about very carefully to avoid damaging the grid screens. Soft sole shoes must be worn. Any screen damage will have to be repaired during the subsequent loading and will add to the total unit downtime.

12. When the grid is clean of sieve, install the precut cardboard and plywood screen covers and secure in place. The plywood should be cut to allow installation and removal around piping flanges, lifting lugs and holddown clips if present. Carefully mark the position of each grid section including the bed number. The grids must be reinstalled in their original locations.
13. Remove the distributor pipe. If it must be cut, make just one thin cut near the center of the chamber. Label each pipe section for location (position, bed number). Unbolt the flanges, saving the bolting. Remove the pipes from the chamber and store with the grids.

NOTE: It may be necessary to empty two top beds before beams or intermediate grids can be removed. Wedges for holddowns may require grinding to make leading/top edge smooth for reinstallation. Keep wedges from holddown clips.

14. To facilitate the removal of grid sections, the middle grid support beams will have to be removed if present.
15. All equipment removed must be marked to facilitate reinstallation. The marking should be permanent so they are not obscured by grid handling.
16. Continue removing grid sections and the remaining adsorbent from the chambers. Note any damage to the chamber, piping, or supports as work progresses.
17. Power or manual wire brushing of the chamber internal surface will remove residual adsorbent or rope packing.

b. Intermediate Grid Cleaning and Inspection

1. As the grids are removed from the chambers, they will have to be cleaned and inspected. Any spent adsorbent remaining on the grids should be removed with a wire brush. It is recommended that the grids also be cleaned

internally. Adsorbent fines and scale from the piping will collect in the grids and plug the screens and internal distributors. Plugging inside the grids will result in increased pressure drop and poor flow distribution during normal operation.

2. The dislodged material can be removed from the grid by drilling holes through the bottom grid flange – use steam and water to help dislodge any collected material and then redry the grid. Flushing with high pressure water followed by steam then nitrogen drying has proven effective. Two 1/4 inch (6 mm) holes will be required, one on each side of the internal baffle. Consult the grid drawings to determine the location of the baffle. When the section is clean, the holes should be covered with 1/8 inch (3 mm) thick carbon steel shims, 1/2 inch (12 mm) in diameter. Seal weld the shims over the holes so no adsorbent can leak into the grid. Figure XIII-38. For pie section grids, it is necessary to drill holes in both ends of the grids for cleaning.
3. The grids must be thoroughly inspected and repaired if necessary. See the section on loading for comments on inspection and repair.

c. Bottom Grid Inspection and Top Grid Reconditioning

1. When all adsorbent and grids, except the bottom grids, have been removed, the chambers should be thoroughly cleaned and inspected. This is also the time to refinish the top grid's seal angle continuous ring bar. Remove any remaining pieces of the old seal angle. The ring should be ready for welding of the new top grid seal angle before the bottom beds are loaded.
2. The top grid sections will have to be refinished at the storage area (unless new top grids have been purchased). Cut off the old T-bars and seal angles. Be careful not to damage the grids. Grind smooth all surfaces. Refinishing the top grids is a time consuming job and should be started immediately after the grids have been removed. It is recommended a specialist from the grid manufacturer supervise grid reconditioning.

3. Check the oxygen concentration of the bottom head before anyone enters.
4. Blind the snorkel and close the manway. Use temporary gaskets.
5. The bottom grid seal welding and bottom collector piping should be checked with a series of two pressure tests. The first test is a bottom grid weld integrity test. The method is identical to that which was used following the original bottom grid installation. See loading instructions.
6. After fixing all seal welding leaks, clean, inspect, and load adsorbent into the bottom bed using the normal loading procedure.
7. The bottom grid collector piping is tested for leaks after the bottom bed has been loaded with adsorbent. Prepare for the bottom grid screen integrity test by closing the top manway and all nozzles. Install a water manometer on the chamber shell. Open the bottom head manway. Carefully pressure the chamber to 500 mm H₂O with clean, dry air. Extreme care must be observed so that the bottom grid piping is not overpressured (see item 6 above). Check the bottom grid for leaks using a soap and water solution. Depressure the chamber and repair any leaks. Repeat the procedure until no leaks are found. Unblind the snorkel flange and install the 2 inch (50 mm) snorkel and the sample probe. Close the bottom head manway with a permanent gasket. Pressure the chamber with air to 5-7 psig (0.3-0.5 kg/cm²g) using a normal pressure gauge and continue with the normal bottom grid screen integrity test.
8. After a successful bottom screen integrity test, continue installing grids and loading adsorbent using the procedures listed for loading.

APPENDIX I



DUR O LOK™ COUPLINGS INSTALLATION PROCEDURE

1. Prepare DUR O LOK Coupling for Welding

- The welding surfaces of the coupling and the mating pipe must be cleaned and prepared for welding. Be sure to remove any dirt, grease, or other surface contaminants prior to welding. Also, be sure to remove plastic protective caps prior to welding, as they will melt. If the coupling has a black oxide finish, lightly grind or sand off the black oxide coating from the welding surfaces of the coupling. Removal of the black oxide coating from the coupling's welding end may have already been done at the factory for your convenience.
- For applications that involve catalyst transfer (e.g. - **UOP** CCR Platforming), it is recommended that the black oxide coating is removed from the entire inner surface of the coupling.

2. Weld DUR O LOK Coupling to Mating Pipe

- It is highly recommended that the DUR O LOK Coupling be welded to the mating pipes as an assembled unit when possible, as this will ensure proper alignment and protect machined surfaces. DUR O LOK Couplings have higher installation tolerances than standard flanges, and extra care must be taken during welding alignment.

Warning: Do not have gasket in place during welding as damage will occur.

- For installation of DUR O LOK Couplings in catalyst lift lines, or similar services where long piping runs are involved, tack weld the assembled coupling to both pieces of pipe positioned horizontally along a flat surface. Care must be taken to ensure that the two pieces of pipe are straight and aligned prior to welding. 80 feet of (shop floor) space is required to position and weld two 40 foot sections of pipe to a DUR O LOK Coupling. The tacked DUR O LOK Coupling may be disassembled prior to completing the circumferential weld.
- For pipes involved in catalyst transfer, a smooth transition at the inside surface for all circumferential welds is mandatory. The inside diameters of the pipes

and welded DUR O LOK Coupling may require grinding to remove all ledges to produce a flush transition in the area of the circumferential welds. The final wall thickness of the pipes and DUR O LOK Coupling must not be less than the minimum required wall thickness.

- For pipes employing precision bends (e.g. CCR catalyst transfer lines and related applications), trial installation in the shop is highly recommended, as it allows for final adjustments to be made. DUR O LOK Couplings have higher installation tolerances than standard flanges, and extra care must be taken during final alignment. Pipe sections must be matched marked after trial fit-up is complete and on-spec. This will facilitate trouble-free installation of the pipe assemblies in the field.
- For non-horizontal coupling installations, ensure that the female hub (hub with gasket cavity) is positioned as the lower half of the coupling.
- Welding of 304SS DUR O LOK Couplings to carbon steel pipe is acceptable.
- Reference Appendix 1, UOP Standard Drawing 8-127- (latest revision), Notes 2 and 5.

Warning: Protect Machined Surfaces of Coupling from Weld Splatter.

3. Position Tapered Retaining Ring

- Place the tapered retaining ring over the male hub end and slide it past the grooves on the hub for the split-coupler. The narrower end of the tapered retaining ring must be orientated toward the mating (gasket) surface of the coupling. The two set screws should be located in the half of the tapered retaining ring closest to the mating surface of the coupling.
- Reference Appendix 1, UOP Standard Drawing 8-127- (latest revision), Note 1.
- Bring the two hub ends welded to the pipe segments into approximate assembled position.

4. Install Gasket

- DUR O LOK Couplings are offered in a standard and high temperature design. Standard design couplings employ an elastomeric type o-ring seal and are used for applications typically below 450°F (232°C). The type of o-ring determines the temperature limit. Materials of construction for o-rings include Viton, Silicone, Kalrez, and many others – depending on application requirements. Although o-rings are typically reusable if not damaged, it is strongly recommended that a used o-ring be replaced. DUR O LOK Couplings employ standard size o-rings, available from your local industrial equipment supplier.
- UOP offers a high temperature DUR O LOK Coupling design which employ a graphite type flat gasket (or equivalent) for nominal service temperatures above 450°F (232°C). A DUR O LOK installation clamp may be required to

compress the installed gasket and properly assemble the high temperature DUR O LOK Coupling. These gaskets are not reusable - they are for one-time use only. These custom sized gaskets are available only through UOP. Please specify DUR O LOK Coupling pipe size and pipe schedule when ordering.

Warning: Low Temperature and High Temperature Design DUR O LOK Couplings and their Gaskets are NOT Interchangeable.

- Closely examine the gasket cavity of the female hub. Ensure that the cavity is free from oil, debris, scratches, or anything that may affect the quality of the seal.
- Place the gasket in the gasket cavity of the female hub.
- Handle gaskets with care in order to avoid damage.

Warning: Damaged gaskets must not be used, as they will not seal properly. Always Replace Used Gaskets to Ensure Proper Seal. High Temperature Gaskets are for One-Time Use Only.

5. Assemble Coupling

UOP offers a special clamp used to bring together hubs that are slightly misaligned. If this clamp is to be used follow step 5b, otherwise proceed with step 5a (*and then skip 5b.*)

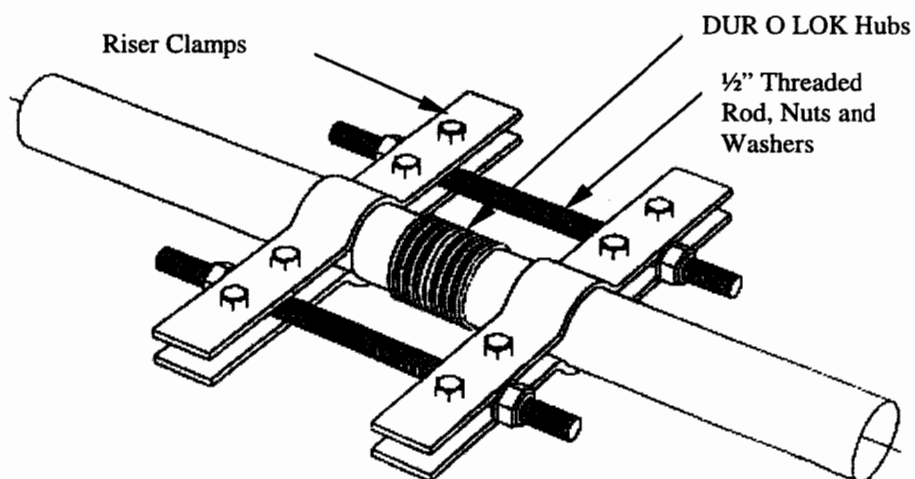
5a. Assemble Coupling

- With the gasket in place, draw the hub ends together. If the welded pipes are not aligned properly, a clamp will be required to draw the two halves of the DUR O LOK Couplings together. If desired, a clamp can be constructed in your shop using standard riser clamps, as depicted in Figure A. Couplings that are excessively out of alignment will require removal, repositioning, and re-welding. It is best to take care to align the piping and couplings prior to welding.
- Place the two split couplers in position with the set screw groove in the split couplers being positioned over the female hub (hub with the gasket).
- Slide the narrow edge of the tapered retaining ring over the split couplers until the tapered retaining ring is in position with the set screw holes directly over the set screw groove in the split couplers. Use a soft faced hammer (e.g. nylon or rubber) to drive the tapered retaining ring into a fully engaged position.

Warning: The tapered retaining ring must be fully engaged to attain the design compression and pressure rating for the desired service. The tip of the set screws must fit completely inside the split coupler's set screw groove. The coupling could become inadvertently disassembled

from vibration during use if not assembled properly. The result may be process material leakage.

Figure A
Shop-made DUR O LOK Coupling Clamp Constructed from Riser Clamps



5b. Assemble Coupling Using Clamp

- Couplings that are excessively out of alignment will require removal, repositioning, and re-welding. It is best to take care to align the piping and couplings prior to welding.
- Select the appropriate size clamp for each application. See Figure C for the location of the DUR O LOK size marking. Use size 1 for 1", 1¼", and 1½" DUR O LOKs. Use a size 2 clamp for 2", 2½" and 3" DUR O LOKs.
- Determine the appropriate adjustable hinge slot for each location. See Figure D for the location and description of the adjustable hinge slots. Set the adjustable hinge to slot 1 for a 1" or 2" DUR O LOK, slot 2 for a 1¼" or 2½" DUR O LOK, and slot 3 for a 1½" or 3" DUR O LOK.
- Remove the secondary driver. Place clamp around DUR O LOK. Hold the lower jaw flush with the lower hub collar on the DUR O LOK and tighten the knurled knob. Loosely engage the upper jaw around the upper DUR O LOK hub. Use the primary driver to lower the upper jaws until they come into contact with the upper hub collar. Replace the secondary driver. Tighten the knurled knob on the upper jaw while holding the upper jaw flush against the upper hub collar.
- Using the primary driver, tighten the clamp until the hubs come into contact. Use the secondary driver to level the hubs as needed.
- Place the two split couplers in position with the set screw groove in the split couplers being positioned over the female hub (hub with the gasket).
- Loosen the jaws using the knurled knobs, and then loosen the clamp and remove.
- Slide the narrow edge of the tapered retaining ring over the split couplers until the tapered retaining ring is in position with the set screw holes directly over the set screw groove in the split couplers. Use a soft faced hammer (e.g. nylon or rubber) to drive the tapered retaining ring into a fully engaged position.

Figure B
DUR O LOK CLAMP

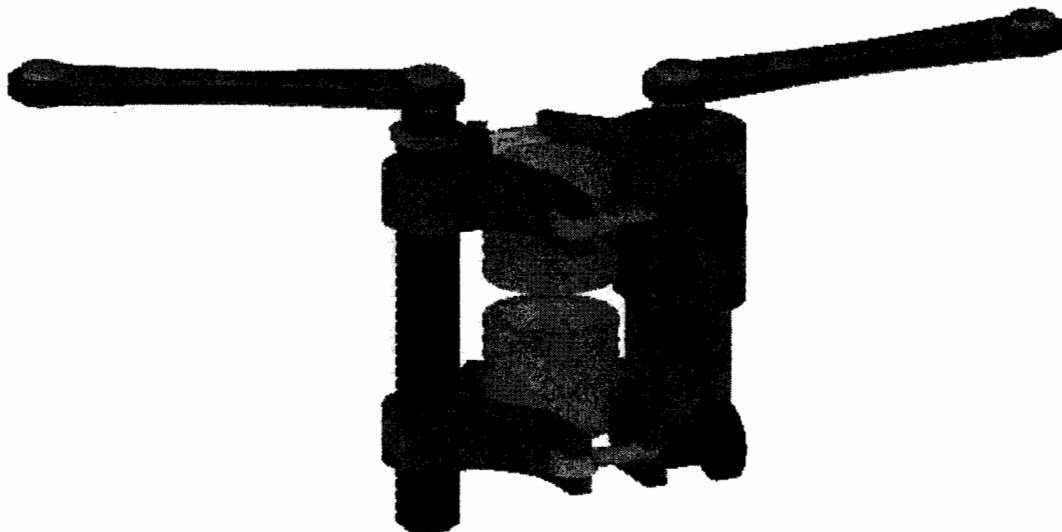


Figure C
DUR O LOK Clamp Part Identification

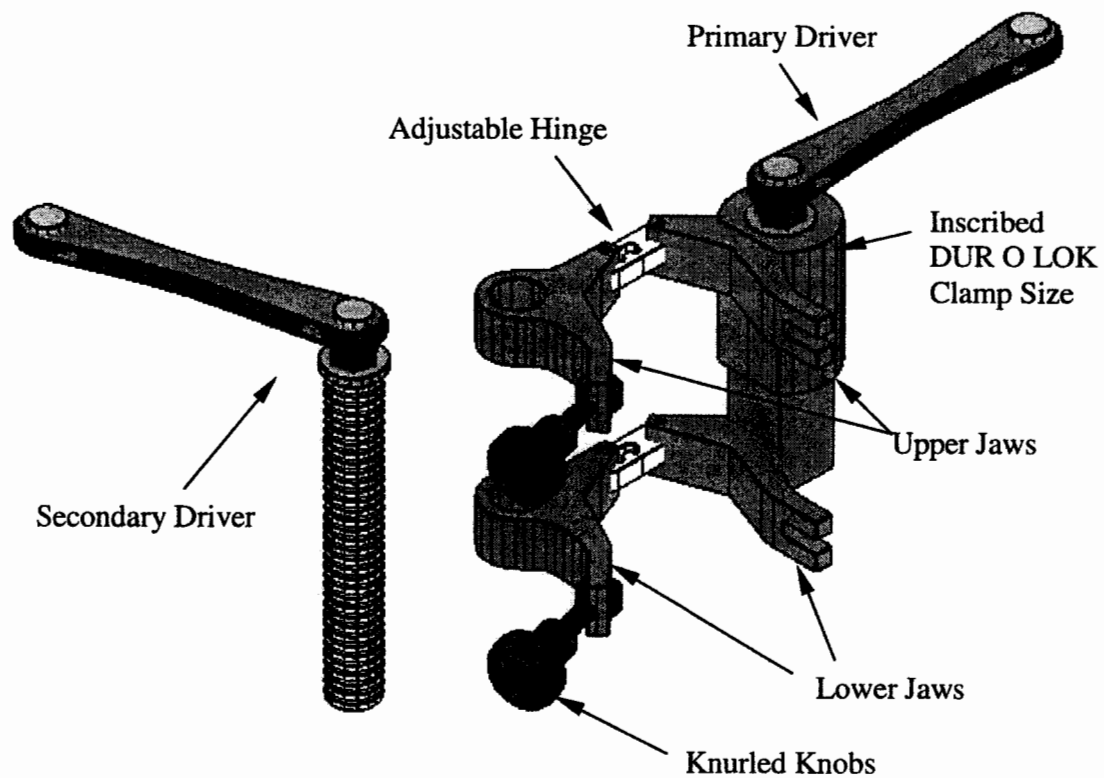
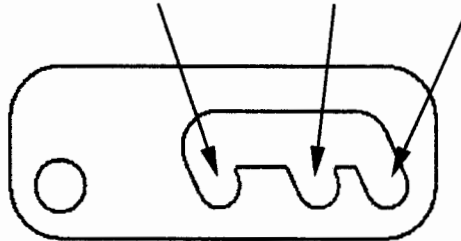


Figure D
Adjustable Hinge

	<u>Slot 1</u>	<u>Slot 2</u>	<u>Slot 3</u>
<u>Size 1</u>	1"	1 ¼"	1 ½"
<u>Size 2</u>	2"	2 ½"	3"



6. Secure Coupling

- Install and hand tighten the set screws with the supplied 3/16" hex key (allen wrench). The function of the set screws are to prevent the tapered collar from becoming disengaged, and to contain the safety wire. The set screws do not function to pull the tapered retaining ring into position. Do not over-tighten, as damage to the threads may occur.
- Install the safety wire through the holes in both set screws and wrap the wire once around coupling.
- Twist the safety wire a minimum of five times such that the set screws can not work loose during operation. Safety wire twisting-cutting pliers are available for this purpose.
- The presence of the safety wire generally is indicative that a gasket is in place. The DUR O LOK Couplings can also be tagged or marked to indicated "gasket installed".

Figure E
Exploded View of DUR O LOK Coupling

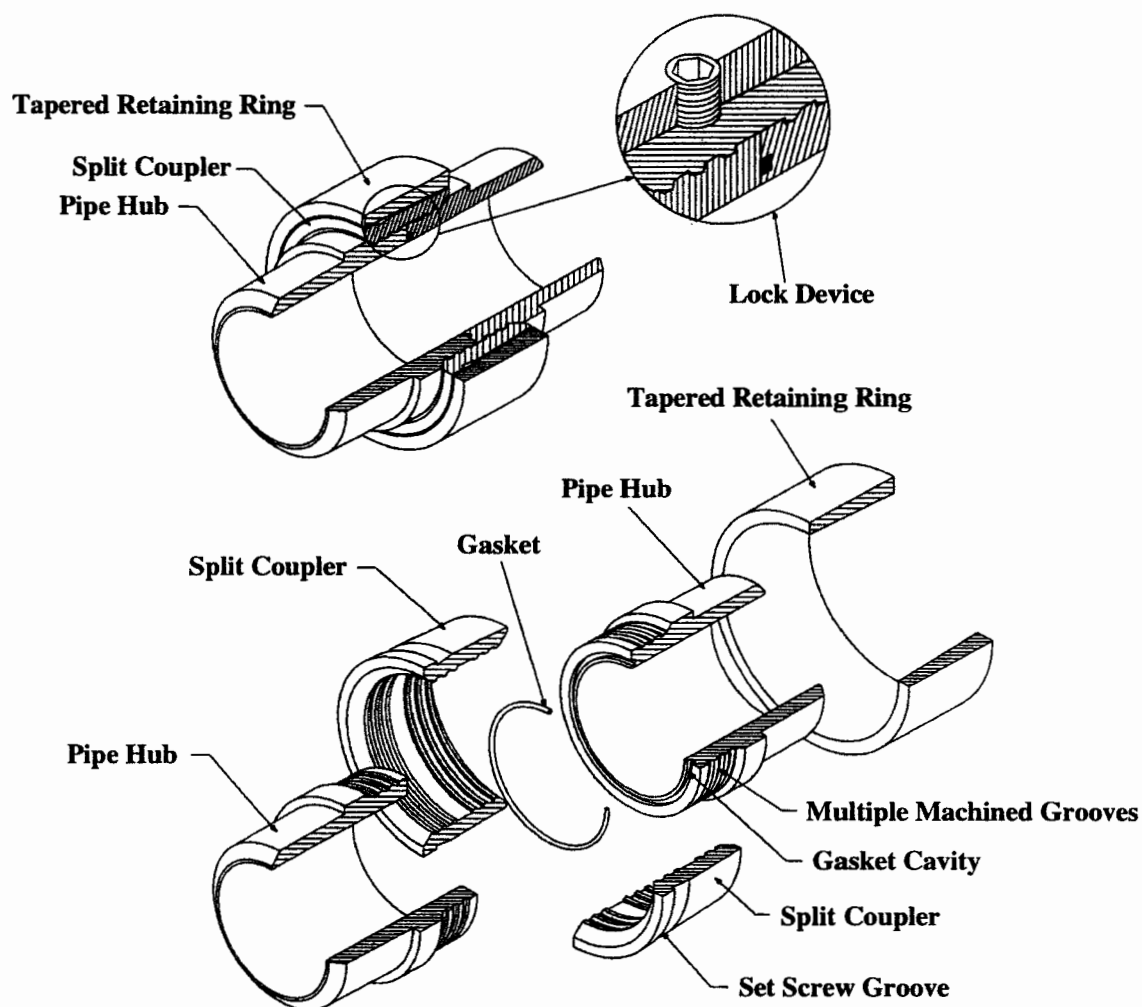


Figure XIII-1
Adsorbent Chamber Layout
Segmental Grid Design

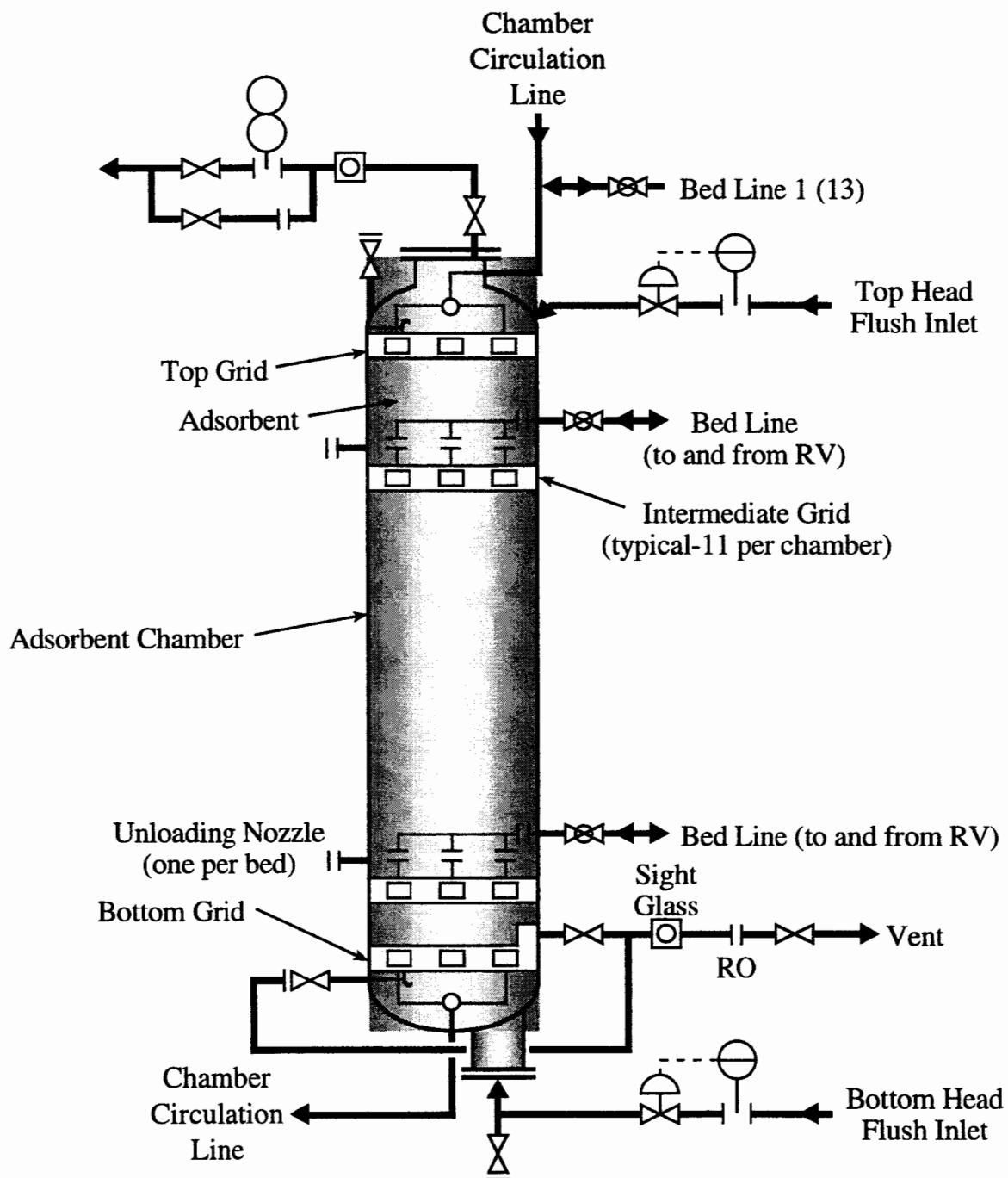
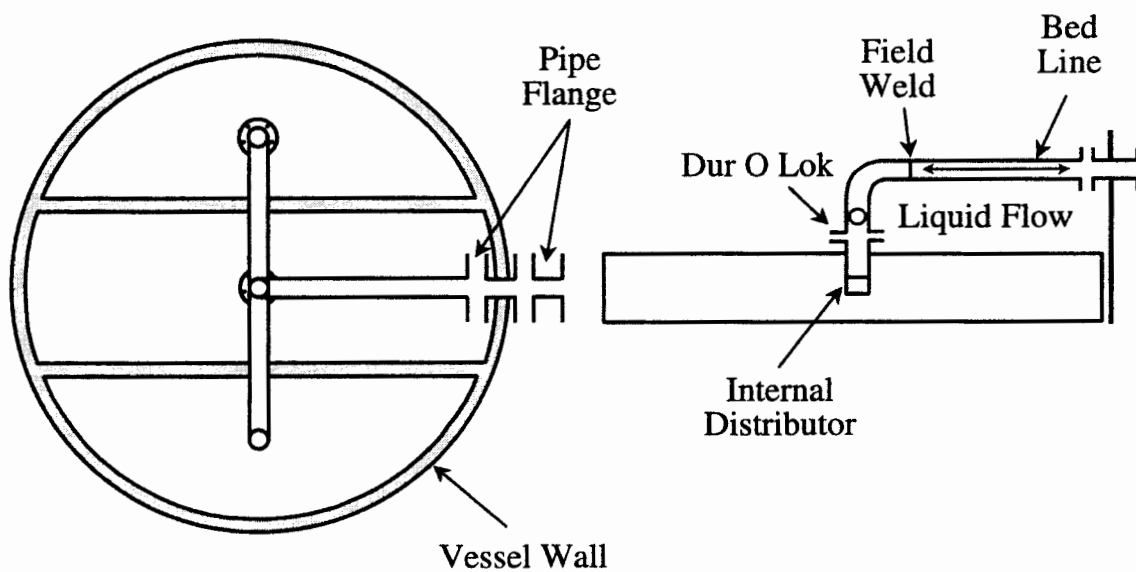


Figure XIII-2
Segmental Grid Design



In the segmental grid design, the grids are connected with external piping, which is then piped to the bed line.

PRX-R00-13

Figure XIII-3
Adsorbent Chamber Internals

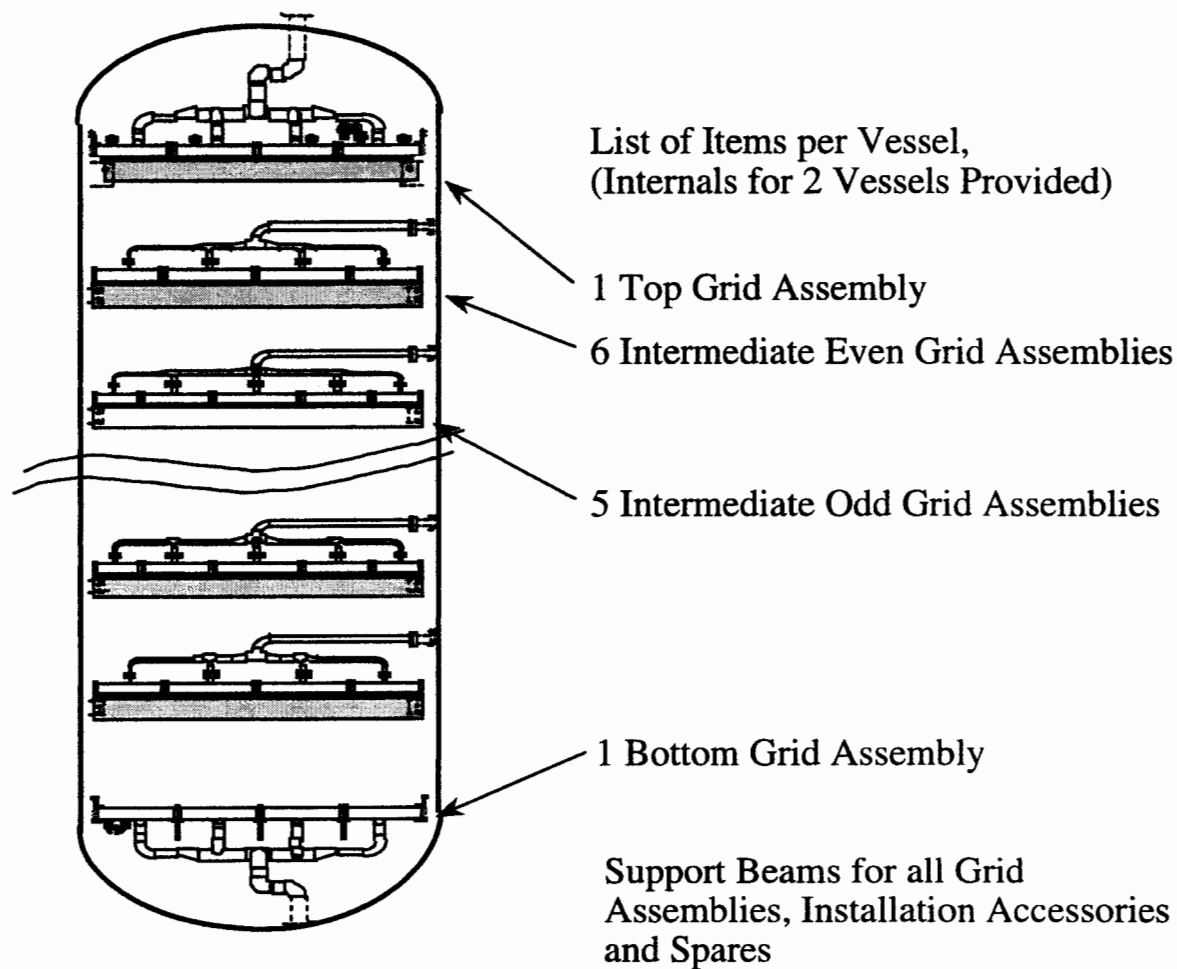
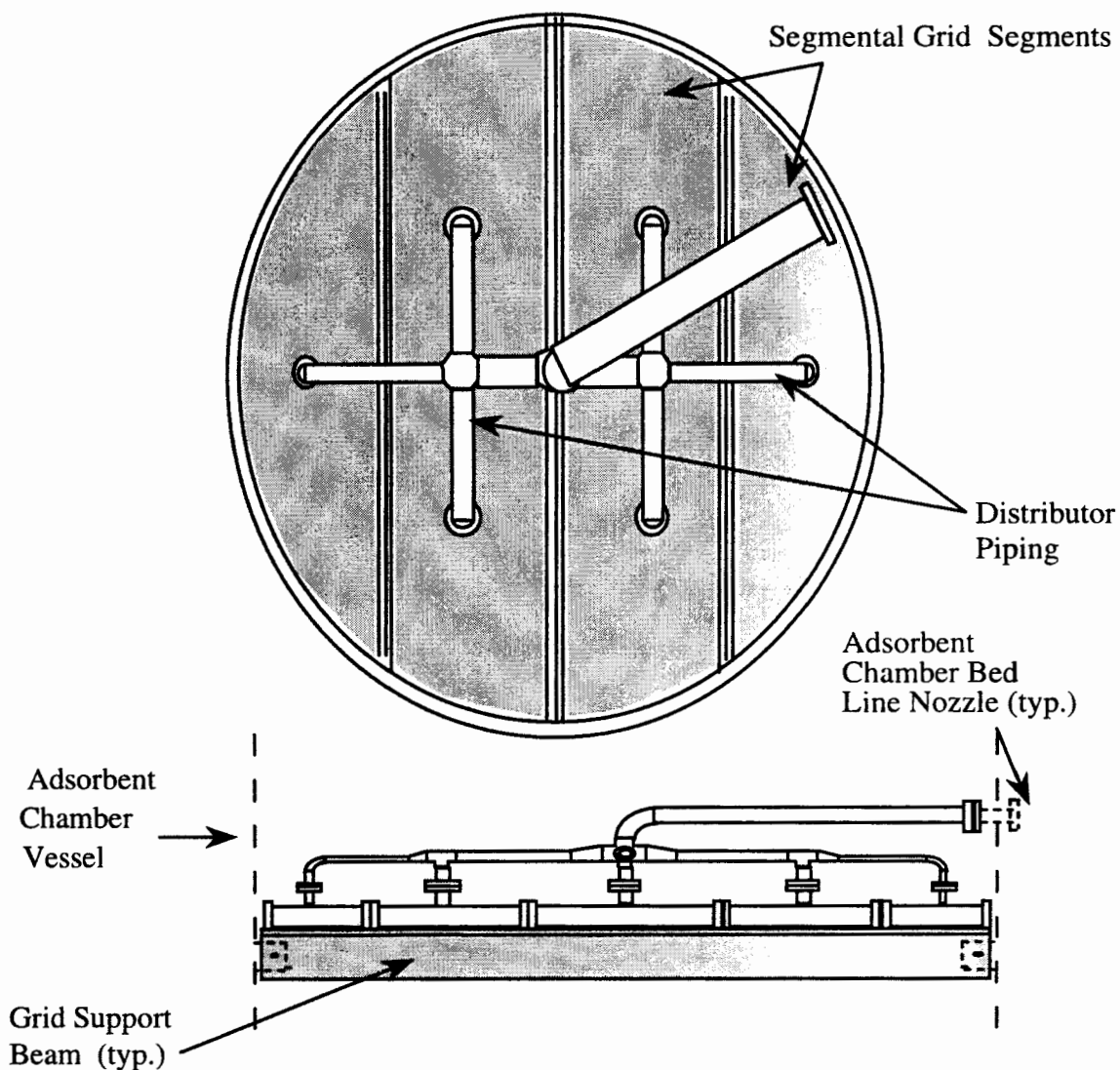


Figure XIII-4
Adsorbent Chamber Internals
Description of Equipment
Intermediate Assembly



Installation Accessories

- Gasketing / Bolting - Internal Flanges and UOP Dur-O-Loks used for piping connections
- Ceramic Rope - Vessel Perimeter
- Beams Installed and Leveled Previously

Figure XIII-5
Grid Lifting Equipment

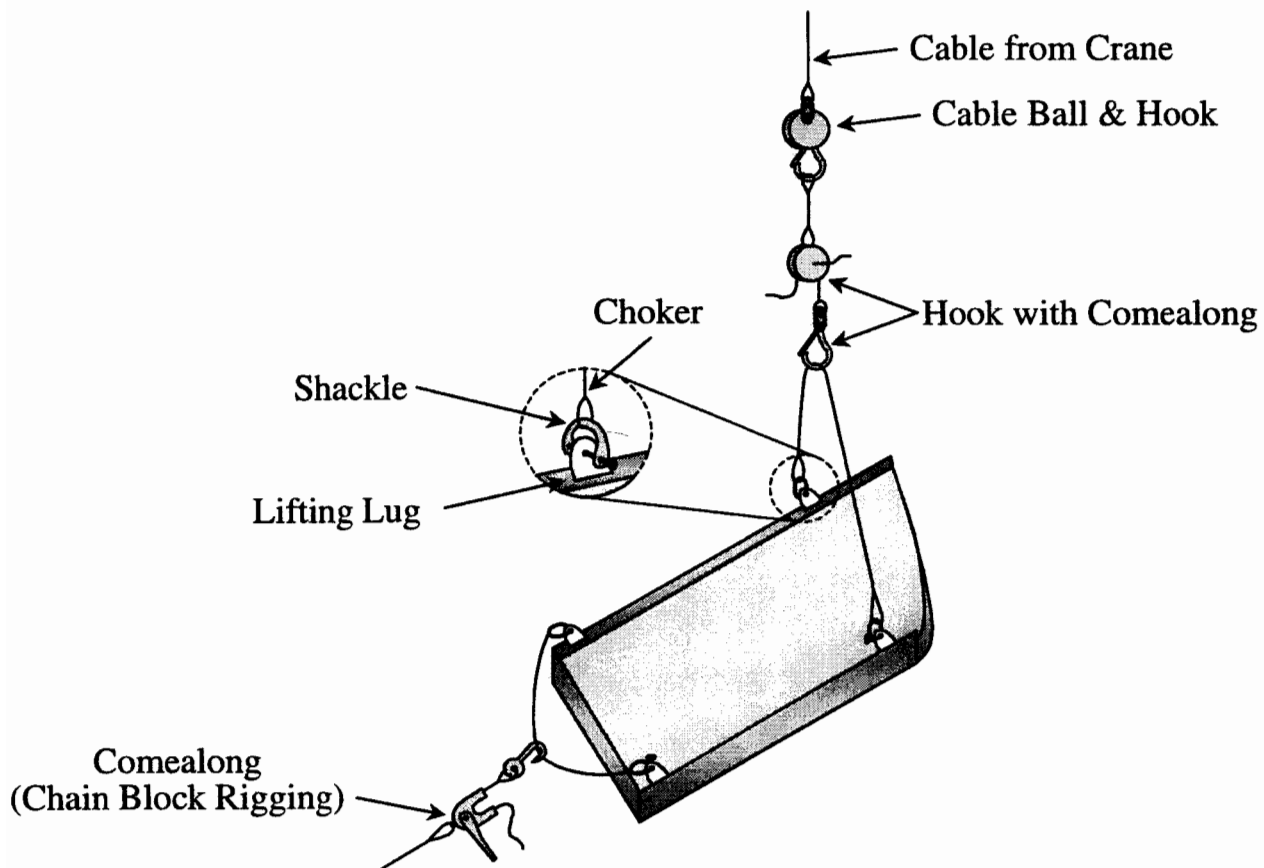
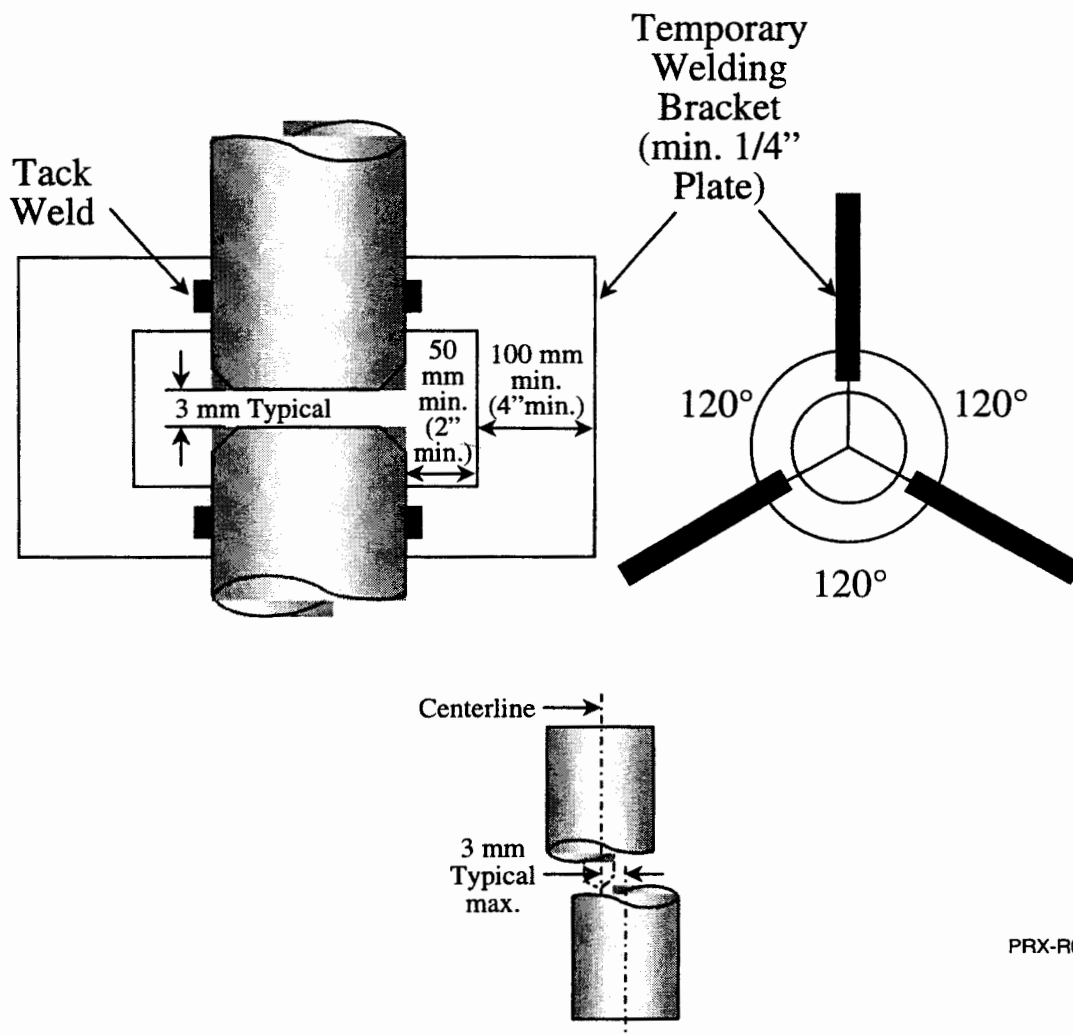
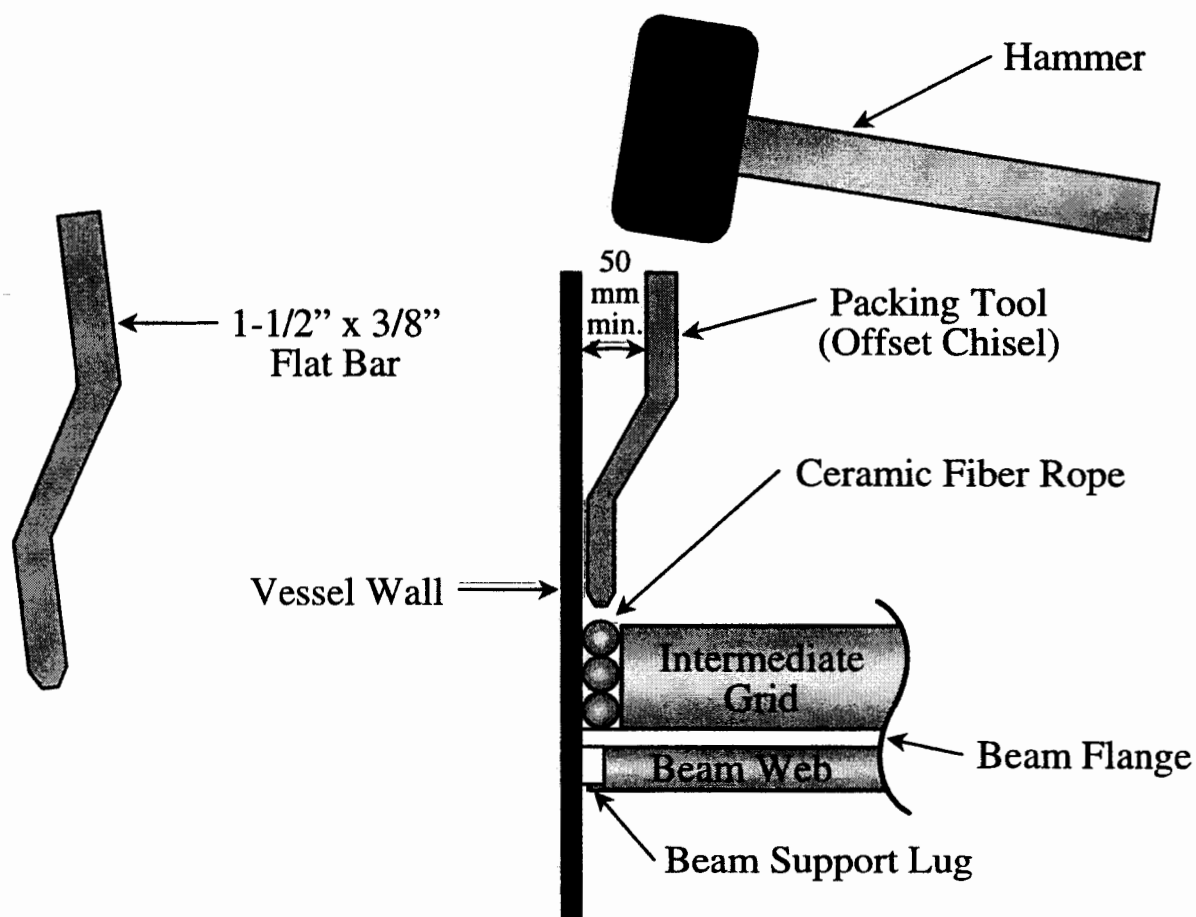


Figure XIII-6
Distributor Pipe Welding Alignment



PRX-R01-29

Figure XIII-7
Ceramic Fiber Rope Packing Tools



PRX-R00-47

Figure XIII-8
Level Checking Apparatus
Water Manometer

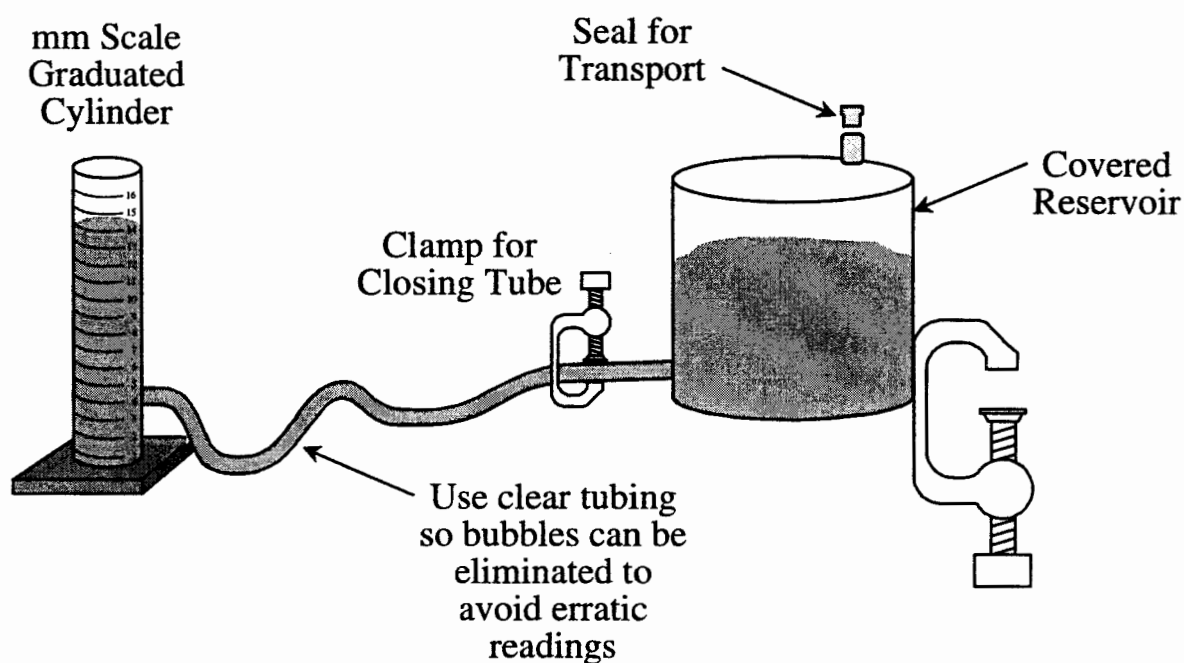
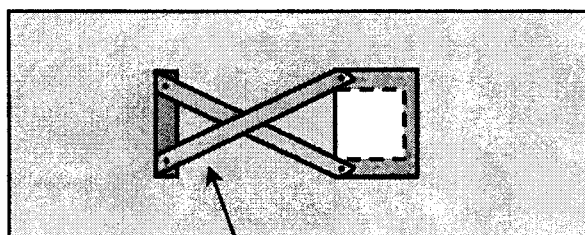


Figure XIII-9
Sieve Loading Shoes



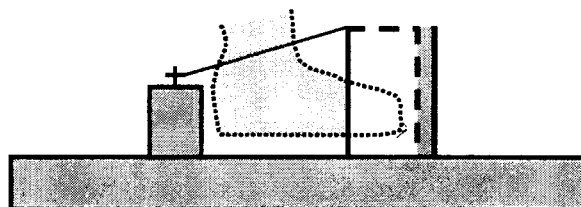
Polyester, Nylon, or
Leather Straps (must
hold sieve loading
shoe to worker's foot)

Surface Area Ave.
Worker's Weight
(lbs) / 0.8 psi = in²

$$\text{Surface Area, in}^2 = \frac{\text{Worker's Weight (lbs)}}{0.8 \text{ psi}}$$

$$\text{Surface Area, cm}^2 = \frac{\text{Worker's Weight (kg)}}{0.056 \frac{\text{kg}}{\text{cm}^2}}$$

1/2" (12 mm) Plywood
typical size 300 mm
x 500 mm



No sharp protrusions on bottom

**Figure XIII-10
Initial Beam Installation
Safe Rigging**

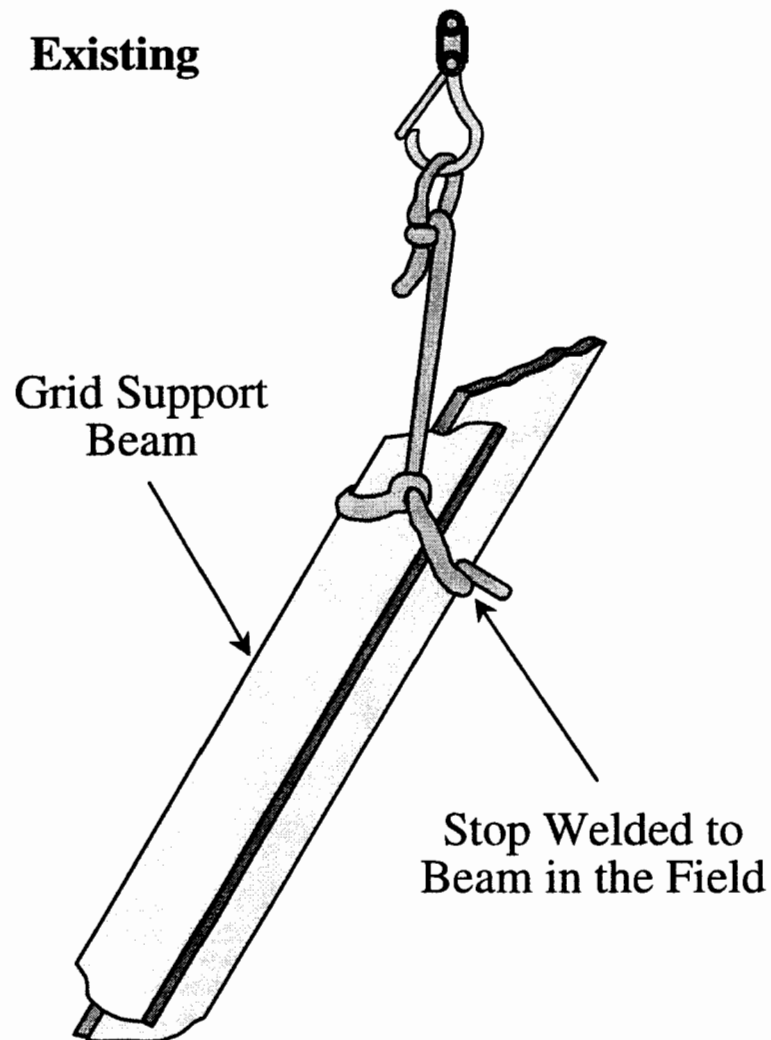


Figure XIII-11
Initial Beam Installation
Safe Rigging

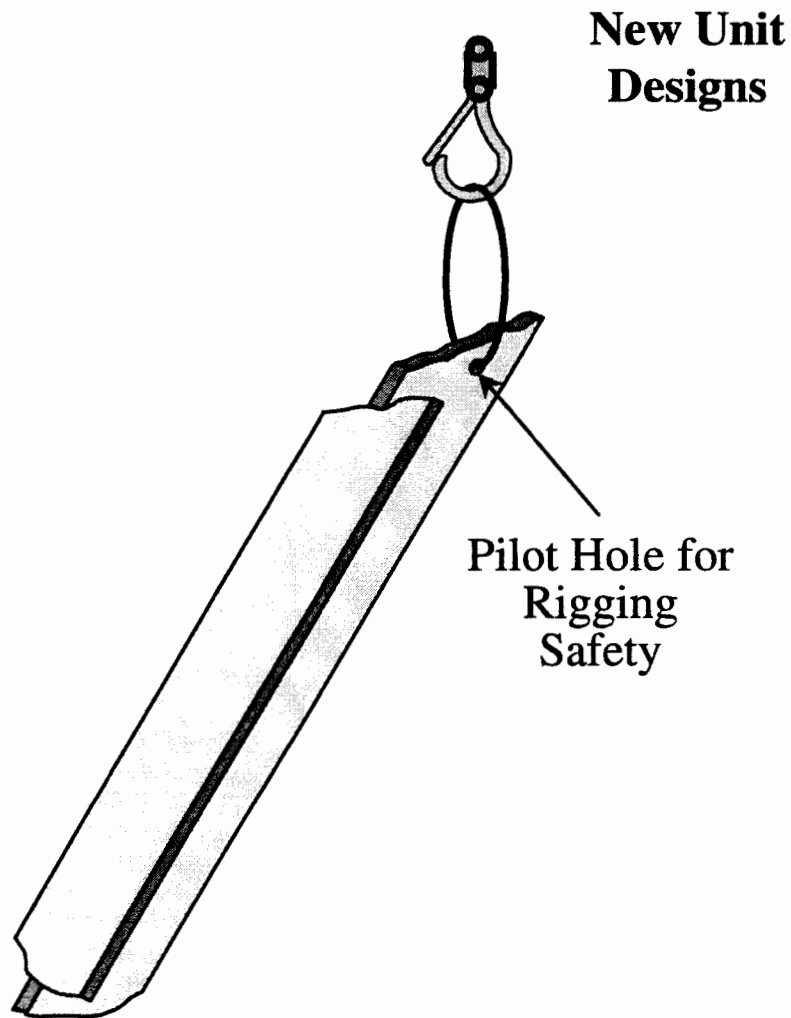
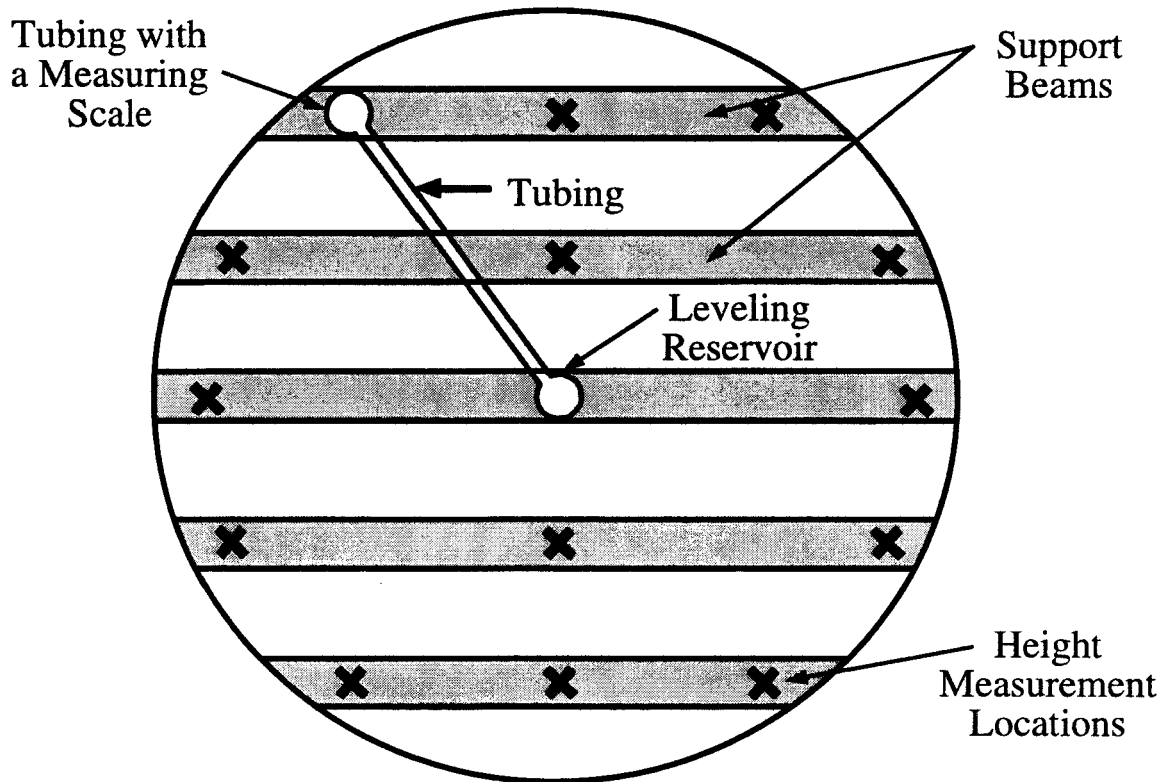


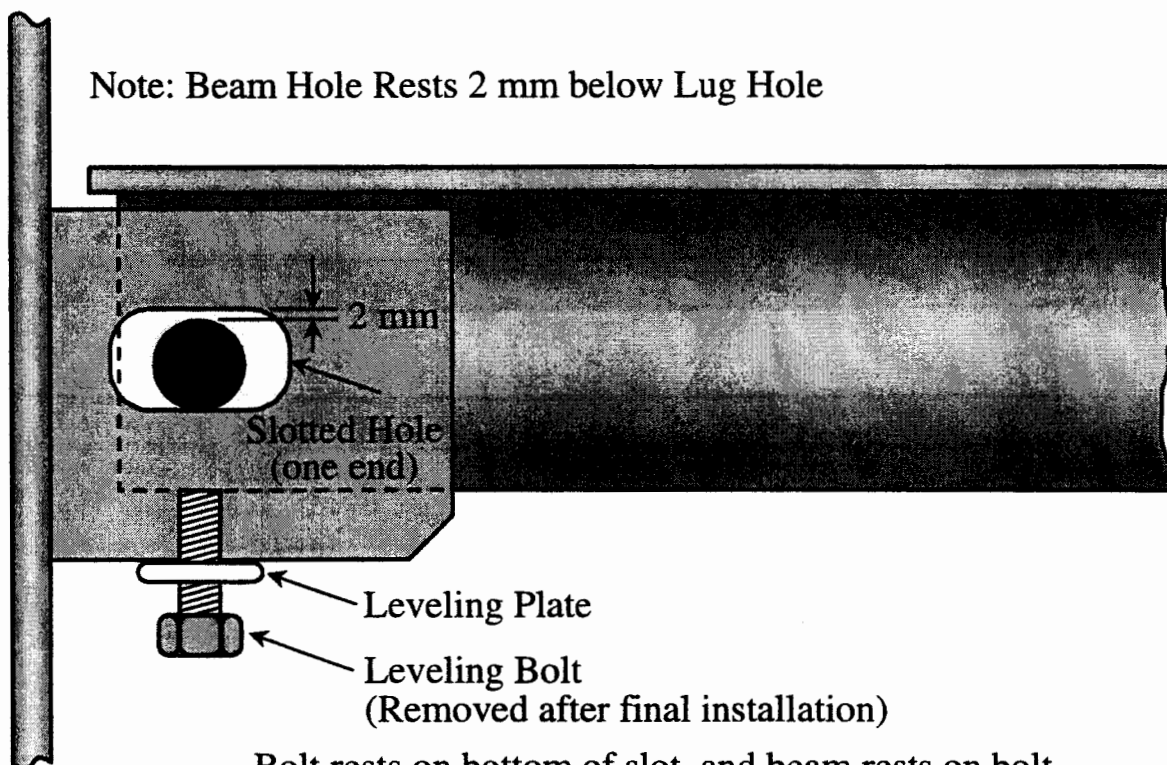
Figure XIII-12
Beam Leveling Chamber (Top View)



PRX-R00-22

Figure XIII-13
Beam Drilling
Segmental Shape Grids Only

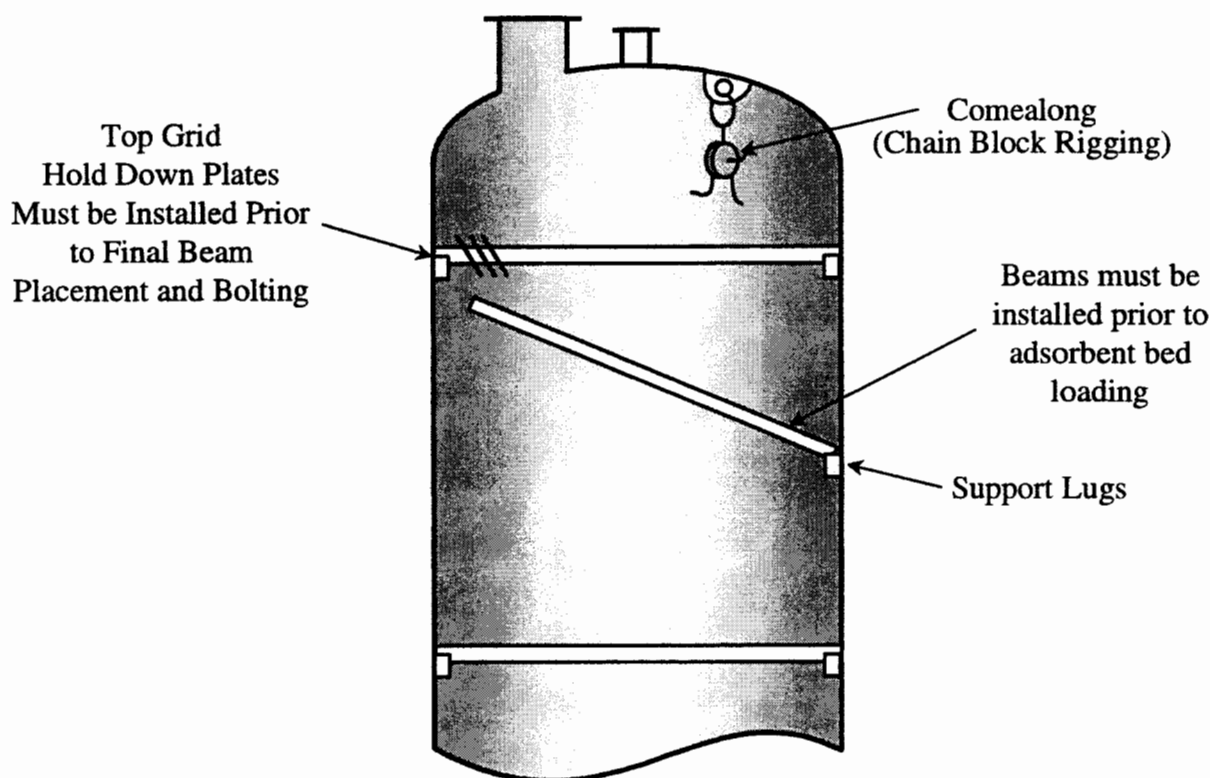
Note: Beam Hole Rests 2 mm below Lug Hole



Bolt rests on bottom of slot, and beam rests on bolt.
The bolt does not fit the slot, but is 2 mm smaller.

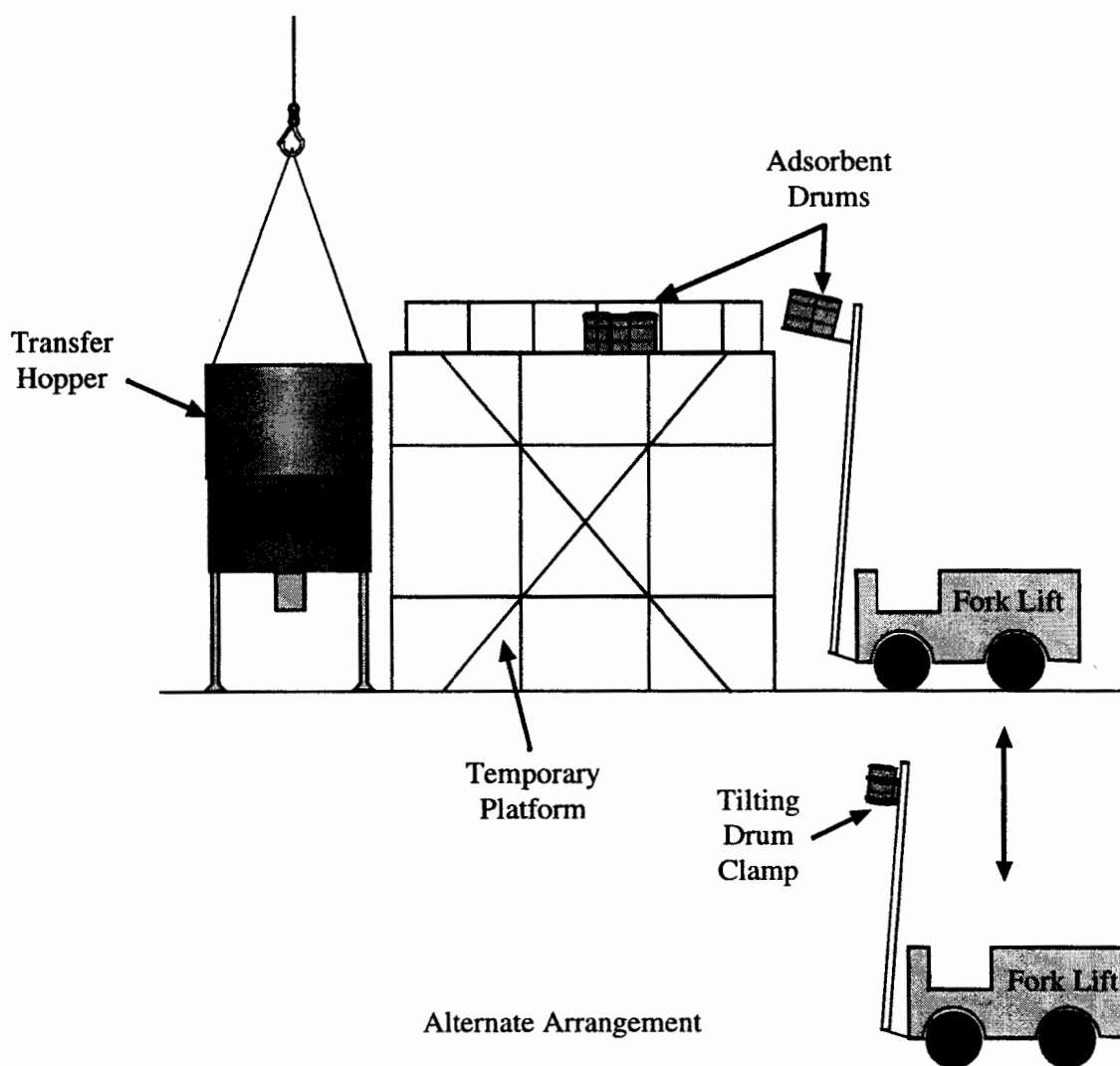
PRX-R00-19

Figure XIII-14
Beam Installation



PRX-R00-18

Figure XIII-15
Filling Transfer Hopper
(Drawing not to scale)



PRX-R00-24

***Figure XIII-16
Inspection Platform***

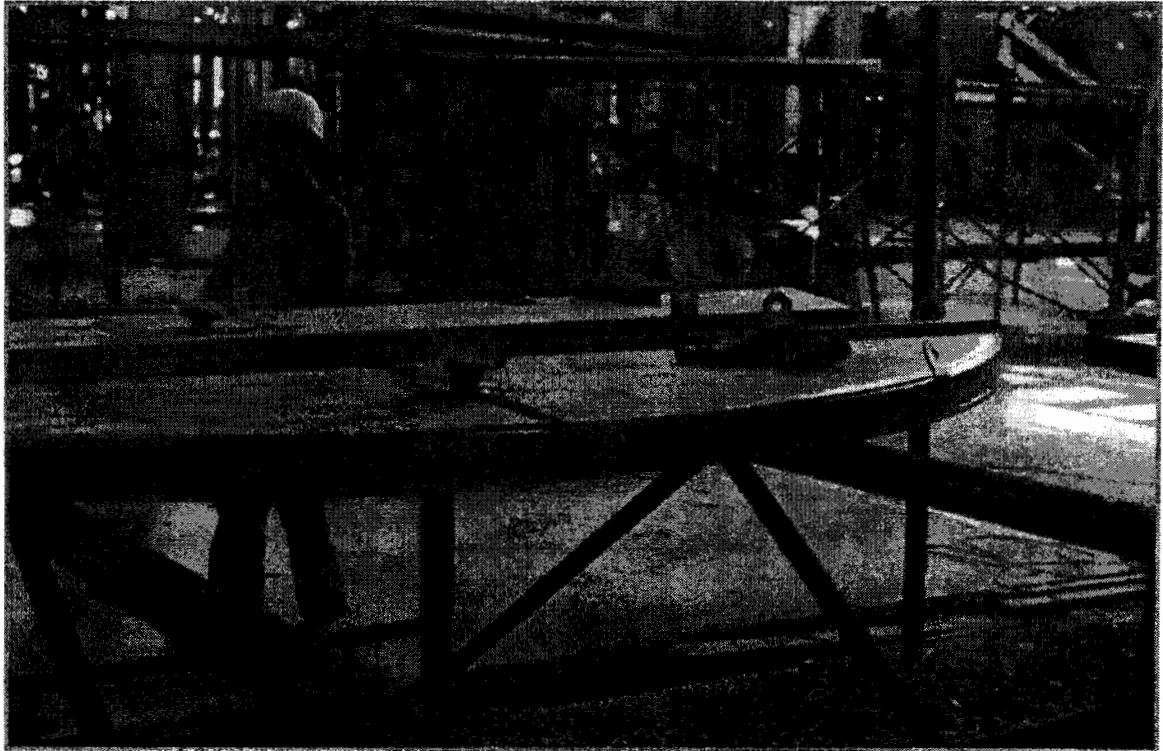


Figure XIII-17
“T”-Bar Welding at Seal Plates

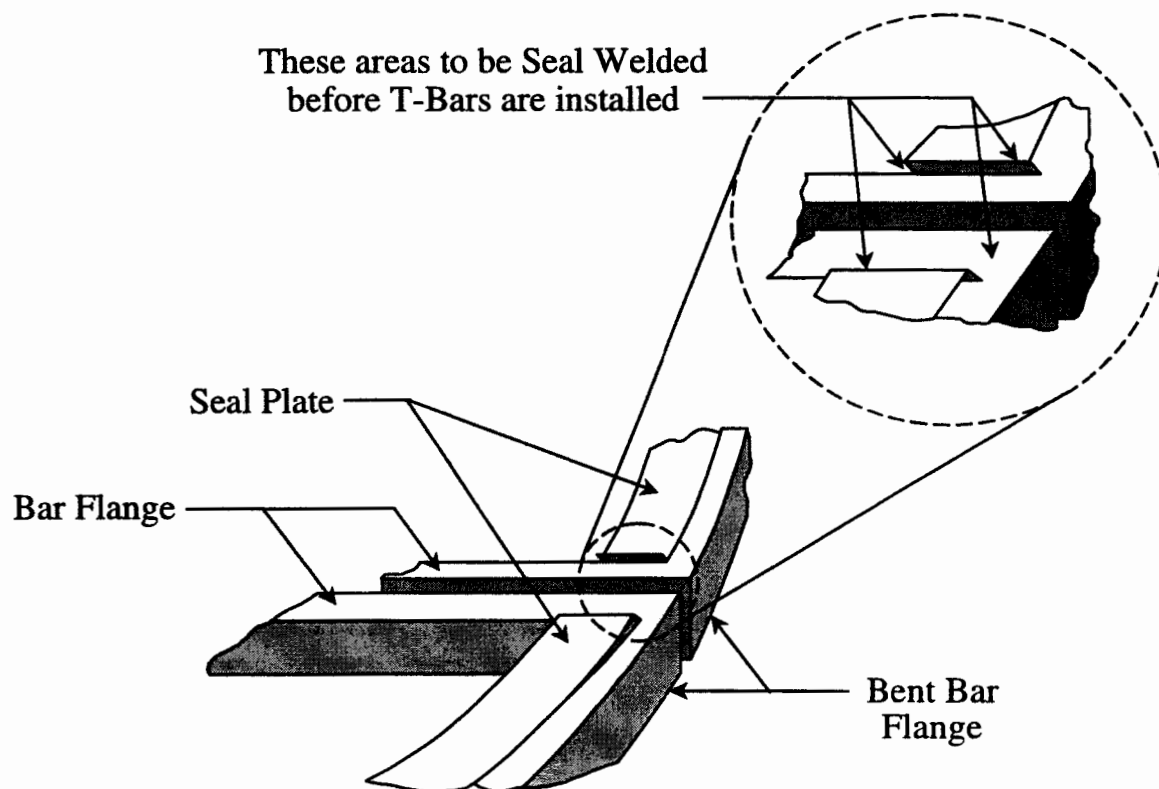


Figure XIII-18
Placing Grid Section

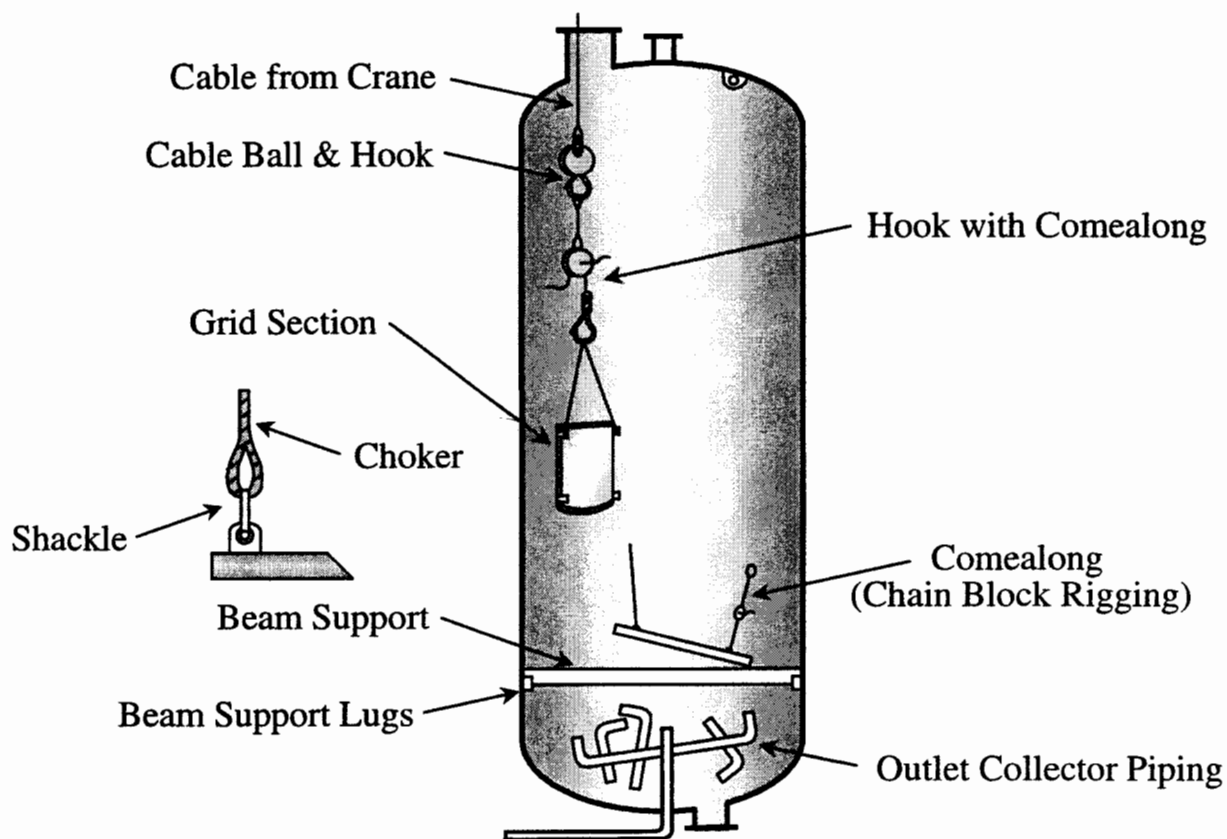
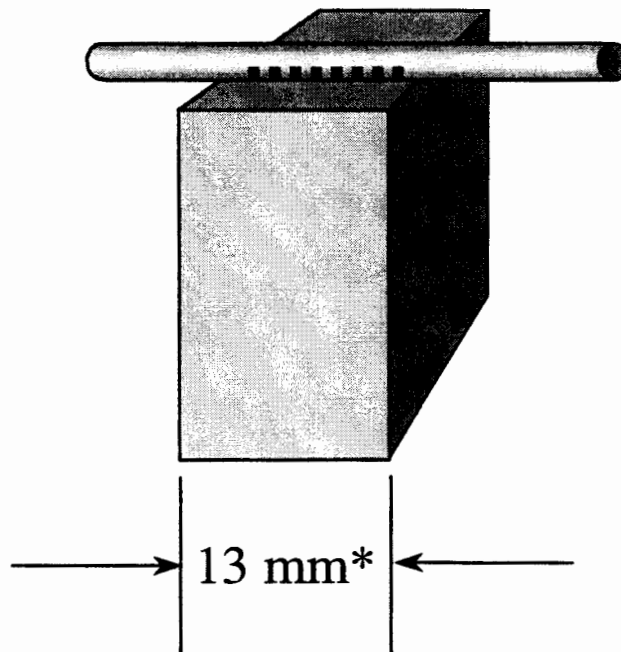


Figure XIII-19
Grid Spacer Bar



* To be confirmed with actual dimension on drawing

Figure XIII-20
Grid Spacer Bars

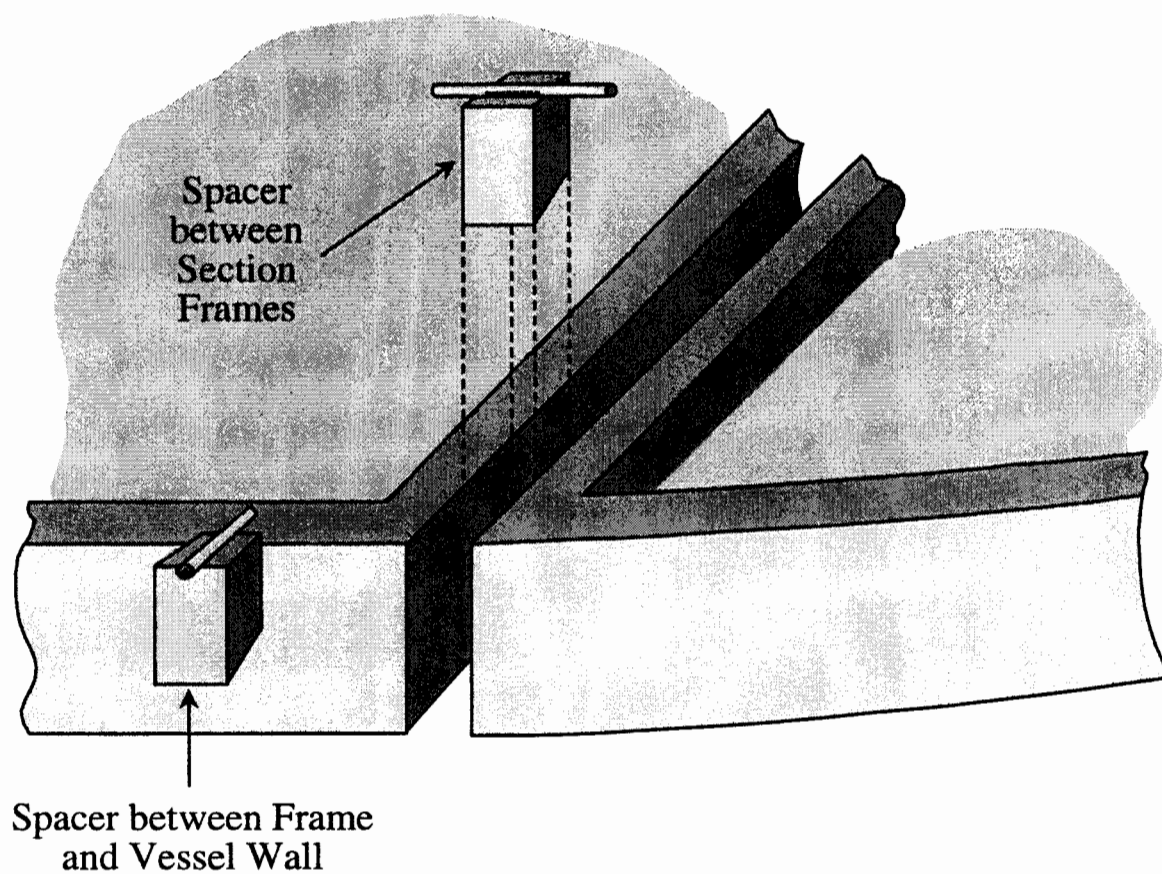


Figure XIII-21
Hold Down Lugs for Bottom Grid

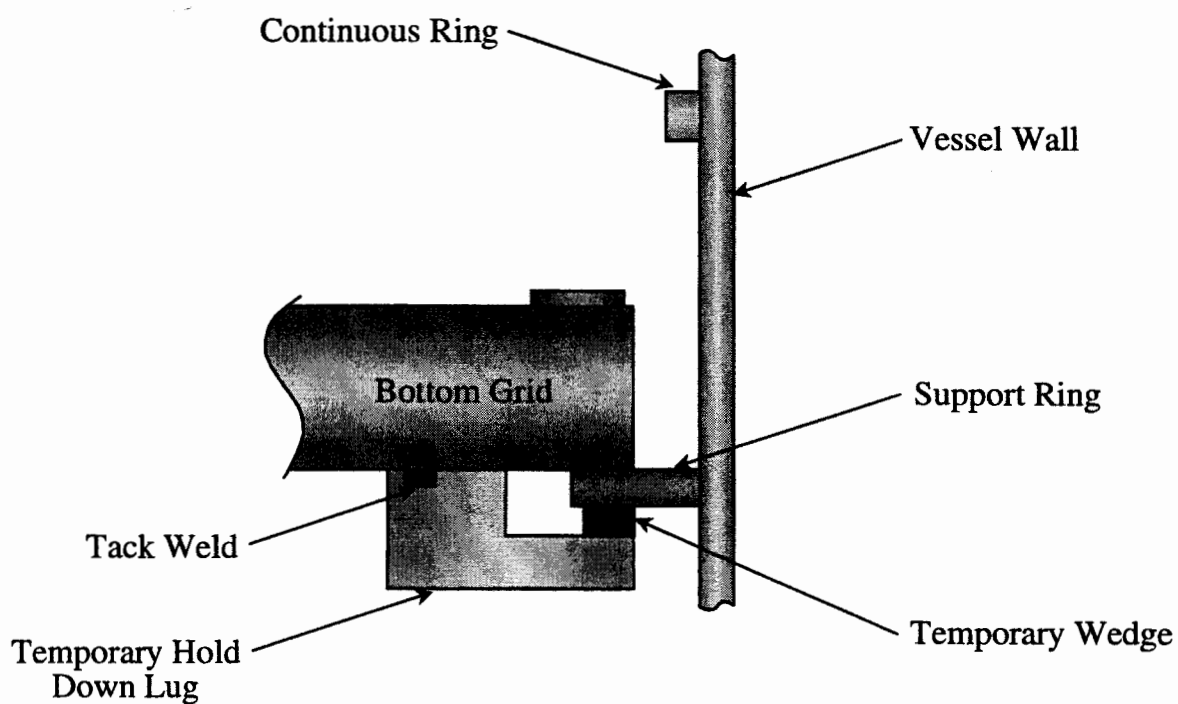


Figure XIII-22
“T”-Bar Welding

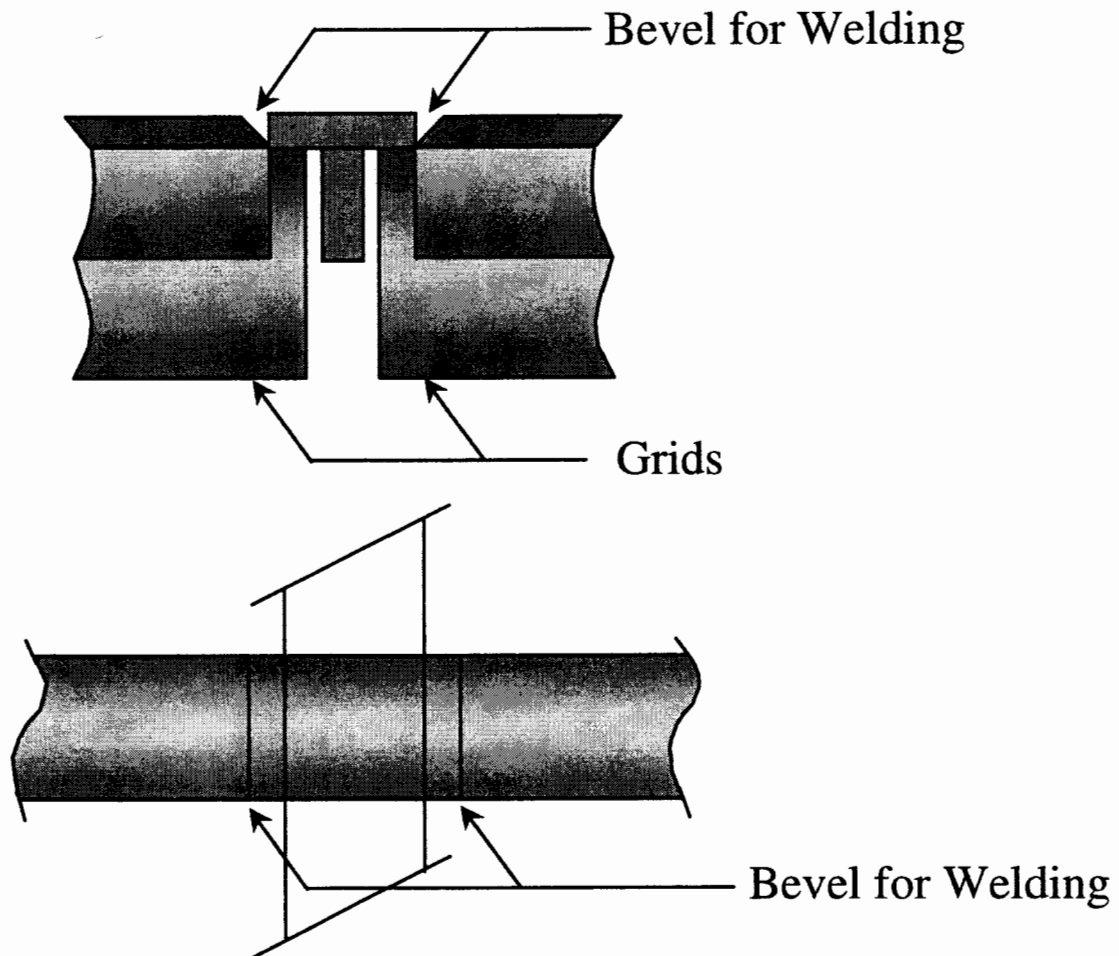


Figure XIII-23
Top and Bottom Grid Installation

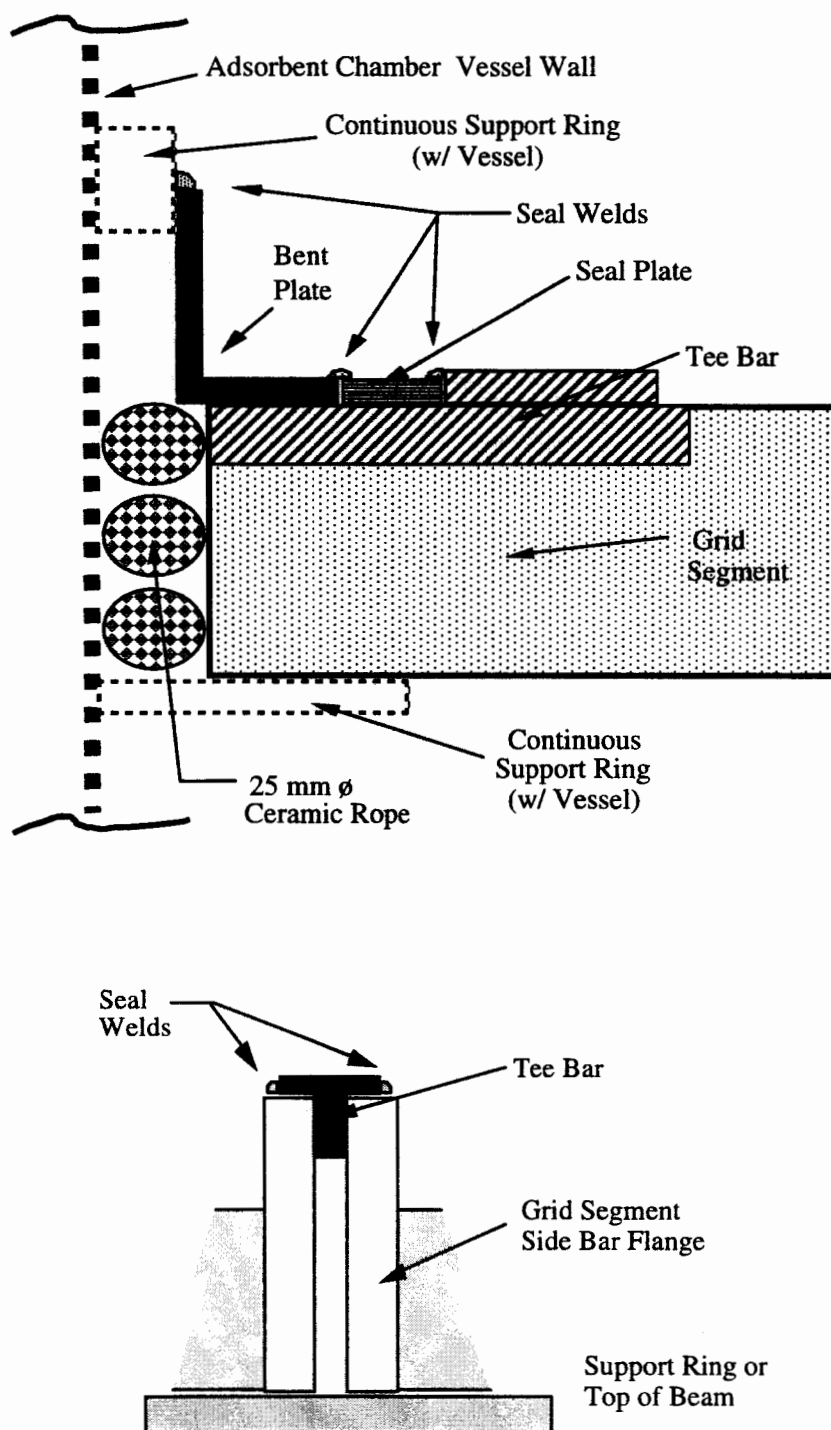
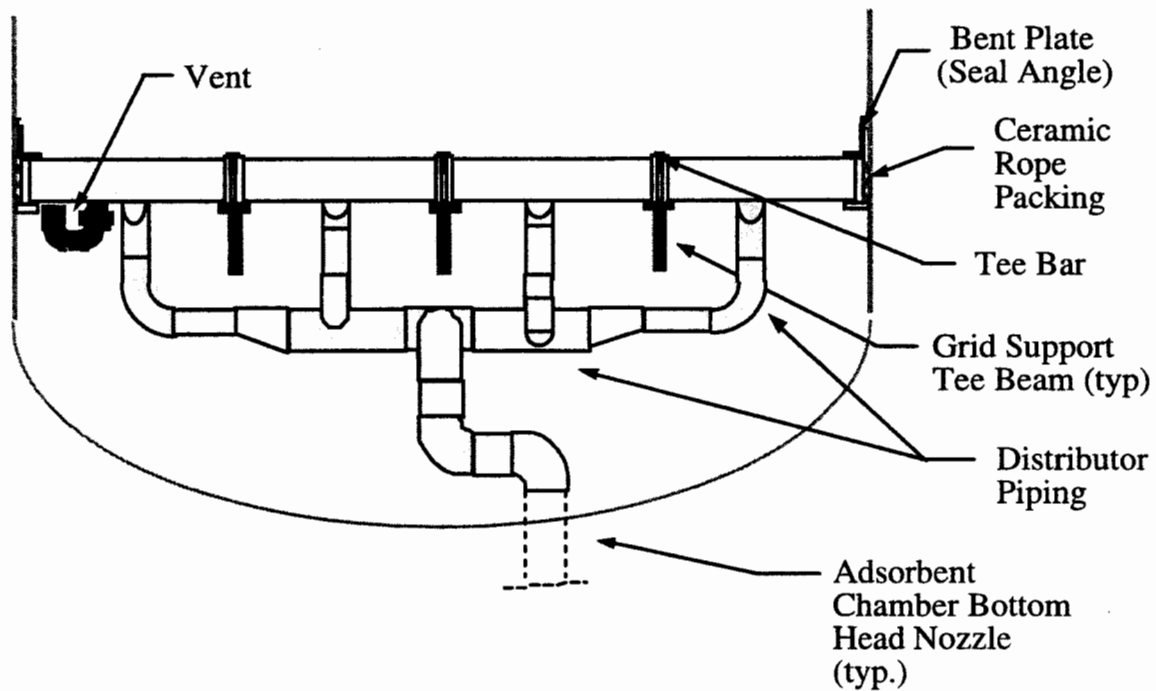


Figure XIII-24
Bottom Grid Assembly (Typical)



Notes:

- Bottom Surface of Bottom Grid is Liquid Tight
- Perimeter Gap: Seal Welded Bent Plate (Seal Angle)
- Between Segment Gap: Seal Welded Tee Bars
- Hydraulic Communication Process/Bottom Head via Vent ("Snorkel")
- Beams Installed and Leveled Previously

Figure XIII-25
Bottom Grid – Welding Leak Test

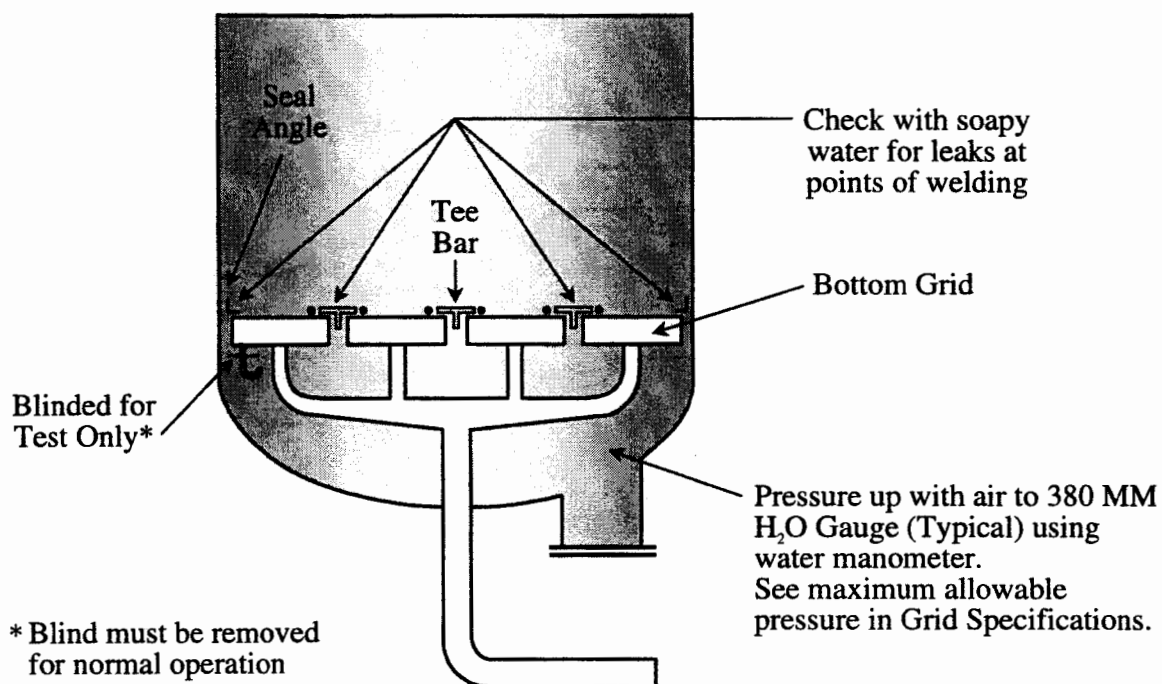


Figure XIII-26
Recommended Air Piping for Leak Test

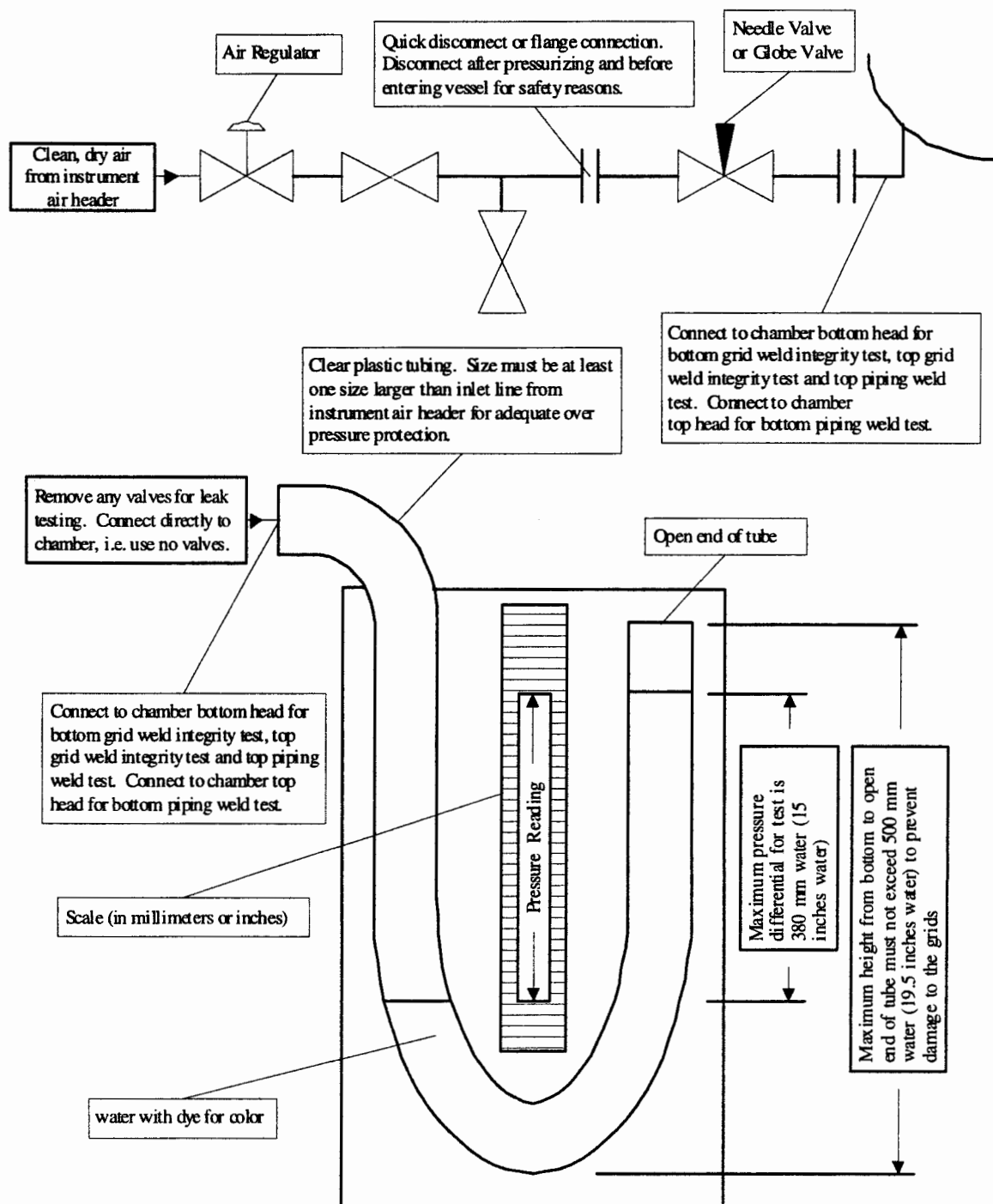


***Figure XIII-27
Manometer Set-up for Grid Leak Test***



Figure XIII-28
Sieve Unloading Nozzle Preparation

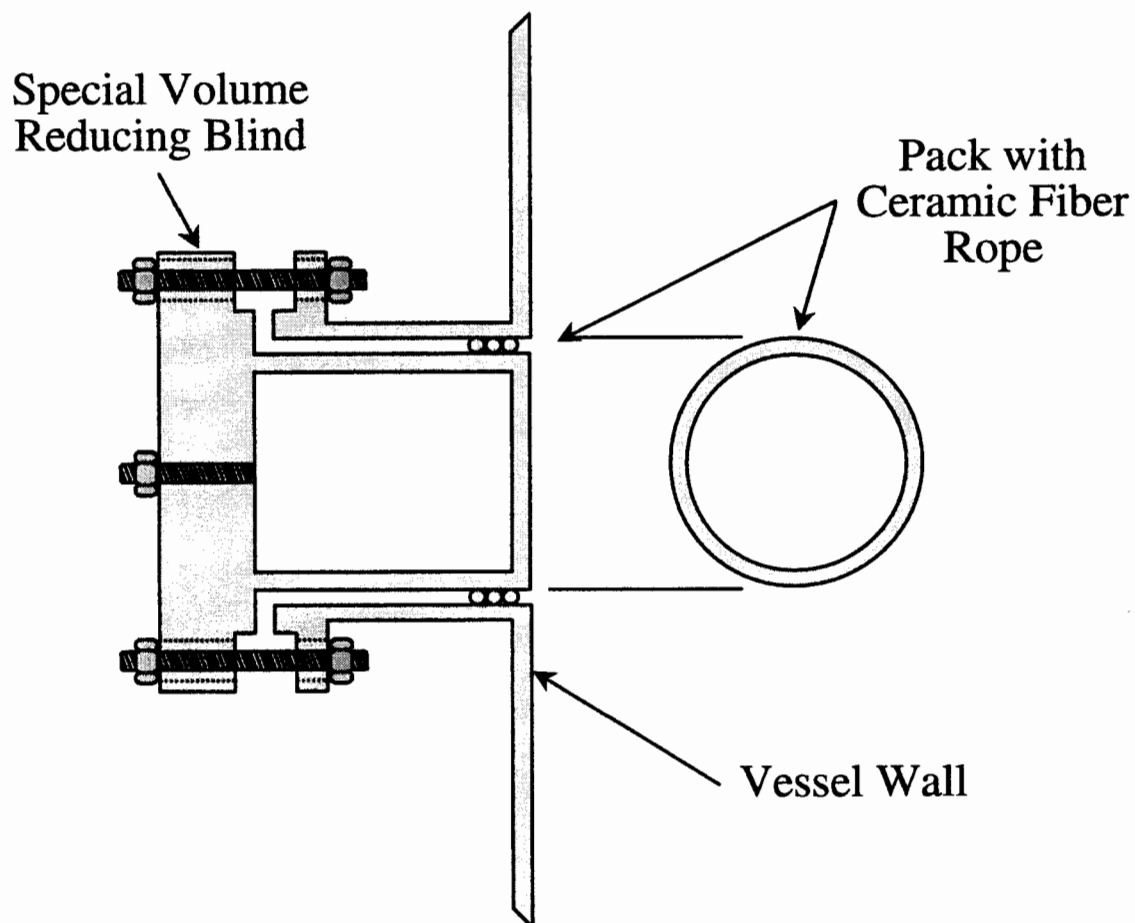
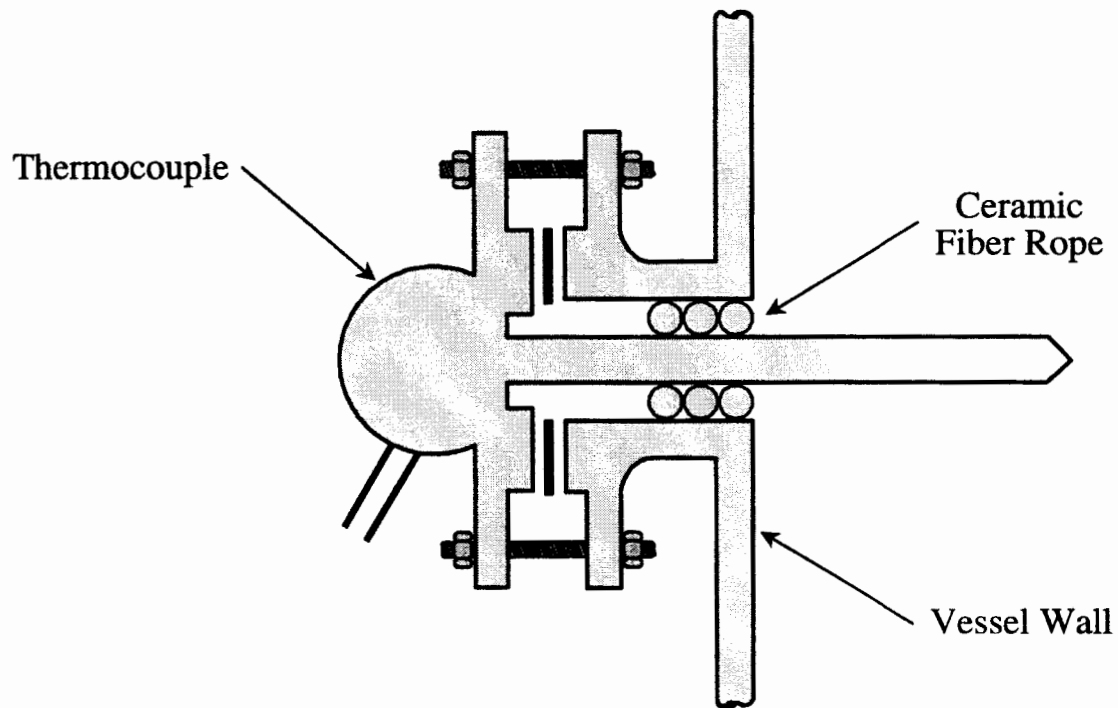


Figure XIII-29
Thermocouple Preparation



Beds 1, 6, 12, 13, 18, 24

Figure XIII-30

Refiner:

[illegible]

Comments

Figure XIII-31
Sieve Loading Arrangement
(Drawing not to scale)

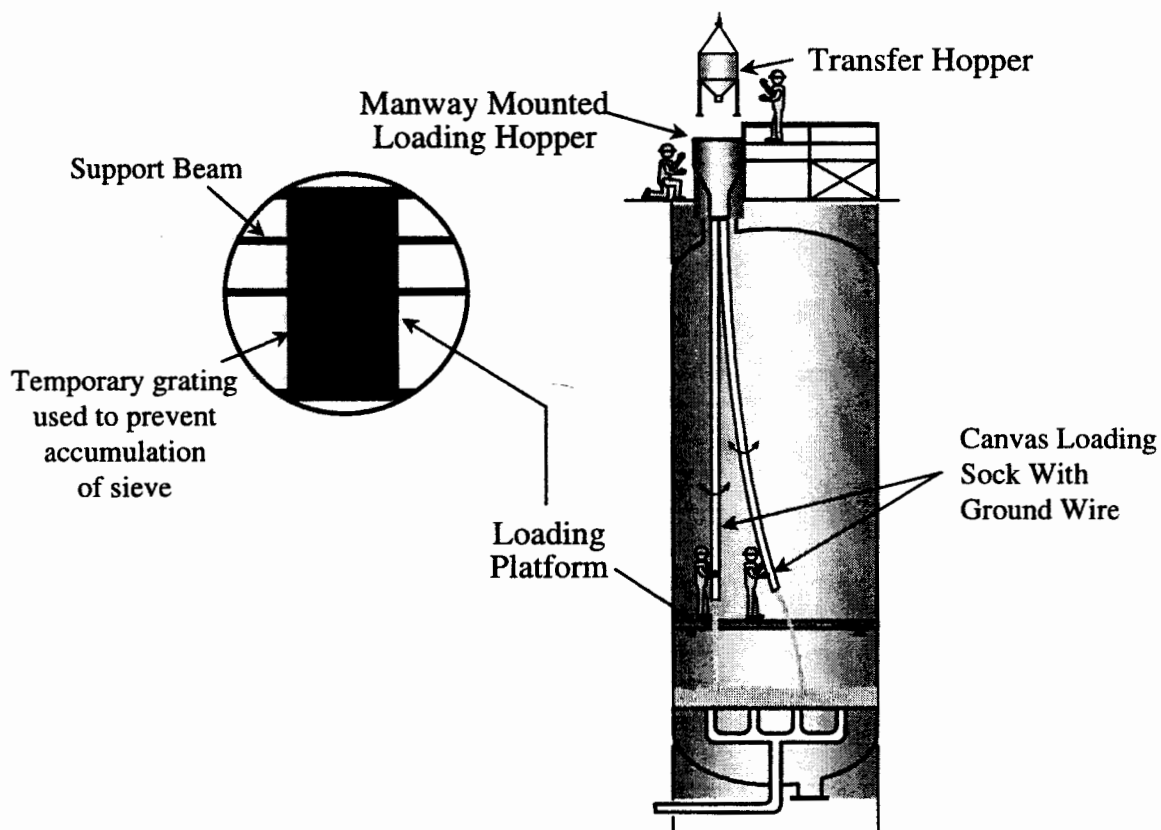
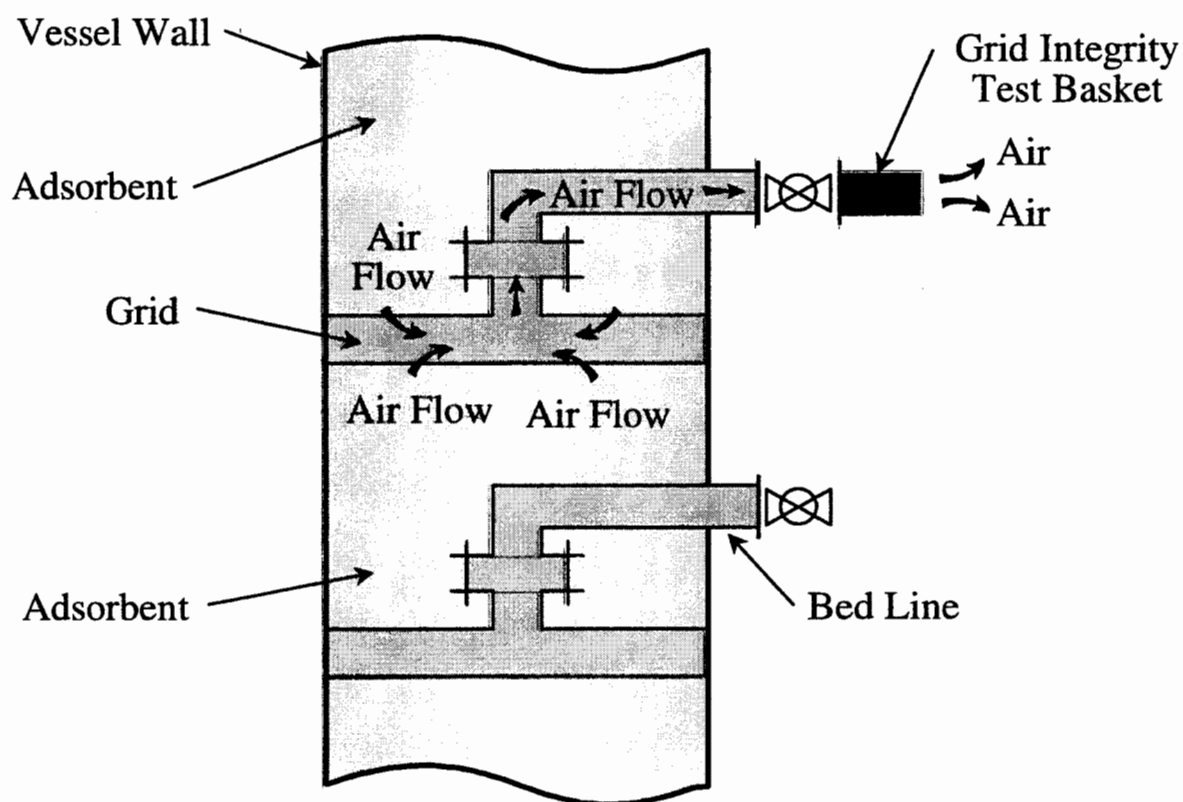


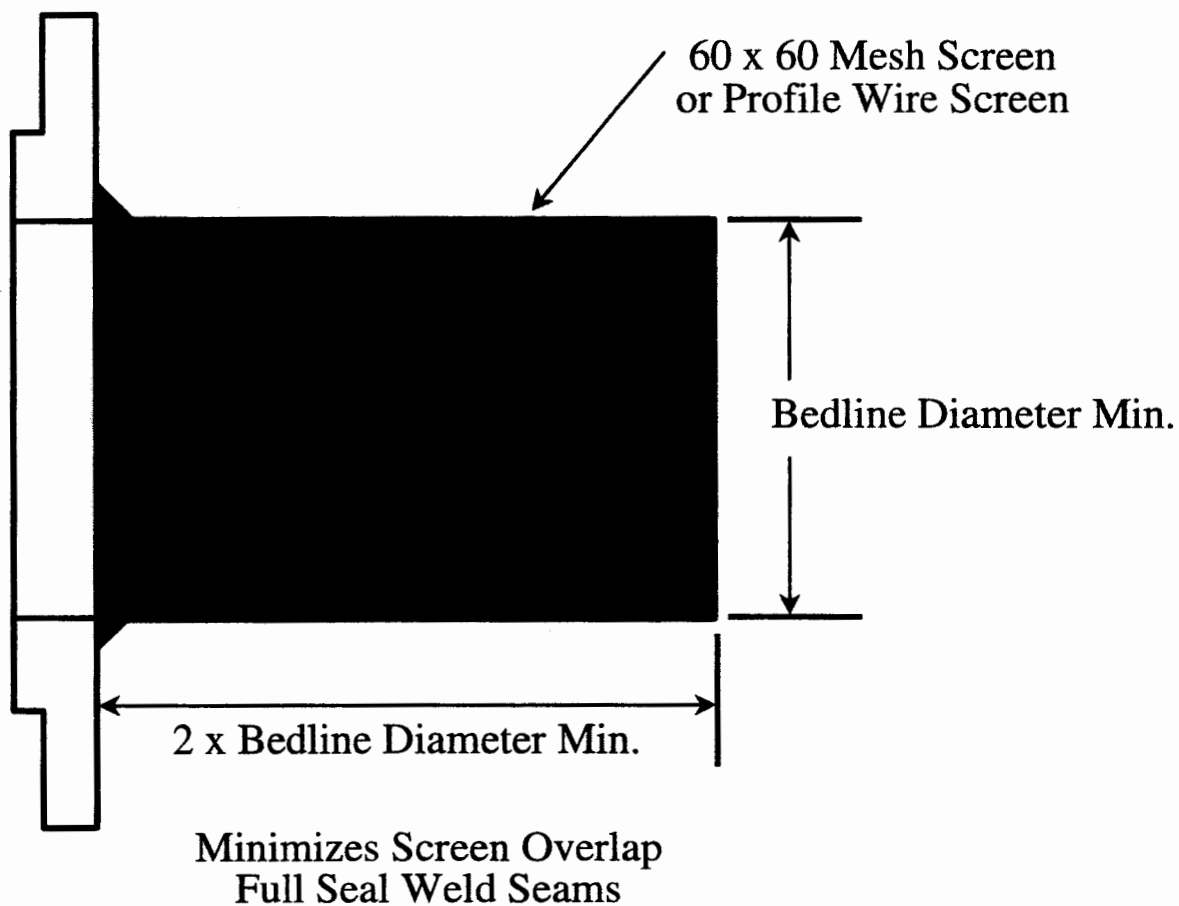
Figure XIII-32
Grid Screen Integrity Test



Maximum Pressure = 0.35 kg/cm²g (5 psig)

PRX-R00-43

Figure XIII-33
Grid Integrity Test Basket



PRX-R00-44

Figure XIII-34
Grid Placement

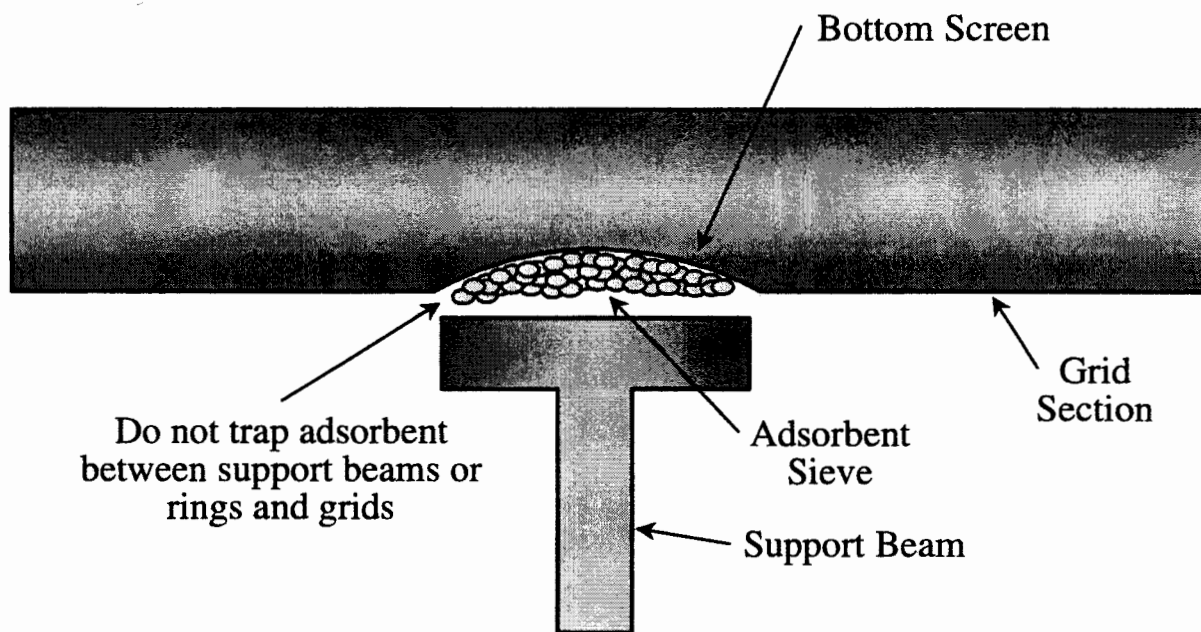


Figure XIII-35
Intermediate Grid Installation

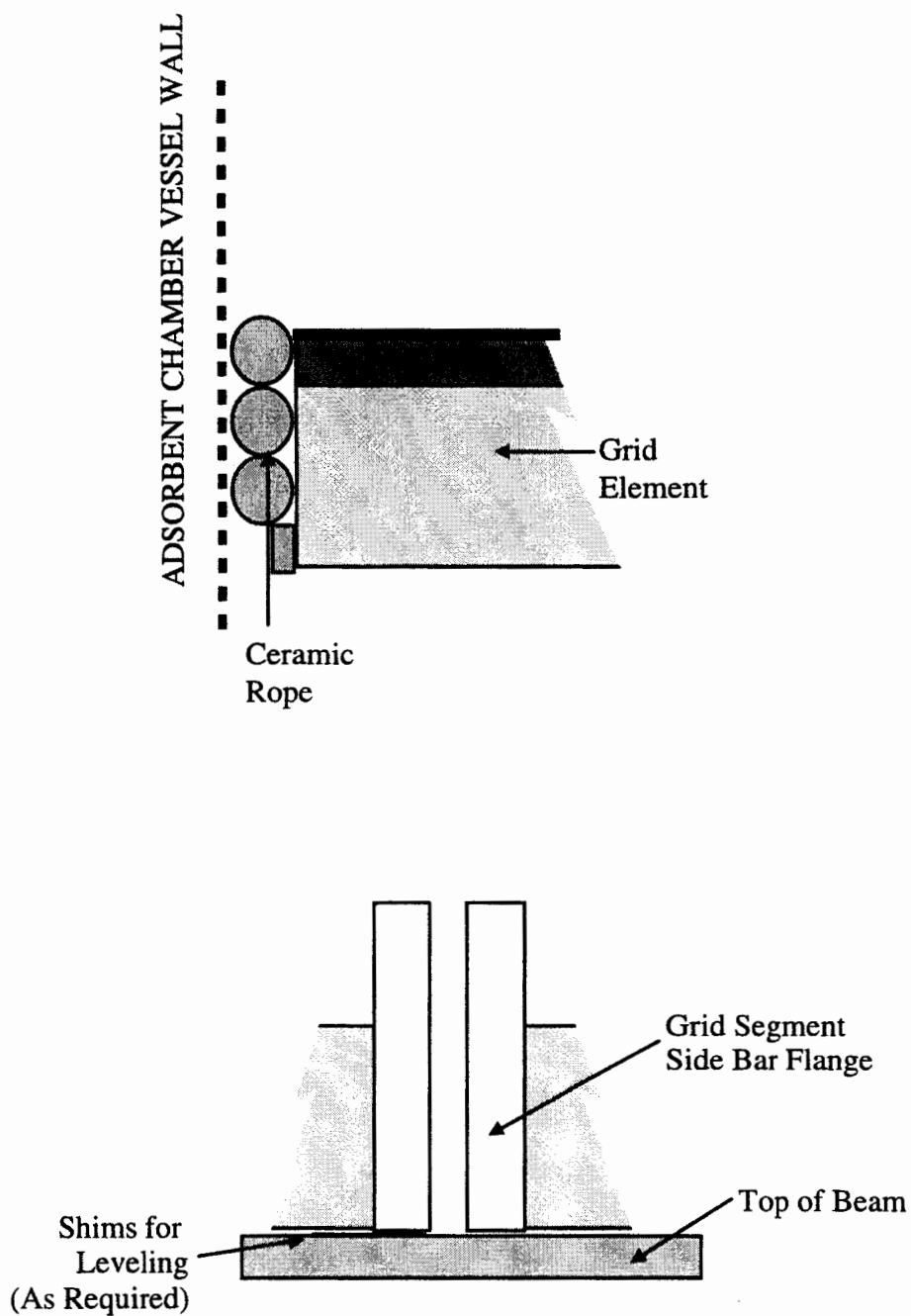


Figure XIII-36
Top Grid Installation
Hold Down Lugs for Top Grid

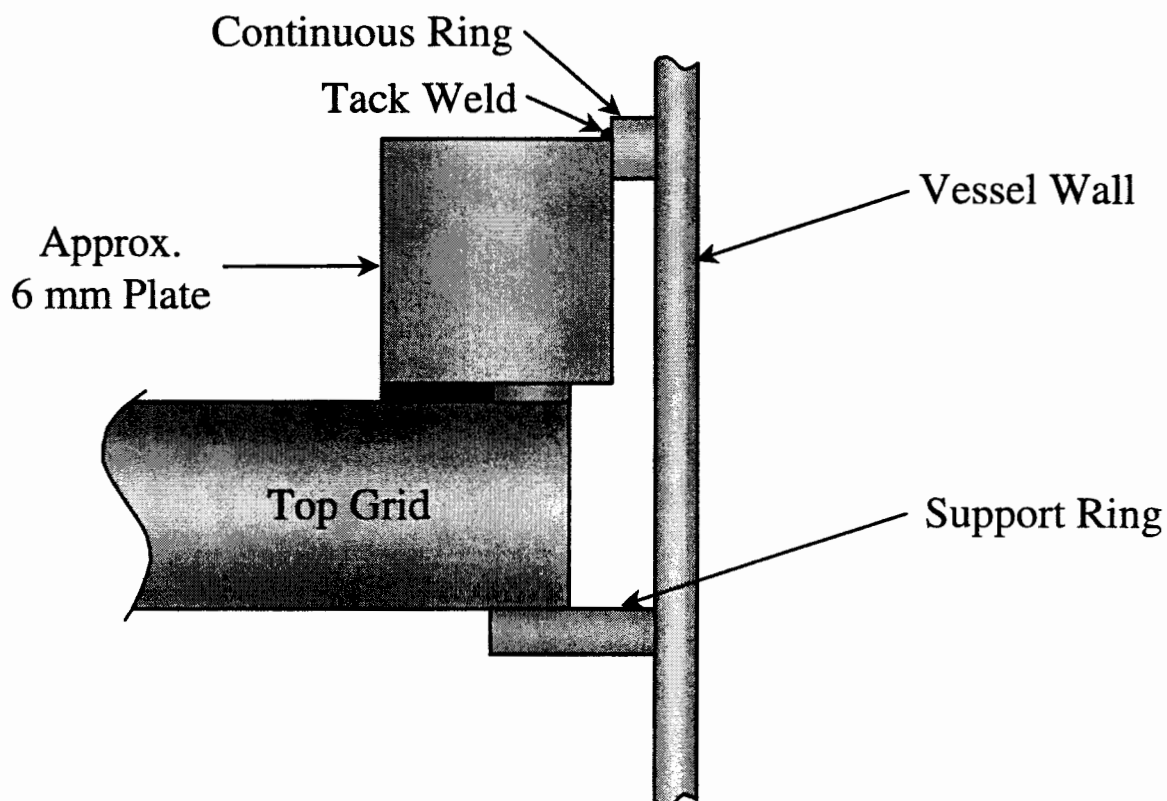


Figure XIII-37
Hold Down Lugs
Top Grids Only

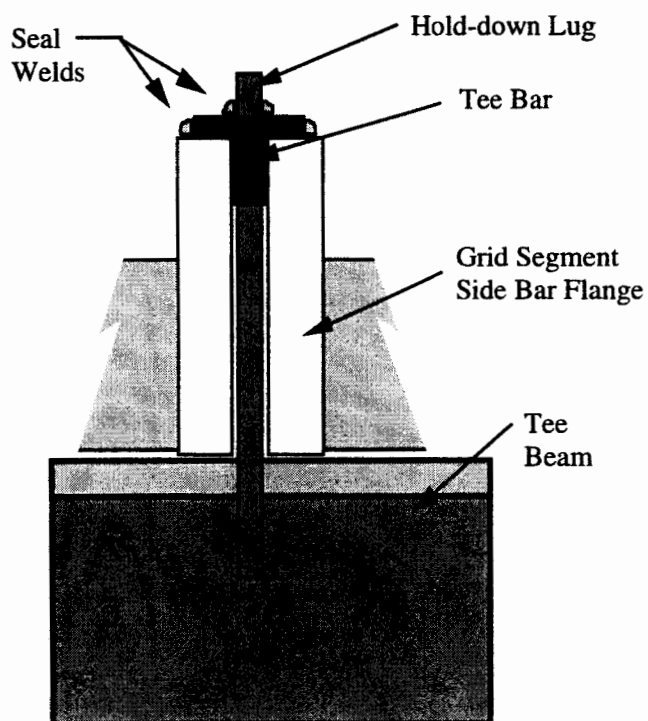
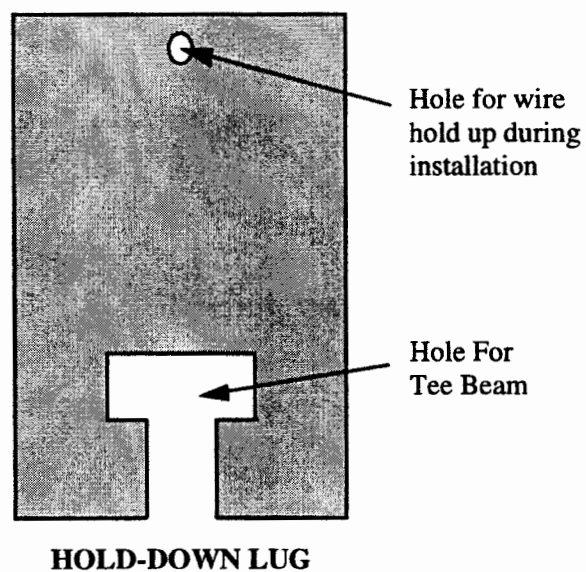
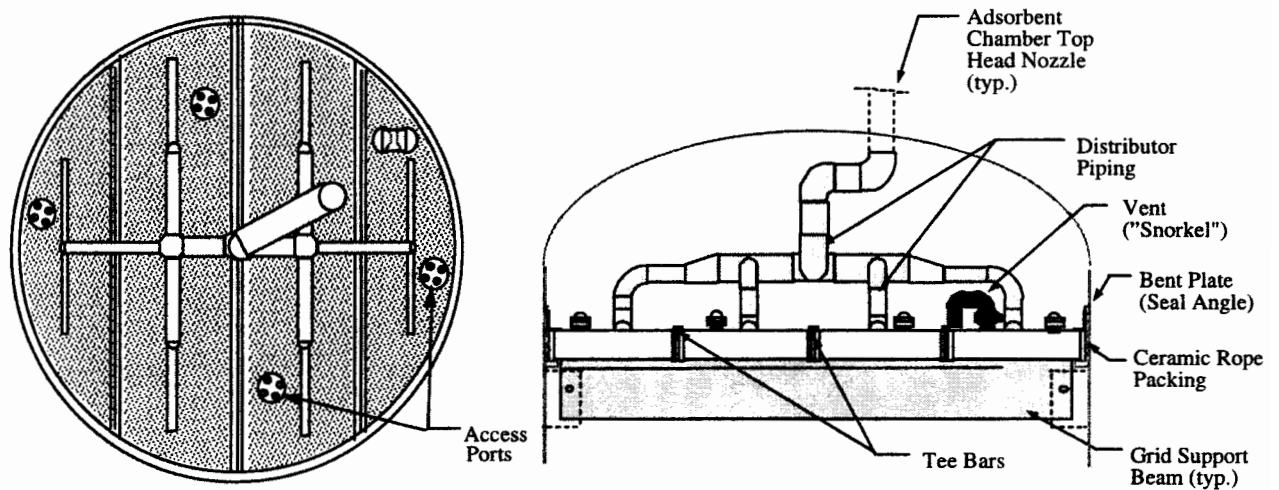


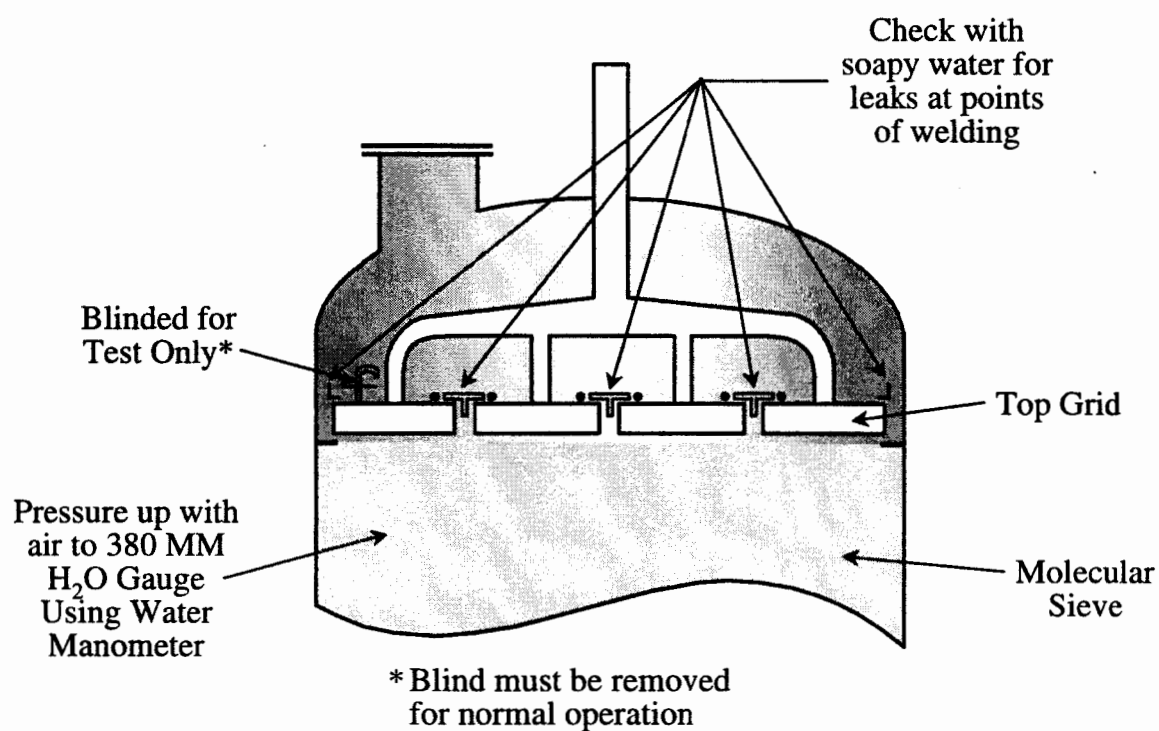
Figure XIII-38
Top Grid Assembly (Typical)



Notes:

- Top Surface of Top Grid is Liquid Tight
- Perimeter Gap: Seal welded Bent Plate (Seal Angle)
- Between Segment Gap: Seal welded Tee Bars
- Hydraulic Communication Process/Top Head via Vent ("Snorkel")
- Beams Installed and Leveled Previously

Figure XIII-39
Top Grid Weld Leak Test



PRX-R00-49

Figure XIII-40 Grid Cleaning

- Four holes should be drilled into each grid section. The holes should very carefully drilled on each side of the internal baffle and on each side of the internal distributor. The holes should be close as possible to the lowest point of each section.

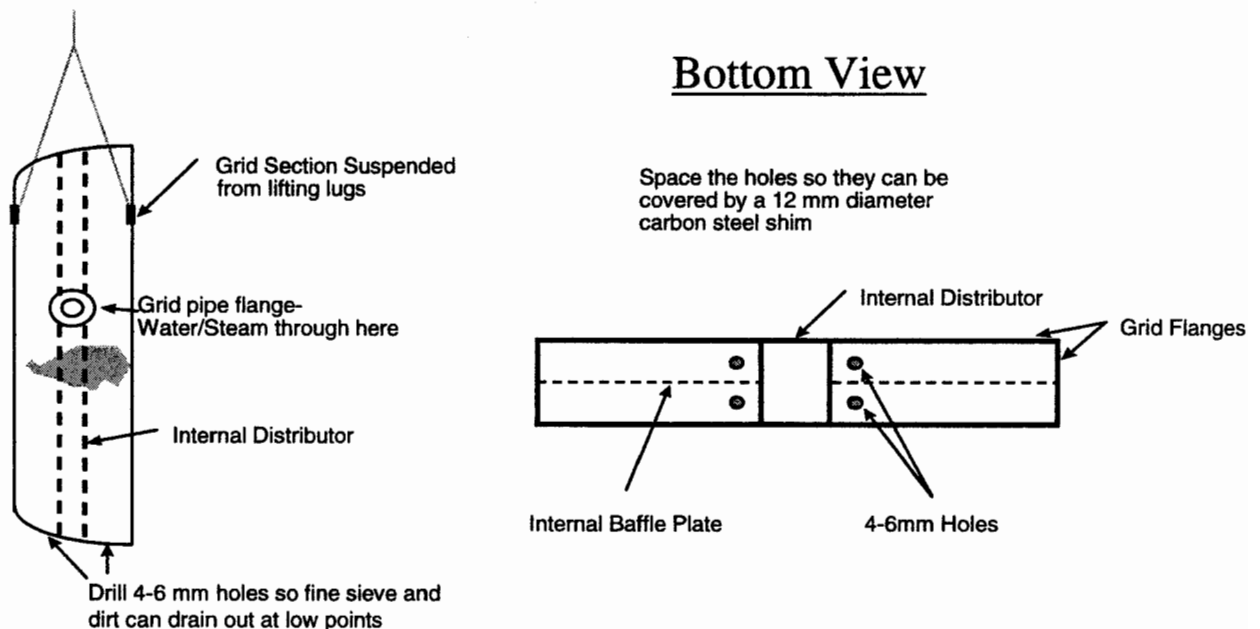


***Figure XIII-41
Grid Cleaning – Hydroblasting***







XIV. SAFETY

The information and recommendations contained in this section have been compiled from sources believed to be reliable and to represent the best opinion on the subject as of 1994. Refer to the references in Table XIV-1 for publications that can be used to supplement this information. However, no warranty, guarantee or representation, expressed or implied, is made by UOP as to the correctness or sufficiency of this information or to the results to be obtained from the use thereof. Each refiner should determine the suitability of the following material for his purposes before adopting them.

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

Government Standards

Below is a description of U.S. Safety Standards (OSHA and NIOSH). It is necessary that these standards or equivalent local regulations be followed.

OSHA Process Safety Management Standard

Beginning with the Seveso Directive in Europe, many countries have adopted regulations or standards that require processing industry companies to implement process safety programs. These programs include such elements as, hazard analyses, procedures, training, mechanical integrity programs, pre-startup safety reviews, and so on. The OSHA Process Safety Management (PSM) Standard, or local PSM requirements, should be followed in order to reduce the potential for process incidents and to improve personnel protection. This OSHA standard

concerning process safety (29 CFR 1910.119) should be read and thoroughly understood before establishing a site PSM program.

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 that are applicable only within the U.S.A. and its territories. Non-U.S. refineries should follow comparable local guidelines and regulations regarding safety in the unit. 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 national, state or local law.

Material Safety Data Sheet (MSDS)

The MSDS requirement falls primarily on chemical manufacturers, 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.

MSDS for UOP Molex Process

Following is a list of Material Safety Data Sheets (MSDS's) which should be considered the minimum for the UOP Molex Process. The most recent and up-to-date versions of these MSDSs that contain exact information relating to the materials used in or resulting from the operation of the unit should be obtained.

Para-xylene
Normal pentane
Iso-octane
Hydrotreated Kerosene
Normal Paraffin Product
ADS-34 Adsorbent

At the end of this section, **sample** MSDS for ADS-34 adsorbent are included **for illustrative purposes only**. This MSDS is current at time of publication of this manual. This MSDS may not apply to each individual unit. UOP assumes no responsibility for the use of any information contained in these MSDSs. **Always** check for the most recent (and applicable) version of the MSDS before using the information therein. MSDSs for UOP's various adsorbents and other UOP-supplied products are available on request.

Safety Information For UOP Catalysts and Adsorbents

It is during periods of handling the adsorbent (chamber loading and unloading) that bodily exposure to UOP Molex Adsorbents may occur and caution must be exercised. **Material Safety Data Sheets should be obtained from UOP at the time of adsorbent supply and should be available to the personnel working in the process unit.**

The major constituents of the UOP Molex Adsorbent is silicon oxide (synthetic), aluminum oxide (non-fibrous), calcium oxide, and sodium oxide which are not currently listed by OSHA as classified or regulated substances. These adsorbents may contain other constituents, in lesser amounts, which might present hazards to human health and/or the environment if handled or disposed of improperly. It is the purpose of this safety section to provide information and recommendations that will allow safe handling and proper disposal of these adsorbents.

The greatest potential for human exposure to adsorbent materials comes from adsorbent dust during adsorbent loading, and from dust, fumes, and vapors during adsorbent unloading operations. For the unprotected worker, hazardous dust, fumes, and vapors may be inhaled, ingested, or contaminate the eyes and skin.

OSHA has established stringent maximum exposure levels for various chemicals, some of which may be found in UOP adsorbents. 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.

Handling UOP Adsorbent Safely

UOP believes that the OSHA exposure limits to adsorbent chemicals will not be exceeded if proper handling procedures and worker protective clothing/safety devices are used. Confined space entry guidelines must be followed. In addition, UOP recommends that the following minimum safety procedures be established and adhered to:

- Handle adsorbents only in a well-ventilated area.
- In areas where natural ventilation is insufficient, use local mechanical exhaust ventilation.
- Wear a NIOSH-approved toxic dust respirator with full face-piece, protective clothing, and gloves for normal handling operations. See Figure 1
- Chamber adsorbent loading and unloading operations present extraordinary health risks, especially to personnel working in the chamber or handling the adsorbents. During loading operations, large quantities of adsorbent dust may be generated. During unloading, the hazards may include inert (nitrogen) atmosphere, toxic feedstock, product, or purge materials present with the adsorbent, or toxic forms of chemicals.
- Personnel should wear a supplied air respirator with a hood or helmet, operated in a pressure-demand or other positive-pressure mode, or in a continuous flow mode (NIOSH Respirator Code SAFE: PD, PP, CF). This respirator should have a primary, secondary, and emergency supply of air. The OSHA regulations concerning use of respirators (29 CFR, Subpart 1, Section 1910.134) should be read and thoroughly understood before undertaking to place personnel in adsorbent loading and unloading operations.
- In addition, personnel entering a confined space should be equipped with safety harnesses and safety lines for rescue and a means for visual, voice or signal line communication with standby personnel, who should be strategically located with suitable rescue equipment. See Figure 2. Protective clothing and all safety devices should be thoroughly decontaminated after each use. Worn-out, broken or defective safety equipment and clothing should be removed from service and repaired or replaced. Good personal hygiene after handling a adsorbent or being exposed to adsorbent dust is an essential part of a responsible adsorbent safety program. Do not eat, drink, or smoke in areas where the adsorbent is being handled or where exposure to adsorbent dust is likely.

Handling Adsorbent Spills and Deposited Adsorbent Dust

Always wear a NIOSH-approved self-contained breathing apparatus or the combination of toxic dust respirator-rubber frame eye goggles, protective clothing, and gloves. Avoid cleaning methods that raise dust. Dispose of all adsorbent wastes properly in containers, which should then be labeled as to the contents and hazards.

Do not dispose of adsorbent wastes in a public water system or in the normal solid waste.

Handling of Hydrocarbons

The safe handling of hydrocarbons requires special precautions.

Fire Hazard

Whether in plant operations or in the laboratory precautions should be taken to minimize the hazard of fires or explosions.

Toxicity Information

Some of the materials used in the UOP Molex unit may have toxic effects upon individuals. Care must be exercised when venting and draining equipment to prevent personnel exposure to toxic compounds. Below are some of the major hazards associated with each of the primary chemicals used in the Molex unit. However, this is not inclusive, so the MSDS and other sources should be consulted.

Ethylbenzene, Toluene, Xylenes and Heavier Aromatics

These aromatic compounds (specifically Para-xylene) are present in the Molex process. These compounds are moderately toxic and are believed to not have the destructive effect on the blood-forming organs as does benzene. If clothing (including gloves, shoes, etc.) becomes wet with such aromatics, remove the

clothing, bathe and put on fresh clothing. Avoid breathing aromatic vapors. Reference information for “The NIOSH Occupational Health Guidelines” for ethyl benzene, toluene and xylene are at the end of this chapter.

Toxicity and Handling Reference Material

Toxicity and safe handling information on most of the materials used in the unit can be found in the following references:

- a. **Dangerous Properties of Industrial Materials**
 N. Irving Sax
 Van Nostrand Reinhold
- b. **Patty’s Industrial Hygiene Toxicology**
 Craley and Craley, eds., 1979
- c. **ASTM D-270, “Standard Method of Sampling Petroleum and Petroleum Products.”**
- d. **Data Sheet D-204 “Xylene and Toluene.”** Published by the National Safety Council.

Minimizing Exposure to Aromatics

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 concentrations exceed allowable limits. This protective equipment is not, however, a substitute for safe working conditions, proper ventilation, safe sampling practices, and proper maintenance of both operating and safety equipment. In all cases, skin contact (especially eyes) and inhalation must be minimized.

Sampling liquid hydrocarbons always requires some care to limit personal exposure and release to the atmosphere. Even greater care is needed when the liquid to be sampled contains aromatic hydrocarbons, especially benzene. UOP's current design calls for flow-through sampling points that utilize closed sample containers whenever aromatics are present.

In order to minimize vaporization, hot hydrocarbon streams must be routed through a cooler before drawing a sample. In all sampling situations, the personnel involved should be instructed to remain at arm's length from the sample container and to situate themselves upwind of the container if at all possible. These simple precautions will greatly minimize exposure to the hydrocarbon vapors.

Normal Paraffins

Normal paraffins, such as n-pentane, iso-octane and kerosene, are present in the Molex process. These compounds are flammable and slightly to moderately toxic, which means that it may be harmful if ingested, inhaled or absorbed. Personal Protective Equipment for personnel should follow recommendations included on Material Safety Data Sheets for such n-paraffins.

UOP Molex Unit Safety Notes

Introduction

The purpose of this section is to acquaint all personnel engaged in the operation and maintenance of the Molex unit with the safety procedures and regulations to ensure safe operation of the unit. Before working in the Molex unit, all personnel must be familiar with the contents of this section of the manual and they must know the location and correct mode of operation of all safety equipment in the unit. The safety equipment includes breathing equipment, eye washes, emergency showers, first aid kits, fire fighting apparatus, and water hoses.

In general, the normal refinery safety rules pertaining to the handling of hydrocarbons under vacuum and pressure and high temperatures also apply to the Molex unit. It is recommended, however, that the items included in this section be brought to the attention of all personnel in the Molex area.

Checkout Prior to Startup

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

Each vessel should be blinded using blinds at each vessel nozzle. However, in the event that many vessels are to be entered in a new unit that is isolated from other plants, the entire unit can be isolated by installing blinds at the battery limits rather than by individually isolating every vessel nozzle.

The oxygen content of the atmosphere inside a vessel should be rechecked before every entry.

Emergency Equipment Checkout

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

Turnaround Inspections

In turnaround inspections, the possibility that vessels will contain dangerous gases is much higher. Equipment that has been in service must be thoroughly drained and purged before entry. The vessel should have been steamed out, unless steam presents a hazard to the internals, and then fresh air circulated through it until the atmosphere is safe to enter. Prior to the introduction of air it is necessary to confirm all the hydrocarbon has been removed. If liquid hydrocarbon remains or if odors persist afterwards, repeat the purging procedure until the vessel is free of hydrocarbon. The service history of the vessel must also be investigated before entry so that appropriate precautions may be taken. The service may require a neutralization step or a special cleaning step to make the vessel safe. Internal scale can trap poisonous gases such as hydrogen sulfide that may be released when the scale is disturbed. If this sort of danger is present, fresh air masks and protective clothing may be required to be worn while working inside of the equipment.

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

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

Chamber Loading

Adsorbent loading has a risk for asphyxiation or injury because some of the safety practices could be overlooked in the rush to complete the loading and get the unit on stream. If the chambers are being loaded, the main concerns are adsorbent

dust and nitrogen. If the loading is a reload, any of the dangerous conditions described for turnaround inspections may also be present.

During loading, dust will always be present. The effect of dust on the lungs is cumulative and even small concentrations with short exposure times should not be tolerated. Whenever possible, fresh air should be supplied from above the adsorbent bed and exhausted from below the bed so that the downward flow of air will hold the dust down and prevent hazardous material from being carried into the working space. This might not be possible when spent adsorbent is present because of residual hydrocarbon on the spent adsorbent. In either case, the fresh air should never be supplied from below the adsorbent bed because the air may carry hazardous gases into the working space of the men inside the chamber.

People who are exposed to the adsorbent should wear OSHA/NIOSH approved dust masks or fresh air respirators, refer to the MSDS for the safety equipment required. Goggles are also recommended. Exposure to adsorbent dust can be minimized greatly by staying outside of the vessel during adsorbent loading and by allowing the dust to settle before entering the vessel for inspection after loading.

Also when handling adsorbents, static electricity may be generated. It is recommended that all adsorbent loading equipment, including the loading sock be grounded.

Chamber Unloading

Chamber unloading can present extraordinary health risks, especially to personnel working with the spent adsorbents. During the unloading, large quantities of adsorbent dust may be generated. Additional hazards may include a contaminated or inert atmosphere in the chamber or during spent adsorbent handling, residual hydrocarbons including toxic forms of adsorbent ,chemicals, or reactants may be present.

Spent adsorbent dust is especially hazardous because it can contain hydrocarbon or other adsorbed species that may be flammable or toxic.

Handling precautions should take into account the potential personnel hazards mentioned above present from both adsorbent dust and the hydrocarbons.

Asbestos

For health reasons, asbestos is not specified for packing material but could be present in older existing units. Fine asbestos particles released into the air in handling this material are especially injurious to the lungs, causing cancer in some cases. All persons in an area or inside a vessel where asbestos is being handled must wear proper protective equipment and be trained in asbestos removal..

NOTE: Gas masks are not to be used for protection from adsorbent and asbestos dust.

TABLE XIV-1 Safety Topic References

Occupational Safety & Health Administration (OSHA), 29 CFR 1910.1200, Hazard Communication, 2000.

OSHA, 29 CFR 1910.134, Respiratory Protection, 2000.

Guide for Inspection of Refinery Equipment; API.

OSHA, 29 CFR 1910.146, Permit-Required Confined Spaces, 2000

ACGIH, Threshold Limit Values for Chemical Substances and Physical Agents & Biological Exposure Indices, 2002.

Occupational Health Guidelines for Chemical Hazards; NIOSH/OSHA; Sept. 1978.

Sax, N. Irving; Dangerous Properties of Industrial Materials; Van Nostrand Reinhold; 6th ed.

ASTM D-270, Standard Method of Sampling Petroleum and Petroleum Products.

International Programme on Chemical Safety, "International Chemical Safety Cards," available through US National Institute of Occupational Safety & Health (NIOSH).

OSHA, Subpart Z, Toxic and Hazardous Substances, 29 CFR 1910.1000, Air Contaminants, 2000.

OSHA, 29 CFR 1910.1001, Asbestos, 2000.

FIGURE 1
Example of Respirator



FIGURE 2 Example of Harnesses



Appendix 1

Molex Unit Summary

Daily Average Data

Unless Indicated

*spot reading

Date

Days on Stream
Daily Sieve Life, MT/kg
Cumulative Sieve Life, MT/kg
Feed Totalizer
Cumulative Sieve Life, MT/kg (By Feed Totalizer)
Feed Normal Paraffins [Fn] m³/hr @ Tf
Bed No. 1 Temperature, °C
Bed No. 6 Temperature, °C
Bed No. 12 Temperature, °C
Bed No. 13 Temperature, °C
Bed No. 18 Temperature, °C
Bed No. 24 Temperature, °C
Chamber 1 Bottom Pressure, kgf/cm² g
Chamber 2 Bottom Pressure, kgf/cm² g
A/Fn
L2/W
L2b/W
Dn/A
L4/W
Cycle Time, minutes
Line Flush, % of Vlbl

Calculations:

Molex Capacity, % of design
Weight Recovery, wt-% of feed rate
Weight Balance Raffinate Column
Weight Balance Extract Column
Volume Balance (vol-% at Tf)
Extract Purity, wt-%
Theoretical Recovery, wt-%
E/F Recovery, wt-%
E/(E+R) Recovery, wt-%
1-(R/F) Recovery, wt-%
n-C10 Theoretical Recovery, wt-%
n-C11 Theoretical Recovery, wt-%
n-C12 Theoretical Recovery, wt-%
n-C13 Theoretical Recovery, wt-%
n-C14 Theoretical Recovery, wt-%
Feed Normal Paraffins, MTD
Paraffins Production, MTD

Appendix 1

Pressures (kgf/cm²g):

Chamber No. 1 Delta P
Chamber No. 2 Delta P
Chamber No. 1 Bottom Pressure
Chamber No. 2 Bottom Pressure
Rotary Valve Dome Pressure
Feed Filter Delta P
Flush Filter Delta P
Desorbent Filter Delta P

Corrected Flow Rates (m³/hr)

Feed to Rotary Valve by T.M. @ 177 °C
Feed to Rotary Valve by O.P. @ Tf
Desorbent to Rotary Valve by T.M. @ 177°C
Desorbent to Rotary Valve by O.P. @ Tf
Extract from Rotary Valve by T.M. @ 177°C
Extract from Rotary Valve by O.P. @ Tf
Raffinate from Rotary Valve @ Tf
Line Flush in to Rotary Valve by T.M. @ 177°C
Line Flush in to Rotary Valve by O.P. @ Tf
Line Flush out from Rotary Valve by T.M. @ 177°C
Line Flush out from Rotary Valve by O.P. @ Tf
Zone Flush to Rotary Valve by T.M. @ 177°C
Zone Flush to Rotary Valve by O.P. @ Tf
Pumparound Flows by Turbine Meter @ Tf (m³/hr)
Pumparound Flows by Venturi @ 15 °C (m³/hr)
Pusharound Flows by Turbine Meter @ Tf (m³/hr)
Dome Seal In @ Tf
Dome Seal Out @ Tf
Chamber No. 1 Top Flush @ 15 °C (Local)
Chamber No. 1 Bottom Flush @ 15 °C (Local)
Chamber No. 2 Top Flush @ 15 °C (Local)
Chamber No. 2 bottom flush @ 15 °C (Local)
Extract Column Bottoms at 15 °C
Raffinate Column Bottoms at 15 °C

Turbine / Orifice Check

Feed
Desorbent
Extract
Line Flush In
Line Flush Out
Zone Flush

Pumparound & Pusharound Flow Rates *

Pumparound Flows by Turbine Meter @ Tf (m³/hr)*
Zone I*

Appendix 1

Zone IIA*
Zone II*
Zone IIB*
Zone III*
Zone IIIA*
Zone IV*

Pumparound Flows by Venturi @ 15 °C (m³/hr)*

Zone I*
Zone IIA*
Zone II*
Zone IIB*
Zone III*
Zone IIIA*
Zone IV*

Pusharound Flows by Turbine Meter @ Tf (m³/hr) *

Zone I*
Zone IIA*
Zone II*
Zone IIB*
Zone III*
Zone IIIA*
Zone IV*

Column Operations:

Extract Column

Total Reflux, m³/hr @ 15 °C
Net Overhead to DSD, m³/hr @ 15 °C
Reboiler Flow (Hot Oil), m³/hr @ 15 °C
Overhead Pressure, kgf/cm² g
PDIC, kgf/cm² g
Bottoms Temperature, °C
Tray 30 Temperature, °C
Tray 20 TIC Temperature, °C
Sidecut Temperature, °C
Overhead Temperature, °C
Condenser Outlet Temperature, °C

Raffinate Column

Net Reflux, m³/hr @ 15 °C
Net Overhead to DSD, m³/hr @ 15 °C
Sidecut Pumparound, m³/hr @ 15 °C
Reboiler Flow (Hot Oil), m³/hr @ 15 °C
Overhead Pressure kgf/cm² g
PDIC kgf/cm² g
Bottoms Temperature, °C
Tray 31 Temperature, °C
Tray 21 TIC Temperature, °C
Sidecut Temperature, °C
Overhead Temperature, °C

Appendix 1

Condenser Outlet Temperature, °C

Desorbent Stripper

Raffinate Sidecut Feed, m³/hr @ 15 °C

Extract Sidecut Feed, m³/hr @ 15 °C

Overhead Vapor to Raff Col m³/hr @ 15 °C

Reboiler (Raff.) Flow m³/hr @ 15 °C

Bottom Temperature, °C

Overhead Temperature, °C

Stream Temperatures (°C)

Feed to Rotary Valve

Desorbent to Rotary Valve

Zone Flush to Rotary Valve

Line Flush to Rotary Valve

Line Flush from Rotary Valve

Extract from Rotary Valve

Raffinate from Rotary Valve

Pumparound

Heaters

Flush Heater Hot Oil, m³/hr @ 15 °C

Flush Heater Outlet Temperature, °C

Desorbent Heater Hot Oil, m³/hr @ 15 °C

Desorbent Heater Outlet Temperature, °C

Laboratory

Feed Composition

nC9-, wt%

nC10, wt%

nC11, wt%

nC12, wt%

nC13, wt%

nC14, wt%

nC15+, wt%

TNP, wt%

TNN, wt%

GC Check

Aromatics, vol%

S, ppm

N, ppm

Br Index

Specific Gravity - Lab

Extract Column Overhead Composition

C5-, wt-%

iC5, wt-%

nC5, wt-%

C6+C7, wt-%

iC8, wt-%

Appendix 1

MCH, wt-%
C8, wt-%
C9+, wt-%
GC Check
Specific Gravity - Lab
Br Index
Water, ppm

Extract Column Sidecut Composition

C5-, wt%
iC5, wt%
nC5, wt%
C6+C7, wt%
iC8, wt%
MCH, wt%
C8, wt%
C9+, wt%
GC Check
Specific Gravity - Lab

Extract Column Bottoms Composition

nC9-, wt%
nC10, wt%
nC11, wt%
nC12, wt%
nC13, wt%
nC14, wt%
nC15+, wt%
Extract Purity, wt% TNP
TNN, wt%
GC Check
Aromatics
Br Index
Specific Gravity - Lab

Raffinate Column Overhead Composition

C5-, wt-%
iC5, wt-%
nC5, wt-%
C6+C7, wt-%
iC8, wt-%
MCH, wt-%
C8, wt-%
C9+, wt-%
GC Check
Specific Gravity - Lab
Br Index
Water, ppm

Raffinate Column Sidecut Composition

C5-, wt%

Appendix 1

iC5, wt%
nC5, wt%
C6+C7, wt%
iC8, wt%
MCH, wt%
C8, wt%
C9+, wt%
GC Check
Specific Gravity - Lab

Raffinate Column Bottoms Composition

nC9-, wt%
nC10, wt%
nC11, wt%
nC12, wt%
nC13, wt%
nC14, wt%
nC15+, wt%
TNP, wt%
TNN, wt%
GC Check
Aromatics
Br Index
Specific Gravity - Lab

Desorbent Composition

C5-, wt%
iC5, wt%
nC5, wt%
C6+C7, wt%
iC8, wt%
MCH, wt%
C8, wt%
C9+, wt%
GC Check
Specific Gravity - Lab
Br Index
Water, ppm

Flush Composition

C5-, wt%
iC5, wt%
nC5, wt%
C6+C7, wt%
iC8, wt%
MCH, wt%
C8, wt%
nC9, wt%
C10, wt%
C11, wt%
GC Check

Appendix 1

Specific Gravity - Lab
Br Index
Water, ppm



XV. EQUIPMENT EVALUATION

The Mechanical Evaluation Test should cover all facets of the unit. All major pieces of equipment should be part of this test, including vessels, pumps, heat exchangers, and piping hydraulics. The procedure for this test is long and involved, but the information can be very useful for the operator and for UOP. If the operator wants to revamp the unit, it is important to determine maximum throughput and actual equipment limitations. Most of the information will be collected only once, although parts of it (such as exchanger surveys) can be repeated to follow fouling or other potential problems.

Chamber Bed Pressure Drop Surveys and Rotary Valve Track Pressure Surveys also provide key information for the evaluation of the unit's hydraulics. Refer to Section X for a description of these surveys and other useful information that may be obtained to benchmark unit performance.

The lists and data sheets included in this section can be used as guidelines in collecting the required data, although the operator may have to modify certain parts for his particular unit. It is important to finish collecting the data within as short a time as possible. A single survey is generally satisfactory and it is not necessary to use long term average data. The unit should be operating smoothly to get realistic and good quality data. Label the data collected and prepare a report in an orderly fashion.

The information generated and collected during the Mechanical Evaluation Test will be used to assess the potential of the unit and to plan for future debottlenecking and optimization studies.

However, for the test information to be of value, the following criteria must be met:

1. The unit **must** weight balance. The weight balance must be consistently between 98 and 102 wt.%, and preferably between 99 and 101 wt.%.

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

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

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

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

A. GENERAL INFORMATION LIST

This list is only a guideline. Please modify or expand as required.

1. Ambient Air Conditions
 - a. Temperature
 - b. Relative Humidity
 - c. Barometric Pressure
 - d. Wind Velocity and Direction (show on rough plot plan)
2. General Description of Unit
 - a. Process Flow Diagram, including flow meter and control valve locations
 - b. Plot Plan showing Layout of Vessel and Equipment
3. Units used (USA, Imperial, Metric) and Standard Conditions (0°C, 760 mm; 60°F, 14.7 psia)
4. Other limiting factors, such as utility capacity (shortage of steam or electricity, etc.) or known existing equipment limitations
5. Performance Data
 - a. Accurate Flow and Weight Balance including sample analyses

B. EXCHANGERS

1. Flow through exchangers on both sides (gas and liquid), composition and mass flow.
2. Temperature in and out on both sides, also between shells, bundles.
3. Pressures in and out on both sides, if possible.
4. If air coolers; air temperature out, air velocity out, motor amps, note any belt slippage, variable pitch positions, louver positions, etc.
5. In preparing data, submit overall heat transfer coefficient, specifics on exchangers.

C. HEATERS

1. Process flows (volume and mass, avg. mol wt., composition, etc.)
2. Process pressures in and out
3. Process temperatures in and out
4. Flue gas composition
5. Fuel gas composition
6. Fuel gas rate, pressure, temperature
7. Fuel oil composition (Specific Gravity, distillation, viscosity, metals, sulfur)
8. Fuel oil rate, temperature

9. Steam pressure for oil atomization
10. Temperatures at bridgewall, any intermediate convection points, stack, tube skins, firebox skins, fuel oil, preheat air
11. Temperature of BFW coils in and out, superheated steam pressure and temperature
12. Steam generation rate, pressure, temperature
13. BFW pressure, rate, temperature
14. Draft, each cell
15. Basic data on process coils (size, number, material, layout sketch, etc.)
16. Basic data on convection coils (size, number, material, layout sketch, etc.)
17. Burner data (rating, design release, etc.)
18. Need sufficient data to calculate heat flux from process side, heat flux from fire side, calculate steam balance, calculate total heat release, calculate heater efficiency.

D. HYDRAULIC SURVEY AND PROCESS SEPARATIONS

1. Single gauge pressure survey of every point available on reactor circuit
2. All control valve positions (including fuel, BFW, etc.)
3. Pump and compressor motor amps

4. Pump suction, discharge pressure, flow rates, composition, temperatures, with manufacturer's curve data for comparison
5. Compressor suction, discharge pressures, flow rates, composition, temperatures, with manufacturer's curve data for comparison
6. Single gauge pressure survey of fractionation systems, with sufficient data to calculate internal reflux, number of theoretical trays, etc.

E. UTILITY CONSUMPTION/PRODUCTION DATA

1. Steam (all pressures)
2. Air (Plant and Inst.)
3. N₂
4. Cooling Water
5. BFW
6. Utility Water
7. Steam Condensate
8. Process Condensate
9. Steam Generator Blowdown
10. Fuel Gas
11. Fuel Oil

F. SAMPLES (TYPICAL)

1. Molex Feed
2. Adsorbent Chamber Head
3. Extract Column Bottoms
4. Extract Column Overhead
5. Extract from Chambers
6. Finishing Column Overhead
7. Finishing Column Bottoms (Paraxylene)
8. Adsorbent Chamber Pumparound
9. Nitrogen Gas Supply
10. Raffinate Column Overhead

11. Raffinate Column Sidecut
12. Raffinate Column Bottoms
13. Raffinate from Chambers
14. Recycle Desorbent
15. Desorbent Rerun Column Bottoms
16. Makeup Desorbent
17. Chamber No. 2 Bed 24 Adsorbent
18. Paraxylene Day Tanks
19. Plant Inventory Tank
20. Water Injection
21. Rotary Valve Dome Sealant Outlet

G. DATA COLLECTION TEMPLATE (TYPICAL)

A template for collection of normal operating data is included in Appendix 1.

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. Liquid	Other
Mass Flow, _____	_____	_____	_____	_____	_____	_____
Temperature, ° _____	_____	_____	_____	_____	_____	_____
Pressure, _____	_____	_____	_____	_____	_____	_____
Composition, _____ %	_____	_____	_____	_____	_____	_____
C ₅ -, wt-%	_____	_____	_____	_____	_____	_____
iC ₅ , wt-%	_____	_____	_____	_____	_____	_____
nC ₅ , wt-%	_____	_____	_____	_____	_____	_____
C ₆ +C ₇ , wt-%	_____	_____	_____	_____	_____	_____
iC ₈ , wt-%	_____	_____	_____	_____	_____	_____
MCH, wt-%	_____	_____	_____	_____	_____	_____
C ₈ , wt-%	_____	_____	_____	_____	_____	_____
nC ₉ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₀ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₁ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₂ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₃ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₄ , wt-%	_____	_____	_____	_____	_____	_____
nC ₁₅ +, wt-%	_____	_____	_____	_____	_____	_____
TNN, wt-%	_____	_____	_____	_____	_____	_____
Aromatics	_____	_____	_____	_____	_____	_____
Avg. Mol. Wt.	_____	_____	_____	_____	_____	_____
Gravity	_____	_____	_____	_____	_____	_____

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

Weight balance _____ Heat balance _____

Deviations from UOP Specifications: _____

CONTROL VALVE SUMMARY

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

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

Deviations from UOP Specification: _____

FLOW METER SUMMARY

page _____

date _____

Item No.: _____

by _____

Service: _____

Type of Fluid: _____ Normal Units of Flow: _____

Type of Meter: _____

Meter Reading: _____

Pressure _____

Temperature _____

Sp. Gr.** _____

Meter Factor _____

Corrected Flow Rate _____

Mass Flow Rate _____

Avg. mol. wt. _____

Molar Flow Rate _____

****Sketch piping layout, showing distances in nominal pipe IDs.**

HEAT EXCHANGER SURVEY

page _____

date _____

by _____

Item No.: _____

Service: _____

Manufacturer: _____

Type, Model: _____

No. of Bundles: _____

No. of Passes/Bundle: _____ Tubes per Pass: _____

Tube Size _____ ID x _____ Gauge x _____ Length _____

Heat Exchange Surface Area/Bundle: _____

Piping Geometry (sketch if necessary): _____

Length of Service: _____

Design Heat Transfer Coefficient: _____

Shell Side	Inlet	Stream A	Pressure	Temperature
	Outlet			
Tube Side	Inlet	B		
	Outlet			
Q (calc.) Shell side				
Q (calc.) Tube side				
Composition, _____ %		A	B	
C ₅ -, wt-%				
iC ₅ , wt-%				
nC ₅ , wt-%				
C ₆ +C ₇ , wt-%				
iC ₈ , wt-%				
MCH, wt-%				
C ₈ , wt-%				
nC ₈ , wt-%				
nC ₁₀ , wt-%				
nC ₁₁ , wt-%				
nC ₁₂ , wt-%				
nC ₁₃ , wt-%				
nC ₁₄ , wt-%				
nC ₁₅ +, wt-%				
TNN, wt-%				
Aromatics				
Mass Flow				
Avg. Mol. Wt.				
Gravity				

 Deviations from UOP Specification: _____

HEATER SURVEY

page _____

date _____

Item No.: _____ by _____

Service: _____

Manufacturer: _____

Type, Model: _____

No. of Passes: _____ Tubes per Pass: _____

Tube Size _____ ID x _____ Wall x _____ Length _____

Geometry (Process): _____

Geometry (Flue Gas): _____

		Stream	Pressure	Temperature
Radiant	Inlet	A	_____	_____
	Outlet		_____	_____
Convection I	Inlet	B	_____	_____
	Outlet		_____	_____
Convection II	Inlet	C	_____	_____
	Outlet		_____	_____
Convection III	Inlet	D	_____	_____
	Outlet		_____	_____
Fuel Gas		E	_____	_____
Fuel Oil		F	_____	_____
Flue Gas Under Convection I		G	_____	_____
Flue Gas Under Convection II		H	_____	_____
Flue Gas Under Convection III		I	_____	_____
Flue Gas Under Stack Damper		J	_____	_____
Flue Gas Above Floor		K	_____	_____

HEATER SURVEY

page _____

date _____

by _____

Stream	A	B	C	D	E	F	G,H, I,J,K
Mass Flow, _____	_____	_____	_____	_____	_____	_____	_____
Composition, _____ %	_____	_____	_____	_____	_____	_____	_____
H ₂	_____	_____	_____	_____	_____	_____	_____
N ₂	_____	_____	_____	_____	_____	_____	_____
O ₂	_____	_____	_____	_____	_____	_____	_____
CO	_____	_____	_____	_____	_____	_____	_____
CO ₂	_____	_____	_____	_____	_____	_____	_____
H ₂ S	_____	_____	_____	_____	_____	_____	_____
SO ₂	_____	_____	_____	_____	_____	_____	_____
C ₁	_____	_____	_____	_____	_____	_____	_____
C ₂	_____	_____	_____	_____	_____	_____	_____
C ₃	_____	_____	_____	_____	_____	_____	_____
IC ₄	_____	_____	_____	_____	_____	_____	_____
NC ₄	_____	_____	_____	_____	_____	_____	_____
IC ₅	_____	_____	_____	_____	_____	_____	_____
NC ₅	_____	_____	_____	_____	_____	_____	_____
C ₆ -205°C (400°F)	_____	_____	_____	_____	_____	_____	_____
205°C (400°F)+	_____	_____	_____	_____	_____	_____	_____
Avg. Mol. Wt.	_____	_____	_____	_____	_____	_____	_____
Gravity	_____	_____	_____	_____	_____	_____	_____
Viscosity	_____	_____	_____	_____	_____	_____	_____
Total Sulfur, _____	_____	_____	_____	_____	_____	_____	_____
Metals, _____	_____	_____	_____	_____	_____	_____	_____
Q (calc.) Absorbed	_____	_____	_____	_____	_____	_____	_____
Q (calc.) Released	_____	_____	_____	_____	_____	_____	_____
Heater Gross Efficiency	_____	_____	_____	_____	_____	_____	_____
Excess Air, %	_____	_____	_____	_____	_____	_____	_____
Tube Skin Temps, ° _____	_____	_____	_____	_____	_____	_____	_____

Burner Pressure _____ % of Rating _____

Provide sketch showing piping and controls for process piping.

Deviations from UOP Specification: _____

CENTRIFUGAL PUMP SURVEY

page _____

date _____

Item No.: _____ by _____

Service: _____

Manufacturer: _____

Type, Model: _____

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

	Pressure	Temperature
Suction	_____	_____
Discharge	_____	

Other Information

Rated Flow (STP)	_____	Seal Type? Single, Tandem, Double, Bellow
Sp. Gr.	_____	Spillback? Yes/No
Viscosity	_____	NPSHR? _____
Static Suction Head	_____	Suction Specific Speed: _____
Speed	_____	
Differential Head (flowing condition)	_____	

Driver Type: _____

Manufacturer: _____

No., Size, Rating and Style (Mfgs. designation): _____

Rating: _____ Insulation Class: _____

Service Factor: _____ Voltage/Phase/Cycle: _____

Motor:

Power consumption _____

Speed _____

Turbine:

Steam consumption	_____	Pressure	Temperature
Steam supply		_____	_____
Steam exhaust		_____	_____
Speed	_____		

Supply copy of Mfgs. pump curve and plot operating point.

Deviations from UOP Specification: _____





