
Uop
DeFine Process Unit
GENERAL OPERATING MANUAL

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I. INTRODUCTION

In the UOP processing route for the production of Linear Alkybenzene (LAB), a precursor for Linear Alkybenzene Sulfonate (LAS), linear paraffins, generally a mixture of nC_{10} - nC_{13} or nC_{11} - nC_{14} , are catalytically dehydrogenated to the corresponding linear olefins in a UOP Pacol process unit. The effluent from this unit is then alkylated with benzene in a UOP/CEPSA Detal Unit or in a Detergent Alkylation unit in the presence of HF catalyst to produce LAB.

Small amounts of di-olefins (DO) are also produced in the Pacol reactor. The di-olefins form undesirable byproducts when alkylated, decreasing the yield of LAB and degrading LAB quality. Di-olefins also if present in Detal Unit feed will have a negative impact on Detal catalyst life. Processing the Pacol Separator liquid through the DeFine Unit selectively converts these undesirable species to the desired mono-olefins (MO). The DeFine Unit consists primarily of a reaction vessel(s) containing one or more beds of the proprietary DeFine catalyst. In the typical UOP designed LAB complexes, the DeFine reactor is placed between the Pacol Separator and the Pacol Stripper. A standard flowscheme is indicated in Figure I-1 and Figure I-2. The DeFine process significantly improves the overall efficiency and profitability of an LAB Complex operation when Heavy Alkylate (HAB) is an undesirable product.

In a Pacol/Olex complex, DeFine increases the mono-olefin content of the Olex Extract while decreasing the di-olefin concentration by over 90%. Figure I-3 shows DeFine integrated with Pacol/Olex.

FIGURE I-1

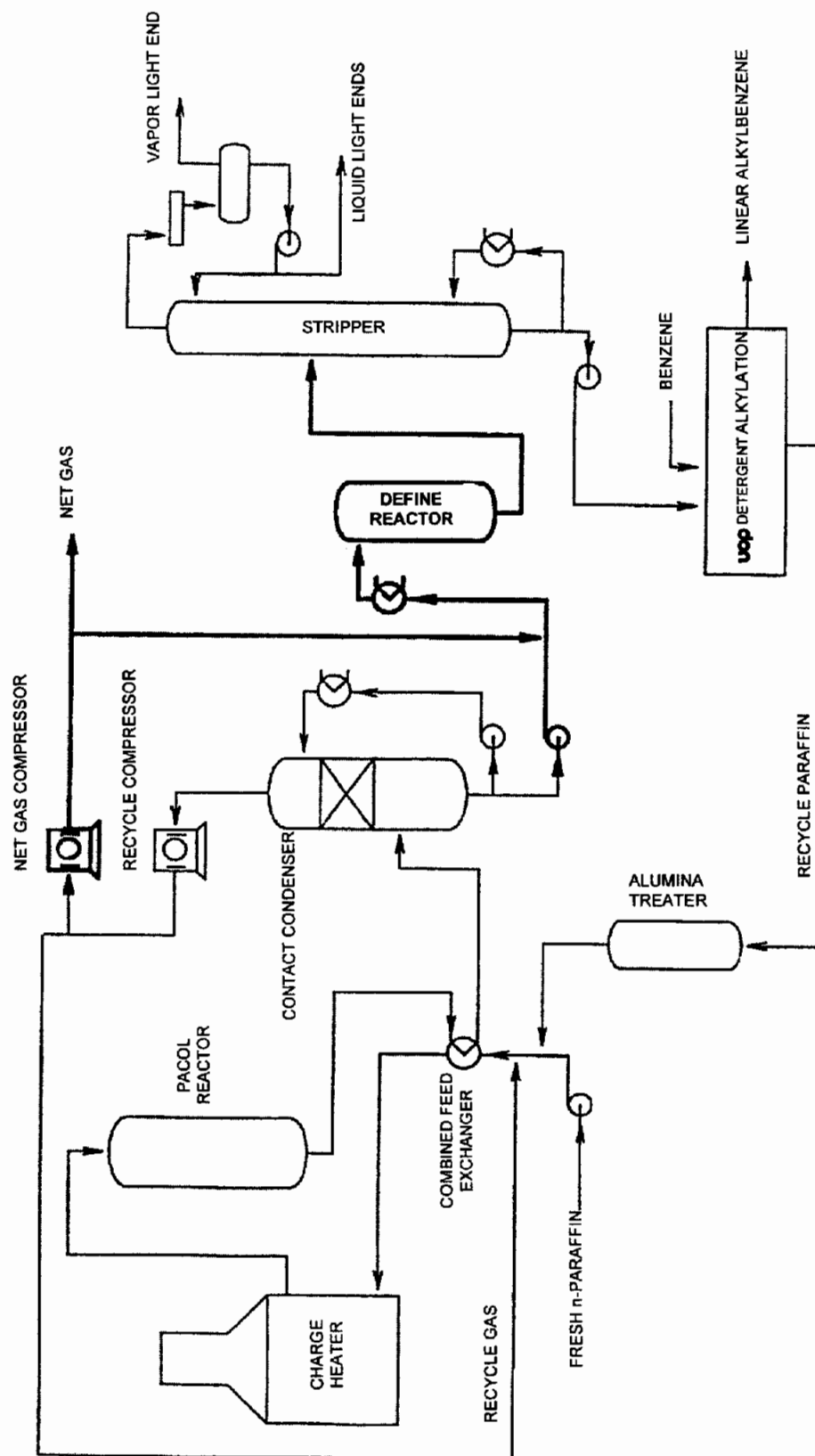


FIGURE I-2

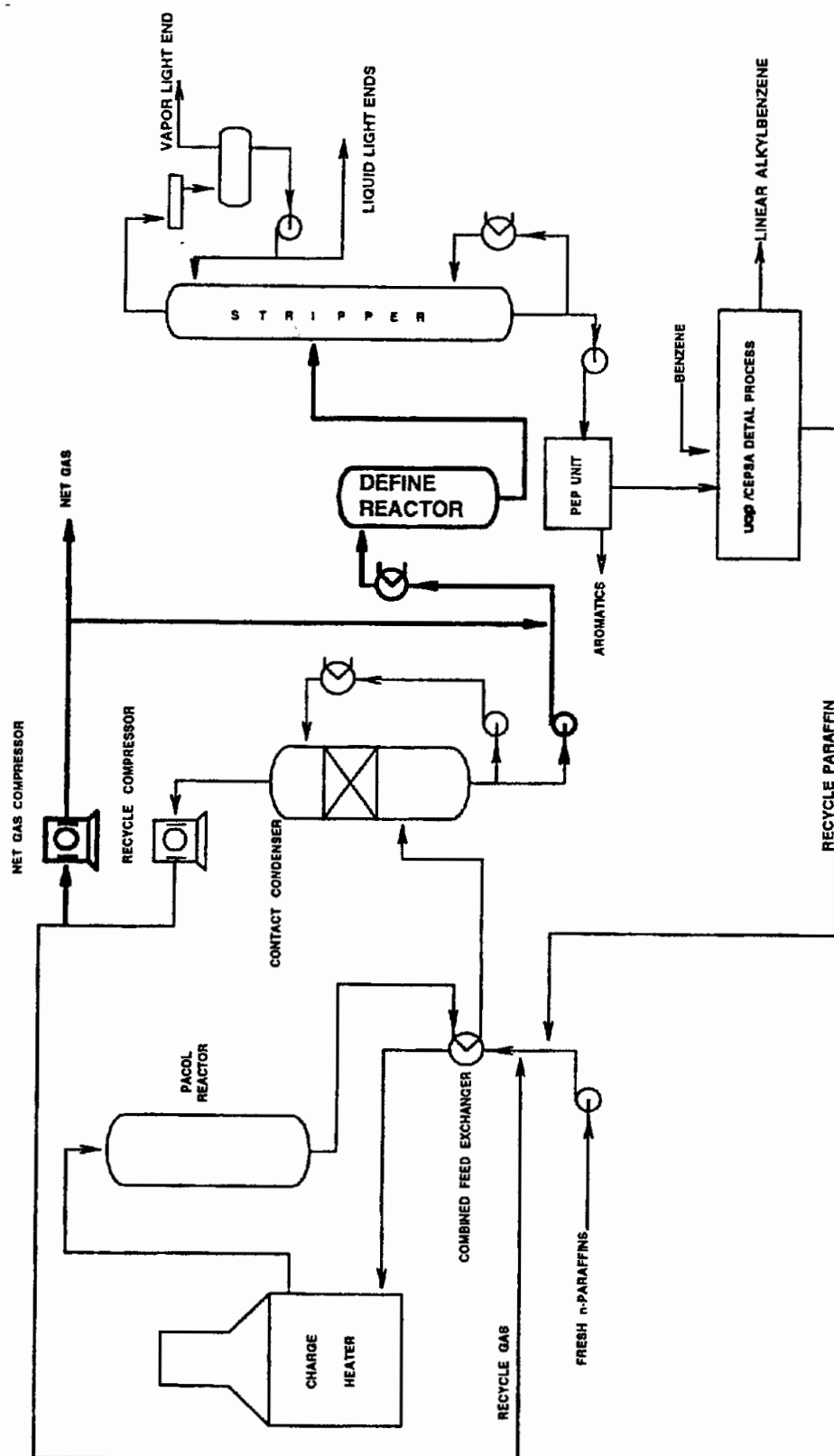
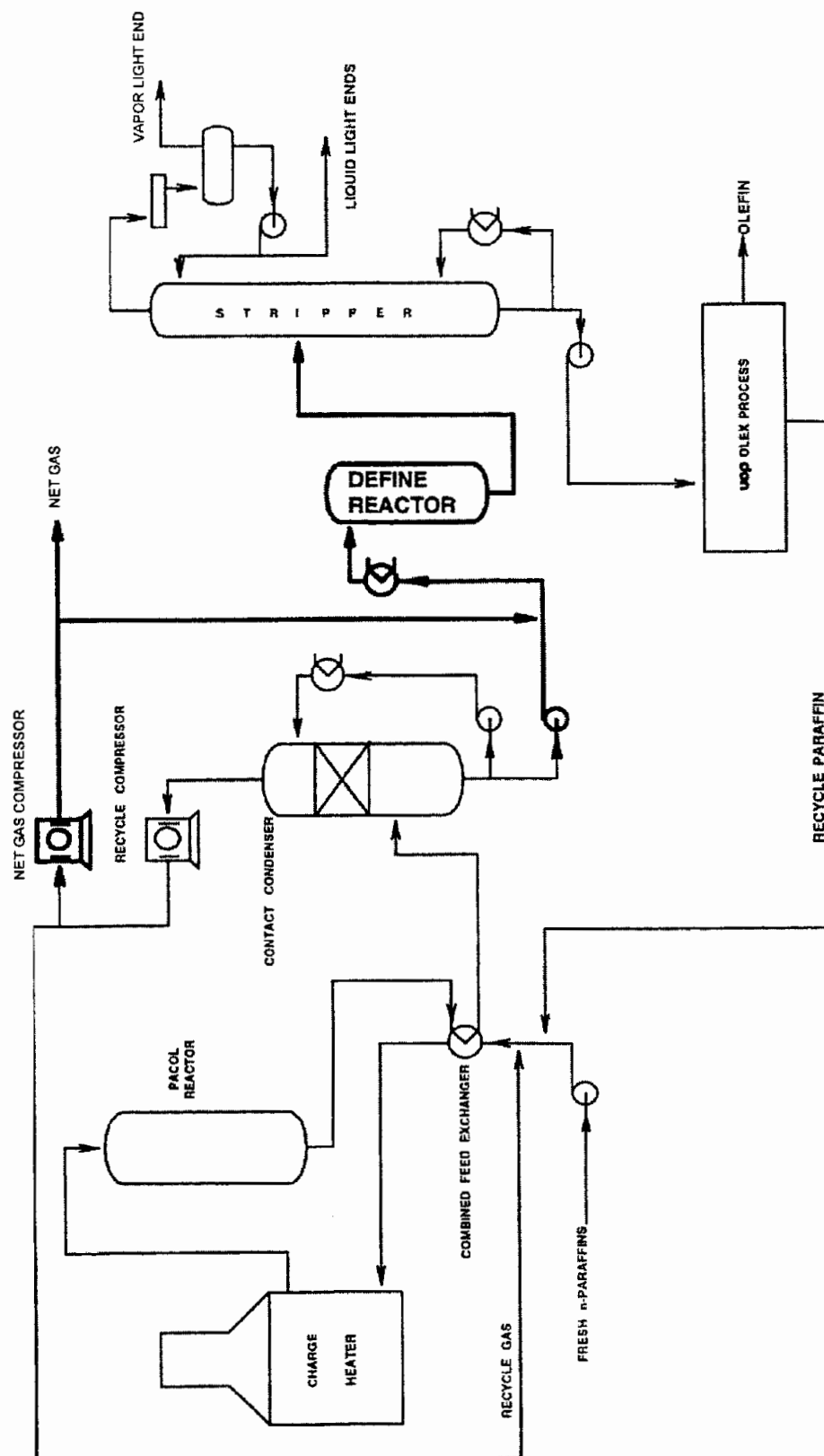


FIGURE I-3



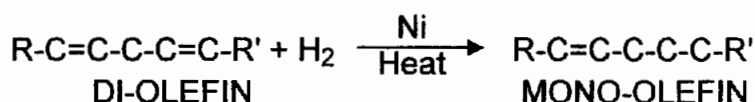
II. PROCESS PRINCIPLES

A. CHEMISTRY

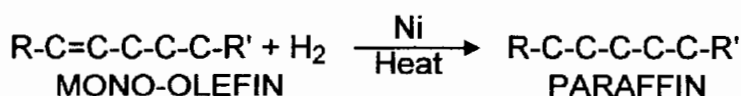
The hydrocarbon chemistry associated with the DeFine Process is straightforward. The major reactions are illustrated in Figure II-1. The DeFine reactions are carried out in the liquid phase, over a fixed bed of catalyst and under controlled conditions of temperature, LHSV, and hydrogen addition rates. Over a proprietary catalyst, a high percentage of the di-olefins present in the Pacol Reactor effluent are selectively hydrogenated to the corresponding mono-olefins in the presence of sulfur. At 90-95% di-olefin conversion, the selectivity to mono-olefins at start of run is 50%.

**FIGURE II-1
DEFINE REACTIONS**

Reaction #1



Reaction #2

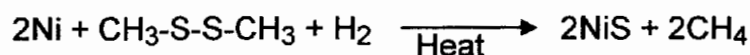


For a Detal Process Unit or a UOP HF Detergent Alkylation Unit the conversion of the di-olefins can result in a significant decrease in heavy alkylate production, and feed consumption. Also high levels of di-olefins in Detal Unit feedstock will negatively impact Detal catalyst life. Lower di-olefins in Olex feedstock result in improved product purities and recoveries in the Olex unit.

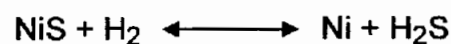
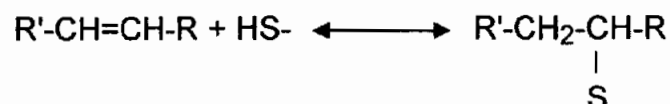
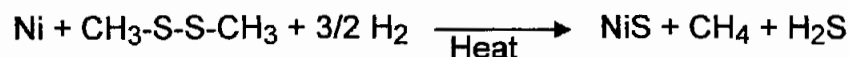
The sulfur chemistry is more complex. Possible reactions appear in Figure II-2. The desired reaction is the formation of NiS which attenuates the Ni atom site as an active reaction site. The presence of sulfur is an additional variable for adjusting the selectivity. Theoretically, sulfur injection is required to selectively hydrogenate DO and reduce MO saturation to paraffin. Without sulfur injection, hydrogenation of MO to paraffin will occur more readily, selectivity to MO will be very low, and high conversion of DO will be difficult to achieve without excessive hydrogen.

**FIGURE II-2
DMDS REACTIONS**

Desired Reaction



Competing Reactions



Figures II-3a and 3b depict the concentration profiles across the DeFine reactor for H₂ and DO respectively. The lines represented by 1 wppm Sulfur injection are the target performance.

FIGURE II-3a
H₂ Concentration Profile in the DeFine Reactor

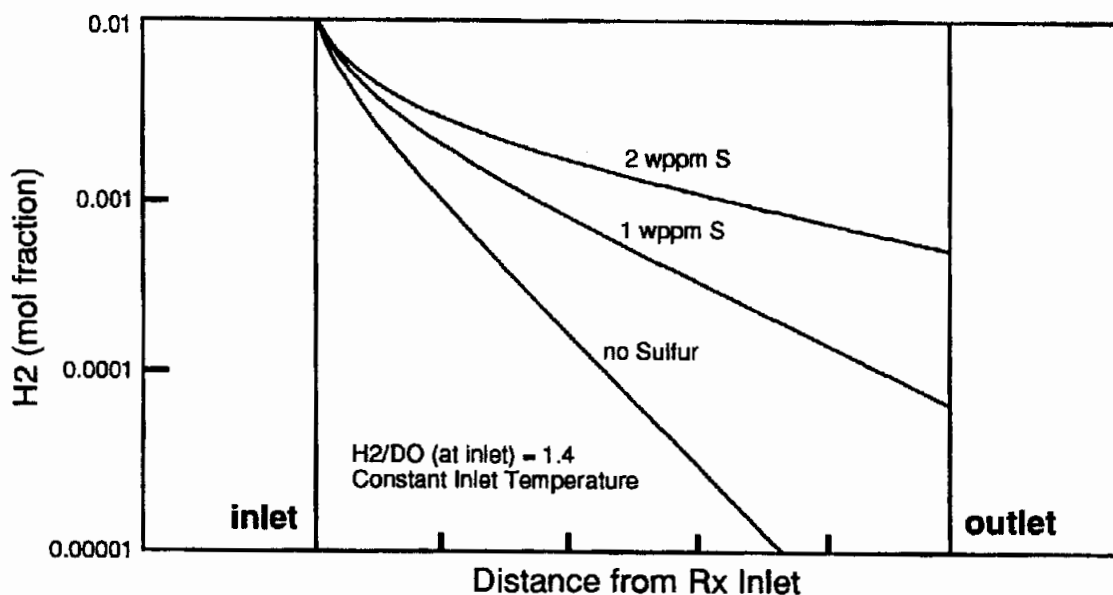
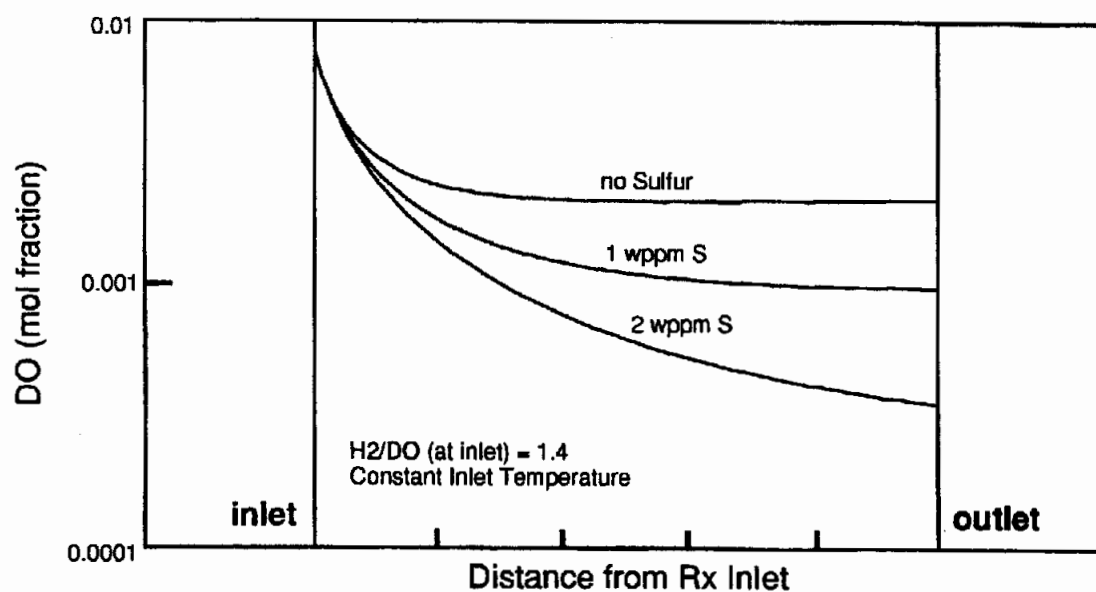


FIGURE II-3b
DO Concentration Profile in the DeFine Reactor



At 1 wppm S, the H₂ is more than 99% reacted at the outlet of the reactor. Under the same conditions, with no sulfur injection, 99% of the reactions take place in the top 40% of the catalyst bed. Conversely, with 2 wppm S injection, 10% of the H₂ remains unreacted.

As long as there is H₂ available, olefins will be converted. The rate of conversion is a function of the di-olefin concentration, [DO], the H₂ concentration, [H₂] and the mono-olefin concentration, [MO].

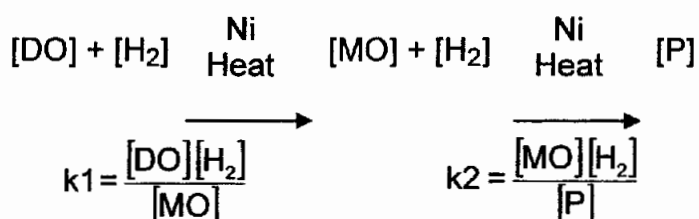
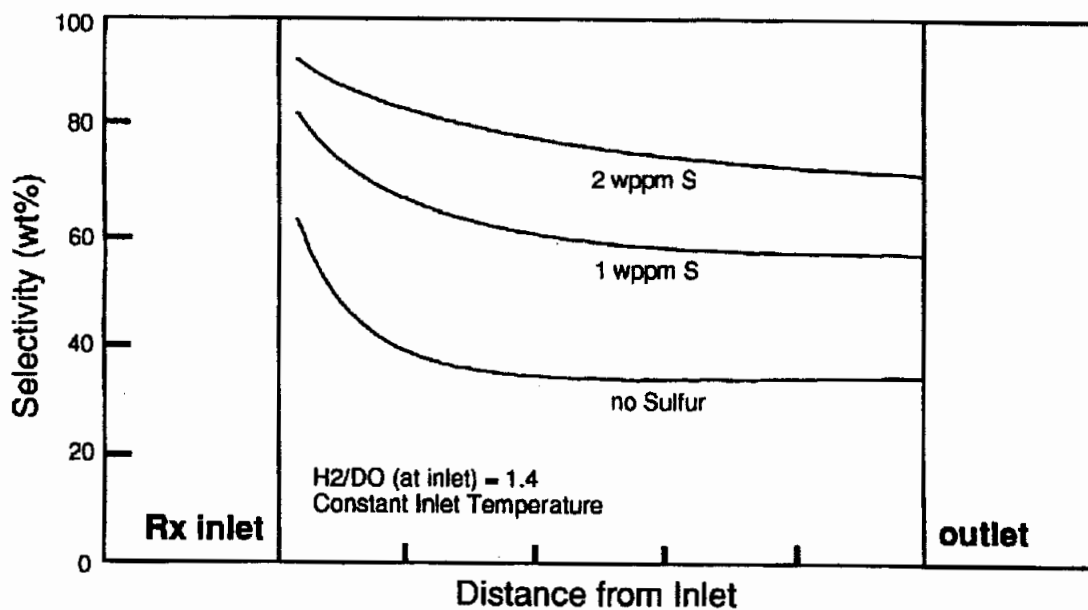


Figure II-4 represents the conversion and selectivity profile across the DeFine reactor based on the above model. As sulfur injection increases, the equilibrium level of NiS in the reactor changes. This has the effect of reducing the MO to paraffin reaction rate relative to the DO to MO rate. Consequently, the di-olefin Conversion increases and mono-olefin Selectivity increases (Figure II-4).

FIGURE II-4
Selectivity to MO Profile in the DeFine Reactor



Figures II-3a through 4 are mathematical constructs intended to convey the trends associated with DeFine. They are not the result of any pilot plant or commercial testing.



III. PROCESS VARIABLES

The process variables that influence the DeFine Unit operations are as follows:

A. PRESSURE

The DeFine Unit pressure is selected to ensure that the hydrogen remains in solution. Pressure is fixed by design and is not varied. The pressure in the reactor is determined by the amount of hydrogen required in solution to achieve the desired conversion (usually 90 to 95%). The latest DeFine Unit design pressures have ranged from 12 to 38 kg/cm². Unit designed with multiple catalyst beds and hydrogen injections are designed to operate at a lower pressure thus avoiding the need for a high head pump.

B. LHSV

The LHSV across the DeFine reactor is defined as the volumetric ratio of the feed rate charged per unit volume of DeFine catalyst. The design LHSV is 5.0. Since the Pacol Unit is always operated at design or higher rates (seldom less than design), the DeFine Unit is also almost always at design LHSV.

C. TEMPERATURE

The reaction temperature is selected to offer the optimum reaction rates for di-olefin conversion. Although the unit is designed for an end of run reactor temperature of 220°C, the initial operation is at 180-185°C. Sometimes heat integration may make it more economical to operate at a certain temperature within the normal operating range.

Theoretically, since the rate of DO conversion to MO is much greater than the rate of MO conversion to paraffin, when H₂ is the limiting reactant, (minimum H₂/DO ratio) an increase in temperature will increase conversion and selectivity. With a fixed amount of hydrogen available, increasing the number of moles of di-olefin converted to mono-olefin leaves less hydrogen to convert mono-olefins to paraffins. Proper optimization consists of a balance between temperature and hydrogen to di-olefin ratio and sulfur injection.

D. H₂/DI-OLEFIN RATIO

Theoretically, hydrogen is the limiting reactant in the DeFine reactions. Operating with a large amount of excess hydrogen will reduce the theoretical maximum selectivity. At a theoretical 100% conversion of di-olefin to mono-olefin, any excess hydrogen will convert the mono-olefin to paraffin, thereby reducing the mono-olefin selectivity.

The following mole balance table illustrates the theoretical relationship between excess hydrogen and mono-olefin selectivity.

The H₂/DO ratio is one, conversion is 75% and selectivity is 67 wt-%. Reaction #1 represents di-olefin converting to mono-olefin, and Reaction #2 is the excess hydrogen converting the mono-olefin. This is for illustrative purposes only. Because of imperfect hydrogen distribution and other physical limitations some excess hydrogen is necessary to maintain high conversions.

	Feed (mol)	Reaction #1	Reaction #2	Effluent (mol)
Paraffin	0	0	25	25
Mono-Olefin	0	75	50	50
Di-Olefin	100	25	25	25
H ₂	100	25	0	0

The other way to increase conversion is to increase the hydrogen to di-olefin ratio. With all other conditions held constant, selectivity to mono-olefins (MO) decreases with increasing conversion by increasing the excess hydrogen to the

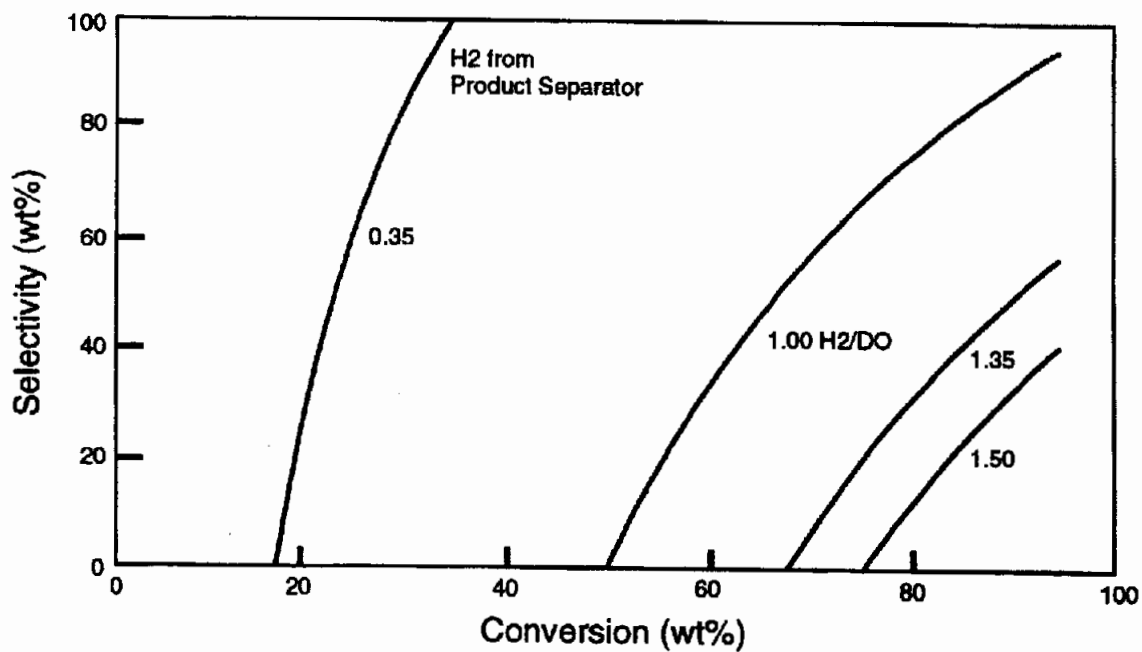
reactor. The position in the conversion/selectivity graph is determined by the amount of hydrogen that is consumed in the reactor. Assuming that all of the hydrogen is reacted, this can be expressed as the molar ratio of Feed H₂ to di-olefins (DO):

$$\text{H}_2/\text{Di-Olefin Molar Ratio} = \frac{\text{Feed H}_2 \text{ (moles)}}{\text{Feed Di-Olefins (moles)}}$$

Theoretically, a H₂/DO ratio of 1.4 will result in about 90% DO conversion with 50% selectivity to MO. For calculation purposes, assume that the dissolved hydrogen coming from the Pacol Separator to the DeFine Unit is 0.3 Nm³ H₂/m³ hydrocarbon. The precise value will be a function of Product Separator temperature and pressure and the Recycle Gas composition. Also assume that all of the hydrogen reacts to form either MO or paraffins.

Hydrogen is the usually the limiting reactant in the DeFine reaction scheme. With all other conditions held constant and a fixed amount of hydrogen available, selectivity to mono-olefins increases with increasing conversion as shown in Figure III-1. The position along this Conversion/Selectivity curve is determined by the relative reaction rates between DO conversion to MO and MO conversion to paraffin. The variable available for adjusting the relative reaction rates is the amount of sulfur as NiS on the DeFine catalyst.

FIGURE III-1
Theoretical Performance



Assumes 100% H₂ utilization

E. SULFUR INJECTION

Sulfur in the form of a sulfiding agent is injected upstream of the Reactor in order to attenuate the activity of the catalyst. Without sulfur, the Selectivity to mono-olefins is relatively low as the active catalyst will hydrogenate mono-olefins to paraffins at a greater rate than the conversion of di-olefins to mono-olefins and high conversion of DO will be difficult to achieve. The recommended sulfur species for DeFine is SA-200. Care must be taken to adjust the sulfur injection calculation to account for different sulfur contents.

UOP recommends a continuous sulfur injection at a low rate. The precise rate will depend upon unit performance. A rate that will provide 1-2 wppm sulfur based on liquid feed has been found to be adequate.

Some mercaptan is also known to form. The H₂S is removed in the Pacol Stripper Column usually in the stripper overhead liquid. The higher molecular weight mercaptans exit the process in the Stripper Column bottom product.

Sulfiding agents are typically chosen based on their handling properties and site considerations. DMDS (Dimethyl Disulfide) or heavier sulfiding agents are most typically used.

F. CALCULATIONS

The performance of the DeFine Unit is characterized in terms of di-olefin Conversion and Selectivity to mono-olefins. The method of calculation of these two parameters is shown below.

Conversion

$$\text{Di-Olefin Conversion, \%} = \frac{(\text{Feed DO wt-\%} - \text{Effluent DO wt-\%})}{\text{Feed DO wt-\%}} \times 100$$

The terms are expressed as the mass flow rate of di-olefins. If samples are taken at the inlet and outlet of the DeFine Reactor, the terms can be expressed

as wt% since there are no other streams to/from the Reactor. If the samples are taken from the Product Separator and the Stripper Bottoms, both the flow rate and the analysis of each stream are required since some hydrocarbon is leaving from the Stripper Overhead. If a quick estimate is required, reasonable accuracy can still be obtained by ignoring the Stripper Overhead flow.

Selectivity

$$\text{Mono-Olefin Selectivity, wt - \%} = \frac{(\text{Effluent MO wt - \%} - \text{Feed MO wt - \%})}{(\text{Feed DO wt - \%} - \text{Effluent DO wt - \%})} \times 100$$

At 0 wt% Selectivity, none of the di-olefins are being converted to the corresponding mono-olefin. Conversely, at 100 wt% selectivity all of the di-olefin is converted to mono-olefin and none is further converted to paraffin.

H₂/DO Molar Ratio

$$\text{H}_2/\text{DO} = [\text{H}_2 \text{ from Pacol Separator} + \text{H}_2 \text{ injection}] / \text{inlet DO}$$

Example:

Calculate the Hydrogen injection rate required to achieve a 1.4 H₂/DO at the DeFine reactor inlet assuming the following conditions:

Basis:

Flow rate = 80 m³/h @ Gb = 0.75

DO MW = 158

DO = 0.8 wt%

1 hour

- 1) H₂ from Pacol Separator Liquid
Assume 0.3 Nm³ H₂/m³ HC at Separator temperature and pressure
80 m³ x (0.3 Nm³ H₂/m³ HC) = 24 Nm³ H₂
24 Nm³ H₂/22.4 Nm³ H₂/kgmol = 1.07 kgmol H₂ from Pacol
- 2) kg/mol DO in DeFine Reactor feed
80 m³ HC x 750 kg/m³ x 0.008 kg DO/kg HC = 480 kg DO
480 kg DO/(158 kg/kgmol) = 3.04 kgmol DO
- 3) Total H₂ Required at DeFine Reactor inlet to Achieve 1.4 H₂:DO Ratio
1.4 H₂/DO x 3.04 kgmol DO = 4.3 kgmol H₂
- 4) H₂ injection rate
H₂ injection = H₂ Total Required - H₂ from Pacol Separator Liquid
= (4.3 kgmol - 1.07 kgmol) H₂ = 3.2 kgmol H₂
= 3.2 kgmol H₂ x 22.4 Nm³/kgmol = 71.7 Nm³ H₂
= 75.5 Nm³/h injection rate @ 95% H₂ purity

DMDS Injection**Example:**

Calculate the DMDS injection rate required to achieve 1.5 wppm S at the DeFine Reactor inlet assuming the following conditions:

Basis:

Flow rate = 80 m³/h @ Gb = 0.75

DMDS sp. gr.(20°C) = 1.0625

DMDS sp. gr.(40°C) = 1.04

DMDS storage vessel @ 28°C

1 hour

- 1) Calculate DMDS specific gravity

$$G_i = mT + G_o$$

$$= (-1.125 \times 10^{-3}) \times T + 1.085$$

$$G_{28} = (-1.125 \times 10^{-3}) \times 28^\circ\text{C} + 1.085 = 1.054 \text{ kg/L}$$

- 2) Calculate Sulfur requirement

$$80 \text{ m}^3 \times 750 \text{ kg/m}^3 \times (1.5 \times 10^{-6}) \text{ kg S/kg HC} = 0.09 \text{ kg S}$$

$$0.09 \text{ kg S} / (0.68 \text{ kg S/kg DMDS}) / (1.054 \text{ kg DMDS/L}) = 0.126 \text{ L}$$

126 mL/h DMDS injection

G. ANALYSES

The wt% total di-olefins and mono-olefins in the feed and effluent streams can be determined by High Performance Liquid Chromatography (HPLC) or mass spectrometry (MS). MS equipment is expensive and difficult to operate; it is seldom feasible to have an instrument at the plant site. HPLC equipment is less expensive and much easier to operate. UOP Methods 869 and 902 have been developed to enable rapid, accurate determination of DeFine Unit performance.

UOP recommends using both methods in parallel for all critical samples where the operation of the unit will be modified.

H. CATALYST POISONS

A catalyst poison is something that will cause a loss in catalyst activity. Most catalyst poisons will deactivate the Pacol Unit catalyst before having any affect on the DeFine Unit. Early indications of contaminants entering the DeFine Unit can be seen in the Pacol or other upstream units' catalyst performance.

Extreme care must be taken in the complex that has a UOP/CEPSA Detal Unit, reduced diolefin conversion in the DeFine Unit could lead to shortened Detal catalyst life. If the DeFine Unit is by-passed for any reason, then the Detal Unit must be by-passed (see Detal Unit General Operating Manual). If the Define Unit is not achieving acceptable di-olefin conversion, some reduction of Detal Unit feed di-olefin content may be achieved by:

- Reduction of Pacol conversion thereby reducing the amount of diolefins produced in the Pacol Reactor which is fed to the Define Reactor.
- Adjustment of the PEP Unit operation to remove more diolefins.

1. Water

Water in amounts normally seen in a Pacol Unit separator bottoms are not a problem for the DeFine catalyst. Higher concentrations of water may suppress some of the DeFine catalyst activity. Once the water rate to the DeFine Unit is back to normal operating levels, the DeFine catalyst activity should recover.

2. Sulfur

Excessive amounts of sulfur in the liquid feed or hydrogen will over attenuate the DeFine catalyst, leading to a loss in activity. Once the sulfur

rate to the DeFine Unit is back to normal operating levels, the DeFine catalyst activity should recover.

3. Chlorides

High chloride levels in the feed or hydrogen to DeFine may cause an increase in isomerization activity. Chloride in the feed to a Pacol Unit will be very noticeable in the form of increased non-normals production and increased cracking. Once the chloride rate to the DeFine Unit is back to normal operating levels, the DeFine catalyst activity should recover.

4. Nitrogen Compounds

Nitrogen compounds will act as a temporary poison to the DeFine catalyst. Nitrogen in the feed to a Pacol Unit will be very noticeable in the form of decreased activity. Once the nitrogen rate to the DeFine Unit is back to normal operating levels, the DeFine catalyst activity should recover.

5. Metals

There should not be any metals in the feed to a DeFine Unit. However, should metal contamination occur, it would be expected that permanent catalyst deactivation would occur. If catalyst containment problems develop in the Pacol Reactor section, Platinum catalyst or catalyst fines could enter the top of the Define Reactor (see X. Troubleshooting).





IV. PROCESS FLOW AND CONTROL

A. INTRODUCTION

Reactor feed is mixed with hydrogen, heated, and then passed over a fixed bed of catalyst. The reactor effluent is then routed to the Stripper Column. The primary systems of equipment in the DeFine Unit are the liquid feed system which includes the booster pump and filter, hydrogen feed system (sometimes includes Hydrogen booster compressor), reactor feed heat exchanger, reactor (single or multi-beds), and sulfide injection system.

B. PROCESS FLOW DESCRIPTION

A simplified process flow scheme is presented in Figure IV-1. Feed to the DeFine Unit originates from the Pacol Unit Product Separator. Material from the Product Separator is sent to the suction of the DeFine Unit booster pump, filtered, mixed with hydrogen, heated then it is charged to the reactor.

Separator Level Control and Define Reactor Pressure Control with a Low Signal Selector (LSS)

The feed rate from the booster pump is controlled. The flow control to the reactor (via FIC) is normally reset by the separator LIC control. There is a PIC on the reactor outlet that sends a signal to a LSS which acts as a low pressure override.

The Feed discharged from the Booster Pump is mixed with a hydrogen-rich (90+ mol %) gas stream that originates either from an outside process unit or from the booster compressor. To provide complete dissolution of the hydrogen, the two streams are thoroughly mixed. The mixing may occur in a static mixer or sometimes mixers are not required if adequate mixing occurs in downstream equipment such as a heat exchanger. Thorough cleaning of the make-up hydrogen line prior to startup is highly recommended to reduce instrument and maintenance related problems in the hydrogen system.

Hydrogen Flow Control (FIC)

The hydrogen feed is either from an outside unit or from the Pacol unit via a booster compressor. The hydrogen is filtered through sintered metal cartridges and sent to each catalyst bed via an independent flow controller (FIC). Hydrogen flow is stopped automatically when the liquid feed flow goes below the trip setpoint.

Define Reactor Temperature Control (TIC)

The Reactor feed is then heated to the desired Reactor inlet temperature. This is accomplished by heat exchanging the DeFine Feed with a hotter process stream (usually Pacol Stripper bottoms). The Reactor inlet TIC typically controls the amount of flow which bypasses the heat exchangers. The heated reactor feed is directed downflow over a fixed bed of catalyst in the DeFine Reactor. Flow distribution is accomplished by an inlet distributor which directs the flow toward the top head of the Reactor.

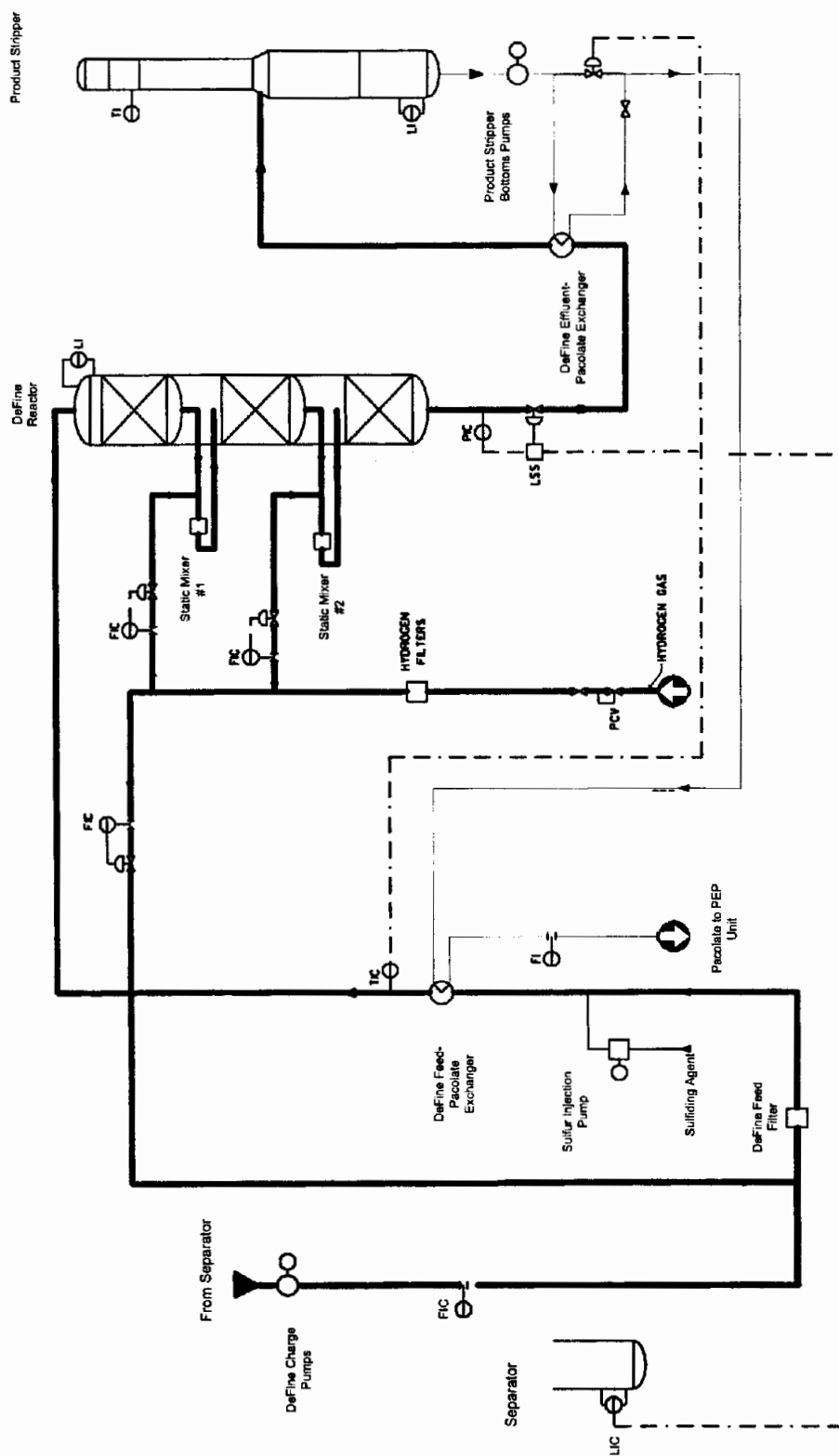
Reactor effluent passes through the control valve on the outlet line and is sent to a downstream fractionation column (usually Pacol Stripper column) where light ends are removed.

The process also incorporates a sulfur injection system. A sulfur compound is injected into the Booster Pump suction or discharge. Suction injection provides better mixing of the sulfur with the hydrocarbon. The sulfur injection pump is a positive displacement pump which injects an adjustable volume of sulfiding agent as required by the process.

In addition to the above mentioned piping and equipment, there are also the following process lines used in the startup or shutdown of the Define Unit:

1. DeFine Reactor bypass. This line can be used for paraffin circulation or a temporary shutdown of the Define Unit.
2. Emptying and Filling line used for the initial liquid fill step, and for draining after a shutdown.

FIGURE IV-1



V. PROCESS EQUIPMENT

A. BOOSTER PUMP

The Booster Pump is a high head centrifugal pump. This pump boosts the feed pressure from the Pacol Unit Product Separator (typically 1.2 kg/cm²) to the DeFine Reactor system pressure. The DeFine Unit is operated at a pressure sufficient to keep the H₂ in solution. For typical design H₂ injection rates and Reactor temperatures, the pressure should be in the range of 12-38 kg/cm² (156-498 psig).

B. HYDROCARBON/HYDROGEN MIXER

The hydrogen mixer (Figure V-1) is constructed from a pipe "T". The gas is injected into the hydrocarbon through a sintered stainless steel element that extends into the flow region. The hydrocarbon flows around the element and is intimately mixed with the fine bubbles of gas. The mixer ensures that the hydrogen is completely and uniformly dissolved in the liquid hydrocarbon. Alternatively some designs have a slotted hydrogen injection quill with a static mixer supplied downstream to ensure the mixing is complete.

If the hydrogen lines from the compressor discharge to the hydrogen mixer are not thoroughly cleaned prior to startup, the hydrogen mixer if supplied with a sintered element will plug, and the DeFine will have to be shut down for cleaning. Purchase new sintered elements or follow the manufacturer's cleaning recommendations.

C. REACTOR FEED HEATER

The Reactor Feed heater is a shell and tube heat exchanger constructed out of killed carbon steel. The process fluid flows through the shell side while the Hot

Oil (or other heating medium) flows through the tube side. The Hot Oil rate is reset by a temperature controller located on the reactor inlet line.

D. DEFINE REACTOR

The DeFine Reactor (Figure V-2) is a vertical-mounted vessel fabricated out of killed carbon steel.

The feed enters the vessel through a side-mounted distributor that distributes the combined feed over the cross-section of the reactor. The liquid travels downflow in a plug flow fashion through the fixed bed of DeFine catalyst.

UOP has designed some multi-stage DeFine Reactors. A three-bed reactor is shown in Figure V-3. In this reactor, feed enters the top similar to a single stage unit. The major difference is that a fraction of the hydrogen is split between each stage. These multi-stage DeFine reactors can be built in one shell as shown or in separate shells. In the single shell design, hydrogen is mixed into the liquid hydrocarbon typically externally to the vessel.

The reactor is intended to operate liquid full. In the event that hydrogen comes out of solution, there is a level glass or level indicator at the top of the reactor that will allow the operators to determine if there is insoluble hydrogen at the top of the reactor. Once out of solution, it is difficult to re-dissolve the hydrogen therefore the gas bubble must be manually vented.

At the bottom of the reactor are one or more catalyst unloading nozzles. The number of nozzles usually depends on the diameter of the vessel. The nozzles are designed to allow the spent catalyst can be removed without disturbing the alumina support spheres.

E. SULFUR INJECTION SYSTEM

A sulfur agent is injected by the Booster Pump (Figure V-4). The sulfur injection provides optimum catalyst selectivity to mono-olefins.

The suction side of the injection system consists of the sulfide drum (provided with flexible metal hose drip free connections, flame arrestor, and N₂ blanket system), site glass connections, and connection to the positive displacement pump suction.

The pump stroke sets the flow rate. The stroke is manually adjusted at the pump. The suction site glass must be calibrated prior to inventory with sulfiding agent to allow accurate determination of the actual flow rate.

In case of high discharge pressure, a spillback safety valve is provided.

F. DEFINE FEED FILTERS

A cartridge filter with fiber wound elements is designed to prevent foreign debris and or Pacol catalyst dust from plugging the Define reactor catalyst bed.

G. DEFINE SHUTDOWN INTERLOCK SYSTEM

The trip philosophy associated with the DeFine Process is illustrated in Figure V-5. A low feed flow, as registered by the feed flow transmitter, will initiate the shutdown of the makeup gas stream entering the DeFine Unit. The DeFine Unit is designed to operate with the reactor liquid full. If the hydrogen comes out of solution, the gas bubble may enter the catalyst bed. The liquid may start to channel, and once begun, it is difficult to eliminate, even after the gas bubble is eliminated.

If the Feed rate falls below a pre-determined value (most likely due to loss of the Booster Pump) the makeup H₂ is automatically blocked in to prevent filling the Reactor with gas.

**FIGURE V-1
HYDROGEN MIXER**

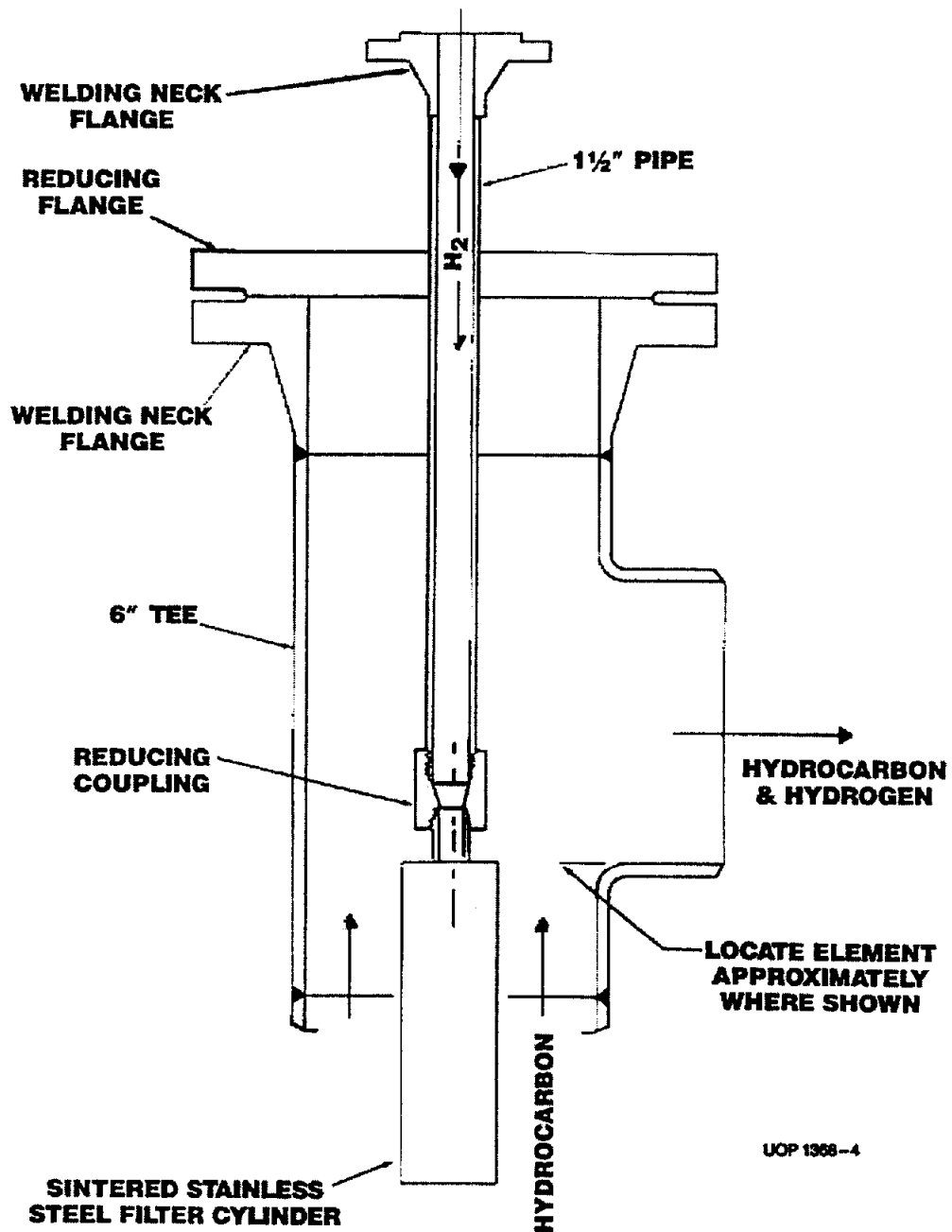


FIGURE V-2
DEFINE UNIT SINGLE BED REACTOR DIAGRAM

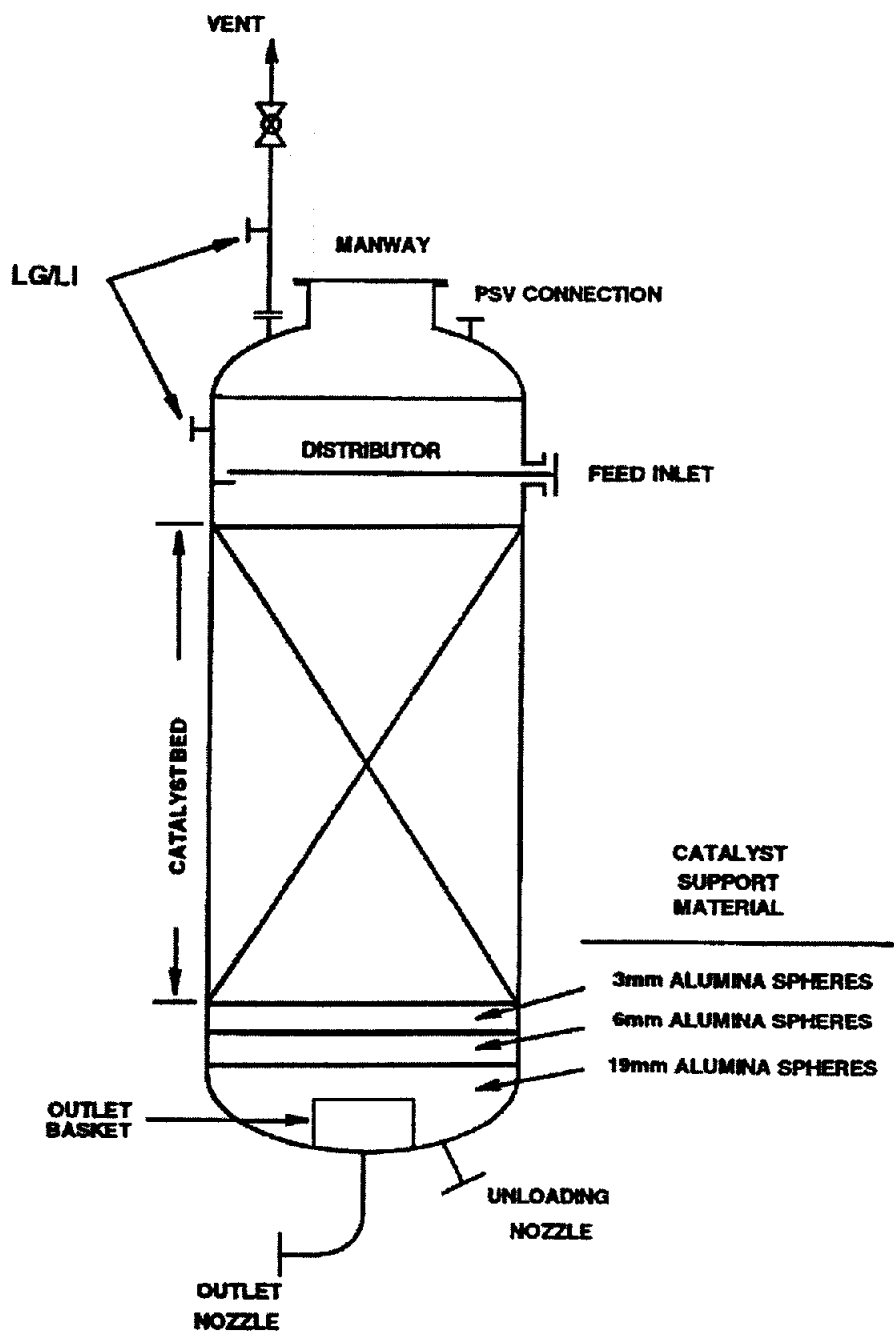


FIGURE V-3 DEFINE UNIT MULTI-STAGE REACTOR

SECTIONAL ELEVATION

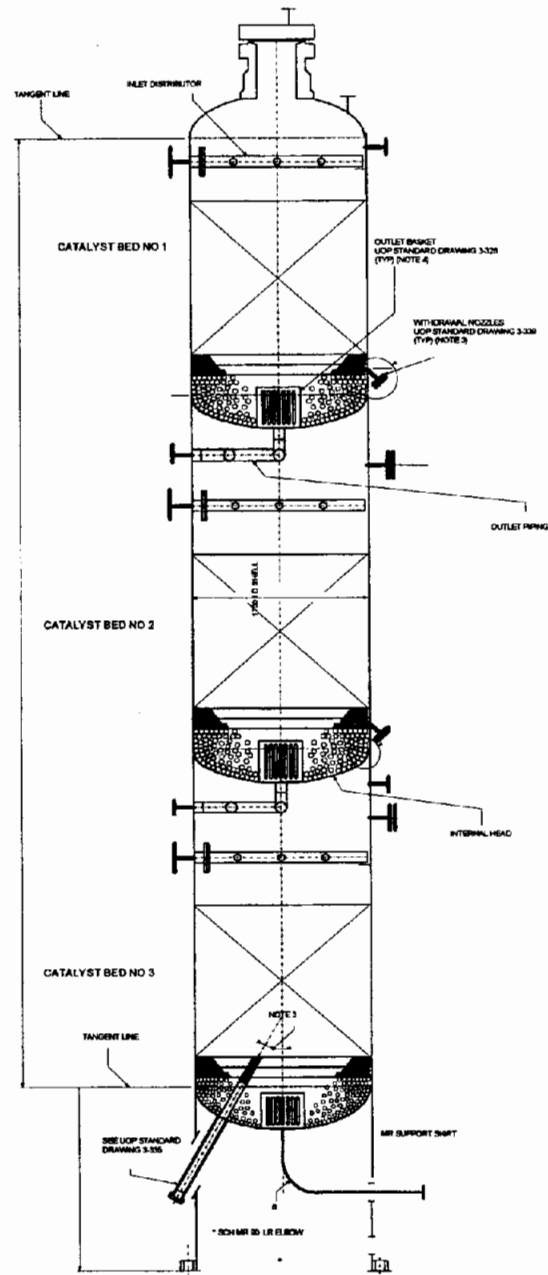


FIGURE V-4

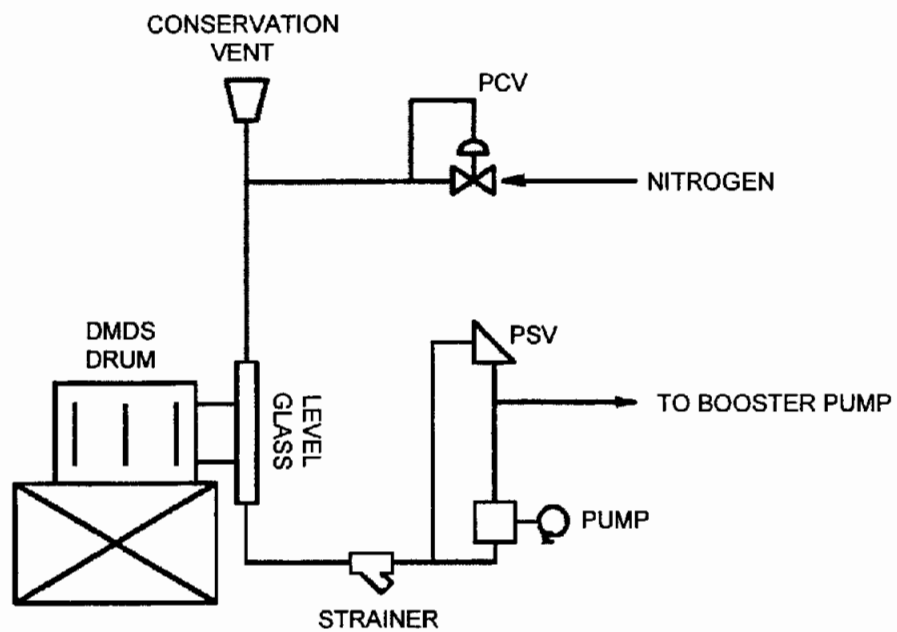
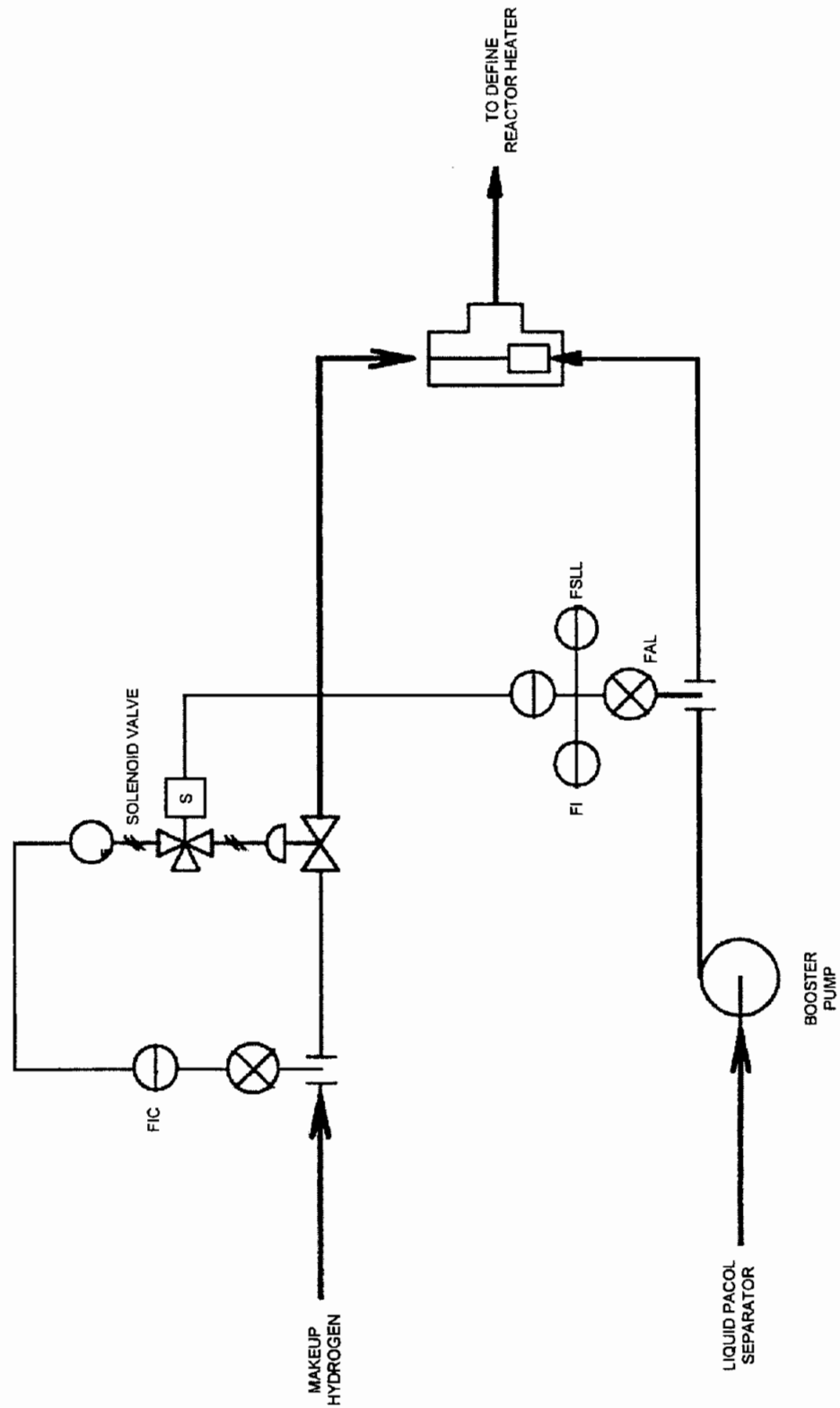


FIGURE V-5





VI. COMMISSIONING

A. 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:

1. Vessel temperature and pressure rating
2. Vessel metallurgy
3. Vessel elevation
4. Vessel lining
5. Trays: number, type, spacing, levelness, liquid seal, weir height, valve number and operation, metallurgy, weep holes, supports
6. Distributors: location, alignment, size and number of openings
7. Nozzles: location and flange rating and facing
8. Vortex breakers
9. Demisters: size, materials, support
10. Thermowells: location and flange rating and facing
11. Level instruments
12. Linings
13. Fireproofing
14. Inspection plates in place
15. 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.**

B. 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:

1. Line sizes and materials
2. Flanges and gaskets
3. Bolting
4. Drains and vents
5. Pump ratings
6. Relief valve settings
7. Lines and equipment supports
8. Equipment accessibility
9. Expansion provisions
10. Utility lines
11. Tie-ins with other units/storage
12. General safety requirements
13. Heaters including burners and internals
14. Control valve operation and failsafe positions

C. 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.

1. Blind (or block and bleed) all safety valves, instruments, and pressure gauges that would be overpressured by the hydrostatic test pressure.
2. Test Gauges must be placed at the low point of a large line vessel so that the liquid head will be taken into account.
3. 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.

4. 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.

5. 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.)
6. 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.
7. 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.
8. 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.

9. 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.

D. FLUSH LINES

All piping should be thoroughly cleaned before startup. 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. Notes applicable to water flushing are:

1. Orifice plates should not be installed prior to flushing.
2. Control valves should be rolled out. Flush to both sides of the valves.
3. Instrument air lines should be thoroughly blown with clean, dry air, then closed off or disconnected.
4. Relief valves should be blinded. (Set pressure can be checked at this time in the shop.)
5. Dirty lines should not be flushed through exchangers. Disconnect lines at exchanger inlet and outlet and flush the lines leading to the exchanger.

6. 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.
7. When filling vessels, open a vent.
8. 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 too rapidly.
9. Lines should be flushed away from vessels. Do not flush debris into equipment.
10. Where possible, flush down or horizontally.
11. Flush headers from source to end, then flush lines away from header to end.
12. Flush through open end lines. When possible, do not restrict flow.
13. Flush through all drains and vents.
14. In all cleaning, use maximum safe volume and velocity.
15. Regulate flushing water from source.
16. Water flushed lines which do not drain freely should be blown clean with air.
17. Upon completion of line flushing for any system, carefully check that all temporary breaks are recognized, control valves replaced, blinds removed and pumps aligned.

E. 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:

1. Check rotation of electric motors uncoupled from the pump. Run in for a minimum of four hours uncoupled, verify operability.
2. Run in, uncoupled from pump, drivers other than electric. Check overspeed trip, bearings, control, etc.
3. Prior to running in the pump, do the following:
 - a. Review the manufacturer's operating instructions, taking note of special precautions such as minimum flow requirements, heat up rates, minimum operating speeds, etc.
 - b. Check that the overall installation is complete.
 - c. Verify that the pump and driver are properly grouted and aligned.
 - d. Verify that cooling water piping is connected and in service where required: pedestals, bearing jackets, stuffing boxes, etc.
 - e. Verify that steam piping is connected where required.

- f. 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. For seal fluid provided by an external source, confirm adequate pressure at seal, normally about 1.4 kg/cm^2 greater than the suction pressure.
 - g. Verify that packing seals are installed.
 - h. Verify that all casing connections which are not utilized are plugged with metallic, not plastic, plugs.
 - i. Verify that temporary strainers have been installed in the suction piping.
 - j. Verify that any required recirculation lines are properly installed and operable.
 - k. Verify that bearings and shafts are clean and properly lubricated.
4. When ready to start the pump, do the following:
- a. Rotate pump and driver by hand, verifying that they will roll freely.
 - b. Check that run-in water supply and delivery circuits are lined up.
 - c. Establish cooling water, where required.
 - d. Check that lubrication is satisfactory, commissioning auxiliary lube and/or seal oil consoles, if present, and confirming oil pressures and temperatures.

- e. Check that pump vent and case drains are closed.
- f. Open suction valves fully, venting air from piping and pump, completely filling with liquid.
- g. Open pressure gauge tapping.
- h. Make sure that electric power is available from the switch gear to the starter of the electric motor drivers.
- i. Barely open the discharge valve of a centrifugal pump and fully open the discharge valve of a positive displacement pump before starting.
- j. Warm up pumps in hot service at approximately 28°C/hr to within 28°C of operating temperature before placing in service. Check pump casing skin couples (if present) to assure that all parts of the casing are within 28°C of the operating temperature. Uneven expansion of the internals and casing may cause seizure and damage when starting.
- k. 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 difficulty is corrected.
- l. When the discharge pressure of a centrifugal pump has increased satisfactorily after starting, gradually open the discharge valve to obtain the desired flow rate.
- m. 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.

- n. 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.
5. 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.
6. The discharge valve of an operating positive displacement pump, rotating or reciprocating, should never be closed as the case can be overpressured.
7. 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.
8. Inspect and clean screens as required.
9. Recheck and realign, if required, after any disturbance of piping such as required for suction screen cleaning.
10. Operate the pump directing flows through all suction and discharge piping.

11. 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.
12. 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.

F. RECIPROCATING COMPRESSORS

Confirm that the compressor is lubricated or non-lubricated consistent with UOP specifications. The compressors will be started and operated according to the manufacturer's instructions. For the initial checkout and startup, a manufacturer's representative should be present. For lubricated machines, the manufacturer's recommended break-in cylinder and packing lubricating rates must be assured.

NOTE: Before starting any reciprocating compressor, the machine should be barred or jacked over by hand to make certain it is free. For reciprocating compressors the scheme of placing the machine on line should be similar to the following:

1. Purge the compressor with nitrogen through the suction purge valve to the flare or the atmospheric vent line.
2. Roll the machine over to ensure complete purge.
3. After nitrogen purging of the machine, introduce hydrogen to the compressor via the hydrogen pressuring line or by cracking open the suction block valve.

4. After partially pressuring the compressor with hydrogen, roll the machine over and vent the hydrogen to the flare or through the atmospheric vent to displace nitrogen in the machine.
5. Gradually open the compressor suction valve to pressure up the machine to line pressure.
6. Start steam to the steam tracing. Drain the suction line and snubbers of any liquid.
7. Make sure that there are no restrictions to the gas flow. Open any upstream flow control valves or spillback control valve prior to starting the machine.
8. As the machine is fully equipped with suction unloader valves, start up the machine as follows:
 - a. After the machine is pressured with hydrogen, close the small bypass-vent line, unload all of the suction valves and open the compressor discharge line.
 - b. Check the compressor's lubricating oil level in the crank case or reservoir.
 - c. Start the compressor and check the oil pressure.
 - d. Let the compressor idle for a few minutes while closely watching the suction temperature. Then close the suction valve loaders to put the machine on line. Follow the manufacturer's loading sequence if he has specified one.
9. After a few days operation, cylinder and packing lubrication rates should be reduced as per manufacturer's instructions.

G. 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 which 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, and 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 freed 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.

H. SERVICE AND CALIBRATE INSTRUMENTS

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

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

In inspecting instruments for compliance with specification, attention should be given to instrument range, pressure rating, connections, materials of construction, size, electrical characteristics, and special features. Special attention should be paid to notes in the specification. Orifice plates should be inspected as soon as possible so they will be ready for installation when line flushing is complete; inspection should include size, material of construction, location of weep hole, if any, counter/bore, overall condition, measurement of the 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 the straight run requirements are met. Instrument piping should be inspected visually for completeness and condition of welds and the 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.

I. 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.

Special care must be taken in the area of the Pacol Reactor and the Heating Medium Loop. See the detailed instructions in Section VII.

The easiest way to assure that all piping is air-free is to divide the unit into sections (off-sites, reactors, 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.

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.

1. 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.

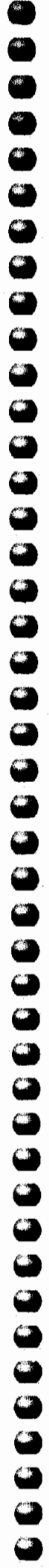
NOTE: Always follow design pressure limitations when air freeing vessels. Most storage tanks are designed for a maximum of 3" (76 mm) of H₂O pressure and some fractionation columns are designed for 3.5 kg/cm² (50 psig).

2. 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.
3. Perform steps 1 and 2, two times.
4. 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 low, the nitrogen displacement step is complete. If the oxygen content is greater than 0.5 mol-% then repeat the nitrogen displacement procedure.

5. 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 a 25 mm (1 in) of H₂O pressure.

All lines to and from storage tanks and the unit battery limits must be air-freed. This includes feed lines, chemical injection lines, product rundown lines and startup bypass lines. By using N₂, the lines can also be blown dry prior to admitting hydrocarbon.



VII. NORMAL STARTUP

After the DeFine reactor has been loaded with catalyst and after ascertaining the proper mechanical condition of all equipment, the proper functioning of all the instruments and the shutdown interlocks, and the availability of utilities, the DeFine Unit may be placed on stream. Other sections of the refinery which may be affected by the startup and subsequent plant operations should be notified of the startup. The following is a general outline of the procedures entailed. More detailed procedures, specific for an individual unit, will be written up by the UOP Chief Advisor.

A. REACTOR AIR FREEING

1. Isolate the DeFine reactor circuit by placing blinds or activating the double block and bleeds on the reactor charge line, reactor effluent line, the reactor top vent gas line and the makeup gas line.
2. Block in all instruments that could be damaged by vacuum.
3. Open up the line connecting the DeFine reactor to the ejector.
4. Commission the ejector and pull a vacuum to about 300 mm Hg absolute and hold for about thirty minutes. If the vacuum does not hold, locate and fix the leaks. Following a successful vacuum test, break the vacuum with nitrogen to about 0.3 kg/cm² pressure.

It is recommended that, prior to air freeing the Reactor, all flanges closed after catalyst loading be taped and be soap tested under elevated N₂ pressure. This will ensure a successful vacuum test.

5. Repeat this procedure 2 more times (without the hold period). Remove any blinds installed. Prior to hydrocarbon introduction ensure that the oxygen content is less than 0.5 mole%.

B. PARAFFIN CIRCULATION AND REDUCTION WITH HYDROGEN

The DeFine catalyst is delivered in reduced form. However, the catalyst has undergone a surface stabilization step during the manufacturing process just prior to shipping. This step entails passing an air stream over the catalyst. Hence, some mild oxidation may have occurred. Therefore, the DeFine catalyst is subjected to an in-situ proof reduction step. Some units are equipped with equipment to allow a gas phase reduction and newer units are designed to complete the proof reduction in the liquid phase.

B.1. GAS PHASE REDUCTION GUIDELINES

1. Initiate a flow of hydrogen from the Pacol Unit's recycle compressor discharge, through the DeFine reduction heater to the DeFine reactor inlet, and out the bottom of the DeFine reactor to the Pacol Product Separator inlet, using the reduction line. The flow path should be lined up accordingly. Establish stable gas circulation through the DeFine Reactor at about 150 GHSV. The higher the rate is better, but since the maximum hydrogen rate is very dependent on the hydraulics of the individual unit, it may not be possible to obtain a GHSV higher than 150.
2. Raise the Rx inlet temperature of the circulating H₂ stream gradually to 205°C (400°F) using the reduction heater, or the purge gas heater.
3. Hold the reactor outlet temperature at least 180°C (356°F) for 8 hours. Very little, if any, water of reduction is expected. The highest gas rate is preferable to sweep any water off the catalyst.

4. Block in the Hot Oil to the Reduction Heater and the circulating hydrogen. Activate the DeFine reactor vent gas system, allowing the DeFine reactor to float on the Pacol Product Separator pressure.

DEFINE REACTOR FILLING

1. This procedure assumes that:
 - DeFine reactor process inlet, outlet and bypass lines are closed.
 - DeFine reactor vent system is activated.
 - DeFine reactor is under a hydrogen-rich atmosphere floating on the Pacol Product Separator pressure.
 - Paraffin circulation is underway.
 - Pacol unit reactor is by-passed with the recycle paraffin liquid routed directly to the Stripper Column.
2. Ensure that the DeFine unit feed line and reactor bypass line have been air-freed and N₂ purged.
3. Start the reactor charge booster pump and divert feed flow through the Reactor bypass line. Slowly close off the DeFine Unit bypass valve.
4. As the unit bypass valve is closed, the Level Control Valve on the DeFine Reactor outlet will open. Establish stable level control and reactor bypass feed circulation.
5. Prepare to liquid fill the DeFine reactor as follows:

If the Pacol Unit is on-stream, and the separator contains olefins, filling the DeFine reactor should be done with care to avoid excessive increases in temperature caused by the exothermic reactions.

 - Block in the reactor vent system.

- Crack open the valve on the filling line around the reactor outlet block valve. The restriction orifice will prevent too high flow rates. Flow can be seen through the sight glass.
 - Slowly fill the reactor from the bottom venting the gas through the PSV by-pass.
 - The reactor is liquid filled in this fashion with PACOL separator liquid. The Stripper pressure and liquid head in the Stripper feed line is sufficient to fill the DeFine Reactor.
 - The filling rate should be very slow so as to ensure that the catalyst bed is not disturbed.
 - When a level is observed at the reactor top site glass, begin to close off on the filling line. Allow the reactor to be liquid full prior to blocking in the filling line and the vent line.
6. With the reactor liquid full, the DeFine Unit is ready to be placed on stream. Start the Define feed booster pump, slowly open the reactor inlet block valve. Since the Reactor is liquid full, the pressure will immediately increase.
- Open the inlet valve completely and then the outlet isolation valves. Gradually close the reactor bypass valve to place the reactor on-line.
7. Gradually raise the temperature of the reactor feed to about 180°C using the reactor charge heater. Calculate the Hydrogen injection rate to achieve a 1.4 H₂/DO at the Reactor inlet. This ratio includes the dissolved H₂ from the Product Separator. The reactor is now ready to receive Pacolate from the separator for normal operations.

B.2. LIQUID PHASE REDUCTION GUIDELINES

DEFINE REACTOR FILLING AND REDUCTION

1. This procedure assumes that:
 - DeFine reactor process inlet, outlet and bypass lines are closed.
 - DeDefine reactor vent system is ready for service.
 - DeDefine reactor is under a positive nitrogen pressure.
 - Paraffin circulation is underway at near design circulation rates.
 - Pacol unit reactor is by-passed with the recycle paraffin liquid routed directly to the Stripper Column.
 - Define feed heater should not be put into service until the reactor is liquid filled.
2. Ensure that the DeDefine unit feed line and reactor bypass line have been air-freed and N₂ purged.
3. Start the reactor charge booster pump and divert feed flow through the Reactor bypass line. Slowly close off the DeDefine Unit bypass valve.
4. As the unit bypass valve is closed, the Level Control Valve on the DeDefine Reactor outlet will open. Establish stable level control and reactor bypass feed circulation.
5. Prepare to liquid fill the DeDefine reactor with cool paraffin as follows:

Filling the DeDefine reactor should be done with care to avoid excessive flow in the upward direction which could disturb the catalyst bed(s).

 - Block in the reactor vent system.

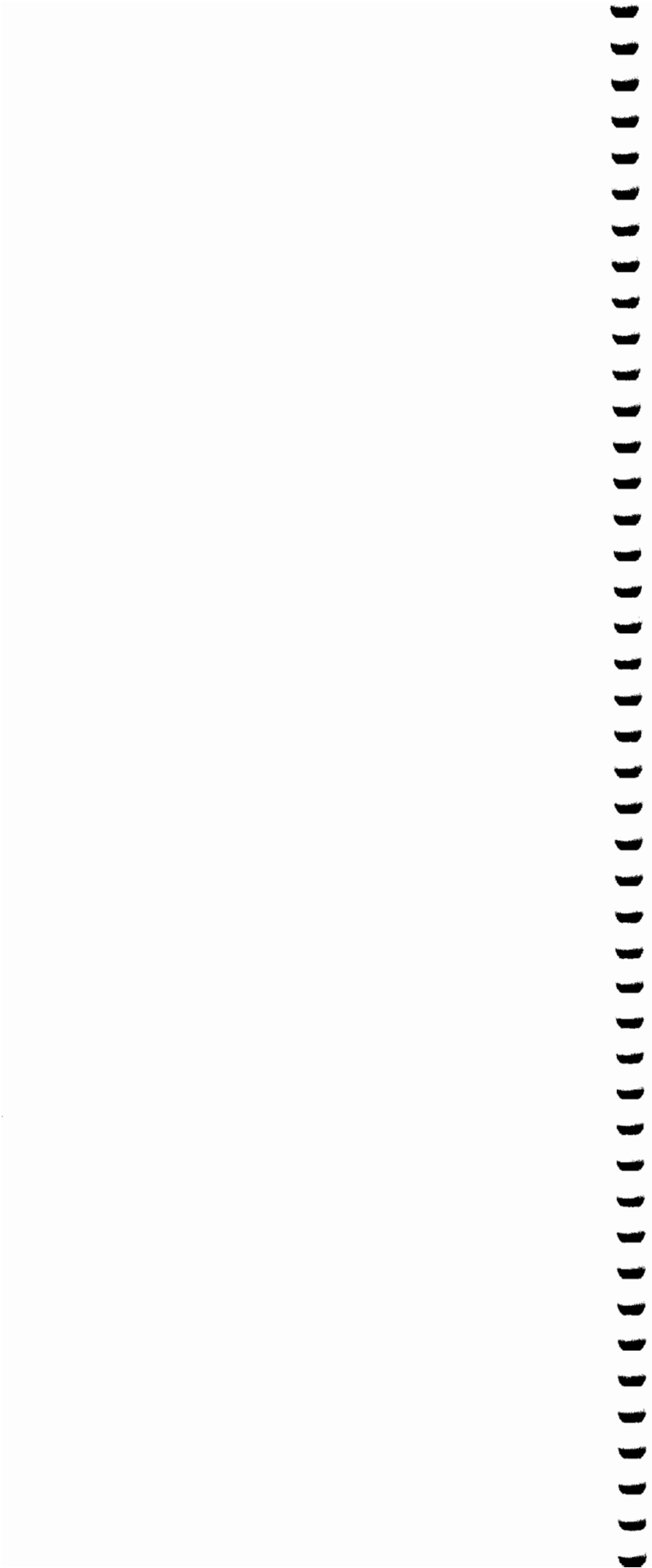
- Crack open the valve on the filling line around the reactor outlet block valve. The restriction orifice will prevent too high flow rates. Flow can be seen through the sight glass.
 - Slowly fill the reactor from the bottom venting the gas through the PSV by-pass or the vent line at the top of the reactor.
 - The reactor is liquid filled in this fashion with PACOL separator liquid. The Stripper pressure and liquid head in the Stripper feed line is sufficient to fill the DeFine Reactor.
 - The filling rate should be very slow so as to ensure that the catalyst bed is not disturbed.
 - When a level is observed at the reactor top site glass, begin to close off on the filling line. Allow the reactor to be liquid full prior to blocking in the filling line and the vent line.
6. With the reactor liquid full, start the define feed booster pumps, slowly open the reactor inlet block valve. Since the Reactor is liquid full, the pressure will immediately increase.

Open the inlet valve completely and then the outlet isolation valves. Gradually close the reactor bypass valve to place the reactor on-line.

7. Gradually raise the temperature of the reactor feed to about 220°C using the reactor feed heater. Establish reactor pressure control at the design operating value. Increase paraffin circulation up to design rate and begin hydrogen injection to all beds at design rate.
8. After 8 hours sample the Product stripper reflux drum gas for hydrogen. If hydrogen is detected then maintain hydrogen injection for an additional 2 hours repeat check for hydrogen every 2 hours. If hydrogen is not detected continue liquid phase reduction up to a maximum of 24 hours.
9. The reactor is now ready to receive Pacolate from the separator for normal operations.

C. Introduction of Pacolate Feed

1. This procedure assumes that:
 - DeFine reactor process inlet, outlet and are closed.
 - DeFine reactor vent system is ready for service.
 - DeFine reactor is liquid full and vented of all gas.
 - Paraffin circulation via the reactor bypass is underway at near design circulation rates.
 - Pacol unit reactor is by-passed with the recycle paraffin liquid routed directly to the Stripper Column and Pacol reactor is ready for service.
 - Define feed heater is ready for service.
 - Catalyst Reduction is complete
 - Feed filters are ready for service
2. Feed is flowing through the Reactor bypass line. Use the flow control valve at the outlet of the Define Reactor to increase the pressure in the flowing line to approximately 10 kg/cm². Slowly open the reactor inlet and outlet valves to allow the reactor to pressurize. Slowly close off the DeFine Unit bypass line valve to establish downflow through the reactor.
3. As the unit bypass valve is closed, the Level Control Valve on the DeFine Reactor outlet will now control the flow. Establish stable level control and reactor feed circulation.
4. Gradually raise the temperature of the reactor feed to start of run temperature about 180°C using the reactor feed heater. Establish reactor pressure control at the design operating value. Increase paraffin circulation up to design rate and begin hydrogen injection to all beds at the desired rate (Hydrogen flow may be increased after feed is established to the Pacol Reactor).
5. The Define reactor is now ready to receive Pacolate from the separator so feed may be started to the Pacol reactor.





VIII. NORMAL OPERATIONS

A. DAILY OPERATIONS

DeFine Unit performance is characterized in terms of di-olefin Conversion and Selectivity to mono-olefins. These parameters are calculated as follows:

$$\text{Di-Olefin Conversion, \%} = \frac{(\text{Feed DO wt-\%} - \text{Effluent DO wt-\%})}{\text{Feed DO wt-\%}} \times 100$$

$$\text{Mono-Olefin Selectivity, wt-\%} = \frac{(\text{Effluent MO wt-\%} - \text{Feed MO wt-\%})}{(\text{Feed DO wt-\%} - \text{Effluent DO wt-\%})} \times 100$$

Here DO stands for di-olefins and MO stands for mono-olefins. The wt-% total di-olefins and mono-olefins in the feed and effluent streams can be determined by high performance liquid chromatography (HPLC) or mass spectrometry (MS). MS equipment is expensive and difficult to operate that it is seldom feasible to have an instrument at the plant site. HPLC equipment is less expensive and much easier to operate. The HPLC methods, UOP Method 869 and 902, have been developed to enable rapid determination of DeFine Unit performance. Both UOP 869 and UOP 902 must be run to get an accurate measurement of di-olefins and mono-olefins.

The process variables influencing the DeFine Unit operation are:

1. Pressure, kg/cm²
2. LHSV, hr⁻¹
3. Temperature, °C
4. H₂/Di-Olefin ratio, mol/mol
5. Sulfur injection rate, wppm

1. Pressure

The DeFine Unit pressure is selected to ensure that the hydrogen remains in solution in the DeFine reactor. Pressure is fixed by design and is not varied. The Reactor pressure floats on the Booster Pump head and should never fall below the setting of the reactor outlet pressure control which acts as a low pressure over-ride of the flow controller.

2. LHSV

The LHSV across the DeFine reactor is defined as the volumetric ratio of the feed rate charged per unit volume of the DeFine catalyst. The design LHSV to DeFine reactor is about 5.0. The Pacol Unit is typically always operated at design or higher LHSV. Consequently, the DeFine Unit LHSV is also always maintained at or above design.

3. Temperature

The reaction temperature is selected so as to offer the optimum reaction rates for di-olefin conversion. At design LHSV the beginning of run reactor temperature is held constant at about 180°C. As the catalyst deactivates due to gum formation, the reactor inlet temperature must be increased to maintain constant performance. End of run reactor inlet temperature is 220°C.

Increasing the Reactor inlet temperature will increase the reaction rate for both DO to MO and MO to paraffin. If di-olefin conversion is low and there is significant **excess H₂** at the outlet of the DeFine reactor, as indicated by analysis of the Stripper Net Overhead gas, the Reactor inlet temperature should be increased.

4. H₂/Di-olefin Ratio

Hydrogen that is available for reaction is expressed as the molar ratio of feed hydrogen to di-olefins:

$$\text{H}_2/\text{Di-Olefin Molar Ratio} = \frac{\text{Feed H}_2 \text{ (moles)}}{\text{Feed Di-Olefins (moles)}}$$

A H₂/di-olefin ratio of 1.4 will result in about 90% di-olefin Conversion with 50% Selectivity to mono-olefins at start of run. The liquid from the Pacol separator contains dissolved hydrogen amounting to approximately 0.3 Nm³/m³ hydrocarbon. The exact value depends upon the Product Separator pressure and temperature as well as the Recycle Gas composition. The value to be used by a particular Licensee should be estimated before the DeFine is brought on-stream.

For a three-stage DeFine unit, the H₂/di-olefin ratio is still 1.4, but 50% goes to the first reactor, 30% goes to the second reactor, and 20% goes to the third reactor.

Typical H₂/di-olefin ratio in the liquid feed from the Pacol Unit separator, at design Pacol Conversion, is on the order of 0.4. At Pacol Conversions other than design, or as the Pacol catalyst life increases, (increased DO/MO) this value is no longer correct.

5. Sulfur Injection Rate

Attenuation of the Nickel catalyst by formation of NiS decreases the MO saturation relative to the DO reactions. Therefore the H₂ is consumed by the mono-olefins more slowly and, consequently, there is more H₂ available for di-olefin Conversion. This increases selectivity and because there is more hydrogen available to react with di-olefins, there is potential to increase conversion.

It is not absolutely necessary to inject sulfur. The DeFine Unit can achieve some DO conversion provided the temperature and H₂/DO are sufficient. Sulfur is only required to maximize the Selectivity to mono-olefins and thereby maximize the Pacol/DeFine MO yield.

B. OPTIMIZATION OF PACOL/DETERGENT ALKYLATION and PACOL/OLEX WITH DEFINE

A true measure of the impact of the DeFine Unit on an LAB complex or an Olefins complex operations can only be realized if the operations of the associated processes are fine tuned.

1. The impact on the Pacol operations will be minor. Three alternatives can be envisioned:

- a. **Constant Pacol Conversion**

In this case Pacol per-pass conversion (Total olefins at Product Separator) should be maintained at the same level as without DeFine. LAB production will increase as di-olefins are converted to mono-olefins (nP/LAB decreases). Pacol fresh feed requirements will still decrease slightly as the Recycle Paraffins will increase, maintaining a constant Combined Feed rate. FF/CF Conversion will decrease, increasing Pacol catalyst life slightly.

- b. **Constant LAB Production**

Pacol conversion is decreased to maintain the same LAB production rate. Pacol Conversion will decrease, decreasing the rate of Pacol catalyst deactivation.

- c. **Constant Pacol Catalyst Life**

Increase Pacol Conversion to maintain a constant MT LAB/kg Pacol catalyst "life" when compared to pre-DeFine operations. LAB production will increase.

2. Detergent Alkylate Unit: Careful fine tuning of the Detergent Alkylate Unit will be required to maximize the benefits of DeFine:

- a. HF Regeneration Rate/LAB Product Quality: If the HF regeneration rate is held constant, DeFine will reduce the amount of indans, tetralins, bromine index-producing materials, and other impurities in the LAB product. If this rate is not reduced to the minimum, losses of LAB and heavy alkylate byproduct to the HF regenerator bottoms will be greater than necessary.

DeFine will reduce the HF Regenerator bottoms rate by a factor of 5-8. A comparison of the bottoms composition can be found in Table VIII-1. Based only on composition, the greatest difference is a five-fold decrease in the fluoride content.

When combined with the flow rate reduction, there is at least a 90% reduction in the Fluorides lost from the system which translates to a decrease in make-up HF requirements.

- b. LAB Rerun/Recovery/Recycle Columns: The heavy alkylate rate will be reduced by about half. The decrease in the amount of potential LAB impurities in the total alkylate may allow closer fractionation or a reduction in reflux while maintaining adequate LAB product quality (i.e. potential Utility savings). The effects of this alternative must be kept in mind while optimizing the HF regenerator as described above.

A comparison of Heavy Alkylate properties, with and without DeFine, can be found in Tables VIII-2a and 2b. Based only on composition, there is a significant reduction in Bromine Number and viscosity.

The reduction in viscosity can cause problems with the Heavy Alkylate customers unless they are advised that the HA quality will change. The HA, after DeFine is more stable (lower olefin content) and has a

significantly narrower boiling range, while the Fluoride content is reduced by half and the Gardener Color is increased by a factor of 2.

The quantity of components which contribute to Gardener Color appears to remain constant (i.e. the Gardener Color increases by a factor of two while the HA rate decreases by half).

- c. Use of Heavy Alkylate: The amount of di-phenyl alkanes and heavy tars in the heavy alkylate will be greatly reduced. The crude heavy alkylate will have a lower bromine number. This may make the heavy alkylate suitable for new uses. There may be an economic incentive to increase heavy alkylate production relative to LAB. If so, reduce the benzene to olefin ratio. This will result in a higher production of di-alkyl benzenes relative to LAB as well as decrease utilities.

Assessing the benefits of DeFine for LAB production:

In order to determine the magnitude of the benefits of the DeFine process, LAB plant performance must be carefully measured and documented both with and without the DeFine Unit in operation. The ideal time to measure plant performance without DeFine is during the Pacol run immediately before bringing the DeFine Unit on stream, so as to avoid having to take it off stream later. However, in order for the comparison to be valid, the following criteria must be met for all cases:

- a. The Pacol operating conditions and the segment of the Pacol catalyst run included in the comparison must be identical or equivalent.
- b. The plant must have been optimized and lined out, with good material balances.
- c. If the benzene to olefin ratio has been decreased to make more heavy alkylate, the comparison should be made in economic terms, including utilities.

C. OPTIMIZATION OF PACOL/DETAL

In a Detal Complex the most important consideration is maintaining Detal feed quality to ensure long Detal catalyst life. It is essential to always operate the Define unit at high conversions of 95% or higher.

TABLE VIII-1
COMPARISON OF HF REGENERATOR BOTTOMS

ANALYSIS	WITHOUT DEFINE	WITH DEFINE
SP.GR.	0.8890	0.8875
BR. NUMBER	65.2	59.7
FLUORIDE, wt%	1.05	0.2
GARDNER COLOR	17.3	16.7
DISTILLATION (by GC), °C		
IBP	200	193
10%	333	331
50%	445	443
90%	557	552
EP	698	666

UOP 4122-15/UOP 1422-15

TABLE VIII-2A
COMPARISON OF HEAVY ALKYLATE PRODUCTS

ANALYSIS	WITHOUT DEFINE	WITH DEFINE
SP.GR.	0.883	0.872
BR. NUMBER	3.4	0.5
FLUORIDE, wt%	0.08	0.04
KINEMATIC VISCOSITY		
CST @ 38°C	36.6	22.3
CST @ 50°C	21.8	14.0
GARDNER COLOR	3.2	6.0
DISTILLATION		
(by GC), °C		
IBP	321	343
10%	365	366
50%	405	389
90%	468	426
EP	580	495

UOP 4122-14/UOP 1422-14

TABLE VIII-2B
COMPARISON OF HEAVY ALKYLATE PRODUCTS

MS ANALYSIS

ANALYSIS	WITHOUT DEFINE	WITH DEFINE
CARBON NUMBER		
C ₁₈ -	0.46	0.37
C ₁₉	0.36	0.26
C ₂₀	1.64	1.31
C ₂₁₋₃₄	91.4	96.93
C ₃₅	1.97	0.46
C ₃₆	1.63	0.37
C ₃₇	0.74	0.20
C ₃₈	0.48	0.09
C ₃₉	0.11	0.01
C ₄₀ ⁺	0.21	0.0
"Z" NUMBER		
6 - ALKYL BENZENE	59.61	81.18
8 - INDANS/TETRALINS	16.43	6.61
10 - DINAPHTHENE BENZENES	6.53	1.03
12 - NAPHTHALENES	5.77	4.72
14 - BIPHENYLS	7.87	4.58
16 - FLUORENES	2.92	1.44
18+	0.87	0.19
AVERAGE MW	361	347

UOP 4122-13/UOP 1422-13

TABLE VIII-3
COMPARISON OF OLEX EXTRACT PRODUCTS

ANALYSIS	WITHOUT DEFINE	WITH DEFINE
n-MONO-OLEFINS	91.0	96.3
DI-OLEFINS	5.0	0.1
OTHER OLEFINS	0.6	0.3
AROMATICS	1.0	1.1
SATURATES	2.4	2.2

UOP 1861-20

TABLE VIII-4
COMPARISON OF LINEAR ALKYL BENZENE PRODUCTS

ANALYSIS	MS ANALYSIS	
	WITHOUT DEFINE	WITH DEFINE
"Z" NUMBER		
6 - ALKYL BENZENE	96.91	98.37
8 - INDANS/TETRALINS	2.00	0.99
10 - DINAPHTHENE BENZENES	0.14	0.00
12 - NAPHTHALENES	0.31	0.16
14 - BIPHENYLS	0.53	0.40
16 - FLUORENES	0.09	0.08
18+	0.02	0.00
CARBON NUMBER		
C ₁₆	8.3	9.0
C ₁₇	35.1	31.8
C ₁₈	30.9	32.8
C ₁₉	24.1	25.2
C ₂₀	1.6	1.2
% UNSULFONATABLE	1.5	1.3
Br INDEX	<5	<5

UOP 4122-12/UOP 1422-12





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 has 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. Local standards, where more stringent than UOP recommendations, must always be followed.

A typical sample connection for a liquid stream is shown in Figure 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 which 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 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 1.5-2.0 kg/cm² 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 DeFine 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 DeFine 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.

The 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. Test Methods which provide the same information do not need to be duplicated. Where accuracy is very important (e.g. UOP 869) the results should be verified periodically by an alternate method.

C. GENERAL GUIDELINES FOR SAMPLE SHIPMENTS TO UOP

SAMPLE PROCESSING PROCEDURES

All Customers:

- 1) All samples are to be shipped at the expense of the customer.
- 2) Prior notification from either the customer or the requesting UOP person is essential. This allows time to calculate costs, get customer signed authorization, locate charge numbers and generally get everything in place so when the samples arrive at UOP we have all the information to begin the processing. Please notify

Ella Silvers
Operating Technical Services Samples Coordinator
Tel.: 1-847-391-2620
Direct Fax.: 1-847-627-8225
e-mail: Ella.Silvers@uop.com
- 3) If the samples are to be done at no additional cost to the customer, a Network and Activity number must be supplied to charge the work. Samples cannot be input into the LIMS system without this information.
- 4) If samples are coming from a Domestic Customer, the customer must contact UOP in advance of shipping the samples. The responsible person at UOP LLC must send to the customer, either by fax or e-mail, a copy of the General Letter for Domestic Samples.
- 5) If samples are coming from an International Customer, including Canada and Mexico, the customer must contact UOP in advance of shipping the samples. The responsible person at UOP LLC must send to the customer, either by fax or e-mail, a signed TSCA Certification Form, as well as the Shipping Instructions for International Customers. The signature must be either original signature or scanned signature. Use of the script font is not acceptable, per our Legal Department. Any UOP LLC employee, who is also a US citizen, can sign this form, but the individuals in the satellite offices, including Guildford, cannot.
- 6) When the International Customer receives the instructions, they must complete the Pro-forma Invoice and the Analytical Requisition Form, and fax them along with a copy of the TSCA Certification Form and the airwaybill to the attention of Ella Silvers, Operating Technical Services Samples Coordinator. This will allow completing the necessary documentation for customs clearance.

MATERIAL SAFETY DATA SHEET

Material Data Safety Sheets (MSDS) must accompany all shipments of sample material regulated as hazardous by IATA, IMO or DOT.

SAMPLE CONTAMINATION – CONTAINER PROBLEMS

Problems relative to the shipment of liquid samples have been found to be associated with standard laboratory vials with conical polyethylene caps. Controlled tests indicate that samples can permeate out of the containers in question, and that the samples 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
- Keep the samples as far apart as possible so they will have less tendency to cross-permeate.

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
Product	
Density	100
Distillation	210 (1)
Composition by GC	150

Notes: (1) May also be used for density



UOP LLC • 25 East Algonquin Road • Des Plaines, Illinois 60017-5017 • Tel:847.391.2000 • Fax:847.391.2253

Sample Shipment Notification

(Complete all shaded areas)

To: Technical Service Sample Coordinator	Date:
To: Technical Service Contact	
Technical Service Contact:	Sender's Name:
Company/Location: UOP – Technical Service 25 E. Algonquin Road Des Plaines, IL 60017-5017	Company/Location:
Send Fax to: 1-847-391-2253	Sender's Phone:

Total Pages: **The following documents must accompany this fax:**

- ☐ Bill of Lading or Master Airway bill with complete vessel/flight information
- ☐ Pro forma invoice
- ☐ TSCA Certification (signed by UOP LLC personnel)
- ☐ Analytical Requisition form
- ☐ MSDS (if materials are regulated as hazardous by IATA, IMO, or DOT)

**Failure to send all of the required documents may
result in delayed Customs clearance and/or refusal of the sample.**



Pro-Forma Invoice
International Shipper/Exporter (complete all shaded areas)

Company:		Invoice No:	
Address:		Invoice Date:	
Address:		Terms: Pro-Forma Invoice	
City, State, ZIP:		Reference No:	
Country:			
Contact:			
Phone:			
Fax:			

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

International Analytical Requisition Form - UOP Technical Service
(Complete all shaded areas)

TO: UOP Technical Service Sample Coordinator Phone 1-847-391-2620 FAX: 1-847-391-2253

UOP Technical Service Contact Phone: Fax: 1-847-391-2253

Customer Refinery:
Address:

Contact Name:
Phone: Fax:

LIQUID & GAS SAMPLE(S)

<u>Description</u>	<u>Analysis Required:</u>	Check if MSDS is included	Rush or Standard
		☐	☐
		☐	☐
		☐	☐

CATALYST & ADSORBENT SAMPLE(S):

Process or unit: Sample Location:

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

Billing Information:

Company:
Address:

Attention: Phone:

Sample Shipping Information:

Shipped Via:
Carrier Phone Number:
Airwaybill Number:

TABLE IX-1
LABORATORY TEST SCHEDULE
DeFine PROCESS UNIT

Stream and Test	Test Number	Frequency	
Pacol Separator Liquid (DeFine Feed)			
Composition (Paraffins and Olefins)	UOP 688	1/WN	1/DS
Composition (by HPLC)	UOP 869 or 902	1/DN	1/DS
Bromine Number	ASTM D 1159	1/DN	3/DS
Hydrogen Injection to DeFine			
Gravity	UOP 948	1/DN	1/DS
Composition	UOP 539	1/DN	1/DS
H ₂ S	Draeger Tubes	Occas	1/DS
CO,CO ₂	Draeger Tubes	Occas	1/DS
Stripper Feed (DeFine Product)			
Composition (by HPLC)	UOP 869 or 902	1/DN	1/DS
Sulfur	ASTM D 4045 or D 5453	1/DN	1/DS
Stripper Overhead Receiver Gas			
Composition	UOP 539	1/WN	3/WS
Gravity	UOP 948	1/DN	1/DS
H ₂ S	Draeger Tubes	Occas	1/DS
Stripper Overhead Liquid			
Relative Density	ASTM D 4052	1/DN	1/DS
Bromine Number	ASTM D-1159	1/DN	3/DS
Composition (Paraffins and Olefins)	UOP 688	1/DN	1/DS
Sulfur	ASTM D 4045 or D 5453	1/DN	1/DS
Stripper Bottoms			
Relative Density	ASTM D 4052	1/DN	1/DS
Bromine Number	ASTM D-1159	1/DN	3/DS
Composition (Normal Paraffins)	UOP 411	1/DN	3/DS
Composition (Paraffins and Olefins)	UOP 688	1/DN	3/DS
Composition (by HPLC)	UOP 869 or 902	1/DN	1/DS
Sulfur	ASTM D 4045 or D 5453	1/DN	1/DS

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





X. TROUBLESHOOTING

The following section details specific troubleshooting guidelines:

A. HIGH DI-OLEFIN CONCENTRATION EXITING THE REACTOR

In complexes utilizing Detal technology, poor di-olefin conversion can lead to irreversible Detal catalyst deactivation. If reactor effluent analysis reveals a higher than expected di-olefin concentration, the following steps may be considered:

1. Careful analysis of the di-olefin concentration into and exiting the reactor is essential in maintaining the proper $H_2:DO$ ratio.
2. If the di-olefin concentration in the DeFine unit feed seems reasonable given the Pacol unit cycle, then adjust the hydrogen flow to achieve hydrogen to di-olefin ratio of 1.4.
3. The next thing to check is the sulfur injection rate. Excessive sulfur injection will suppress the activity of the DeFine catalyst.
4. If the $H_2:DO$ ratio and sulfur injection rate are correct, then consider a slight increase in reactor inlet temperature.

B. LOSS OF UNIT PRESSURE

If unit pressure decreases as indicated by a decrease in pressure at the reactor outlet, possibly resulting in a decrease in the overall conversion of di-olefin across the reactor, the following steps may be considered:

1. Identify where the decrease in pressure has taken place. Verify that the PIC on the reactor outlet is working correctly. Verify that the hydrogen addition flow is as specified. Ensure that the hydrogen supply PI and FI are as expected.
2. Check the pressure upstream and downstream of the hydrogen mixer. If this is the cause of low pressure, then the mixer will have to be removed and cleaned. Removing the mixer may entail shutting down the unit or the whole complex.

C. SULFUR INJECTION SYSTEM PROBLEMS

As discussed in earlier sections a sulfiding agent such as DMDS is a selectivity enhancer. It functions to slow the rate of mono-olefin saturation, so operating without sulfur injection will cause more mono-olefins to be saturated. This may result in a lower di-olefin conversion, as more hydrogen is saturating mono-olefin. Careful attention must be paid to the inlet and outlet di-olefin concentration while there is no sulfur injection.

D. LOW HYDROGEN:DI-OLEFIN RATIO

If there is limited hydrogen available, compressor, or hydrogen mixer problems, the H₂:DO ratio may fall below 1.4.

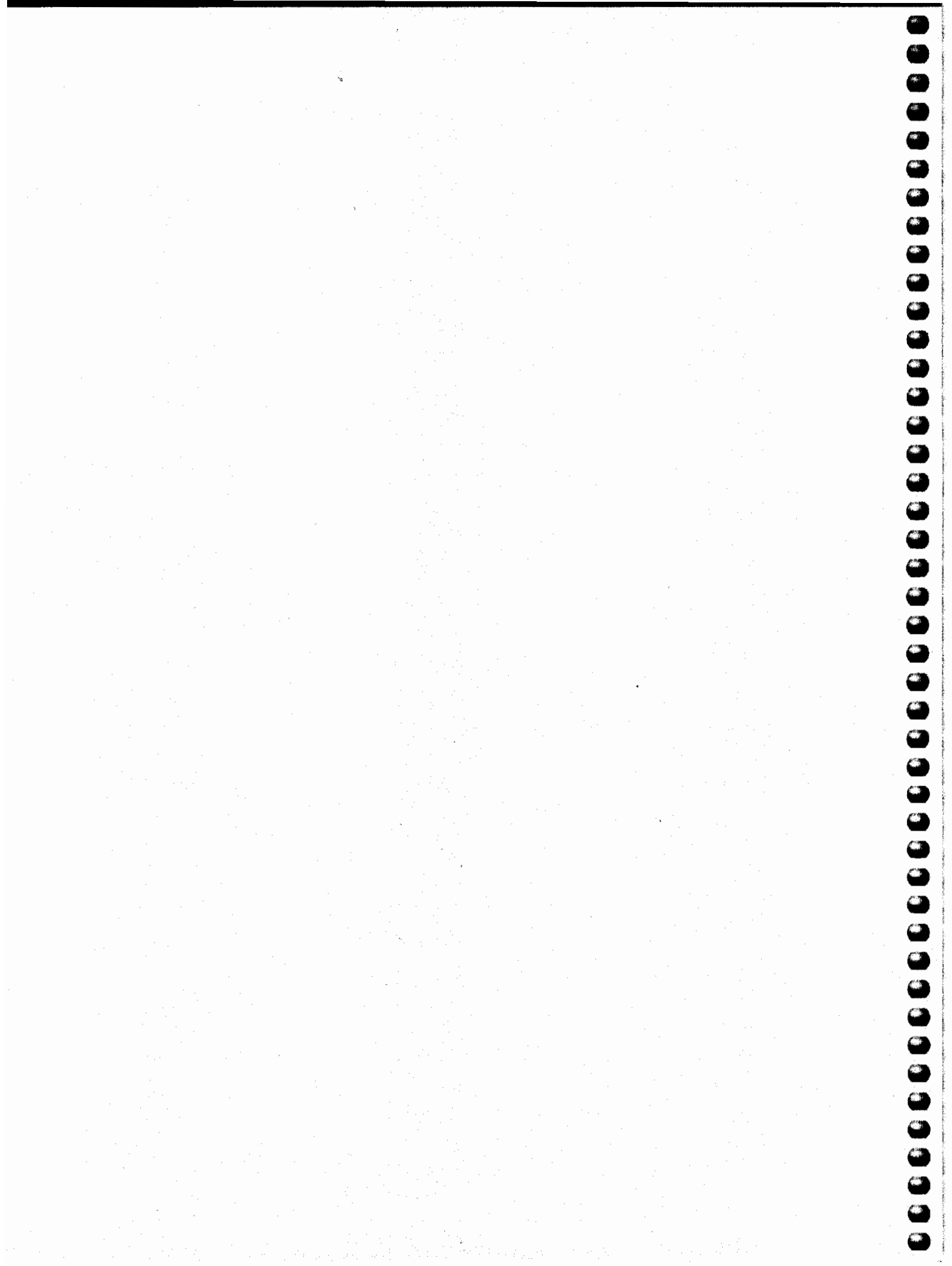
There is no DeFine specific problem in running the DeFine unit short of hydrogen. If di-olefin conversion falls, then Alkylation byproduct production will increase, complex feed consumption will increase. In complexes utilizing Detal technology, poor di-olefin conversion can lead to irreversible Detal catalyst deactivation.

E. HIGH PRESSURE DROP ACROSS REACTOR BED

If an increasing or higher than normal pressure drop is detected across one or more of the Define Reactor catalyst beds it might be a sign of the bed plugging. Operating the catalyst bed with very high pressure drops could result in further catalyst damage or damage to the reactor internals. The filters should be checked to confirm there is adequate filtration. If poor filtration is the root cause of the problem, bed skimming or even reloading may be necessary.







XI. NORMAL SHUTDOWN

The DeFine unit shutdown should proceed as described in the following steps. Please note: These procedures are of a general nature and will be supplemented by the UOP Chief Advisor during the initial startup sequence.

A. TOTAL SHUTDOWN FOR CATALYST CHANGEOUT

The DeFine unit shutdown operation entails the following steps. These steps assume a total unit shutdown and preparation for catalyst unloading and vessel entry. Commercial experience has shown that catalyst replacement is very infrequent. Some of the equipment required for total catalyst replacement is typically not installed but it is more economical and practical to use temporary equipment to facilitate the catalyst unloading.

Local regulation and ultimate disposal or recovery of the spent catalyst will have some impact on what the detailed methodology of unloading will be. If the catalyst is not to be re-used often the nickel from the catalyst is worthwhile to reclaim.

1. In unloading used catalyst that has not been regenerated, special precautions must be taken to prevent the ignition of pyrophoric iron sulfide scale deposits which can be found in the reactor and catalyst beds. The temperature generated by this combustion can be quite high, and if left unchecked, can result in severe damage to the catalyst, reactor internals, and catalyst handling equipment. For these reasons, the entire catalyst dumping and screening operations should be conducted under a nitrogen blanket. All personnel involved with the unloading should be properly informed of the dangers involved, and of the proper safety precautions to be followed.

2. Inform other units of impending shutdown. Bypass the Pacol reactor and allow sufficient time to purge out all of the olefinic material from the separator and Define Reactor. Stop DMDS injection.
3. Gradually decrease the heats to the Reactor Charge Heater to decrease the reactor temperature to 200 C for standby.
4. When olefin content has dropped to a low value (less than 0.2 wt %) stop makeup hydrogen flow and block in the control valve.
5. Switch feed flow through the DeFine Reactor bypass line. If the upstream Unit is also being shutdown, stop and block in Booster Pump.
6. Block in the reactor inlet and outlet isolation valves.
7. Pressure out the liquid in the reactor with Pacol recycle gas or nitrogen. Control the emptying rate by cracking open the valves on the emptying line. Effect this operation very gradually so as to not upset the Pacol Stripper. Monitor the reactor de-inventorying through the emptying line sight flow glass.
8. When the reactor is empty, as evidenced by no liquid flow being observed at the emptying line sight flow, Block in the emptying line. Continue with the gas sweep through the catalyst bed until all the hydrocarbons have been purged off the catalyst and the catalyst bed is sufficiently cool, to permit unloading operations. At this time the reactor feed line should also be drained and purged free of hydrocarbons.

If time is available or it is desirable to reduce the catalyst hydrocarbon content before dumping use portable equipment to hot nitrogen strip the catalyst before cooling the catalyst to handling temperatures. This will

remove even more hydrocarbon from the catalyst surface.

9. With reactor system isolated at the feed booster pump, the reactor outlet and the reactor top, evacuate the system using the Pacol ejectors. Evacuate three times and break each time with N₂.
10. After oil has been cut out of the reactor(s) and the catalyst has been thoroughly flushed, decrease the pressure to a nominal 10psi, then cool the catalyst with a nitrogen flow, de-pressure and purge to a safe location. The catalyst should be cooled to less than 40-50 C (100-125 °F). At temperatures above this level, the combustion of iron sulfide scale in air is greatly accelerated, and, in addition, as the catalyst in the drums cools there will be a greater tendency to draw air into the drums.

If the top of the catalyst bed is to be inspected, further cooling will be required. All personnel should be equipped with fresh air for this operation. With a nitrogen purge going in the bottom of the bed open the manway at the top of the reactor and visually inspect the top of the catalyst bed. Remove any obvious debris or areas of the bed that may be causing pressure drop.

11. Prepare to unload catalyst.
 - a. Blind the reactor at the inlet and outlet.
 - b. Additional blinds should be placed at the battery limits and between various pieces of equipment, as required for safety.
 - c. Remove catalyst from the DeFine reactor. Under no circumstances should air be pulled through the reactor since spontaneous combustion is possible. Wood, canvas or other combustible

materials should not be used in the catalyst dumping operation because of the danger of fire. In addition, the general area around the reactors should be free of combustible materials.

NOTE: The DeFine catalyst has been exposed to sulfur and hence could be pyrophoric. The presence of CO₂ type fire extinguishers is desirable during unloading operations a precaution in case of spontaneous combustion. Any ignition of catalyst contained in the drums can be smothered by inert gas or by closing the container.

The removal of potential pyrophoric material from the reactors has been accomplished safely using a number of different techniques. Below is a method that has proven itself useful in field.

- (1) Attach an unloading nozzle such as that depicted in Figure XI-1 to the reactor to be unloaded.
- (2) Begin a nitrogen purge to the unloading nozzle and to the reactor.
- (3) The reactor catalyst bed should be cooled to less than 100°F.
- (4) Line up several drums and their lids. Make sure the drums are mechanically sound.
- (5) Purge the drum to be loaded with nitrogen or steam to purge the air out of the drum itself.
- (6) There could be "sparking" of the pyrites.
- (7) Open the slide valve on the unloading nozzle and fill the first drum. Maintain the nitrogen purges to the reactor and the slide valve assembly. Further keep nitrogen or steam flow into

the drum. Note: Gradually pull the purge hose out of the drum as the drum is filled with catalyst.

- (8) Continue to maintain a nitrogen blanket in the reactor, and unload the catalyst from the bottom. If the catalyst is to be reused, it should first be screened to remove dust and fines which could cause excessive pressure drop in the reactor, resulting in maldistribution. This can be conveniently done during unloading by dumping the catalyst from the reactor directly into the screening apparatus. The screening apparatus should be enclosed and maintained under a nitrogen blanket to prevent air from entering. Use the proper size screens to separate the catalyst from the ceramic balls, and from the dust and fines. The catalyst should be dumped directly from the screening apparatus into nitrogen blanketed metal drums. This can be accomplished by using a copper tubing line at the unloading site to fill each drum with nitrogen before putting in catalyst. Allow the tubing to remain in the drum as catalyst is being dumped into it to keep a steady stream of nitrogen flowing over the catalyst. (See Figure XI-2)
- (9) When the drum is near full, close the slide valve, place the lid on the drum, and seal it. The catalyst should pose no problems while sealed in the drums.
- (10) Continue unloading all the free flowing catalyst in this manner.
- (11) When catalyst stops flowing from the reactor, purge the reactor upflow with a high flow of steam. This will dampen the remaining catalyst rendering it easier to handle.

- (12) Removal of this material should be done via inert gas entry.
 - (13) Clean the reactor per normal practices.
 - (14) Reload per start up section of this manual.
- d. The spent catalyst should be disposed of in a safe manner. It may be economical to send the spent catalyst out for metals recovery.
 - e. The top heads of the reactors may be opened intermittently so that removal of the catalyst from the reactor may be observed. However, they should be kept closed normally during catalyst withdrawal to prevent natural draft.
 - f. It is recommended that catalyst be dumped into drums at the Unit for transfer to a temporary storage area.
 - g. Catalyst is ordinarily removed from the DeFine reactor through unloading nozzles on the bottom head. The blind flange is removed from this nozzle and a dumping spout allows the flow of catalyst to be stopped as required when such items as changing of catalyst drums must be taken care of. Empty drums can be located under the outlet of unloading spout.

After catalyst has been removed, the alumina supports remaining below the catalyst bed may be removed from the bottom head through the outlet nozzle. There should be a lug on the "elephant stool" for lifting it out of the outlet nozzle. After screening for fines removal and separation of the different sizes, all alumina removed from the reactors can be reused. The alumina spheres should be stored in a dry place.

- h. Spent catalyst from the unit should be free-flowing. If the Unit has been on stream for a long time, or if there are indications that there has been significant gum formation, the hot H₂ purge mentioned in Step 8 should be performed.
- i. After filling, the catalyst drums should be sealed with the proper cover and rubber gasket to keep out moisture and oxygen.
- j. With the catalyst removed, the reactors are now ready for cleaning and internal inspection. Always sample the internal atmosphere of the vessel prior to entry.

B. PARTIAL SHUTDOWN

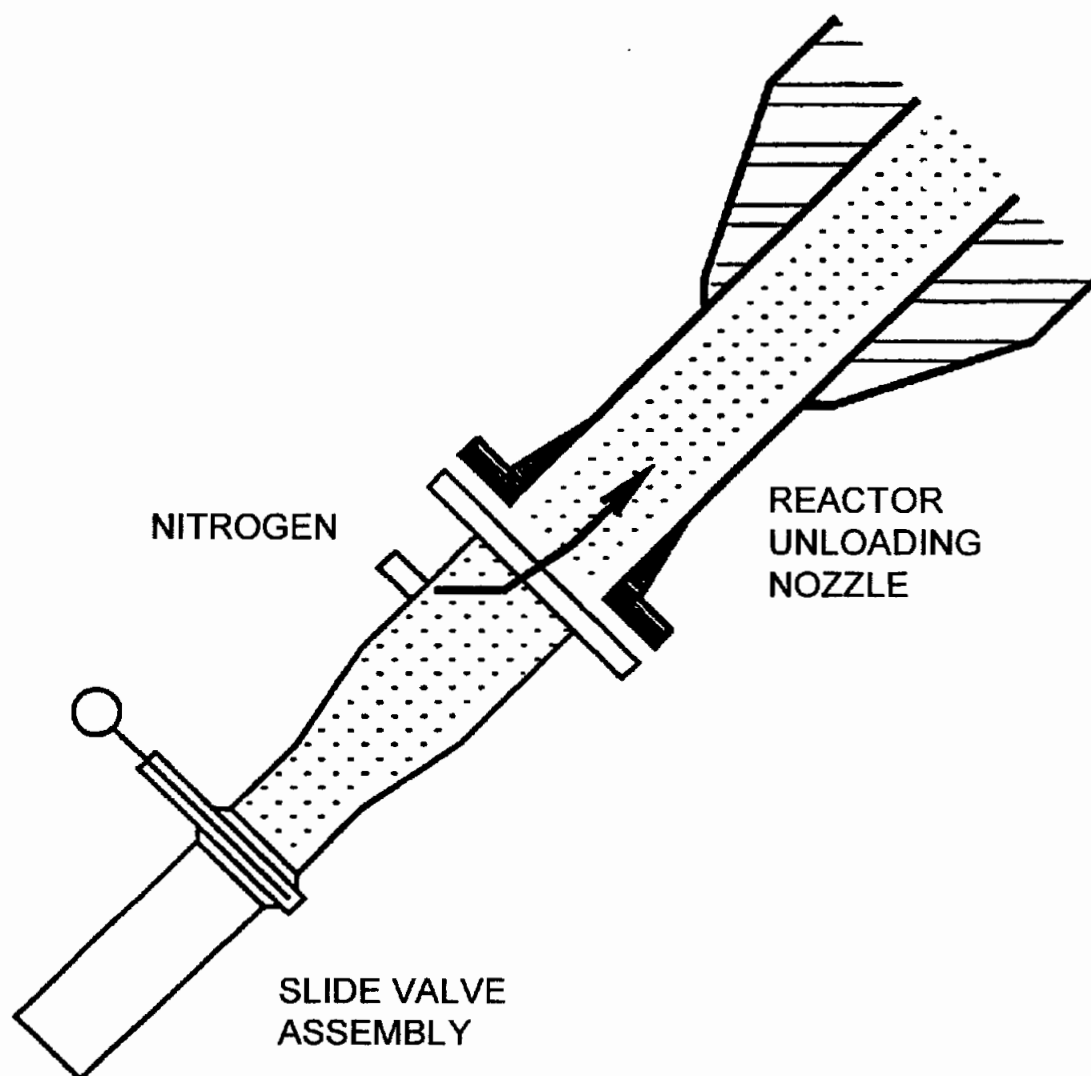
On occasion it may be necessary to shutdown the DeFine Unit for a short period of time for routine maintenance. An arbitrary definition of "short period of time" could be anything over 8 hours. The important factor to remember is that di-olefins at high temperature may tend to polymerize to form gums which could potentially deactivate the catalyst. In the absence of hydrogen, lower temperatures are better. During a normal shutdown always purge all of the olefinic materials from the Pacol and Define Units before shutting down the Define reactor.

A planned, partial shutdown is identical to the complete shutdown through Section A.6. The intent is to eliminate the reactants by purging paraffins through the system and lowering the reactor temperature to lower than 200 C.

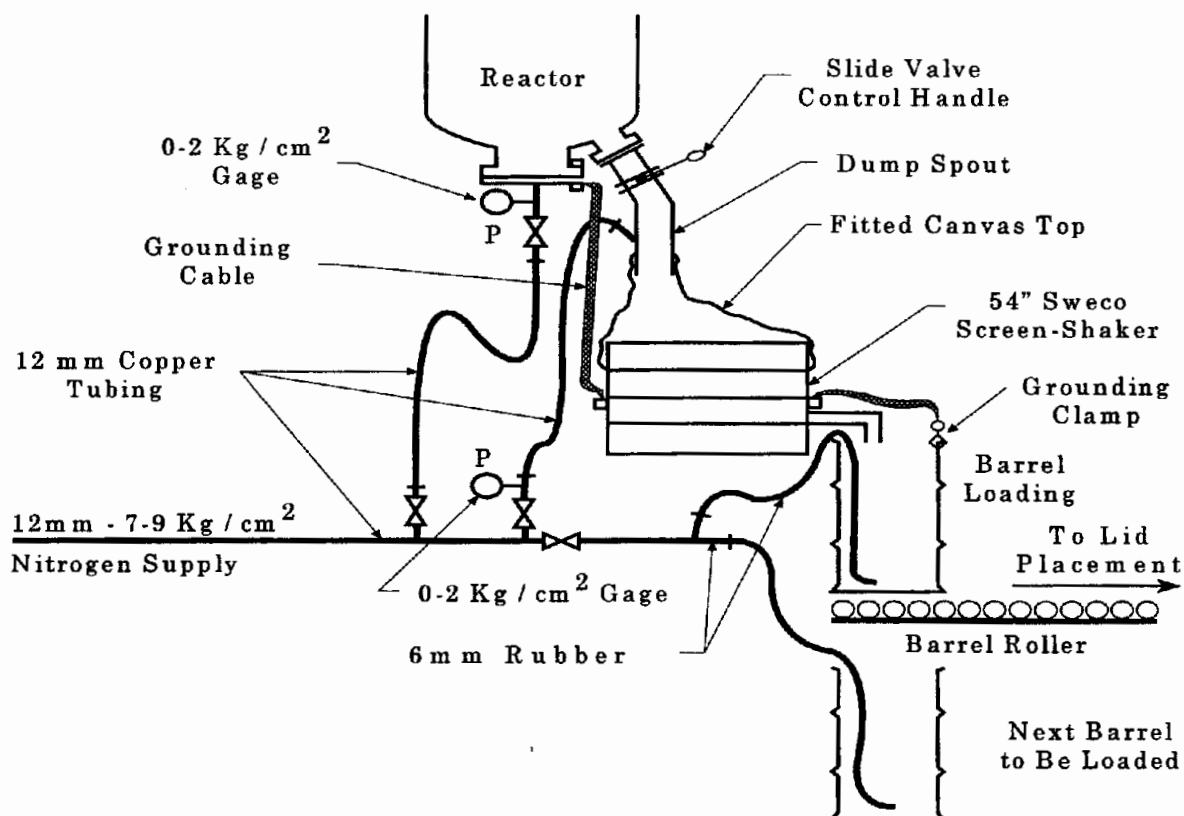
1. Inform other units of impending shutdown. Stop sulfur injection.
2. Gradually decrease the heats to the Reactor Charge Heater to decrease the reaction rates.

3. Gradually phase out m/u hydrogen flow and block in the control valve. This step is optional since the hydrogen will be removed in the Stripper overhead.
4. Switch feed flow through the DeFine Reactor bypass line.
5. Block in the reactor inlet and outlet isolation valves.
6. Purge the olefinic material in the reactor with fresh n-paraffins brought in through the Pacol reactor by-pass line to the product separator and to the DeFine reactor feed line. If the Pacol unit is not shut down, then proceed to drain the DeFine reactor by slowly pressuring the liquid to the stripper with Pacol recycle gas either through the reduction line or through the normal hydrogen feed line to the reactor. Control the rate by cracking open the valves on the emptying line. Effect this operation very gradually so as to not upset the Pacol Stripper. Monitor flow rate of the fresh n-paraffins. Purge at least 1.5 times the empty Reactor volume (a little more than two times the Reactor void volume). There is no reason to try to conserve the fresh n-paraffins. They will be recycled to the Pacol and there will be a temporary decrease in the fresh n-paraffin rate to that Unit.
7. When finished, block in the Reactor emptying line and the n-paraffin line. The Reactor can remain in this condition for extended periods of time since there are no olefins to react.

**FIGURE XI-1
INERT GAS CATALYST UNLOADING**



**FIGURE XI-2
UNLOADING USED CATALYST**



Procedure

1. Normal Cooling and Purging.
2. Install Dumping Spout and Nitrogen Purge Facilities.
3. Commission Nitrogen Purge to Reactor.
4. Open Reactor Top, Remove Baskets, Break-Up and Remove any Surface Crust.
5. Close and Blind Reactor Top
6. Set Nitrogen Purges on Dumping Spout and Barrels.
7. Dump and Screen Catalyst at 3 to 5 Minutes per Barrel to Obtain Good Separation of Catalyst and Fines.
8. Roll Away Barrels and Install Gaskets, Lids and Clamps as Each Barrel Is Filled.

PLT-R00-





XII. EMERGENCY PROCEDURES

A. EMERGENCY SHUTDOWN

In all scenarios, what would constitute an emergency shutdown for the DeFine will also entail an emergency shutdown of the upstream and/or downstream Units. The DeFine Unit can be safely addressed at a later time when the overall situation is under control and operational staff is available to attend to the DeFine Unit. Note that one must restart the Define operation before beginning Detal Unit operation.

Makeup hydrogen to the DeFine Unit will automatically stop on low feed flow. The control valve should be isolated as a precaution against leakage. It may be necessary to stop sulfiding agent injection during a prolonged shutdown.

During a shutdown scenario there are steps which can reduce the chance of any formation of gum-like materials. The directional relationship is that higher di-olefin concentrations and higher reactor temperatures increase the chance of gum formation. Therefore the general shutdown philosophy is to lower the reactor temperature and purge the olefinic material out of the reactor.

The expected duration of the shutdown will determine what should be done. If the shutdown will be for less than 8 hours (for example) it is not critical that the Reactor be purged of olefins. If the duration is expected to be much longer than 8 hours, the following steps are recommended.

1. Purge the olefinic material in the reactor with n-Paraffins brought in through the line to the Pacol reactor by-pass product separator then to the DeFine reactor feed line. Effect this operation very gradually so as to not upset the Pacol Stripper. Monitor flow rate of the fresh n-paraffins. Purge at least 1.5 times the empty Reactor volume (a little more than two times the Reactor

void volume). There is no reason to try to conserve the fresh n-paraffins. They will be recycled to the Pacol Unit and there will be a temporary decrease in the fresh n-Paraffin rate to that unit. There is no loss of n-Paraffins.

2. When finished purging, block in the Reactor emptying line and the n-Paraffin line. The Reactor can remain in this condition for extended periods of time since there are no olefins to react.
3. Block in the reactor inlet and outlet isolation valves. Isolate the Makeup Hydrogen control valve. Stop sulfur injection.

1. Loss/Reduction of Feed

When the feed rate suddenly decreases or stops altogether, the hydrogen is still entering the reactor circuit at the normal flow rate. This rate was set by the expected di-olefins in the total feed. Therefore, upon loss or reduction of feed, stop or reduce hydrogen injection. Once stable conditions return, proceed with a normal startup, and remember to ensure the reactor is liquid full before re-establishing flow through the reactor if the reactor was bypassed.

2. Loss of Hydrogen

The complete loss of hydrogen increases the ability of the dienes and olefins to react with each other. If hydrogen is unavailable it will be necessary to bypass the Pacol and Detal Reactors and purge the Define reactor with paraffin feed. Decrease Define Reactor temperature to 200 C during the purge step. If possible paraffin circulations should be maintained.

3. Electrical Power Failure

If there is a loss of electrical power, all the pump motors and air exchanger fans will stop. Most likely steam supply will be lost as well. This will require a total shutdown

of the unit. Cooling water supply should automatically switch over to the turbine-driven pump.

Initial action should be to stop steam or Hot Oil to the feed heater. Operators should have on-hand an ordered list of items to be taken care of.

4. Instrument Air Failure

Loss of instrument air will cause the unit to shut down. Control valves will fail open or closed as shown on the mechanical flow diagrams and in the instrument summary. All operating personnel must know the action of each control valve on air failure to ensure taking proper action during an emergency. Monitor the reactors for exothermic reactions. If the temperatures rise to near alarm status, proceed to shut down as outlined in Section XI.







XIII. SPECIAL PROCEDURES

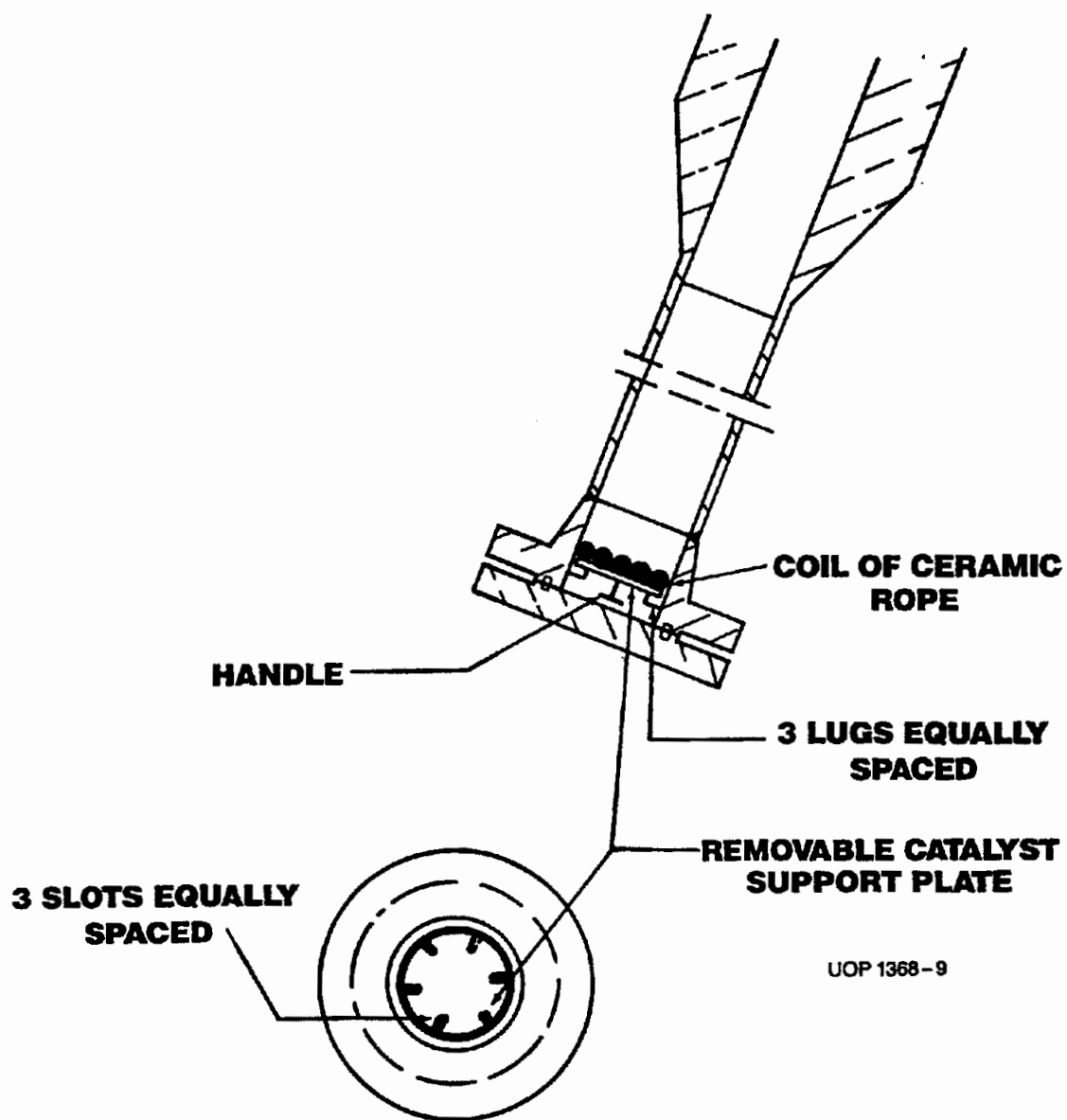
A. REACTOR LOADING

The personnel involved with the loading of the DeFine Reactor should follow all the refinery safety rules and practices. In addition, the workers should wear proper dust masks, eye protection (goggles) and gloves.

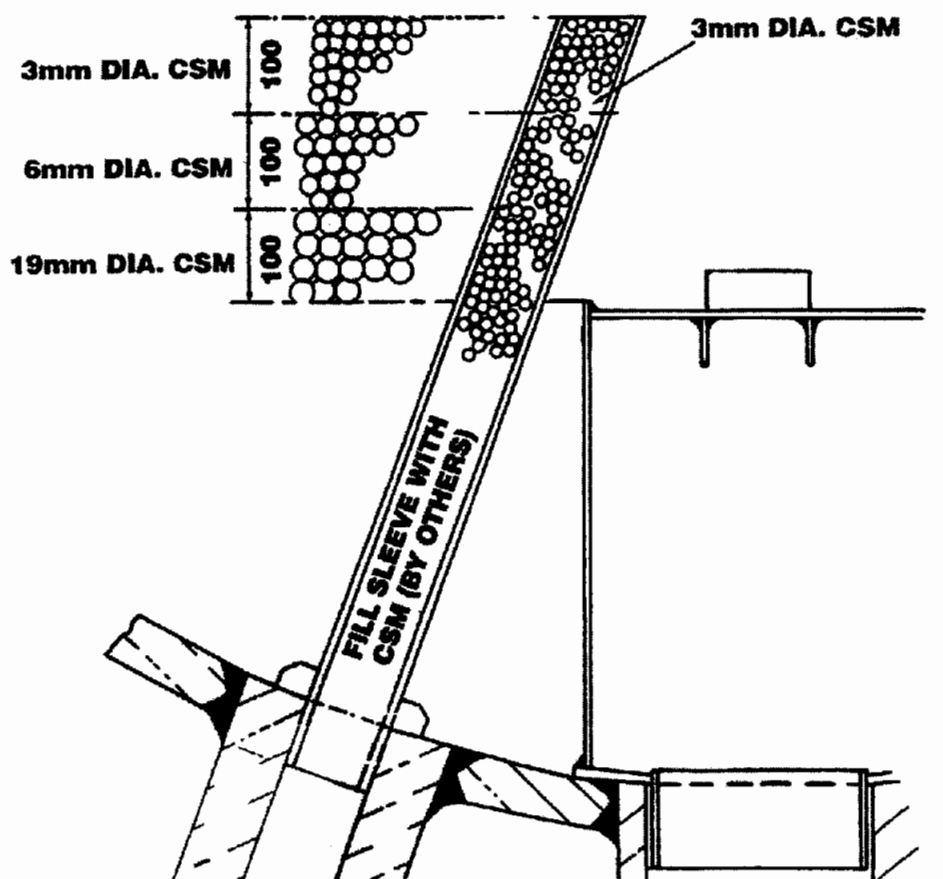
1. Preparation

- a. Install blinds at the reactor outlet and inlet lines. This is necessary to safely load the catalyst and also to prevent damage to the catalyst.
- b. Clean dirt and any foreign matter from the reactor.
- c. Install a removable catalyst support plate in each of the catalyst unloading nozzles. Install a coil of ½" ceramic fiber rope on the top of the support plate after it has been positioned in the unloading nozzles (see Figure XIII-1).
- d. Clean the mating surface and install a blind flange on the catalyst unloading nozzle. If possible, install an ejector (with dust filter) on the unloading nozzle or the outlet nozzle in order to maintain down-flow air movement during loading. This will prevent dust from leaving the top of the vessel where people are working and also aid in settling the dust prior to visual inspections.
- e. Install the reactor outlet basket screen.
- f. Install an extension sleeve in the catalyst unloading nozzle (see Figure XIII-2). Install the outlet basket.

FIGURE XIII-1 REMOVABLE CATALYST SUPPORT PLATE



**FIGURE XIII-2
CATALYST UNLOADING NOZZLE SLEEVE
EXTENSION AND OUTLET SCREEN**



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2. Loading Reactor with Alumina Support Balls

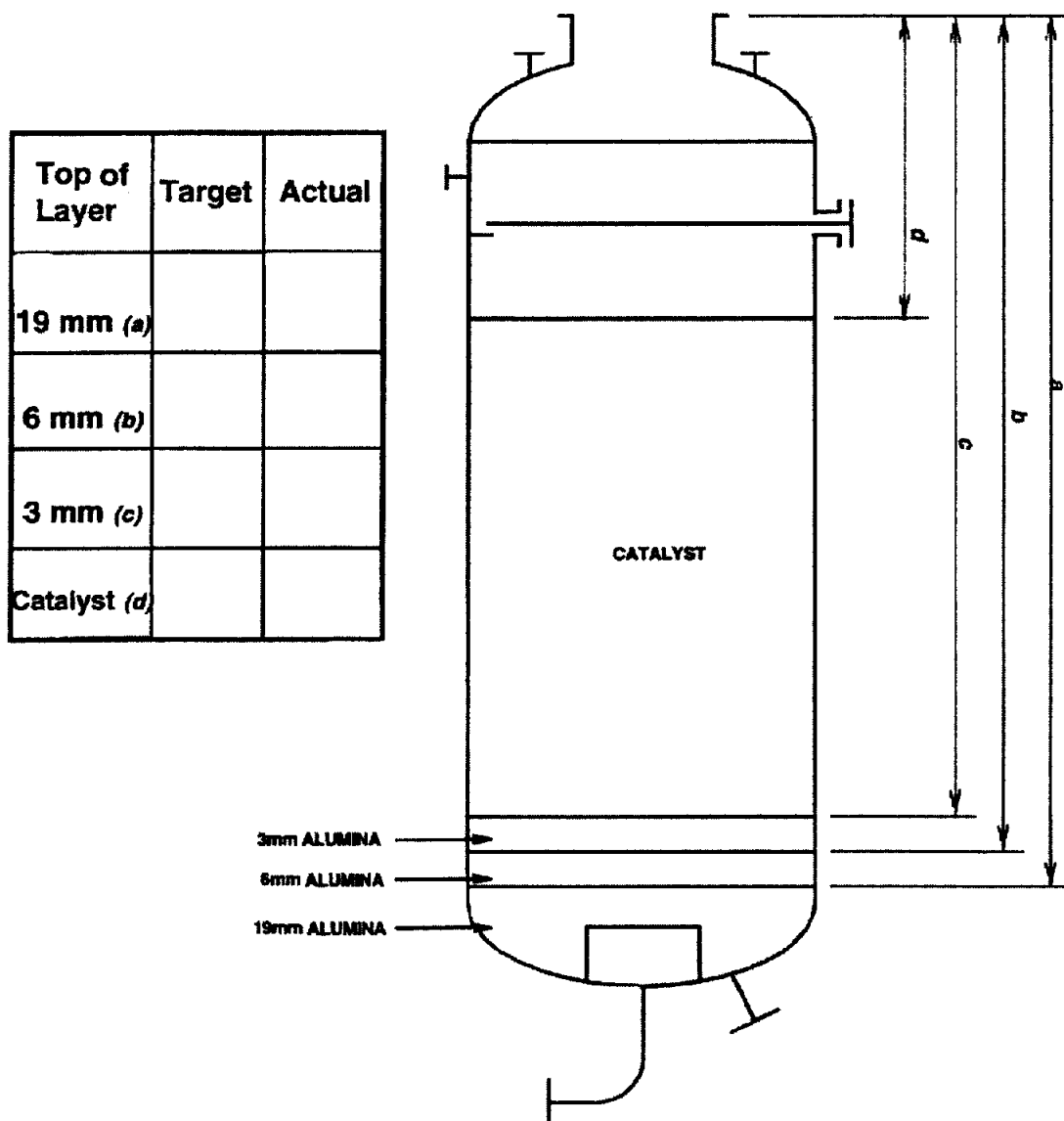
- a. Fill the catalyst unloading nozzle and sleeve with 6 mm catalyst support material (CSM) up to 100 mm from the top of the sleeve.
- b. With the help of a manometer (or other level device) and chalk inscribe circles to represent the depth of the 19 mm, 6 mm and 3 mm alumina support balls.
- c. The alumina loaded should be dry. Load the alumina balls into the Reactor with buckets through the top manway (or bottom manway if available). The alumina balls should be spread uniformly across the reactor cross-section. After each layer is loaded it should be leveled and the dimensions referenced against a point higher up in the reactor. For the DeFine reactor the top manway can be employed as the reference point. (See loading diagram presented in Figure XIII-3.) The various dimensions should be measured with a tape and recorded. The target values should have been pre-recorded utilizing the reactor fabricator's detailed drawings. After all the support balls are in, the bottom manway can be permanently closed.

3. Loading Catalyst

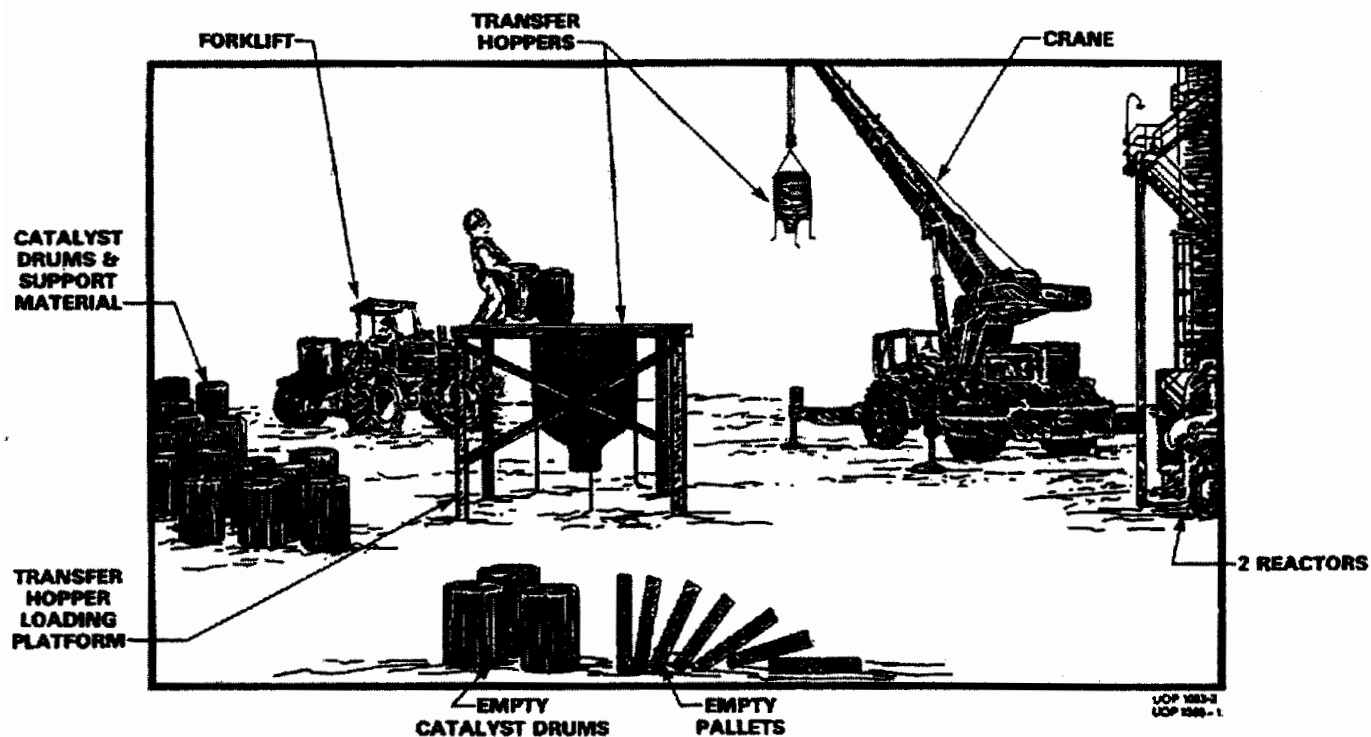
- a. Transfer catalyst from the drums into transfer hoppers at ground level. This can be accomplished by lifting the drums with a forklift truck onto an elevated platform and then dumping them into the transfer hoppers (see Figure XIII-4).

NOTE: The hoppers and socks used throughout the loading operation must be clean to avoid contaminating the alumina balls and catalyst. The loading hoppers should be cleaned. The loading socks should be brand new.

**FIGURE XIII-3
DEFINE UNIT REACTOR LOADING DIAGRAM**



**FIGURE XIII-4
TYPICAL LAYOUT FOR CATALYST LOADING**



- b. Lift the transfer hopper to the top of the reactor. Empty the contents into the loading hopper anchored on or above the reactor top manway. On the bottom of the loading hopper, install a 1000 mm long, 4 inch diameter flexible rubber hose. This hose should be fixed to the hopper in such a way as to avoid it dropping off into the reactor during the loading. The manway gasket mating surface should be protected from damage.
- c. Swing the flexible rubber hose in the manway as the catalyst flows through it. This will eliminate the formation of a mound in the center of the catalyst bed. The bed will be loaded more evenly and the possibility of flow channeling during normal operation will be reduced.
- d. Record the lot and drum number printed on each drum as they are loaded into the transfer hopper. Record the net catalyst weight in each drum.
- e. Take a 10 ml sample from each drum of catalyst loaded and combine them into a 1000 ml bottles. Label the samples to indicate what position of the bed they represent. These samples will be held for future reference.
- f. After emptying the contents of each drum of catalyst, replace the foam rubber seal and re-bolt the top back on the drum. It is important to use these original drums during future handling operations to avoid contamination of the catalyst. Store the drums with the bottom side up. Do not remove the drums from the site until the loading has been completed.
- g. Stop the loading operation after each meter of catalyst. Allow the dust to settle and then lower lights into the reactor. Observe the surface of the catalyst bed. The surface should be flat and level. If it is not, an adjustment in the technique of swinging the loading hose must be made so as to eliminate any mounding.

Severe mounding would require vessel entry for leveling. Proper precautions including, but not limited to, full face mask and outside air source, gloves, coveralls and head covering, should be followed at all times. Further details are provided via MSDS (Material Safety Data Sheet) in Section XIV. Safety.

- h. Load catalyst according to outage measurements and the catalyst loading diagram. Smooth and level the surface of the catalyst bed. This should then be checked with a carpenter's level. Measure and record, on the form provided, the distance between the top surface of the catalyst bed and the reference point.
- i. The loading diagram should serve as the basis of calculating the volume of loaded catalyst. The loaded ABD can then be calculated based on total catalyst weight loaded.

4. Close the Reactor

- a. Check the inside of the reactor to ensure that no tools or foreign matter has been left.
- b. Clean the surface of the top manway flange and ensure the feed inlet distributor is positioned properly and free of any catalyst. Close the top manway.
- c. Remove the blinds at the outlet of the reactor and the outlet of the Reactor feed heater (or Reactor inlet). Pressure the entire reactor section to $>1.0 \text{ kg/cm}^2$ with dry nitrogen. Maintain this positive pressure until the plant is ready for startup.

Check the flanges from which the blinds were removed with soapy water for leaks.

The following materials are required for the loading of the DeFine reactor:

- 19 mm alumina support balls
- 6 mm alumina support balls
- 3 mm alumina support balls
- 6 500 ml sample bottles with screw tops
- 8 1000 ml sample bottles with screw tops

The supplier of the DeFine Catalyst is:

UOP
25 E. Algonquin Road
Des Plaines, Illinois 60017
U.S.A.

The supplier of the catalyst bed alumina support balls should be from the list of suppliers (refer to project specification).

The following loading equipment is typical:

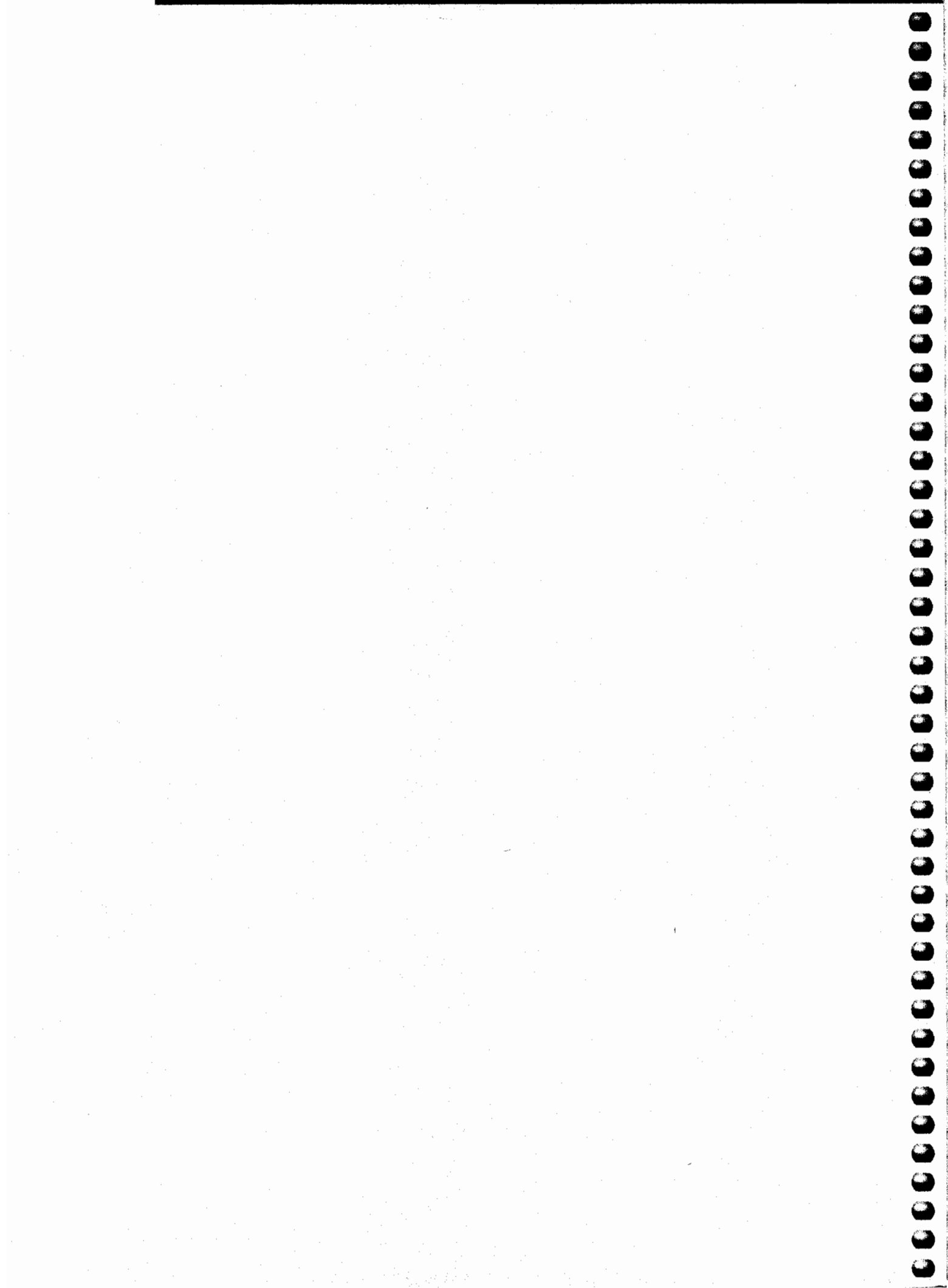
- 2 transfer hoppers (1-2 m³ capacity)
- 1 stationary loading hopper (0.2-0.5 m³ capacity)
- 1 loading platform
- 1 forklift truck
- 1 crane
- 1.0 m length of new, flexible rubber hose
- 2 or more electric lamps for use inside of the reactor
- 15 m tape measure
- rake for smoothing the surface of the various materials
- 1 to 2 m long carpenter's level
- ladder up to 10 m long for entering the reactor
- safety rope harness

B. REACTOR UNLOADING

For reactor unloading, see Chapter XI. Normal Shutdown.







XIV. SAFETY

This chapter on safety includes the following sections:

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

The information and recommendations contained in this manual have been compiled from sources believed to be reliable and to represent the best opinion on the subject as of 1999. However, no warranty, guarantee or representation, expressed or implied, is made by UOP as to the correctness or sufficiency of this information or to the results to be obtained from the use thereof. Each refiner should determine the suitability of the following material for his purposes before adopting them.

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

A. OSHA HAZARD COMMUNICATION STANDARD

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

exceptional conditions or circumstances, or because of applicable federal, state or local law.

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

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

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

1. Written Hazard Communications Program

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

company will use to inform its employees and contractors of the hazardous chemicals.

2. Training and Information

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

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

3. Labels and Other Forms of Warning

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

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

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

Labels can also be color coded according to the following:

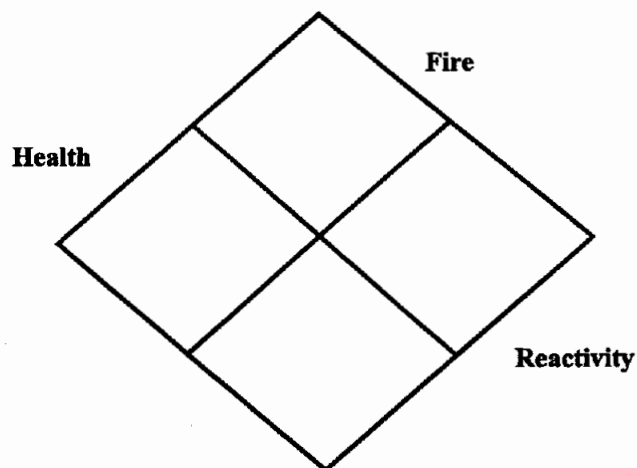
Orange	Carcinogen Hazard (i.e.: Benzene)
Red	Chemical Burn Hazard (i.e.: Acids, Bases)
Yellow	Toxic Vapor Hazard (i.e.: H ₂ S)
White	All Others

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

4. Material Safety Data Sheet (MSDS)

The MSDS requirement falls primarily on chemical manufacturer, importers and distributors. The OSHA standard requires them to develop and provide a MSDS for each hazardous chemical they produce or handle. These manufacturers, importers and distributors are required to provide the MSDS to the purchasers of the hazardous chemical. Refer to section F for MSDS sheets on Define catalyst and chemicals.

Figure XIV-1
NFPA 704 Diamond



CAUTION: CONTAINS HAZARDOUS CHEMICAL
Transportation Emergencies Call
Health Emergencies

HAZARD RANKING

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

PLT-R00-99

TABLE XIV-1

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

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

TABLE XIV-1
(cont'd)

National Fire Protection Association
Identification of Color Coding

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

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

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

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

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

B. HYDROGEN SULFIDE POISONING

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

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

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

1. Acute Hydrogen Sulfide Poisoning

Breathing air or gas containing more than 500 mol-ppm H_2S can cause acute poisoning and possibly be fatal, concentrations of H_2S in the Pacol Stripper after the Define reactor are always much less than 500 mol-ppm.

2. Subacute Hydrogen Sulfide Poisoning

Breathing air or gas containing H_2S anywhere between 10 to 500 mol-ppm for an hour or more may cause subacute or chronic hydrogen sulfide poisoning. H_2S in the Pacol Stripper after the Define reactor would be expected to be below or at the low end of this range.

5. Symptoms of Subacute Poisoning

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

6. Treatment of Subacute Poisoning

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

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

7. Prevention of Hydrogen Sulfide Poisoning

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

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

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

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

TABLE XIV-2

RESPIRATORY PROTECTION FOR HYDROGEN SULFIDE

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

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

8. Further Information

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

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

C. NICKEL CARBONYL FORMATION

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

The potential for forming nickel carbonyl does exist with the nickel-containing H-14 catalyst during the handling of spent or unregenerated catalyst. Care must be used to ensure that the procedures used will prevent the formation of nickel carbonyl. Data has been published showing the equilibrium concentration of $\text{Ni}(\text{CO})_4$ versus temperature, pressure and CO concentration in a gas. The nickel carbonyl concentration drops rapidly with increasing temperature and decreasing CO concentration. At 7 kg/cm²g (100 psig) with 0.5 mol-% CO in the gas, the nickel carbonyl concentration is at the maximum recommended spot level of 0.04 ppm at 149°C (300°F), and 0.001 ppm at 182°C (360°F).

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

1. Once a reactor containing a nickel catalyst has been exposed to oxidizing conditions (regeneration), a measurable concentration of oxygen must be maintained until the combustion of all carbon ceases and all CO_2 has been purged from the system.

2. Once a reactor containing a nickel catalyst is in a reducing atmosphere and regeneration is not desirable, maintain the system in a reducing or inert atmosphere until all the catalyst has been cooled to at least 66°C (150°F). Unregenerated catalyst should be unloaded with N₂ purge before receiving used catalyst. Oxidation (burning) must be avoided.

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

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

D. PRECAUTIONS FOR ENTERING ANY CONTAMINATED OR INERT ATMOSPHERE

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

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

Refinery personnel who do have to enter a contaminated or inert atmosphere should follow all prescribed standard safety precautions and regulations that apply for the refinery. OSHA regulations concerning the use of respirators (29 CFR Subpart 1, Section 1910.134) should be read and thoroughly understood.

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

E. PREPARATIONS FOR VESSEL ENTRY

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

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

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

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

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

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

1. Positive Vessel Isolation

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

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

2. Vessel Access

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

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

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

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

3. Wearing of a Safety Harness

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

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

4. Providing a Manway Watch

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

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

5. Providing Fresh Air

The vessel must be purged completely free of any noxious or poisonous gases and inventoried with fresh air before permitting anyone to enter. The responsible department, usually the safety department, must test the atmosphere within the

vessel for toxic gases, oxygen and explosive gases before entry. This must be repeated every 4 hours while there are people inside the vessel. When possible the UOP technical advisor should personally witness the test procedure. Each point of entry and any dead areas inside of the vessel, such as receiver boots or areas behind internal baffles, where there is little air circulation should be checked.

Fresh air can be circulated through the vessel using an air mover, a fan, or, for the cases where moisture is a concern, the vessel can be purged using dry certified instrument air from a hose or hard piped connection. When an air mover is used, make certain that the gas driver uses plant air, not nitrogen, and direct the exhaust of the driver out of the vessel to guarantee that this gas does not enter the vessel. When instrument air is used, the UOP technical adviser must confirm that a check of the supply header is made to ensure that it is properly lined up and that there are no connections where nitrogen can enter the system (nitrogen improperly used as a backup for instrument air by some refiners). The fresh air purge should be continued throughout the time that people are inside of the vessel. The responsible control room should be informed that instrument air is being used for breathing so that if a change to nitrogen is required the people are removed from the affected vessel.

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

6. Preparation of Vessel Entry Permit

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

7. Checkout Prior to New Unit Startup

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

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

8. Inspections During Turnarounds

In turnaround inspections, the possibility that vessels will contain dangerous gases is much higher. Equipment that has been in service must be thoroughly purged before entry. The vessel should have been steamed out unless steam presents a hazard to the internals and then fresh air circulated through it until all traces of hydrocarbons are gone. If liquid hydrocarbon remains or if odors persist afterwards, repeat the purging procedure until the vessel is clean. The service history of the vessel must also be investigated before entry so that appropriate precautions may be taken. The service may require a neutralization step or a special cleaning step to make the vessel safe. Internal scale can trap poisonous gases such as hydrogen sulfide or hydrogen fluoride 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 at the vessel with absolutely no exceptions. There will always be process material at the low and high points in the lines connecting to the vessel because it is not possible to purge them completely clean. The blinds must all be in place before the vessel is purged.

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

9. Reactor Loading

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

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

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

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

Section XIII in this manual contains detailed reactor loading procedures.

10. Reactor Unloading

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

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

- Personnel working in reactors being unloaded should wear a fresh air respirator with a hood or helmet, operated in a pressure demand or other positive pressure mode, or in a continuous flow mode (NIOSH Respirator Code SAFE: PD, PP, CF). This respirator should have a primary, secondary, and emergency supply of air.
- Personnel in the reactor should be equipped with safety harnesses and safety lines for rescue and a means for visual, voice or signal line communication with standby personnel, who should be strategically located with suitable rescue equipment.
- The OSHA regulations concerning use of respirators (29 CFR, Subpart 1, Section 1910.134) should be read and thoroughly understood before undertaking to place personnel in reactors during catalyst loading and unloading operations.
- Protective clothing and all safety devices should be thoroughly decontaminated after each use. Worn-out, broken or defective safety equipment and clothing should be removed from service and repaired or replaced. Good personal hygiene after handling a catalyst or being exposed to catalyst dust is an essential part of a responsible catalyst safety program. Do not eat, drink, or smoke in areas where the catalyst is being handled or where exposure to catalyst dust is likely.

F. MATERIAL SAFETY DATA SHEET (MSDS)

1. MSDS SHEETS FOR DEFINE CATALYSTS

An MSDS Sheets for the Define H-14 catalyst is included.

2. HEALTH AND SAFETY GUIDELINES FOR SULFIDING AGENTS

Material safety data sheets for the sulfiding agents should be provided by the vendor of the sulfiding agent.



MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY INFORMATION

Product Name: H-14 DEFINE CATALYST

Product Use: Catalyst

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

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

Emergency Assistance - 24 hour Emergency Telephone Numbers:

USA (UOP LLC) : + 1-847-391-2123
USA (CHEMTREC) : + 1-800-424-9300
Canada (CANUTEC) : + 1-613-996-6666
Outside USA (CHEMTREC) : + 1-703-527-3887

2. COMPOSITION/INFORMATION ON INGREDIENTS

INGREDIENT & CAS NO.	% WEIGHT	ACGIH TLV-TWA	OSHA PEL-TWA	UNITS
Aluminum oxide (non-fibrous) 1344-28-1	>94	10	15(TD) 5(R)	mg/m ³
Nickel oxide 1313-99-1	<10	0.2 as (I) Ni	1 as (I) Ni	mg/m ³
Nickel 7440-02-0	<6	1.5 (I)	1	mg/m ³

NOTE: UOP Internal Exposure Limit for Nickel (elemental/metal) 1.0 mg/m³
UOP Internal Exposure Limit for Nickel, insoluble/soluble compounds, as Ni 0.1 mg/m³

Abbreviations:

N.A.	- Not Applicable	RD	- Respirable Dust	Fu	- Fume	IS	- Insoluble
N.E.	- None Established	R	- Respirable Fraction	I	- Inhalable	FuD	- Fume and Dust
SC	- Soluble Compounds	F	- Respirable Fibers	TD	- Total Dust		

3. HAZARDS IDENTIFICATION

Emergency Overview:

Repeated or prolonged exposure may irritate eyes, skin and respiratory system. May cause sensitization by skin contact. Nickel and nickel compounds are classified as carcinogens.

Form: Spheres

Color: Black

Potential Health Effects:

Primary Routes of Exposure: Contact with skin and eyes. Exposure may also occur via inhalation or ingestion if product dust is generated.

Skin Contact: Repeated or prolonged exposure may cause skin irritation. Prolonged or repeated exposure may cause sensitization.

Eye Contact: Repeated or prolonged exposure may cause eye irritation.

Ingestion: Available data suggest no adverse toxic effects.

Inhalation: Exposure to dust particles generated from this material may cause irritation of the respiratory tract. Prolonged or repeated inhalation may cause lung injury/cancer.

Chronic Effects: Nickel and Nickel compounds are classified as carcinogens.

Carcinogenicity Classification:

International Agency for Research on Cancer (IARC):

Nickel - Possibly Carcinogenic to Humans (Group 2B)

Nickel oxide - Carcinogenic to Humans (Group 1).

U.S. National Toxicology Program (NTP):

Nickel oxide - Known to be a Human Carcinogen.

Nickel and Insoluble Nickel compounds - Known to be a human carcinogen.

U.S. Occupational Safety and Health Administration (OSHA):

Nickel and Certain Nickel Compounds - Possible Select Carcinogens

American Conference of Governmental Industrial Hygienists (ACGIH):

Aluminum oxide - Not Classifiable as a Human Carcinogen (A4).

Nickel - Not Suspected as a human Carcinogen (A5).

Nickel oxide - Confirmed Human Carcinogen (A1).

4. FIRST AID MEASURES

Eye contact: Flush immediately with plenty of water for at least 15 minutes. If eye irritation persists, consult a physician.

Skin contact: Wash off with soap and plenty of water. If skin irritation persists, call a physician.

After inhalation: Remove the victim into fresh air. If symptoms persist, call a physician.

After ingestion: Do not induce vomiting. Obtain medical attention.

Notes to physician: If the product is inhaled, clean the nasal passages. Skin and eye contact with the product or, more probably, product dust, may produce irritation. Repeated or prolonged exposure may cause skin irritation and dermatitis. Hydrocarbons and other materials that contact the product during normal use can be retained on the product. The used product can contain material of a hazardous nature. Identify that material and treat symptomatically.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media: The catalyst is non-combustible. Use extinguishing media for surrounding fire.

Unsuitable extinguishing media: N.A.

Fire and explosion hazards: The product itself does not burn. The used product can retain material of a hazardous nature. Identify that material and inform the fire fighters.

May form nickel carbonyl, which is very toxic and potentially lethal, on contact with carbon monoxide.

Special protective equipment: Wear protective clothing. In case of respirable dust and/or fumes, use self-contained breathing apparatus.

Flash Point: N.A.

6. ACCIDENTAL RELEASE MEASURES

Personal protection: See Section 8.

Environmental precautions: No special environmental precautions required.

Clean-up: Sweep, shovel or vacuum spilled product into appropriate containers (do not use a vacuum if material has contacted a hydrocarbon material). Pick-up and arrange disposal without creating dust. Never use spilled product. Spilled product should be disposed of in accordance with all applicable government regulations. (See Section 13).

7. HANDLING AND STORAGE

Handling: Handle and open container with care. Avoid formation of dust particles. Avoid contact with skin and eyes. Take precautionary measures against static discharges.

Storage: Store in original container. Keep tightly closed in a dry and cool place.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Engineering measures: Ensure adequate ventilation, especially in confined areas.

Personal protection equipment: Handle in accordance with good industrial hygiene and safety practice.

Eye protection: Safety glasses.

Hand protection: Protective gloves.

Skin and body protection: Lightweight protective clothing.

Respiratory protection: In case of insufficient ventilation, wear suitable respiratory equipment. Air-purifying respirator with NIOSH classification N-100 filter or P-100 (or equivalent) if oil/liquid aerosols are present (42 CFR 84).

9. PHYSICAL AND CHEMICAL PROPERTIES

Info These data do not represent technical or sales specifications.

Form: Spheres

Color: Black

Odor: None

pH: N.A.

Boiling point/range: N.A.

Melting point/range: >1000°C

Flash point: N.A.

Autoignition temperature: N.A.

Bulk density: 21±2 lbs/ft³ (0.34±0.03 g/cc)

Explosion limits: N.A.

Vapor pressure: N.A.

Relative density/Specific Gravity: N.A.

Vapor density: N.A.

Viscosity: N.A.

Water solubility: Insoluble

Solubility: N.D.

Abbreviations:

N.D. - Not Determined
N.A. - Not Applicable

10. STABILITY

Stability: Stable at normal conditions.

Hazardous decomposition products: Hydrocarbons and other materials that contact the product during normal use can be retained on the product. It is reasonable to expect that decomposition products will come from these retained materials of use.

Conditions/Materials to avoid: Contact with carbon monoxide under certain conditions can form very toxic and potentially lethal nickel carbonyl. Free water may damage the product.

II. TOXICOLOGICAL INFORMATION

Acute toxicity:

LD50/oral/rat = > 5 000 mg/kg *

LD/dermal/rabbit = > 2 000 mg/kg *

LC50/inhalation/rat = > 2 mg/l *

Chronic toxicity: Classification of Ingredients

EC Carcinogenic:

Cat. 1 (Nickel oxide)

Cat. 3 (Nickel)

Carcinogenicity (ACGIH):

A4 (Aluminum oxide)

A5 (Nickel)

A1 (Nickel oxide)

EC Mutagenic:

Not listed.

IARC classification:

Group 2B (Nickel)

Group 1 (Nickel oxide)

EC Toxic for Reproduction:

Not listed.

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

Irritation:

Skin (rabbit): Not classified as a skin irritant. *

Eye (rabbit): Not classified as an eye irritant. *

Additional product information:

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

May cause skin sensitization.

Avoid repeated exposure.

Additional component information:

No data available.

12. ECOLOGICAL INFORMATION

Mobility:

No data available.

Biodegradation:

No data available.

Bioaccumulation:

No data available.

Aquatic toxicity:

No data available.

Further Information:

No information available.

13. DISPOSAL CONSIDERATIONS

Provisions relating to waste: EPA - Resource Conservation and Recovery Act (RCRA) Hazardous and Solid Waste Management Regulations.

Disposal information: This product (in its fresh unused state) is not listed by generic name or trademark name in the U.S. EPA's RCRA regulations and does not possess any of the four identifying characteristics of hazardous waste (ignitability, corrosivity, reactivity or toxicity). The used product can contain material of a hazardous nature. Identify that material and dispose accordingly.

14. TRANSPORT INFORMATION

UN-No.:

N.A.

Proper shipping name:

Not applicable.

Packing group:

N.A.

<u>Transport Mode</u>	<u>Class</u>	<u>Additional Information</u>		<u>Remarks</u>
U.S. DOT:	Not regulated.	Reportable Quantity:	Nickel - 100 lbs.	N.A.
		Marine Pollutant DOT:	No	
ADR/RID:	Not regulated.	Danger Code:	N.A.	N.A.
IMDG:	Not regulated.	Marine pollutant:	No	N.A.
		EmS:	N.A.	
IATA:	Not regulated.	Instr. Passenger:	N.A.	N.A.
		Instr. Cargo:	N.A.	

15. REGULATORY INFORMATION

United States

Toxic Substances Control Act (TSCA): All the ingredients of this mixture are registered on the TSCA Chemical Substance Inventory.

CERCLA (Comprehensive Environmental Response, Compensation, and Liability Act) Reportable Quantity:

The following component(s) of this product is/are subject to release reporting under 40 CFR 302 when release exceeds the Reportable Quantity (RQ):

Nickel - RQ of 100 lbs. (No reporting of releases of this hazardous substance is required if the diameter of the pieces of the solid metal released is equal to or exceeds 100 micrometers (0.004 inches)).

SARA Title III (Superfund Amendments and Reauthorization Act of 1986):

Section 302 (Extremely Hazardous Substances):

The following component(s) of this product is/are subject to the emergency planning provisions of 40 CFR 355 when there are amounts equal to or greater than the Threshold Planning Quantity (TPQ):

-- None --

Section 313 (Toxic Chemicals):

The following component(s) have been specified as Toxic Chemicals under SARA Section 313 and may be subject to the Toxic Release Inventory (TRI) reporting requirements under 40 CFR 372:

Nickel compounds - Category N495

Nickel

The following components are listed in U.S. State Regulations:

State Reg Reference:

California - Proposition 65:

State Reg Reference:

Nickel

Nickel oxide

Massachusetts Right-To-Know:

Aluminum oxide

Nickel oxide

Nickel

New Jersey Right-To-Know:

Aluminum oxide

Nickel

Nickel oxide

Pennsylvania Right-To-Know:

Aluminum oxide

Nickel

Nickel oxide

Note: Other U.S. State Regulations may exist, check your local sources if available or contact the UOP Product Stewardship Manager (see Section 16).

Canada

Canadian Hazardous Products Act:

This product is classified as a material causing Other Toxic Effects, carcinogenicity and embryotoxicity - Class D, Division 2, Subdivision A.

This product is classified as a material causing Other Toxic Effects, sensitization - Class D, Division 2, Subdivision B.

The above classification(s) is/are in accordance with the regulations pursuant to the Federal Hazardous Products Act (e.g. WHMIS)

Canadian Environmental Protection Act: All the ingredients of this mixture are notified to CEPA and on the DSL (Domestic Substances List).

European Union (EU)

European Inventory of Existing Commercial Chemical Substances: All components of this product are included in EINECS/ELINCS.

Council of European Communities Directive on Classification, Packaging and Labelling of Dangerous Substances/Preparation (67/548/EEC & 1999/45/EC, as amended):

The following label information applies:

Symbol(s):	T - Toxic
Contains:	Nickel oxide Nickel
Risk Phrases:	R49 May cause cancer by inhalation. R43 May cause sensitisation by skin contact.
Safety phrases:	S22 Do not breathe dust. S53 Avoid exposure - obtain special instructions before use. S45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible). S36/37/39 Wear suitable protective clothing, gloves and eye/face protection.

Additional Governmental Inventories

Australia - Inventory of Chemical Substances (AICS): All the ingredients of this mixture appear on the AICS.

China: All the ingredients of this mixture appear on the China Inventory.

Japan - Existing and New Chemical Substances (ENCS): All the ingredients of this mixture are registered in accordance with Japanese regulations.

Korea - Existing and Evaluated Chemical Substances (ECL): All the ingredients of this mixture appear on the ECL.

Philippines - Inventory of Chemicals and Chemical Substances (PICCS): All the ingredients of this mixture appear on the PICCS.

16. OTHER INFORMATION

Summary of changes: 3, 9
Supersedes: April 2004
Prepared by: UOP Health, Safety & Environmental Department

HMIS™ - Hazardous Material Information System:

HMIS™ Ratings: 0-minimal hazard, 1- slight hazard, 2- moderate hazard, 3- serious hazard, 4- severe hazard.

HEALTH : 1* - * may cause cancer
FLAMMABILITY : 0
REACTIVITY : 0

For additional information concerning this product, contact the following:

For health, safety and environmental information, please contact:

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

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

For technical or purchasing information, please contact:

Customer Order Management
UOP LLC
25 E. Algonquin Road
Des Plaines, IL 60017-5017
USA
Tel: +1-800-243-6757
Fax: +1-847-391-3690

PRODUCT EMERGENCIES

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

24-Hour EMERGENCY NUMBER (UOP LLC) : + 1 - 847 - 391 - 2123

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



SAFETY DATA SHEET

(according to Directive 91/155/EEC as amended)

1. PRODUCT AND COMPANY IDENTIFICATION

Product Name: H-14 DEFINE CATALYST

Product Use: Catalyst

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

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

Emergency Assistance - 24 hour Emergency Telephone Numbers:

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

2. COMPOSITION/INFORMATION ON INGREDIENTS

INGREDIENT & CAS NO.	EINECS NO. ELINCS NO.	% WEIGHT	EC SYMBOL	EC R-PHRASES
Aluminum oxide (non-fibrous) 1344-28-1	215-691-6	>94	N.E.	N.E.
Nickel 7440-02-0	231-111-4	<6	Xn	40, 43
Nickel oxide 1313-99-1	215-215-7	<10	T	49, 43, 53

Note:

- Full text of R-phrases is given in Section 16.

- UOP Internal Exposure Limit for Nickel (elemental/metal) 1.0 mg/m³
- UOP Internal Exposure Limit for Nickel, insoluble/soluble compounds, as Ni 0.1 mg/m³

3. HAZARD IDENTIFICATION

Classification/Overview:

Classified as hazardous according to Directive 1999/45/EC as amended.
May cause cancer by inhalation. May cause sensitisation by skin contact.

Potential Health Effects:

- **Eye Contact:**
Repeated or prolonged exposure may cause eye irritation.
- **Skin Contact:**
Repeated or prolonged exposure may cause skin irritation. Contains a component which is a known or suspected skin sensitizer.
- **Inhalation:**
Exposure to dust particles generated from this material may cause irritation of the respiratory tract. Prolonged or repeated inhalation may cause lung injury/cancer.
- **Ingestion:**
Available data suggest no adverse effects.

Potential Environmental Effects:

If used as directed, no hazardous effects to the environment are to be expected.

4. FIRST AID MEASURES

Eye contact:

Flush immediately with plenty of water for at least 15 minutes. If eye irritation persists, consult a physician.

Skin contact:

Wash off with soap and plenty of water. If skin irritation persists, call a physician.

After inhalation:

Remove the victim into fresh air. If symptoms persist, call a physician.

After ingestion:

Do not induce vomiting. Obtain medical attention.

Additional information:

Symptomatic treatment is advised. Repeated or prolonged exposure may cause skin irritation and dermatitis. If the product is inhaled, clean the nasal passages. Hydrocarbons and other materials that contact the product during normal use can be retained on the product.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media:

Non-combustible. Use extinguishing media for surrounding fire.

Unsuitable extinguishing media:

N.A.

Special exposure hazards:

The product itself does not burn. May form nickel carbonyl, which is very toxic and potentially lethal, on contact with carbon monoxide. The used product can retain material of a hazardous nature. Identify that material and inform the fire fighters.

Special protective equipment:

Wear protective clothing. In case of respirable dust and/or fumes, use self-contained breathing apparatus.

6. ACCIDENTAL RELEASE MEASURES

Personal protection:

See Section 8.

Environmental precautions:

No special environmental precautions required.

Clean-up:

Sweep, shovel or vacuum spilled product into appropriate containers (do not use a vacuum if material has contacted a hydrocarbon material). Pick-up and arrange disposal without creating dust. Never use spilled product. Spilled product should be disposed of in accordance with all applicable government regulations.

7. HANDLING AND STORAGE

Handling:

Handle and open container with care. Avoid formation of dust particles. Avoid contact with skin and eyes. Provide an electrical ground connection during loading and transfer operations to avoid static discharge in an explosive atmosphere and to prevent persons handling the product from receiving static shocks.

Storage:

Store in original container. Keep tightly closed in a dry and cool place.

Specific use:

Handle in accordance with good industrial practices.

8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

International Exposure Limits (mg/m³):

* In case no specific national exposure limit is established, the national exposure limit for general dust applies.

INGREDIENT & CAS NO.	EC Exposure Limit	UK OES/MEL	Germany MAK	The Netherlands MAC	France VME
Aluminum oxide (non-fibrous) 1344-28-1	N.E.	10(I) 4(R)	3(R)	10	10
Nickel 7440-02-0	N.E.	0.5	0.5(I) STEL: 2(I)	1	1
Nickel oxide 1313-99-1	N.E.	0.5(as Ni)	0.5(I) STEL: 2(I)	N.E.*	1(as Ni)

Occupational exposure controls:

Ensure adequate ventilation, especially in confined areas.

Personal protection equipment:

Handle in accordance with good industrial hygiene and safety practice.

- **Eye protection:** Safety glasses or goggles.
- **Hand protection:** Protective gloves.
Type of material: Dustproof (Vinyl-PE-PVC-Nitril-Latex)
Protection Category: Category 1 (EN420)
Breakthrough time: N.A.
- **Skin and body protection:** Dust impervious protective suit.
- **Respiratory protection:** In case of insufficient ventilation wear suitable respiratory equipment:
Disposable filtering half mask Class FFP3 (EN149).
Half mask with a particle filter P3 (EN143).

Environmental exposure controls:

Handle in accordance with local environmental regulations and good industrial practices.

9. PHYSICAL AND CHEMICAL PROPERTIES

These data do not represent technical or sales specifications.

Form:

Spheres

Vapor pressure:

N.A.

Color:

Black

Relative density/Specific Gravity:

N.A.

Odor:

None

Vapor density:

N.A.

pH:

N.A.

Viscosity:

N.A.

Boiling point/range:

N.A.

Melting point/range:

N.A.

Water solubility:

Insoluble

Solubility:

N.D.

Flash point:

N.A.

Bulk density:

21±2 lbs/ft³ (0.33±0.03 g/cc)

10. STABILITY AND REACTIVITY

Stability:

Stable at normal conditions.

Hazardous decomposition products:

Hydrocarbons and other materials that contact the product during normal use can be retained on the product. It is reasonable to expect that decomposition products will come from these retained materials of use. If the product is subject to extreme temperatures or chemical conditions, decomposition may occur and the product will include the oxides shown in Section 2.

Conditions/Materials to avoid:

Contact with carbon monoxide under certain conditions can form very toxic and potentially lethal nickel carbonyl.

11. TOXICOLOGICAL INFORMATION

Product toxicity:

LD50		LC50	Irritation (rabbit)	
oral/rat	dermal/rabbit	inhalation/rat	eyes	skin
> 5 000 mg/kg *	> 2 000 mg/kg *	> 2 mg/l *	Not classified as an eye irritant. *	Not classified as a skin irritant. *

Additional product information:

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

May cause skin sensitization.

Avoid repeated exposure.

Ingredient information:

INGREDIENT & CAS NO.	LD50 (mg/kg)		LC50 (mg/l)	Irritation (rabbit)	
	oral/ rat	dermal/ rabbit	inhalation/ rat	eyes	skin
Aluminum oxide (non-fibrous) 1344-28-1	>10000	N.D.	N.D.	None	None
Nickel 7440-02-0	>5000	N.D.	N.D.	N.D.	N.D.
Nickel oxide 1313-99-1	>5000	N.D.	N.D.	N.D.	N.D.

INGREDIENT & CAS NO.	EC Category			US Classification		International
	C	M	R	NTP	OSHA	IARC Group
Aluminum oxide (non-fibrous) 1344-28-1	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
Nickel 7440-02-0	3	N.E.	N.E.	NTP-K	OSHA-PSC	2B
Nickel oxide 1313-99-1	1	N.E.	N.E.	NTP-K	OSHA-PSC	1

Notes:

NTP-K : Known To Be A Human Carcinogen.

OSHA-PSC : Possible Select Carcinogen.

12. ECOLOGICAL INFORMATION

Product ecotoxicity:

LC50/96hr Fish	EC50/48hr Daphnia	IC50/72hr Algae	Log Pow	BCF
N.D.	N.D.	N.D.	N.D.	N.D.

Ingredient information:

INGREDIENT & CAS NO.	LC50/96hr Fish	EC50/48hr Daphnia	IC50/72hr Algae	Log Pow	BCF
Aluminum oxide (non-fibrous) 1344-28-1	N.D.	N.D.	N.D.	N.D.	N.D.
Nickel 7440-02-0	N.D.	N.D.	N.D.	N.D.	N.D.
Nickel oxide 1313-99-1	>100	>100	>100	N.D.	N.D.

13. DISPOSAL CONSIDERATIONS

Provisions relating to waste:

EC Directives 75/442/EEC; 91/689/EEC as amended.

EC Regulation No. 259/93 as amended.

Disposal information:

Where possible recycling is preferred to disposal or incineration. Can be landfilled or incinerated, when in compliance with the local regulations. Materials of a hazardous nature that contact the product during normal use may be retained on this product. The user of the product must identify the hazards associated with the retained material in order to assess the waste disposal options.

14. TRANSPORT INFORMATION

UN-No.: N.A.	Proper shipping name: Not applicable.	Packing group: N.A.
------------------------	---	-------------------------------

Transport Mode	Class	Remarks		
ADR/RID:	Not regulated.	Danger Code:	N.A.	N.A.
IMDG:	Not regulated.	Marine pollutant:	No	N.A.
		EmS:	N.A.	
IATA:	Not regulated.	Instr. Passenger:	N.A.	N.A.
		Instr. Cargo:	N.A.	

15. REGULATORY INFORMATION

The product is classified and labelled in accordance with the EC Directives 67/548/EEC and 1999/45/EC, as amended.

The product needs to be labelled in accordance with EC-Directives.

Symbol(s): T - Toxic

Contains: Nickel oxide
Nickel

Risk Phrases: R49 May cause cancer by inhalation.
R43 May cause sensitisation by skin contact.

Safety phrases: S22 Do not breathe dust.
S53 Avoid exposure - obtain special instructions before use.
S45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).
S36/37/39 Wear suitable protective clothing, gloves and eye/face protection.

16. OTHER INFORMATION

Summary of changes: New Format. Sections 8, 15
Supersedes: July 2000
Prepared by: UOP Health, Safety & Environmental Department

Abbreviations:

N.A.	Not applicable
N.E.	None established
N.D.	Not determined
OES	Occupational Exposure Standard
MEL	Maximum Exposure Limit
MAK	Maximale Arbeitsplatz-Konzentration
MAC	Maximale Arbeidsplaats Concentratie
VME	des Valeurs limites de Moyenne d'Exposition
STEL	Short-Term Exposure Limit
EC	European Community
IARC	International Agency for Research on Cancer
OSHA	Occupational Safety and Health Administration
NTP	National Toxicology Program
SC	Soluble Compounds
FuD	Fume and Dust
Fu	Fume
D	Dust
I	Inhalable
R	Respirable
AS	Aqueous slurry
IS	Insoluble Compounds

R-phrases in Section 2:

R40	Limited evidence of a carcinogenic effect.
R43	May cause sensitisation by skin contact.
R49	May cause cancer by inhalation.
R53	May cause long-term adverse effects in the aquatic environment.

For additional information concerning this product, contact the following:

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

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

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

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

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

PRODUCT EMERGENCIES

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

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

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





XV. EQUIPMENT EVALUATION

The Mechanical Evaluation Test should cover all facets of the DeFine Unit, including the reactor, and Make-up Gas compressor. All major pieces of equipment should be part of this test, including vessels, pumps, compressors, 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.

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

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.

D. UTILITY CONSUMPTION/PRODUCTION DATA

1. Air (Plant and Inst.)
2. Hot Oil
3. Cooling water

E. SAMPLES (TYPICAL)

1. DeFine Effluent
2. Pacol Separator liquid
3. Make up gas

RECIPROCATING COMPRESSOR DATA

page _____

date _____

Item No.: _____ by _____

Service: _____

Manufacturer: _____

Type, Model: _____

No. of Stages: _____

OPERATING CONDITIONS/PERFORMANCE

Flow Rate: _____ Suction Temperature: _____ °F

Suction Pressure: _____ psig Discharge Temperature: _____ °F

Discharge Pressure: _____ psig Power: _____ hp

Differential Head: _____ MW: _____

Polytropic η : _____

Operating Speed: _____ rpm

Type of Seal: _____

Lube/Seal Oil System: _____

Buffer Gas: (yes/no) Buffer Gas Rate: _____ SCFH

Automatic Surge Control: (yes/no)

DRIVER

Motor Manufacturer: _____

Rating: _____ Service Factor: _____

Insulation Class: _____ Voltage/phase/cycle: _____

Turbine Manufacturer: _____

Speed: _____ Steam Supply: _____ psig _____ °F

Steam Rate: _____ Steam Exhaust: _____ psig _____ °F

Gear Manufacturer: _____

Rating: _____ Service Factor: _____

Type: _____ Power Loss: _____

Deviations from UOP Specification: _____

CONTROL VALVE SUMMARY

Item No.: _____ page _____
Service: _____ date _____
Description of Valve: _____ by _____
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: _____

		Stream	Pressure	Temperature
Shell Side	Inlet	A	_____	_____
	Outlet		_____	_____
Tube Side	Inlet	B	_____	_____
	Outlet		_____	_____

Q (calc.) Shell side _____

Q (calc.) Tube side _____

Composition, _____ %

	A	B
H ₂	_____	_____
N ₂	_____	_____
H ₂ S	_____	_____
H ₂ O	_____	_____
C ₁	_____	_____
C ₂	_____	_____
C ₃	_____	_____
C ₄	_____	_____
C ₅	_____	_____
C ₆	_____	_____
C ₇	_____	_____
C ₈	_____	_____
C ₉	_____	_____
C ₁₀	_____	_____
C ₁₁	_____	_____
C ₁₂	_____	_____
C ₁₃	_____	_____
C ₁₄	_____	_____
C ₁₅	_____	_____
Mass Flow	_____	_____
Avg. Mol. Wt.	_____	_____
Gravity	_____	_____

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: _____

REACTOR SECTION PRESSURE SURVEY **(delete unused equipment)**

page _____
 date _____
 by _____

			Pressure	Temperature
Feed pump discharge			_____	_____
Feed CV	Inlet		_____	_____
	Outlet		_____	_____
Charge Heater	Inlet		_____	_____
	Intershell	A	_____	_____
		B	_____	_____
		C	_____	_____
		D	_____	_____
		E	_____	_____
		F	_____	_____
	Outlet		_____	_____
Sulfur Injection Pump Discharge			_____	_____
RX #1	Inlet		_____	_____
	Outlet		_____	_____
RX #2	Inlet		_____	_____
	Outlet		_____	_____
RX #3	Inlet		_____	_____
	Outlet		_____	_____
Separator Gas to Booster Compressor			_____	_____
	Suction		_____	_____
	Discharge		_____	_____

