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PPG INDUSTRIES
INDUSTRIAL CHEMICAL DIVISION

LAKE CHARLES, LOUISIANA

OXYHYDROCHLORINATION (OHC-EDC)
OPERATIONS MANUAL

MANUAL No. 23

ASSIGNED TO: [redacted]
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CONFIDENTIAL

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OHC-EDC PROCESS

INTRODUCTION

This operations manual has been assembled to serve the following objectives:

A. As a training guide for engineers and operators in learning the OHC-EDC plant operations.

B. To serve as a ready reference for the operating personnel.

C. To provide a standard approach for operating the plant so that continuity of operations is maintained.

D. To serve as a place where current data, information, procedures, etc., relating to the OHC-EDC plant are compiled.

This manual is designed to provide a logical approach to the start-up and initial operation of the OHC-EDC plant. As operational experience is gained, it is expected that some of the Standard Operating Procedures (SOP's) will have to be altered. The operators can help to keep this manual up to date by making recommendations to change SOP's that appear to be no longer in use. The information contained in this manual is considered confidential. This manual is the property of PPG and may be recalled at any time.

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I. COMPOUND ABBREVIATIONS

Abbreviation	Name	Formula	Atm. Boiling Point °F
CO	Carbon Monoxide (Light)	CO	-378.0
N_2	Nitrogen (Light)	N_2	-320.4
O_2	Oxygen (Light)	O_2	-297.4
C_2H_4	Ethylene (Light)	C_2H_4	-219.0
C_2H_6	Ethane (Light)	C_2H_6	-190.9
CO_2	Carbon Dioxide (Light)	CO_2	-173.0 (Subl.)
Cl_2	Chlorine (Light)	Cl_2	-29.3
VC	Vinyl Chloride (Light)	C_2H_3Cl	7.9
$C_2H_5Cl_2$	Ethyl Chloride	C_2H_5Cl	55.4
F-11	Trichloromonofluoromethane (Light)	CCl_3F	77.4
VDC	Vinylidene Chloride (Light)	$C_2H_2Cl_2$	88.9
CH_2Cl_2	Methylene Chloride	CH_2Cl_2	104.2
Trans-DCE	Trans-1,2-Dichloroethylene (Light)	$C_2H_2Cl_2$	119.1
1,1-EDC	1,1-Dichloroethane (Light)	$C_2H_4Cl_2$	135.1
Cis-DCE	Cis-1,2-Dichloroethylene (Light)	$C_2H_2Cl_2$	140.4
CHCl	Chloroform (Light)	$CHCl_3$	143.1
Carbon Tet.	Carbon Tetrachloride (Light)	CCl_4	170.2
1,2-EDC	1,2-Dichloroethane (Key)	$C_2H_4Cl_2$	182.2
TRI	Trichloroethylene (Heavy)	C_2HCl_3	188.7
Chloral	Chloral	CCl_3CHO	207.7
H_2O	Water	H_2O	212.0
1,1,2-TCE	1,1,2-Trichloroethane (Heavy)	$C_2H_3Cl_3$	236.8
PER	Perchloroethylene (Heavy)	C_2Cl_4	249.8
Unsym-Tet.	Unsym-tetrachloroethane (Heavy)	$C_2H_2Cl_4$	266.9
Sym-Tet.	Sym-tetrachloroethane (Heavy)	$C_2H_2Cl_4$	295.3
Tars or HvCl	Any heavy chlorinated compounds		

NOTE: "Heavy" indicates compounds having higher boiling points than EDC.
"Light" indicates compounds having lower boiling points than EDC.

II. GENERAL DESCRIPTION OF THE OHC PROCESS

A. Uses of Product (EDC)

The chief uses of the EDC produced in the OHC plant are as feed to the Per-Tri Plant and Vinyl Chloride Plant (via the heavies still in EDC Plant #2). At the VC Plant the EDC is cracked to form vinyl chloride and hydrogen chloride, which in turn is used as a feed to the OHC plant. EDC is chlorinated in the Per-Tri plant to form perchloroethylene, a dry-cleaning agent, and trichloroethylene, a degreasing solvent.

B. Chemistry

In the past EDC has been produced by PPG in liquid-phase reactors by direct chlorination of ethylene, which is as follows:

$$\underset{\text{Ethylene}}{\overset{H\qquad H}{HC = CH}} + \underset{\text{chlorine}}{Cl_2} \xrightarrow[\text{Catalyst}]{FeCl_3} \underset{\substack{\text{Ethylene Dichloride}\\ \text{(EDC)}}}{\underset{Cl\qquad Cl}{\overset{H\qquad H}{HC - CH}}} + \text{Heat}$$

Mol. Wt.		
28.052	70.914	98.966

The OHC Plant will produce EDC by the following method, however:

$$\underset{\text{Ethylene}}{\overset{H\qquad H}{HC = CH}} + \underset{\substack{\text{Hydrogen}\\ \text{Chloride}}}{2\ HCl} + \underset{\text{Oxygen}}{1/2\ O_2} \xrightarrow{\text{Catalyst}} \underset{\substack{\text{Ethylene}\\ \text{Dichloride}}}{\underset{Cl\qquad Cl}{\overset{H\qquad H}{HC - CH}}} + \underset{\text{Water}}{H_2O} + \text{Heat}$$

Mol. Wt.				
28.052	72.930	16.000	98.966	18.008

In the reactors, oxygen is reacted with HCl to liberate Cl_2 by the "Deacon" reaction as follows:

$$2\ HCl + 1/2\ O_2 \xrightarrow{\text{Catalyst}} Cl_2 + H_2O + \text{Heat}$$

The free chlorine thus formed can then be reacted with the ethylene to form EDC. The catalyst in this reaction is a carrier coated with cupric chloride and potassium chloride.

Side reactions occur to form lighter and heavier compounds in addition to EDC. These are limited by controlling the reaction temperature, ethylene conversion, and the excess oxygen (it is not practical to use enough oxygen for complete HCl recovery). If too much oxygen is used, then burning of the organics will take place and the CCl_4 in the product will increase. This represents a loss in yield of ethylene. Thus, a balance will have to be made between recovery of Cl_2 from HCl and undesirable side reactions. The reactors are designed for 75% ethylene conversion per pass; this is accomplished by recycling most of the reactor vent gas, which is rich in ethylene content, back through the reactor.

C. Process Description

1. <u>Reactors</u>: HCl, C_2H_4 and recycle gas are heated, premixed, and fed through a series of nozzles at the bottom of each of the two reactors. Oxygen is fed separately and enters the reactor's fluidized catalyst bed 19 inches above the bottom feed plate through several "risers." The end of each riser has a single small hole pointing vertically upward. Sixteen banks of several hairpin loops of 2" piping are suspended in the catalyst; liquid Dowtherm "A" is circulated through these banks to remove the heat of reaction. As required, a portion of the heated Dowtherm is sent to a cooler. The amount of heated Dowtherm that by-passes the cooler is controlled to give an average Dowtherm return temperature necessary to control the reactor temperature.

Condensate is heated to form 30 psig steam in the Dowtherm coolers. All of the steam used in the OHC plant is 30 psig, but the reactors generate more steam than the plant can use. The 2 EDC strippers in the EDC plants will use some of this steam, but the majority will be used by the stripper in the vinyl chloride plant.

The superficial gas velocity, based on reactor temperature, pressure, and feed rates, will be maintained at 0.8 ft/sec. Poor mixing (because of plugged feed distributors), less than 0.8 ft/sec velocity, or insufficient Dowtherm cooling can cause hot spots and catalyst clinkering, which could burn through the Inconel hairpins.

The reaction gases then pass through about 15 ft. of vapor space, where most of the catalyst drops out. The remaining catalyst dust is removed by a Centrifix, from which it drops to and is collected in a heated vessel. This catchpot is periodically dumped.

2. Condensers: The hot gases then go to a series of 4 graphite heat exchangers. The first is a water-cooled up-flow condenser. Because graphite can tolerate temperatures only up to 330^{o}F, this condenser, and the next one also, have control valves to feed flush water to the top of the tubes. The primary condenser water flush will come on automatically if the bottom condenser head temperatures becomes too high. The Fluorogreen-lined tee under the primary condenser is also heat sensitive and needs the primary water flush whenever there is not enough normal condensate falling out of the primary condenser. In addition, the water flush on the first two condensers serves to flush out tube wall catalyst deposit.

The second and third condensers are downflow and water-cooled. The 4th condenser is downflow and brine-cooled. The vent temperature from this refrigerated condenser controls the brine flow.

Nearly all of the virtually EDC-free gas remaining then goes to the recycle system; however, a portion is vented automatically (by pressure control) to one or both of two scrubbers, depending on the size of the stream. The recycle system will be covered in more detail later.

The condensate from all four heat exchangers collects in a degasser where a liquid level is maintained automatically by a level control valve; this valve also maintains a seal on the reactor system. The degasser is vented back to the refrigerated reactor condenser.

Liquid from the degasser goes to an atmospheric pressure flasher, which is a small vessel from which all pressure-dissolved gases are flashed off as the pressure is reduced. In the next vessel, the phase separator, the upper aqueous HCl layer overflows to the acid pit and the heavier EDC layer flows to the intermediate crude storage tanks through an interface level control valve.

3. <u>Venting:</u> Vent gases from the flashers, phase separators, intermediate crude tanks, and the drying still primary condenser contain appreciable quantities of recoverable EDC; therefore, they are fed to a brine-cooled vent condenser. The brine flow to this exchanger is controlled by the process exit temperature. Condensate from the vent condenser normally goes to one of two Haveg driers before passing to the Per-Tri feed tanks. The vent condenser exit

gases discharge to the stack vent; this is a small, unpacked vessel into which water is sprayed.

The "normal" reactor vents also discharge to the stack vent; however, there are times when the total vent stream is too large (during start-up or system upsets) for this vessel. Therefore, large vent streams are discharged to a large start-up vent scrubber. During reactor air fluidization and during start-up with a vent high in HCl, condensers 2, 3, and 4 will give a high back pressure due to the inert gas load. Consequently, a control valve (remotely operated) on the primary condenser vent can be opened to offset the high pressure drop. It, too, discharges to the start-up vent scrubber. Because it must absorb quite a bit of HCl, this Haveg scrubber contains ceramic packing.

4. <u>Recycle Scrubbing</u>: The gases from the condensers normally contain only small amounts of HCl, which must be removed due to the materials of construction in the recycle system. Therefore, this stream is sent to the recycle scrubber through which a $NaHCO_3$ solution is pumped to neutralize the HCl.

Because the bicarbonate surge tank blowdown will flash off ethylene as soon as the pressure is reduced, it is undesirable to feed this stream directly to the sewer. Therefore, it goes to the degassing tank first, where the ethylene is flashed off. A positive N_2 flow is maintained on this vessel.

5. <u>Recycle Compression</u>: Three reciprocating compressors serve both reactors; the middle one can serve as a spare for either reactor.

The compressor control system will stop the compressor if the suction pressure drops to 0 psig. Abnormally high discharge pressures are controlled by individual safety relief valves. The discharge pressure is controlled with an automatic compressor by-pass control valve.

Because moist CO_2 (carbonic acid) is slightly corrosive, all of the recycle system after the scrubber is built of stainless steel.

The forward recycle stream is heated and flow-controlled into the reactor. A continuous on-stream chromatographic analyzer receives its samples from the heated compressor discharge while continuous oxygen analyses are taken on the compressor suction header.

6. Chloral Treatment: The crude EDC in the intermediate crude storage tanks is pumped to the chloral treatment tank through a flow control valve. Dilute cell liquor is also added to this tank (through a flow control valve) and the two are mixed by an agitator which is installed inside the tank. This step must remove all of the chloral (CCl_3CHO), which is corrosive to the vinyl chloride cracking furnaces and also inhibits the cracking of EDC to vinyl chloride. Chloral is very difficult to remove from EDC by distillation, but the reaction with caustic forms a water-soluble sodium formate (NaOCOH) and chloroform ($CHCl_3$). Chloroform can be removed in the lights still.

The EDC-caustic mixture next passes to the chloral treatment phase separator. Here the caustic-water phase overflows to the sewer and the chloral-free EDC is pumped through an interface level control valve to the DH still feed tank.

7. <u>DH Still</u>: The function of this still is the removal of the dissolved water in the product from the chloral treatment system. This material may, at times, contain enough hydrochloric acid to be quite corrosive; therefore, the still is constructed of Haveg 41 and filled with a ceramic packing. The reboiler, however, is of Monel construction and it is essential that none of the wet feed to this column ever be allowed to accumulate in the reboiler.

The overhead stream passes through the DH still condenser where partial condensation takes place. The condensed material from this exchanger flows by gravity to the DH reflux drum. The organic then flows back to the top of the still while the aqueous HCl level overflows to the sewer. In order that all of the low-boiling compounds which concentrate in the top of the DH still may be recovered, the vent stream from the DH still condenser passes through the refrigerated vent condenser.

Steam is supplied to the DH still reboiler through a flow control valve. The bottoms withdrawal stream, which should contain a maximum of 20 ppm water, is regulated by a level control valve; this valve is controlled by the DH still reboiler level. ~~The dry EDC is then pumped through a product safety drier, which is filled with calcium chloride, and a filter, which should prevent any calcium chloride carryover~~.

The dry EDC crude can now go to one of two places: 1) If it is to be used in the Per-Tri plant, it will pass through the DH still bottoms cooler and then go to the Per-Tri feed tanks in the storage area. 2) If it will be required in the EDC Plant No. 2, then it will flow to the lights still feed tank through a level control valve according to the lights still feed tank level.

8. Lights Still: This still separates the low-boiling compounds, particularly chloroform, from the EDC and all of the compounds which boil at a higher temperature than EDC. The feed to this still is pumped through a flow control valve. The overhead stream is condensed and the condensate is collected in the lights still reflux drum. The DH still bottoms product contains a few parts per million of dissolved water. This water passes through the lights still with the overhead product where it is concentrated many-fold. Therefore, the overhead stream is sent through dryers to remove this water. These dryers, which are charged with calcium chloride flake, are much more efficient when drying cool liquid. Since the liquid in the reflux drum is near the boiling point, it is necessary to install two heat exchangers to cool the reflux before it enters the dryers. A filter downstream of the dryers is for the prevention of calcium chloride carryover. From here reflux is pumped back to the column through a flow control valve while the excess is sent to the Per-Tri feed tanks through a level control valve.

Steam is added to the lights still reboiler through a temperature control valve as determined by the temperature on the 20th tray.

9. Storage Tanks: The bottoms stream is pumped through a level control valve and a bottoms cooler enroute to the product storage

tanks. There are two tanks for storing EDC for the Per-Tri plant and two for the EDC Plant No. 2. The Per-Tri feed tanks can receive any dry material from the OHC plant. The EDC plant feed tanks, however, should receive material only from the bottom of the lights still; this material will be pumped to the heavies still, where the higher-boiling components are removed, and then to the one of the EDC-consuming units. The material from any of these four tanks can be pumped back through a rework line into several vessels within the process area. Approximately one million gallons of EDC can be stored in the storage tank west of the OHC process area. Product EDC can be transferred to this tank and stored for future use in the VC plant, Tri-Ethane plant, barge shipments, etc. Also, the EDC plants can vary their rates according to the chlorine availability without affecting the other plants which consume EDC.

III. SAFETY FOR OHC-EDC PLANT

A. General

Due to the nature of the EDC operations and the chemicals involved, the OHC Plant is restricted to authorized personnel only.

Company vehicles will be permitted to travel the roads skirting the plant area during normal operations. Special permits will be required for vehicles to enter any of the other areas in this plant.

Never cross a chain, regardless of whether it is up or down, with a vehicle without a pass. Passes for vehicles will be issued by the operating supervision or lead operators of the area. All passes are immediately revoked if the Area "B" evacuation whistle sounds. If you are operating a vehicle and the emergency horns sound, shut down the vehicle and evacuate the area as quickly as possible on foot. A new pass will be issued to remove the vehicle.

The control room and electrical starter rooms are pressurized for safety precautions; therefore, they are to be utilized by authorized personnel only. The doors and windows must be kept closed.

B. Definitions

Some of the terms used in the following discussion are defined below:

1. Flash Point: The flash point of a solvent is the lowest temperature at which vapor is given off in sufficient quantities so that the vapor-air mixture above the surface of the solvent will propagate a flame away from the source of ignition. It is the temperature below which a solvent may be used or stored in open containers without formation of an explosive vapor-air mixture.

2. Explosive Limits: When combustible vapor is mixed with air in the proper proportions, ignition will produce an explosion. The vapor-air mixtures which will form this proper proportion is called the explosive range. The explosive range includes all concentrations of a mixture of flammable vapor or gas in air in which a flash will occur or a flame will travel if the mixture is ignited. The lowest percentage at which this occurs is the lower explosive limit and the highest percentage is the upper explosive limit.

Explosive limits are expressed in percent by volume of vapor in air.

3. Maximum Allowable Concentration (MAC): The maximum allowable concentration for a material is the maximum concentration of that material that can be tolerated by personnel for a continuous 8-hour exposure with no ill effects (given as MAC numbers).

C. Chemicals in the OHC-EDC Plant

The chemicals that are required are listed on the next page:

D. Electrical Equipment

The characteristics of EDC and ethylene are such that the materials are classified in Group D, Class I by the U. S. National Electrical Code. The method and materials of installation are those recommended by Factory Insurance Association. In general, the installation is Class I, Group D, Division 2. Motors are TEFC (Totally Enclosed, Fan Cooled), lighting is vapor-tight, and all arcing devices are explosion-proof with seal-offs. Since the Control Building is pressurized and isolated from the process area, general purpose equipment is used. The Laboratory hood is not pressurized and is essentially furnished atmospheric air by a dual fan arrangement (one supplies air and one exhausts the fumes); this requires all explosion-proof equipment inside the hood. The laboratory itself is pressurized along with the rest of the control room.

1. Relamping and Receptacles: Vapor-tight fixtures are used in the process area. The relamping procedure will be to first determine the lamps which need changing by turning on all lights. Then, turn off all lights and relamp. Globes and guards must be replaced after relamping.

2. Grounding: Grounding in the plant has been given special attention due to problems peculiar to the handling of hydrocarbons. Equipment change-outs should be checked to see that the ground has been replaced.

Due to the tendency for hydrocarbons to build up a static electricity charge as a result of movement, agitation, or free fall through a gas space, a system of jumpers for pipe flanges has been installed to provide continuity of the piping system containing flammable materials. All vessels are grounded at two points. The result is a system of lines, tanks, and vessels operating at ground potential. This will not prevent the generation of static charges but should provide adequate leakage to ground to prevent the accumulation of dangerous charges.

A particular hazard is created by the use of cranes with long booms. Under certain conditions, depending on boom length and crane location, it is possible to pick up a voltage high enough to burn the person handling a load that is suspended from the crane. This will occur even if the crane is grounded. It is also conceivable that an arc would occur if the crane hooks touch a grounded object.

It is obviously important that the grounding system be maintained intact. This should be kept in mind when performing maintenance work on any equipment in the area.

E. Tools

Special spark-proof or alloy tools are not required in the area except where special precautions must be taken. However, precautions should be utilized in the handling of tools and equipment even under normal conditions. In other words, do not strike a metal object that could cause a spark in the presence of EDC, or ethylene, and air.

F. Pumps and Process Equipment

Any piece of equipment that is removed from the process must be thoroughly cleaned and inspected at a specified work area before it is permitted to leave the area for the plant shop or other work areas.

G. Safety Rules for the OHC Plant

The following list of safety rules is applicable to the OHC Plant. These safety rules are designed to provide a systematic approach to safety in the area. They are general enough so that the operating personnel, if they have a fundamental grasp of the reasons for the rules, will be able to extend their knowledge to situations not covered in this Manual.

1. Personnel entering the Area will deposit lighters, matches, regular flashlights (as opposed to explosion-proof flash-lights), etc., at the gate to the area.

2. Smoking will be permitted in the Control Building only.

3. The usual safety equipment will be required in the area, as follows:

   a) Safety hats

   b) Safety glasses

   c) Respirators

   d) Neoprene-coated gloves

4. The established general plant procedure will apply for locking-out electrical equipment and for safe-to-work permits on process equipment.

5. Only Company vehicles will be allowed to travel without a permit from the main east-west Area B road.

6. A vehicle permit signed by the operating supervisor or Lead Operator will be required for any vehicle to go into the OHC plant proper.

7. An equipment permit signed by the operating supervisor will be required before the following equipment can be carried into the area:

   a) Welding machines

   b) Cutting torches

   c) Electrically-driven drills

   d) Any electrical equipment except explosion-proof flashlights

e) Gasoline engine driven equipment not previously covered

f) Lighters, blow-torches or any flame producing equipment

g) Grinders, chipping equipment, or sand blasting equipment

8. Only explosion-proof flashlights will be permitted in the area.

9. Regular rubber plant utility hoses are not to be used for organic-chemical transfer or handling purposes.

10. All light circuits outside the Control Room or Electrical Starter Room must be off and tagged-out before relamping is undertaken.

11. Do not dump flammable organics into trapped sewers or openings where vapors could be evolved that would be either harmful to health or create an explosion hazard.

12. Do not leave an open sample or container of organics sitting around to give off vapors.

13. Use Full-Face, Chemox or Scott Air-Pac mask for protection against organic vapors.

14. Clothing that has been wet with organics should be removed immediately and the body thoroughly washed with soap and water. These clothes should be properly laundered before they are used again.

15. Do not permit air to enter any of the process equipment that contains organics or ethylene.

16. In the case of an emergency warning, all vehicles and equipment in the area on permits will be shut off immediately.

17. All steam-out nozzles or hoses and N_2 gas purge equipment must be properly grounded to prevent the possibility of an arc from accumulated static charge.

18. No smoking or open flames will be allowed in the Control Laboratory.

19. The Laboratory hood fans are to operate continuously. Do not attempt to run analyses unless the fans are in service.

20. Always put liquid organics into tanks through stand legs or through nozzles in the bottom of the vessels. Falling liquid can generate static electricity.

H. Cleaning of Tanks and Process Vessels

The area supervisor and maintenance supervisor will see that all vessels or tanks are cleaned and checked with an explosion meter before declaring them suitable for maintenance.

The recommended cleaning procedure is:

1. The tank or vessel will be emptied and all valves will be closed and tagged.

2. The vapor contents of the tank will be purged with nitrogen.

3. Blinds will be inserted in all connecting lines.

4. The equipment will be steam purged where possible to vaporize and remove all flammable materials. If steam cannot be utilized, nitrogen will be used.

5. Purge the equipment with plenty of air.

6. The equipment will then be checked with an explosion meter before work is begun.

7. Safety belts and safety lines will be required in top-entry tanks.

8. A sufficient oxygen check will be made before man-entry of any vessel, whether purged with N_2 or not.

I. AREA B - SAFETY PERMIT FORM

CONFIDENTIAL:
Subject to Protective Order
of 14th Judicial District Court
No. 91-1145

43A-STD-4-66
(Rev. 3-7)

AREA B SAFETY PERMIT

To be used to pass restricted equipment into Area B and to be endorsed for the use of such equipment in the

________________________________ Unit.

To: ____________________ Date: ____________________

Location: ____________________ Equipment: ____________________

Please Check	Yes	No
1. Have workmen been provided with proper safety equipment?		
2. Are adjacent areas and equipment safe?		
3. Has adequate fire protection been provided?		
4. Is presence of operator required?		

Statement: I have personally checked the area covered by this permit and find it safe for the job described.

Signed: ____________________ ____________________
Production Supervisor or Lead Operator(routine vehicle entry only)

If a siren or emergency horn sounds, I am to turn off my equipment immediately and evacuate the area on foot.

____
Employee's Signature

WELDING OR BURNING PERMIT

This permit, when signed, allows only necessary welding or burning at:

____
(Location)

It is good for: Date ____________ Time ____________ AM PM

To:(Max.4 hrs.)Date ____________ T ne ____________ AM PM

Special Comments: ____________________

Statement: I have personally checked the area covered by this permit and find it safe for the job described.

Signed: ____________________ ____________________
Area Production Supervisor Maintenance Supervisor

J. Fire Protection

Fire protection in the area is provided in three forms--water, dry chemicals, and CO_2. The dry chemical is the most effective way of fighting an EDC fire. Water is good to partially contain the fumes and to keep the vessels and structural steel cool. The CO_2 cylinders are only good for small fires, primarily small electrical fires. CO_2 cylinders are located in the switchgear room. Thirty-pound dry chemical units are located throughout the area. A large portable unit is available at the perimeter of the process area and a truck unit serves the whole organic area.

The main water protection is a manual or automatic sprinkling device, tripped by a deluge valve. The process area and the cooling tower are each protected with their own deluge system. Heat Activated Devices (HAD's) are located throughout each area and a sudden rise in the HAD temperature will cause the deluge valve to open and the respective area to be covered with a water spray. The process area sprinklers may also be tripped manually from behind the control board or at the Automatic Sprinkler deluge valve building. You are encouraged to manually trip the sprinklers if there is a large leak, spill, or other condition that would cause a dangerous situation, even though there may not yet be a fire. The deluge valve must be manually reset each time it is used before further automatic protection is available.

There also will be a hose house and two water hydrants due south of the main area. In addition, there are hydrants near the catalyst storage building and SE of the area. The Per-Tri hose and hydrant house will serve the north side of the OHC Plant.

Fire Extinguishers:

1. In control roon near lab, one 15# CO_2.
2. Switch room upstairs of control building, one 15# CO_2.
3. Catalyst storage building, one 15# CO_2, one A-30-D 30# Ansul.
4. Ground floor near stairwell--one A-30-D 30# Ansul.
5. Second floor near stairwell--one A-30-D 30# Ansul.
6. Third floor near stairwell--one A-30-D 30# Ansul.
7. Fourth floor near stairwell--one A-30-D 30# Ansul.
8. South of process pad near crude storage tanks, one 125# A-150-B Ansul.

K. Warning Devices

A control room switch will activate a "clear the area" siren. This should be used initially to clear all personnel from the upper decks and personnel not required for operation completely from the OHC area. If an evaluation of the situation shows that no people should be in the area, allow the siren to operate continuously. If there is no danger to life and limb, the operating personnel should remain, however, to regain control of the plant. Remember that an impending disaster (a bad ethylene leak, for an example) would be sufficient to clear the area.

If the disaster, or impending disaster, is great enough to affect all of Area B, call the Shift Engineer at 445 and/or the EDC control room at 441 so that the Area B alarm can be actuated in the EDC control room. The plant guard at 328 can also sound the Area B alarm at EDC.

If Area A would also be affected call the plant guard at 328 or the shift supervisor at 476 for sounding the alarm for the whole plant.

L. Emergency Area Exits

East - Main truck gate.

North - 1) Emergency gate just east of the ethylene meter station.

2) Locked gate just north east of the EC-VC control building.

Key is in EC-VC control room.

South - Emergency gate south of the maintenance building.

West - Emergency gate west of the VC tank car loading station.

M. Gas Masks

In the OHC and adjacent areas, there are several gases that could necessitate the use of gas masks. Chlorine, hydrogen chloride, ammonia, and organic vapors are the most likely gases to escape. Since the York unit is charged with Freon, the possibility of this gas escaping also exists. Dowtherm is the only other gas that could be released into the area.

Following is a list showing the type, location, and number of gas masks for the OHC Plant:

1. Scott Air Packs (30 minute units)

   a) Control room, two (2) with wall case.

   b) Foreman's office, one (1) with wall case.

2. Chemox

   Control Room, two (2) stored in special cabinet.

3. Full-face all purpose

   a) Control room, six (6) stored in special cabinet.

   b) Process area, 2nd deck near stairwell, one (1) stored in special cabinet.

   c) Process area, 3rd deck near stairwell, one (1) stored in special cabinet.

d) Process area, 4th deck near stairwell, one (1) stored in special cabinet.

e) Catalyst building, one (1) stored in special cabinet.

4. Cylinder type with connecting regulator, hose, and mask

Control room, two (2) complete units, located behind the panel board, one on each side. One is mounted on wheels.

Operators are expected to know and remain familiar with the locations of the various types of masks. Also, complete familiarity with the various masks and the proper technique for their use is an absolute must.

The following is a run-down on the type situations for which each of the masks will be used:

1. Scott Air Pack - This unit is good for any concentration of gas and can be used for periods up to thirty (30) minutes. The unit can be put on in a few seconds, and because of this, its primary use is for major gas breaks requiring quick response to get valves closed or injured men out of contaminated areas. The point to remember with this unit is that it has a life of only 30 minutes and when using it one should not remain in a contaminated area for a period of more than 20 minutes.

2. Chemox - This unit is good for any concentration of gas and can be used for periods up to one hour. It is a unit which chemically generates breathing oxygen and because of this, several minutes are required to get this unit operating properly. The primary advantage of this unit is its long life. Because of this, the unit should be used in cases where one is required to enter and remain in a contaminated area for longer periods. With this unit one should not remain in a contaminated area for more than 45 minutes.

3. Full-face All Purpose - This unit is good for weak concentrations of gas. One can equip himself with this mask in a few seconds. It should be used only in weakly contaminated area and for escape purposes.

4. Cylinder type with connecting regulator, hose and mask - These units are good in any concentration of gas. They are to remain in the control room and are to be used in the event of gas being pulled into the control room. The important point about using these units is to get the units on before the room becomes excessively contaminated. Since the control room is pressurized, contaminated gases can enter only through the intake of the pressurizing unit. When contamination is detected, the pressurizing unit should be shut down and instrument air bleeds behind the panel board and in the laboratory should be opened.

IV. DETAILED PROCESS AND EQUIPMENT DESCRIPTION

A. Raw Materials, Feeds

The raw materials for the OHC process (HCl, ethylene and oxygen) are fed to the plant from different suppliers.

1. Hydrogen Chloride: The HCl is sent to the OHC plant from the vinyl chloride area via three available reciprocating compressors located at the HCl Plant compressor area. The compressor feed comes from the vinyl chloride plant direct at 40 psig or from the same supply after a pressure reduction to 20 psig (this pressure reduction is for use in HCl distribution only). When the OHC plant operates at full capacity, 40 psig suction is required. Two of the three compressors (numbers 6, 7 and 8) are required for both OHC reactors operating at full rates. The third compressor (No. 6) is a spare; however, this compressor also acts as a spare on the supply to Ancon and may not be available. Communications between HCl and OHC are essential as to the status and availability of this compressor. The power supply for these compressors comes from the HCl feeder, not OHC's. It should be understood by all concerned that prime consideration should be given to maintaining an HCl flow to the OHC plant.

The total HCl flow, with temperature and pressure corrections, is metered as it enters the OHC plant. An alarm sounds if the pressure drops below 70 psig (normal is 75 psig). The incoming line contains a large diameter tank containing "Hi-Sil" pellets, which removes trace quantities of undesirable materials (oil, organics, etc.). Any carryover Hi-Sil will be trapped in a Ful-flo cartridge-type safety filter. This filter should be put into service slowly so as to prevent plugging and

Hi-Sil carryover. The pressure drop across the filters should be routinely observed.

2. Ethylene: Ethylene is supplied to PPG by PCI at a high pressure (about 600 psig). The ethylene is filtered, heated, and reduced in pressure at the ethylene control station north of the No. 1 EDC plant. The ethylene for the OHC plant is taken off the 175 psig header by the EDC cooling towers, and is metered to OHC by a mass flowmeter located at the OHC battery limits. The flow indicated by the mass flowmeter will be used to charge ethylene usage by OHC and should be read at 7:00 a.m. each morning. The ethylene pressure is further reduced to 95 psig before being metered again. This flow will be continuously recorded on the OHC panel board.

3. Oxygen and Nitrogen: Two companies supply both oxygen and nitrogen: Big-3 and Gulf Oxygen. Since Big-3 gets preference up to its contract limitations, all first gas comes from them until which time a pre-set flow control valve throttles their flow. At this time, if higher O_2 and N_2 use continues, the main header pressure drops. This causes Gulf Oxygen's control valve to open so that the common header pressure is maintained. It would be best for Gulf Oxygen if we do not operate right at this break-point. If this case occurs, a foreman will have to decide between; 1) deliberately increasing or decreasing the O_2 or N_2 rate, or 2) decreasing the consumption (lower the set-point) from Big-3.

Low header pressure for either gas will sound an alarm in both the Per-Tri and OHC control rooms. A high nitrogen draw from Big-3 will sound an alarm at Per-Tri only. A high nitrogen draw from Gulf Oxygen will sound an alarm at OHC only. A four-way direct telephone line will be installed between Big-3, Gulf Oxygen, the Per-Tri Plant, and the OHC Plant to allow for instant communication to all concerned with O_2 or N_2.

Both Big-3 and Gulf Oxygen maintain back-up supplies of liquid gases that can be vaporized and fed to us should their main supplies fail; therefore, complete loss of either gas is unlikely. Their back-up supply is limited, however, so you must take appropriate action regarding cutting back or shutting down, depending on what their difficulties are.

Whenever a drastic N_2 or O_2 change is to be made (up or down) inform Per-Tri, Big-3, and Gulf Oxygen. The supplier controls are such that if you "upset" them too rapidly, the response of the instruments may overshoot and cause a complete shutdown.

The oxygen pressure is reduced to 80 psig and metered by an orifice to the plant. The nitrogen is used at the header pressure; the total consumption by OHC is continuously metered and recorded.

B. Preheating, Mixing

Each reactor has a separate feed system. The ethylene, oxygen, and HCl for each reactor are heated in individual spiral heat exchangers. Each stream is heated to 250°F with 30 psig steam; the steam is supplied through a temperature control valve, which is controlled by the process outlet temperature. The unreacted gases which pass through the reactor condensing system are compressed, heated to 250°F in a spiral heat exchanger, and recycled to the reactor feed system. The HCl and ethylene are premixed in a mixing jet. The recycle gas is then premixed with the ethylene-HCl mixture in another mixing jet. The resulting ethylene-HCl-recycle mixture is known as "mixed feed" and is fed into the bottom head of the reactor through two inlets. Oxygen is fed into an internal oxygen manifold in the bottom reactor head through a separate feed inlet. The oxygen is not premixed in the mixed feed because an uncontrolled reaction could occur if the streams come in contact before reaching the fluidized catalyst bed. For this reason, the feed system must be thoroughly purged with nitrogen before addition of the feeds. Each of the feed streams is metered through orifice runs. These flows are automatically calculated and recorded as standard cubic feed per hour based on a common pressure and temperature (STP).

C. Reactors

Each reactor consists of a vertical vessel 85.5 inches inside diameter with a straight side length of 40 feet. The entire reactor assembly is constructed from Inconel Alloy 600. The bottom head of the reactor serves as the feed manifold. Mixed feed enters the head directly through two 4" nozzles on the bottom of the head. Each nozzle has an 8" diameter impingement baffle immediately above the inlet. The mixed feed is distributed across the reactor by a 3 3/4" thick distribution plate fitted with 353 mixed feed nozzles. The 3/8" inch i.d. nozzles are mounted on a 3" x 2 1/2" pitch. The orifice opening in the bottom of each nozzle is 1/8". The oxygen enters the bottom reactor head through a 3" pipe which forms a single pipe manifold across the head about 5 1/2" below the distributor plate. Twenty 2" nozzles connect the main 3" manifold to twenty 2" half-pipes which are welded to the bottom of the distributor plate. The oxygen is distributed into the reactor by 150 oxygen feed nozzles which sparge the oxygen into the reactor 19" above the distributor plate (just above the 180° ells of the cooling tubes). Each nozzle consists of a 1/2" schedule 80 pipe with a 1/16" orifice opening. The mixed feed and oxygen nozzles are designed so that 25% of the total pressure drop across the reactor will occur across the distributor plate and feed nozzles; this pressure drop is necessary in order to insure that the feeds are distributed evenly across the reactor.

The OHC reaction is catalyzed by a 7.5% copper-5.3% potassium catalyst. The copper and potassium compounds are applied as a coating to a clay carrier. The catalyst is fluidized in the reactor by the reacting gases. A superficial fluid velocity of 0.8 feet/sec is required in the reactor at the height of the outlet of the oxygen nozzles in order to maintain fluidization with a minimum catalyst carry-over. This velocity must be maintained at all production rates. Loss of fluidization would result in plugging of the feed orifices and require complete reactor shutdown, catalyst removal, cleaning, and recharging. Since varying production rates require varying feed rates, the velocity must be maintained constant by changing the reactor conditions so as to maintain a constant volume flow of gases in the reactor. This is accomplished by changing the reactor pressure (temperature will be essentially constant) with varying production rates. Thus, the reactor pressure will be decreased as the production rate is decreased. Each reactor is designed to operate at 46 psig at full rates (156 TPD crude EDC per reactor). The pressure required for corresponding production rates is shown in Figure 10-1. The feed rates required for varying reactor pressures are given in Table 10-1 It should be noted that the feed rates for zero, one, and two psig operating rates are the same. This is a result of the requirement that a minimum of 25% of the reactor pressure drop occur across the distributor. Otherwise, at flow rates lower than 2 psig rates, this criteria would not be met.

The Deacon and oxyhydrochlorination reactions are highly exothermic (give off heat) and make heat removal from the reactor necessary. This is accomplished by circulating liquid Dowtherm A through 16 parallel banks of 2" pipe hairpin loops suspended in the fluidized bed. The pipe loops are suspended from a level 25 feet above the distributor and extended down to 1 1/2' from the distributor plate. The reactor temperature is controlled at 525°F by varying the inlet Dowtherm temperature (with constant Dowtherm flow rates for all production rates). The hot liquid Dowtherm is circulated to a Dowtherm cooler. The cooler is a thermosiphon-type reboiler with the Dowtherm acting as the heat source in the shell while steam is formed in the tubes. The steam passes overhead from the Dowtherm cooler into a steam drum. Condensate at about 150°F is fed to the steam drum through a level control valve. The steam generated is discharged from the steam drum through a pressure control valve at a rate sufficient to maintain 35 psig on the steam drum. The outlet Dowtherm temperature from the cooling system is maintained at the desired set point by automatically bypassing Dowtherm around the cooler at the rate necessary to maintain this temperature. The Dowtherm then flows to Dowtherm circulation pumps (which also can pull from a Dowtherm surge tank), which pump the Dowtherm to the reactor. These Dowtherm pumps are jacketed and cooled by cooling tower water. The seal flush on these pumps is cooled and filtered before entering the seal gland.

D. Reaction Mechanism

Several variables enter into the performance characteristics of an OHC reactor. The basic reactions are:

1. The Deacon reaction:

$$2\ HCl + 1/2\ O_2 \longrightarrow Cl_2 + H_2O$$

2. The subsequent chlorination reaction:

$$C_2H_4 + Cl_2 \longrightarrow C_2H_4Cl_2\ (EDC)$$

Reaction "1" must be going before chlorine is available to give reaction "2". Other, undesirable reactions also occur:

3. "Burning" - complete:

$$C_2H_4 + 3\ O_2 \longrightarrow 2\ CO_2 + 2\ H_2O$$

"Burning" - partial:

$$C_2H_4 + 2\ O_2 \longrightarrow 2\ CO + 2\ H_2O$$

Burning of EDC may also occur:

$$C_2H_4Cl_2 + 5/2\ O_2 \longrightarrow 2\ CO_2 + H_2O + 2\ HCl$$

4. EDC chlorination:

$$C_2H_4Cl_2 + Cl_2 \longrightarrow 1,1,2\ C_2H_3Cl_3\ (TCE) + HCl$$

5. Formation of carbon tetrachloride is another undesirable side reaction. This is formed by too large an excess of oxygen in the presence of the organic within the reactor.

Our process provides for recycling of unreacted ethylene and oxygen, but not HCl. Thus, any HCl passing through the reactor unreacted is lost, which is, of course, undesirable. The Deacon reaction follows the basic law of chemistry which states that any excess of reacting components (those on the left hand side of the equation) will tend to drive the reaction of the remaining reacting components to completion. Thus, if an excess of oxygen is fed to the reactor with respect to the HCl, the HCl will tend to be more completely reacted. If very large excesses of O_2 are fed, essentially all of the HCl will be used up; however, the formation of CCl_4 then becomes excessive. Thus, a balance must be made between HCl loss due to incomplete reaction and C_2H_4 loss due to CCl_4 formation. Our plant design calls for 11.5% (on a mole or volume basis) excess oxygen (above theoretical for Deacon reaction) in the feeds when recycling gas to the reactor. During the initial startup, a lower excess will be used to establish a more firm knowledge of the reactor.

The amount of TCE formed by reaction "4" is controlled by the reactor temperature, the amount of EDC in the recycle, and the percent ethylene conversion per pass. It is expected that at a reactor temperature of 525°F the TCE content in the reactor crude material will run approximately 1.28%. Increasing the temperature would result in higher TCE formation. Too low a reactor temperature, however, will lower the reactor's capacity due to the decreased cooling capacity. The refrigerated reactor condenser should lower the reactor vent temperature sufficiently to insure that our recycle EDC content is low. By controlling percent ethylene in the recycle at a relatively high value, a lower recycle gas flow will result for a specified percent conversion. The smaller volume of recycle gas results in improved condenser efficiency so that the amount of EDC in the recycle is decreased, thus lowering the rate of TCE formation.

Chloral (CCl_3CHO), carbon tetrachloride, and chloroform will be the largest organic impurities other than TCE. The calculated reactor off-gas composition (with recycle, 10% excess O_2 and 75% ethylene conversion per pass) is given in Table 4-1. This analysis, however, will rarely, if ever, be made.

Table 4-1
Reactor Off-Gas Composition

Component	Mole %
O_2	1.64
N_2	0.38
CO	0.49
CO_2	9.46
C_2H_6	2.27
C_2H_4	12.38
HCl	0.92
C_2H_3Cl	0.01
$CHCl_3$	0.03
1,2 EDC	36.12
CCl_4	0.03
CCl_3CHO	0.13
1,1,2 $C_2H_3Cl_3$	0.33
H_2O	35.80

The OHC-EDC reactor is designed for a conversion of 75% of the ethylene per pass through the reactor. Unreacted ethylene is recycled back to the reactor. Pilot plant studies showed that the catalyst bed height in relation to reactor diameter had great effect on the actual ethylene conversion necessary to achieve a desired yield and product purity. Since our catalyst bed level is fixed at a point slightly above the Dowtherm tubes, only a short time will be required to determine the actual conversion per pass which can be achieved in the full scale reactor.

E. Catalyst Removal System

The top 15 feet of the reactor is open space in which most of the catalyst particles are separated from the reactor gases. These gases then pass overhead into a centrifix system where the remaining catalyst is knocked out. The centrifix is a 2 feet diameter vertical vessel 9 feet high. The centrifix removes catalyst particles from the reactor overhead gases by centrifugal action which is imparted to the gases by a tuyere in the top. This tuyere is a set of vanes formed into a cone. The lower section of the centrifix is steam jacketed to prevent condensate from forming, with subsequent corrosion taking place.

Immediately below the centrifix is located a catalyst collection drum which is used in the removal of catalyst from the system. It is a cone-bottomed steam jacketed vessel, and is separated from the centrifix by a rod-out valve.

The frequency of dumping the catalyst catch pot will have to be determined after operation has started. Initially, it will be twice per shift. Due to the materials involved, the catch pot blowdown operation must be carefully done. The gases in the catch pot will be flammable upon air exposure; also, the blowdown of entrained catalyst particles can set up static electricity generation. The procedure for removing this catalyst will be discussed in the Special Procedures Section.

F. Condensing System

The organics and water in the reactor off-gases are condensed in a series of four graphite heat exchangers. Each reactor has a separate series of condensers and associated vessels. The first (primary) condenser is a vertical up-flow shell and tube exchanger with cooling tower water in the shell. All surfaces in contact with the organic are graphite, while the rest is steel. ~~The cooling water flow rate is controlled with a manual loading control valve so as to maintain an outlet gas temperature of about 225°F (this temperature will vary directly with system pressure).~~ Graphite cannot withstand temperatures above 338°F;

therefore, care must be taken to prevent damage to the exchangers from overtemperature. The top tube sheet of the primary exchanger is designed for cooling the graphite by a water flush distribution down through all the tubes. This is accomplished by threading a 3" Teflon weir tube into each graphite tube. High pressure well water is injected onto the top tubesheet, overflows the weir tube, and runs down through the tubes. This well water flow is remotely controlled by a manual loading station; however, if the gas temperature in the bottom head exceeds 280°F, the remote valve is automatically opened wide open. The bottom tee under the primary condenser, while not made of graphite, is lined with Fluorogreen. This material is also heat sensitive and needs the water flush whenever there is too little condensate falling out of the primary. The water flush also serves to remove tube wall catalyst deposits.

The secondary condenser is a vertical downflow exchanger very similar in construction to the primary condenser. A similar well water flush system is available on the top head. The tertiary (third) condenser is also a vertical downflow exchanger, but does not have provision for well water flush. The secondary and tertiary condensers are cooled by cooling water, but do not have automatic temperature control on outlet gases.

The fourth condenser is a downflow exchanger with refrigerated calcium chloride brine on the shell side. The brine flow is automatically controlled to give an outlet gas temperature of 60°F; however, it is desirable to lower this temperature as much as possible.

The refrigerated condenser outlet gas is essentially free of EDC, but contains both ethylene and oxygen. The gas flows to an entrainment separator where the entrained liquid is removed. Most of this gas goes to the recycle system; however, part of the gas is vented through a stack scrubber. The amount of vented gas is controlled automatically so as to maintain a set-point back pressure on the reactor. This is the pressure which is adjusted with varying production rates so as to maintain the 0.8 feet/sec superficial gas velocity in the reactor. A differential pressure switch is provided to indicate when the pressure drop across the centrifix and condensers is excessive. An alarm from this switch would probably indicate an excessive pressure drop across either the centrifix or the primary condenser due to catalyst carryover.

G. Recycle Scrubbing

The gas from the condensing system contains ethylene, ethane, oxygen, nitrogen, CO_2, CO, and a small amount of HCl. The actual composition will depend on reactor operation. The normal HCl content should be low; however, this HCl must be removed because of the materials of construction in the recycle system. This is accomplished in a recycle gas scrubber (one for each reactor). The scrubbers are porcelain-packed Haveg 61 towers, through which a $NaHCO_3$-NaOH solution is pumped to neutralize HCl.

The major reactions are as follows:

1. $NaOH + CO_2 \longrightarrow NaHCO_3$
2. $NaHCO_3 + HCl \longrightarrow CO_2 + H_2O + NaCl$

Other side reactions will take place also, but they should be comparatively minor. The first reaction will be the major one during initial start-up. However, it takes place merely to replace the depleted bicarbonate ($NaHCO_3$) after that. CO_2 from a cylinder will be used to make the initial $NaHCO_3$ solution to avoid upsetting the recycle system. Operation of the system will be tested after startup to determine the exact ratios necessary. The caustic make-up to the system must be at a rate sufficient to insure neutralization of all of the HCl; too much caustic make-up, however, would be an unnecessary operating expense.

The effluent $NaHCO_3$-NaOH aqueous solution flows from the scrubber through a pH meter into a surge tank. The bicarbonate solution is pumped from the surge tank back to the scrubber. Make-up condensate (cooled) and cell liquor are added to the scrubber feed stream. The cell liquor make-up rate must be manually adjusted to obtain the proper (to be determined after startup) effluent pH. The purpose of the cooled condensate make-up is to keep the solution concentration below the NaCl saturation point and thus prevent "salting out" in the scrubber packing. The condensate make-up rate should initially be about the same as the cell liquor make-up. The scrubber is also equipped with an alternate cell liquor source which automatically opens if the effluent pH drops below 5.0. This is necessary to neutralize HCl surges which occur with reactor upsets.

The scrubber is provided with a pressure drop transmitter so as to indicate when plugging of the packing occurs. Proper make-up of cooled condensate should prevent this plugging; however, should plugging occur the scrubber may be bypassed for a short time and unplugged with high condensate flow. Bypassing of the scrubber means that HCl could be sent into the recycle system. This must be prevented or limited because of the corrosive effect of HCl in the recycle system; therefore, do this only when the reactor is lined out with a high HCl utilization.

A side stream is taken off the $NaHCO_3$ pump discharge at a rate sufficient to maintain a constant level in the $NaHCO_3$ surge tank. This aqueous stream will contain dissolved C_2H_4 (absorbed in the scrubber). The C_2H_4 is flashed off to a stack in the C_2H_4 degassing tank before being sewered. The degassing tank has on N_2 purge flow to it; this N_2 flow must be maintained at all times. The vent from this tank is equipped with snuffing steam for fire extinguishing.

The recycle gas leaving the recycle scrubber goes to a recycle compressor system. Carryover liquid from the scrubber is removed in an entrainment separator. This separator is a vertical vessel 10 3/4" o.d. and 3'10" long with a demister in the top. The liquid is drained back to the bicarbonate surge tank.

H. Recycle Compression:

Three specially-constructed reciprocating compressors are available to compress the recycle gas. The No. 2 compressor serves as a spare for the other compressors. The compressors are designed to compress 300 cu. ft/min of the recycle gas from 70°F and 44 psig (full rate suction conditions) to 80 psig, or 281 cu. ft/min at 70°F and 0 psig (atmospheric rate suction conditions) to 36 psig. The compressor has a 12" bore with a 9" stroke and turns over at 300 rpm. Power is provided by a 60 hp, 1775 rpm motor with a v-belt drive to the compressor. The compressors are designed to compress the gas completely free of oil. The cylinders are the non-lubricated type with Teflon piston rings and Teflon wear rings. The valves are all stainless steel, the piston is steel, and the cylinder liner is Ni-resist. The cylinder head is jacketed and cooled with cooling tower water.

The cylinder head is separated from the crankcase by a double gas-tight distance piece. Nitrogen is metered through a rotameter into the distance piece closest to the cylinder head, and lines are available to put nitrogen into the compressor suction and the other distance piece. The nitrogen will be vented off the bottom of the front distance piece; the vent rate and the N_2 purge rate must be manually adjusted so as to maintain about 5-10 psig greater than discharge pressure on the distance piece. This will prevent an air buildup in the distance piece and possible flammable mixtures of air and recycle gas in the distance piece of the compressor. It also insures that any leakage through the shaft packing will be <u>into</u> the compressor. As a secondary precaution, a slow nitrogen sweep should be kept flowing through the rear distance piece.

The crankshaft bearings are lubricated by an oil dipper type system. The main bearing, crosshead guide, and crosshead bearing are lubricated by oil vapors and residual oil splashing. It is essential that the proper oil level be maintained in the compressors at all times.

The compressor valves are the spring loaded disc type. The springs are designed to deliver a minimum 36 psi pressure increase across the compressor.

The recycle gas from each reactor is compressed and flows to a recycle surge tank (one for each reactor). All equipment from the recycle gas scrubber back to the reactor are constructed of stainless steel to protect against corrosion from moist CO_2. A stream is taken off the top of the surge tank and circulated through a recycle cooler-entrainment separator combination back to the compressor suction. The recycle cooler is a spiral heat exchanger with well water as the coolant. The separator is an expansion chamber 30 inches diameter by 7 feet high with a demister over the vapor outlet.

The amount of gas circulated back to the compressor is automatically controlled so as to maintain a constant set-point discharge pressure. The set point will be adjusted by the operator at a point high enough to insure steady feed back to the reactor. The recycle gas passes through a steam-heated spiral superheater before being flow controlled back to the reactor. The steam rate to the superheater is automatically controlled to maintain the recycle gas at 250°F.

The point should be made that the recycle compressors do not have internal valve unloaders. The compressor is a constant displacement machine in which the pressure control is accomplished by varying the amount of artificial load being applied to the compressor. This artificial load is obtained by a pressure control valve which maintains a constant discharge pressure by recirculating a portion of the gas back to the suction. Abnormally high discharge pressures (above 100 psig) are controlled by individual safety relief valves from the discharge of the compressor back to the suction. This safety relief valve system is for emergency discharge pressure relief only; if the valve operates, corrective action must be taken at once because this vented gas is not cooled and will cause both overpressuring of the system and overheating of the compressor.

The inlet and outlet valves for the spare recycle compressor are equipped with alarms to indicate when improper valve positions occur. The compressor should be operated with suction and discharge on the same reactor recycle system. The alarms sound if the valves allow interchange of recycle gases between the two reactors.

I. Recycle Gas Analysis

The recycle gas streams are perhaps the most hazardous streams in the OHC plant. The expected composition is as follows:

Table 4-2
Recycle Gas Composition

Component		Mole %
O_2	32	5.71
N_2	28	1.28
CO	28	1.72
CO_2	44	32.78
C_2H_6	30	7.98
C_2H_4	28	43.25
EDC		6.17
H_2O		1.11

Most of the condensible components have been removed in the condensing system and thus the oxygen and combustible components are concentrated in the vent streams. The <u>most important operating point</u> to remember in the OHC plant is to keep this stream out of the explosive range. The explosive curve is presented in Figure 10-3 (appended). This curve is based on ambient conditions. Higher pressures and temperatures decrease the oxygen content required for the explosive range. The guide to be used as to explosibility of this stream is the oxygen content. We will consider the maximum O_2 content as 8 to 9%, which is slightly below the actual maximum as dictated by the explosive curve. This safety margin will give the operator time to correct the cause of high O_2 before reaching the danger point.

The O_2 content of the recycle gas will be continuously analyzed by a Hays Model 632C-II Oxygen Analyzer. The sample is taken off the line downstream of the recycle scrubber entrainment separator. The sample pressure at the analyzer is maintained at 7 inches of water by a diaphragm type pressure regulator. The O_2 recorder is provided with an alarm if the O_2 content exceeds 9%. The recorded O_2 concentration should be closely followed by the operator, and extra caution taken to insure that the oxygen content is well within the safe range.

The more complete analysis of the recycle gas will be determined by a continuous gas chromatograph. The G.C. analyses for ethane, CO_2, ethylene, oxygen, nitrogen and carbon monoxide in that order. The analysis will be in volume percent and be on $HCl-H_2O$-EDC free basis. Samples will be analyzed every 30 minutes. The primary function of the G.C. will be in reactor feeds control; however, the results of this instrument will serve as a check on the oxygen analyzer.

As a further check on the content of the recycle gas stream, the operator will be required to run Fischer Gas Partitioner or Orsat analyses periodically. The normal frequency of these tests will be determined by the foreman. In times at which the O_2 analyzer and/or G.C. is out of order, these alternate tests will be run quite frequently. This is necessary to keep tabs on the recycle stream. Any failure of or discrepancies between the three methods of recycle gas analysis must be reported to the foreman <u>at that time</u>. During the night shifts this should be reported to the Area "B" Shift Engineer.

J. Condensed Crude Processing

The condensed organics and water from all the reactor condensers flows to a Haveg Degasser tank (one for each reactor-condenser system). Some dissolved gases come out of the solution in this tank and are equalized into the inlet of the reactor refrigerated condenser. Condensed crude flows from the degasser to the flasher through a level control valve at a rate sufficient to maintain a constant level in the degasser. The flasher is maintained at a low pressure (1-2 psig) so that dissolved ethylene, CO_2, and other gases are flashed off. These gases flow to the refrigerated vent condenser and then to the stack scrubber.

The liquid from the flasher flows to a glass-lined steel phase separator. The organics are separated from the water phase in this vessel. The upper aqueous HCl layer overflows to the sewer and the lower crude EDC layer is level-controlled off to the intermediate crude storage tanks. The interface level is sensed by an interface level transmitter. Fine catalyst dust will tend to build up at the organic-aqueous interface and/or the bottom of both the phase separators and the intermediate crude tanks. Draw-off valves are provided on the vessels and catalyst build-up should be flushed out routinely. In addition, an aqueous layer and catalyst dust layer will slowly accumulate in the top of the intermediate crude tanks and should be routinely decanted off.

K. Detailed Reactor-Condenser-Recycle Operation

The operation of the reactor-condenser-recycle system should be considered all together because of the close interrelation of variables involved. Any changes in the reactor feed rates will cause changes in the recycle gas composition. The condensing system must be properly operated in order to help maintain reactor EDC purity.

The operating rate of the OHC plant will be fixed by the HCl available from the vinyl chloride plant. The HCl flow to the OHC plant will be maintained constant with the exception of major rate changes; that is, OHC has first chance at the HCl produced. Other users will absorb any routine

HCl fluctuations. This approach is necessary because of the fluidization and recycle gas composition problems inherent in the OHC process.

As mentioned in the "Reaction Mechanism" section, the O_2 to HCl ratio must be controlled with enough O_2 excess to insure good HCl conversion, but not so high as to give high CCl_4 formation. The exact ratios to be used will be worked out during the initial operation of the reactor. The following guides will be available to determine the feed ratios required and should be thoroughly learned by operating people:

1. HCl: A minimum HCl utilization of 98.5% should be expected. Lower HCl utilizations can be compensated for by increasing the O_2:HCl ratio (assuming O_2 content in the system is within the above described limits). HCl yields also suffer at higher ethylene conversions. The HCl conversion is determined by analyzing the acidity in the condensed aqueous phase. See Fig.10-4 for this relationship. A high HCl utilization is very important to the economics of this process because an overall low chloride cost is thus obtained.

2. Oxygen: The upper limit on O_2 to HCl ratio must be based on the amount of carbon tetrachloride formed (pilot plant studies indicated increased CCl_4 formation with high O_2:HCl ratios).

The CCl_4 composition in the condensed EDC crude should be about 0.2%. CCl_4 is an undesirable impurity in the EDC crude because if forms an azeotrope with EDC and complicates later purification of EDC. Again, operating experience will be necessary to determine the acceptable levels more closely. In general, high burning or CCl_4 content will require a cut in oxygen flow. Maldistribution of feeds may also have an excess oxygen effect and give a high CCl_4 content in the reactor vent.

3. Ethylene: The ethylene feed rate will be mainly dependent upon HCl feed rate, utilization, ethylene in the recycle stream, and vent purge rate. At fixed HCl feed rate and utilization, the burning should be relatively constant. Thus, the CO_2 and CO inert load should be fixed. These inerts are purged from the system to a refrigerated condenser and vented to the atmosphere through a scrubber at a rate just sufficient to keep the reactor system pressure constant. Since this vent stream is simply a side stream off the recycle gas system, it must carry ethylene and the other recycle gas components with it. Therefore, the ethylene feed rate is controlled at a rate which maintains a satisfactory recycle composition. It should be apparent at this point that high inerts in the feed (ethane, nitrogen, etc.) require a high vent rate and subsequent high ethylene loss.

Another inert, ethane, is present in this stream. The amount of ethane present in the ethylene feed varies and will require close monitoring. The ethane is blended into the ethylene at PCl. At the present time, the contract calls for a minimum ethylene concentration of 98.5%; however, the existing PCI ethane blending compressor does not have sufficient capacity to meet this specification. The actual ethane content will vary with total ethylene demand from PCI and with the ethane compressor maintenance. PCl informs the EDC control room of major changes in blending. Good communication should be maintained between the EDC and OHC control rooms.

Still another inert load exists in the nitrogen purge rates to the reactor head, top pressure transmitters, and other sources. These purges should be maintained at minimum, since any additional increase in inert loading results in more ethylene loss.

A further consideration in C_2H_4 feed rate lies in the actual composition of the recycle stream. The critical importance of maintaining a non-explosive gas mixture will override any economic consideration. In other words, if the O_2 content of the recycle gas is near the 8-9% level, do not hesitate to add ethylene to move away from this range (in addition to a decrease in oxygen). The response of the percent oxygen in the recycle is much greater due to an ethylene increase than an oxygen decrease.

4. Recycle: The composition of the recycle stream must be kept in the non-explosive range. In addition, it is an important indication of the reactor operation; the recycle stream composition is therefore closely followed. The recycle analyses, vent purge rate, crude product composition and HCl utilizations will normally give enough information to specify any feed adjustments to the reactor. Conversely, operation at design recycle composition, purge rate, and HCl utilizations is a strong indication that the feeds are properly balanced.

The design recycle ethylene content is approximately 50%. Thus the ethylene feed should be adjusted to allow the ethylene content to line out at this value. If the ethylene drops below this value enough excess ethylene will not be present in the reactor to allow good HCl utilizations and high purity product. Too little ethylene feed will cause the ethylene to be consumed from the recycle stream. A decrease in ethylene in the recycle combined with a decreased purge means that too little ethylene is being fed. A slight increase in ethylene feed will correct this situation. An increasing ethylene content in the recycle with an above design purge means that the ethylene feed is too high, and too much ethylene is being lost.

It is possible that the ethylene concentration will change because of other reasons. A sudden increase in the inert loading will cause the ethylene composition to decrease. This will be associated with an increased purge. Thus a decreasing ethylene concentration with an increasing purge means that some other non-reactive gas is entering the system. Inerts addition was discussed in the ethylene section.

If too much caustic is added to the recycle scrubber, the CO_2 will be adsorbed from the reaction system. Both ethylene and O_2 will increase, CO_2 will decrease, and the purge rate will decrease. The pH in the recycle scrubber will also increase. This would be corrected by reducing the caustic makeup. Immediate action such as increasing ethylene should also be taken to prevent the O_2 concentration from reaching the explosive limit.

The oxygen concentration must be closely watched for safety as well as process control reasons. It is stressed again that no feed adjustment should be made that will bring the oxygen content of the recycle into the explosive range. Oxygen content can be quickly reduced by increasing either ethylene or nitrogen or by decreasing oxygen.

Normal control of oxygen feed while operating outside the explosive range can be done by considering the HCl utilizations and the analysis of the crude product. This has been discussed under O_2 in the section on reactor feeds. High oxygen in the recycle, combined with very high HCl utilizations and excessive carbon tetrachloride in the product all are indications of too much oxygen feed. Low HCl utilizations combined with exceptionally low oxygen in the recycle are indications of too little oxygen feed.

The amount of chlorinated organics in the recycle will be dependent upon the exit temperature of the refrigerated reactor condenser. This should normally be about 6% at atmospheric rates and about 1.5% at full rates (46 psig).

L. Reactor Temperature Control

The basic method of reactor temperature control will be the control of the Dowtherm inlet temperature to the reactor. The total Dowtherm flow will be maintained constant. The sixteen tube banks in each reactor may have slightly different pressure drops. For this reason, the Dowtherm flow to some tube banks will have to be restricted so as to maintain relatively equal Dowtherm outlet temperatures. The reactor temperatures will be indicated at six levels in the reactor and at 120^{o} intervals around the reactor. No appreciable temperature difference in the catalyst bed is expected. The reactor temperatures are continuously recorded on board mounted temperature recorders and must be closely followed. Any indication of hot spots or unexpected changes in temperature must be investigated and corrected for. Unfortunately, because of the cooling coil layout, the reactor temperatures are limited to the perimeter (maximum penetration is only 24"); therefore, the only indication of trouble near the center will be the composition of the reactor vent gas.

Localized hot spots will be considered as indication of clinker formation. These localized hot spots can result in quick deterioration of the Inconel tubes. This, of course, must be prevented. More probable indications of clinkering are high burning and high trichlor and perchlor formation. Severe clinkering may also produce a dark-colored crude product. If clinkering occurs, consideration must be given to immediate shutdown.

The existence of a cooling tube leak may be detected by analysis of the proper stream. Normally, the reactor will be under more pressure than the Dowtherm; therefore, any leak would be from the reactor into the Dowtherm. The Dowtherm acidity tests should be faithfully run. In cases in which the Dowtherm is under greater pressure than the reactor, leaks may be found by such phenomena as unknowns showing up in the crude EDC analysis or loss in head tank Dowtherm level.

Design temperature is 525°F. Pilot plant experience has shown that this represents a good compromise between heat removal and TCE content of the crude.

Reactor Feed Temperatures: Each reactor feed temperature is controlled at 250°F. The actual feed temperature at the inlet to the reactor may be lower because of expansion across the flow control valves. A close watch on the actual feed temperatures must be maintained.

M. Reactor System Pressure Control

The primary purpose of controlling reactor pressure is the maintenance of a specified superficial gas velocity in the reactor fluidized bed. This specified velocity is 0.8 ft/sec at the level of the oxygen nozzles and at the bed temperature and reactor pressure at the top. Fluid velocity less than 0.8 ft/sec could result in slow catalyst movement, clinkering and loss of fluidization. Greater fluid velocities will result in increased carryover of catalyst and a higher attrition rate of catalyst due to fines generation. For these reasons the fluid velocity must always be maintained at 0.8 ft/sec.

The actual throughput of feeds to the reactor must, of course, change with varying production rates. The velocity is held constant by changing the reactor pressure which changes actual volume rate of flow. High throughputs require high pressures and vice versa.

The normal pressure controlled vent stream is taken off downstream of the refrigerated reactor condenser and goes to the stack scrubber. The reactor pressure is changed by simply changing the set point on the pressure controller. Changes in pressure should be made slowly, since rapid changes could cause changes in catalyst fluidization and carry-over. Rapid changes may also upset the recycle control system.

Other vent points are available for special situations. The reactor off-gas may be vented immediately after the primary condenser to a large "start-up" vent scrubber. This valve is remotely operated from the board. This vent route will be used during reactor heat-up and initial feed addition. Another vent to this scrubber is located just downstream of the normal purge vent. This vent valve automatically opens when the controller output pressure exceeds 9 psig.

Pressure control will be very touchy during start-up of the recycle compressors. This technique will be discussed in the start-up section.

N. Reactor Instrumentation

1. <u>Computer System</u>: The OHC plant is equipped with many instruments which are tied into the IBM 1800 computer in the main office. Important temperatures, pressures, and flows are printed out on the typewriter console

in the control room. Temperature, pressure, and flow instruments are available, as normal, on the control board. The flow instruments on feeds to the reactors are automatically corrected for pressure and temperature changes by individual local computers.

The IBM 1800 computer will be used to make routine calculations of reactor operating conditions, as well as OHC plant production. The following list of data will be calculated by the computer and printed on the control room typewriter.

Items to be Reported by the Computer Daily:

a. Ethylene feed (each reactor) - lbs.

b. Oxygen feed (each reactor) - lbs.

c. HCl feed (each reactor) - lbs.

d. Recycle feed (each reactor) - lbs.

e. EDC production (total) - lbs.

f. Transfers to EDC plant (total) - lbs.

g. Transfers to Per-Tri plant (total) - lbs.

h. Monthly production (total) - tons

i. Average daily production for month - tons

j. Product inventory - tons

k. HCl utilization (total) - %

l. Ethylene yield (total) - %

m. Excess oxygen (total) - %

n. Percent burning (each reactor) - %

o. Ethylene lost in vent (each reactor) - %

p. Unaccountable carbon losses (total) - %

q. Ethylene conversion per pass (each reactor) - %

r. Reactor superficial velocity (each reactpr) - FT/SEC.

Items reported on demand:

1. Percent burning (each reactor) - %

2. Ethylene lost in vent (each reactor) - % of feed

3. Ethylene conversion per pass (each reactor) - %

4. Reactor superficial velocity (each reactor) - FT/SEC.

2. Reactor Shutdown System: It is imperative that there never be too much O_2 in relation to HCl in the reactor feeds. This is true because unreacted O_2 in too great an excess would drive the system into the explosive range in the reactor vapor space or condensing system. For this reason each reactor is equipped with an O_2 to HCl ratio-recorder-controller. If HCl flow should drop (which would increase O_2 concentrations in the system), the ratio recorder would sense the temporary flow ratio change and automatically decrease the O_2 flow. This type of control is labeled "cascade" control on the instruments and should always be used during normal operation. During rate changes, this control can be changed to "auto" whereby each flow (HCl and O_2) can be controlled independently; this procedure is particularly good when decreasing rates as the oxygen flow can be cut first. During times when these flows are off "cascade" control, an operator should always be present in the control room to watch these instruments. It is extremely critical that such care as this be taken in order to keep the system out of the explosive range during upsets.

If oxygen flow fails during routine operation, an automatic control system is available to introduce nitrogen into the O_2 feed line to keep the O_2 nozzles open. This system also should always be in the automatic shutdown position or have operator attendance. This system would also automatically cause N_2 addition to the oxygen feed header should HCl flow be lost.

Each reactor is equipped with an emergency shutdown system actuated by a board-mounted automatic shutdown button. Operation of this button gives the following simultaneous action:

a. The C_2H_4 valve closes.

b. The O_2 valve closes and N_2 flow is opened into the O_2 feed header. This N_2 addition system consists of two automatic valves with an automatic bleed valve between. The purpose of this arrangement is to guard against contamination of the nitrogen system by leaking valves. When there is no N_2 flow, the two double block valves will be closed while the automatic bleed will be open. This arrangement is reversed with a N_2 flow.

c. HCl flow is not affected. It remains flowing until which time the operator can phase the HCl out by adding N_2 into the HCl feed header.

d. The open/close remote operated valve on the gas flow to the recycle gas scrubber will automatically close. This will cause low suction pressure on the recycle compressor, which automatically shuts down on low suction pressure.

e. Any reverse flow in the recycle line to the reactor will be sensed which then shuts the recycle feed flow control valve.

O. Intermediate Crude Storage

The Intermediate crude tanks are glass-lined steel horizontal tanks. The piping is arranged so that the production from the reactors can be split (i.e., each reactor crude flow to a separate tank or all production to a single tank).

Each tank is provided with a rupture disc set for 5 psig burst pressure. This low burst pressure is necessary because the vessel is connected to the flasher and vent condenser driers (which have 5 psig design pressure) during normal operation.

Each tank is provided with a nitrogen purge, metered by a rotameter, and a vent line going to the vent condenser. The vent line must be kept open at all times during operation. The N_2 purge must also be kept flowing during operation, but must <u>always</u> be shut off if the vent line is closed off. The nitrogen comes from the 90 psig header and could easily exceed the rupture disc pressure if not properly controlled.

P. Chloral Treatment System

Chloral (CCl_3CHO) is an oxygen bearing compound that inhibits the cracking of EDC to vinyl chloride. The chloral is formed in the reactor in small quantities. It would be very difficult to remove from EDC by distillation but is readily reacted with caustic soda to form water soluble sodium formate (NaOCOH) and chloroform $CHCl_3$. The sodium formate is removed from the EDC in the aqueous phase by a phase separator. Chloroform is then relatively easy to remove by distillation. The sodium formate is removed from the EDC in the aqueous phase by a phase separator.

1. <u>Feeds</u>: The crude chloral-bearing EDC is pumped to the chloral treatment system by special corrosion resistant Duriron pumps. These pumps are constructed of Durcon 6, an epoxy resin which is resistant to acid and abrasive fluids.

Caustic soda is pumped to the chloral treatment tank from a caustic storage tank. Cell liquor is diluted with condensate in the caustic tank before being pumped to the treatment tank. Regular cell liquor is strong enough to give two potential problems if mixed with EDC; first, emulsion formation with EDC and, second, it is so close in specific gravity with EDC that it might invert with EDC in phase separation. For these reasons, the cell liquor is diluted by one-half by holding equal settings on the water and cell liquor rotameters. These flows must be manually set so as to maintain the tank level at about two-thirds full. The caustic should be checked for strength periodically and any adjustments necessary to hold 5-6% NaOH made. If inversion does take place, then further dilution of the caustic may be necessary.

The caustic feed pumps serve two purposes; first, feed to chloral treatment at 3 gal/min and low pressure and second, emergency caustic feed to the recycle gas scrubbers at 50 gal/min and high pressure. Thus, during normal operation the pumps operate much below their normal capacity and could cavitate. For this reason, part of the discharge from the pumps is recycled back to the caustic storage tank. The amount of recycle should be normally controlled at a rate such that the pump discharge pressure is sufficient to drive cell liquor into the recycle gas scrubbers. Do not overlook this point when raising reactor rates.

2. Chloral Treatment Tank and Phase Separator: The treatment tank is constructed of steel. The tank is agitated by a Chemineer turbine type agitator turning at 120 rpm. All wetted parts of the agitator are constructed of Monel. The agitator has a stuffing box type seal.

The reacted caustic and chloral-EDC solution overflow from the chloral treatment tank into the chloral treatment phase separator. The aqueous layer is decanted off to a small catchpot and then flows to the sewer. The crude EDC is pumped off the bottom at a rate automatically regulated to control the interface level in the phase separator.

Nitrogen is metered into both the treatment tank and the phase separator. The nitrogen is vented from the treatment tank through the overflow liquid line into the phase separator and through a vent on the overflow line to the sewer. This nitrogen flow should be added slowly so as to prevent vapor locking the overflow line. The nitrogen in the phase separator is vented into a catchpot.

The normal ratio of EDC to caustic feeds to the treatment tank should be about 18 to 1. The actual ratio used will be determined by testing the phase separator effluent for chloral removal. The 18:1 ratio represents a 20% caustic excess. It is very important that a caustic excess by maintained so as to insure complete reaction. A chart showing the relationship of cell liquor flow and crude EDC flow is shown in Figure 10-5.

Q. DH Still System

1. Feed Tank: The chloral-free EDC is pumped to the D-H feed tank. This feed tank is a vertical steel atmospheric design tank. The tank is padded with nitrogen which is fed at a rate sufficient to hold 2 inches of water pressure on the tank. The tank is also provided with a 12 inch pressure-vacuum vent valve, which has a built-in relief feature at 6 ounces/square inch pressure and 2 ounces/square inch vacuum.

2. DH Still: The crude EDC in the D-H feed tank is wet; this water is to be removed in the D-H still. The separation between water and EDC is a psuedoazeotropic distillation with the water being removed out the top of the still. Any HCl formed is also removed in the D-H still in the overhead. The EDC leaving the bottom of the still should contain less than 20 ppm water.

The D-H still is constructed of Haveg 41 and is 3' diameter and 22'4" high. It is packed with 12 feet of 1 1/2 inch Intalox saddles. The feed and reflux are distributed over the packing by a ceramic weir-riser distributor plate. The reboiler is all Monel on the process side. The liquid and vapor lines to the reboiler are steel.

The D-H still overhead condenser is graphite on the tube (process) side with a steel shell. Cooling is provided by cooling tower water. The exchanger is mounted vertically with the inlet gases entering at the top. The reflux drum is constructed of glass-lined steel.

3. Instrumentation and Operation: The D-H still is provided with a top pressure and pressure drop recorder in addition to a recorded temperature profile. The feed rate and steam rate are flow controlled. Water, EDC, and light organics are condensed out of the overhead and are collected in the reflux drum. The water phases out in the drum and overflows to the acid sewer. The loop in the reflux line is designed to hold sufficient level in the reflux drum to allow all the water to phase off the organics before the reflux gravity flows back to the still. The low boiling compounds are concentrated in the top of the drying still and are not recoverable in the water cooled D-H condenser. Therefore, the condenser is vented to the refrigerated vent condenser.

It is essential that the water and HCl be removed in the D-H still. The corrosive capabilities of the crude material make close control of this still very important. The reboiler and bottoms piping are quite susceptible to corrosion. The steam flow rate is controlled to maintain the amount of boilup necessary for the water and HCl removal. The water-EDC "azeotrope" is not a true azeotrope and can be thought of as removing water by stripping action. For this reason, the steam rate should be maintained high even during times when the feed rate is low in order to provide sufficient vapor for stripping. The design calls for 50% boilup at full rates.

The pressure drop recorder will give visual indication of any plugging or buildup in the packing. Buildup from an unknown source was encountered in the pilot plant, so this must be watched carefully. The design pressure drop of the packing at full rates with 50% boilup is 0.32 psi.

4. D-H Still Bottoms Flow: The EDC from the bottom of the D-H still is acceptable feed material for the Per-Tri plant if it is dry. The EDC passes through a dryer and filter to remove any residual water or water contamination due to D-H still upsets. The EDC to be sent to Per-Tri then passes through the D-H still bottoms cooler before being sent to Per-Tri feed storage tanks. This EDC being sent to the tanks must be free of water so this moisture test should be run diligently.

R. Refrigerated Vent Condenser, Stack Scrubber, and Vent Condenser Driers

Several vent streams in the OHC plant go to the vent condenser system:

1. Off-gases from the flashers and phase separators.
2. Off-gas from the D-H still condenser and reflux line.

3. Gases flashed off in the intermediate crude tanks.

4. Pad gas from the D-H still feed tank.

The vent condenser is a vertical up-flow graphite exchanger cooled by refrigerated brine. The refrigerant flow is automatically controlled to give an outlet gas temperature of 60°F. The condensate flows to two Haveg 41 vent condenser driers charged with calcium chloride. These driers are necessary because the condensate contains considerable dissolved water at this low temperature. The condensate flows from the driers to the intermediate crude tanks. The driers are equipped with a nitrogen pad. This pad system has a safety relief valve which opens if the pressure exceeds 5 psig. Also, a 5 psig graphite rupture disk is mounted on each drier.

The outlet gas from the vent condenser flows to the stack scrubber before being vented through a stack to the atmosphere. The normal reactor operating vents and the vents from the recycle gas headers also enter this scrubber. The scrubber is constructed of Haveg 41. Well water is continuously sprayed into the tower. The effluent water overflows to the acid sewer. The stack is equipped with a steam snuff.

S. Lights Still System

1. Feed System: During full rate operation, about one-half of the OHC-EDC will go to the Per-Tri plant. The remaining EDC is purified further before being sent to storage. This purified EDC may be used in the vinyl chloride plant, the Tri-Ethane plant, or for sales.

The first stage of this purification (lights removal) is done in the OHC plant. The EDC product from the D-H still bottoms dryer-filter is split; part is sent to Per-Tri storage while part is sent to the lights still feed system. The amount of EDC fed to the lights still system is automatically controlled so as to maintain a constant level in the lights still feed tank. This EDC flows to the suction line of the lights still feed pumps which is also equalized to the feed tank. This feed tank is nitrogen padded to maintain 4 inches H_2O pressure. It is protected by a pressure-vacuum vent valve which relieves at 16 ounces/square inch of pressure and 2 ounces/square inch of vacuum.

The feed to the lights still is flow controlled. Thus, if the lights still feed rate is increased, the feed tank level drops and more D-H still product is diverted to the feed tank, and vice versa.

2. Lights Still: The lights still is a steel vessel 42 inches diameter and 72 1/2 feet tall. The still has 50 trays; the tray spacing is 12 inches for the top 17 trays and 18 inches for the rest. Each tray is equipped with 54 Nutter type "B" float valves. All materials are steel with the exception of the valves which are type 410 stainless steel. Six feed points (located at trays 34, 32, 30, 28, 26 and 24) are available. Heat is introduced in a steel thermosiphon reboiler.

The overhead vapors from the column are condensed in a horizontal steel condenser with cooling tower water. The condensate flows to a vertical reflux drum. The condenser and reflux drum are padded with nitrogen so as to maintain a 1 psig pressure. The condensate is pumped from the reflux drum to the lights still reflux economizer and then to a water cooled reflux cooler. The cooled reflux flows through one of two available lights still reflux dryers and a filter. The flow of this cooled, dried overhead is split; part goes through the cold side of the reflux economizer and the remaining goes to the Per-Tri feed storage tanks. The reflux to the still is flow controlled. The amount of overhead sent to the Per-Tri feed tanks is then automatically controlled to maintain a constant level in the reflux drum.

Lights-free EDC is pumped from the bottom of the lights still through a bottoms cooler and to storage. This flow is automatically controlled to maintain a constant reboiler level. This EDC can go to the Per-Tri feed storage tanks; however, it normally will flow to two tanks (identical to the Per-Tri feed tanks) which serve as feed tanks for further purification. This EDC contains heavy (high boiling) components. The new EDC still in the No. 2 EDC plant was designed to distill the product from OHC in addition to its regular load. Thus, the EDC product from OHC is further purified in the No. 2 EDC plant.

3. Lights Still Operation and Control: The primary function of the lights still is to separate light (low boiling) components, primarily chloroform, from the EDC. See Table 4-3 for anticipated feed composition. However, carbon tetrachloride and EDC form an azeotrope and this carbon tetrachloride will pass overhead out of the lights still. The information available during the lights still design was very limited and several assumptions had to be made during the design. Subsequently, the data for design has been obtained and back calculations show that the existing lights still will not perform as designed. The new data shows that more reflux is required for the design separation. All this means is that more EDC will be driven overhead and sent to the Per-Tri feed tanks via the lights still overhead. This is not harmful, since this feed is primarily EDC anyway. The design reflux ratio on the lights still is 20 to 1;

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Table 4-3

Expected Lights Still Feed Composition

Component	Wt. %
Methylene Chloride	0.01
Vinyl Chloride	0.01
Ethyl Chloride	0.01
DCE	0.01
Chloroform	0.31
1,1 EDC	0.01
1,2 EDC	98.20
Carbon Tetrachloride	0.13
TCE	1.28
Perchloroethylene	0.02

however, the new design information indicates that approximately 200 to 1 will be required. The still will probably be operated with a high steam rate at all times in order to maintain a high reflux ratio. This steam rate can be automatically controlled so as to maintain a constant temperature on tray number 20. The set point temperature to be used will be determined after startup. Several temperatures in the still will be logged on the IBM console.

The temperature profile should be watched closely and steam and reflux adjustments made so as to prevent lights break-through out the bottom of the column. This bottom stream will be checked by chromatographic analysis. The key component may be either carbon tetrachloride or chloroform in the bottom stream, depending on how much carbon tetrachloride is made in the reactor. The still will be fed at the upper feed point initially to insure against contamination of the bottoms stream.

Operation of the reflux cooling-economizer-drier system should be centered around one-point; any moisture fed to the still will concentrate in the overhead. The reflux drier is designed to remove this moisture. This drier is most effective when the EDC is cool. The purpose of the economizer-water cooler is to lower the temperature of the EDC for drying. The reflux to the still is preheated in the reflux economizer so as to increase its heat content going back to the still. The flow of cooling tower water to the reflux cooler should be regulated to give about 100° outlet temperature.

T. EDC Storage

Four tanks are available for OHC-EDC product; the two Per-Tri feed tanks and the two feed tanks for No. 2 EDC plant still. Each tank has a capacity of 180 tons (90% full). Each set of tanks has a nitrogen pad system which provides a pad pressure of 2 inches of water. Each tank is equipped with a 16 inch pressure-vacuum tank vent which relieves at 6 ounces/square inch pressure and 2 ounces/square inch vacuum. Each tank has a Varec level indicating gauge.

The normal procedure for using these tanks will be to produce into one tank of each set while the other is being emptied to the user. During changing of these tanks, always record starting and ending levels by the Varec gauge and temperatures. This information will be used to determine production and transfer rates.

The EDC flow to Per-Tri from the feed tanks is critical to Per-Tri operation. This EDC supply must be consistent and any measures necessary to do this must be taken. The tanks should never be emptied less than 4 feet so as to always have EDC available in the tanks and to give plenty of time to switch tanks. The pumps should be checked periodically for bearing and motor temperatures, seal leakage, and vibration. Any loss of these pumps must be handled on an emergency basis. An alternate EDC source is available (the No. 1 EDC plant); however, this procedure takes time and should not be relied upon except in an emergency.

The flow of EDC to the No. 2 EDC still is not as critical as the Per-Tri feed; however, this flow should be consistently maintained if at all possible. Again, hold 4 feet minimum level in the tanks on switching. A cross over line between the Per-Tri and EDC still feed lines is available so that lights-free EDC may also be fed to Per-Tri.

The material from any of the four tanks can be pumped back through a rework line into the OHC process. This rework line may be used when off-specification EDC is produced to the tanks. This line allows feeding

EDC to the D-H feed tank, lights still feed tank, and intermediate crude storage. The line also allows bypassing of the entire purification system after chloral removal; it also allows bypassing of the lights still only.

U. Cooling Tower System

The OHC cooling tower is an extension of the Per-Tri tower. The basis of the two towers will be equalized at all times. The OHC tower is designed to cool 2750 gpm of water from 115°F to 90°F. Two pumps will be available to circulate the water through the OHC plant. The pumps have Garlock packing--this packing should be tightened so that a slow drip occurs. The suction and discharge piping on these pumps is independent of the Per-Tri pumps.

Equip List

A single fan pulls air through the crossflow type tower. Loss of this fan will sound an alarm. The fan is driven by a 60 h.p. motor with a gear reducer. A cut-out switch will stop the motor if vibration is excessive. This vibration could result from misalignment of the drive system or wear in this system. The oil level in the gear reducer must be checked periodically.

The cooling action of the tower creates a loss of water by evaporation. This evaporation loss will cause hardness salts (particularly silica and calcium) to concentrate in the circulating water stream. Concentration to the saturation point will cause plating out of silica and calcium in the heat exchangers. This must be avoided. The degree of concentration is best indicated by the ratio of chloride in the cooling water to the chloride in the make-up water. This concentration ratio should be held between 3 and 4 by regulating the amount of blowdown. The following blowdown points are available:

1. Cooling on Dowtherm pumps
2. Vent scrubber seal pot
3. Centrifix collecting drum vent pot
4. Steam drum condensate sample condensers
5. Chloral treatment vent seal pot
6. Lights still condenser vent seal pot

The Per-Tri operator will run all tests on the cooling tower and will make any necessary adjustments in the treatment chemicals. The control of blowdown is the OHC operator's responsibility since the bulk of the blowdown must be done in the OHC plant in order to insure adequate cross-flow between the cooling tower basins. Good communications must exist between the OHC and Per-Tri operators.

If the pH test by the Per-Tri operator indicates a declining OHC water pH, or consistently lower pH than the Per-Tri water, the possibility of a condenser leak exists. Such a condenser leak would allow HCl to migrate into the cooling water. This possibility must be thoroughly checked.

V. Dowtherm System

As discussed in the reactor section, the heat of reaction is removed by pumping Dowtherm A through cooling coils in the reactor. The Dowtherm is subsequently cooled by generating steam in a thermosiphon reboiler-type cooler. The cooled Dowtherm is pumped back to the reactors; the suction of these pumps is equalized with a Dowtherm surge tank.

Dowtherm A is a eutectic mixture of two organic liquids, diphenyl and diphenyl oxide. New Dowtherm A is a clear straw-colored liquid that darkens with use. Its freezing point is 53.6°F and its atmospheric boiling temperature is 495.8^{o}F. All Dowtherm piping is steam traced to prevent freeze-up in cold weather. Although it is a flammable material, Dowtherm A is not considered particularly hazardous at less than boiling temperature; however, the higher the temperature, the more easily it will ignite. Dowtherm A presents no appreciable hazard to health and has only a very slight irritating effect on the skin. The distinct odor of Dowtherm A has a lingering quality which makes it wise to avoid getting it on clother or shoes.

The OHC plant will share the 8868 gallon Dowtherm storage tank at Per-Tri. The tank is vented to the atmosphere and is heated internally to avoid freezing of the Dowtherm in cold weather. The Dowtherm can be pumped from the tank to any of the OHC or Per-Tri reactors with the make-up pump.

The cooling system for each reactor contains about 3000 gallons of Dowtherm. Since Dowtherm A expands greatly with an increase in temperature, a 2000 gallon surge tank is provided to absorb the changes in volume. When the Dowtherm is cool, the tank must not be overfilled so that sufficient expansion room remains. The liquid Dowtherm is sometimes circulated at temperatures above its boiling point. For this reason a nitrogen pad-pressure relief system is available to suppress boiling. ~~The nitrogen pad-pressure is automatically regulated at 10 psig. This pressure will prevent boiling during normal operation. The safety relief valve operator~~
There is a SRV on the [illegible] that operates
when the vessel pressure exceeds 150 psig, which could occur during some emergencies (such as power failure). The safety valve discharges into the sewer.

1. Dowtherm Heater: During startup of the reactor it is necessary to raise reactor bed temperature to reaction temperature. This is achieved by circulating Dowtherm through a heater before going to the reactor tubes. The Dowtherm heater is a direct gas fired furnace through which the Dowtherm passes. The Dowtherm splits into four tube passes before going through the heater. Each tube pass consists of 5 u-bend tubes 12 feet long. The heater is designed to deliver 2 million BTU per hour, which is sufficient to heat 1600 gal/min of Dowtherm to 700°F. The heat energy is provided by burning natural gas in a single John Zink burner. The furnace is natural draft with a self-supporting stack.

The John Zink type VBM burner is a premix burner in which a portion of the air for combustion is mixed with the gas before emerging into the main combustion chamber. The mixer is the aspirating type and uses the energy of the gas to pull air into the mixer body. The air pulled into the mixer body is called "primary air". The amount of primary air drawn in is controlled by a small threaded "primary air door" located at the bottom of the burner where the main gas line ties in. The mixture of gas and air flows up through the gas-air tube and is evenly distributed into a spider gas distributor. Additional air for combustion flows around and between the spider arms and is controlled by means of lower type secondary air door.

The adjustment of the primary air door depends on the gas pressure, type of gas, and type of flame desired. In our case, this primary air flow should be relatively small so as to give a radiant (yellow) flame. The adjustment of the secondary air door depends on furnace draft and primary air adjustment. Furnace draft draws in secondary air, so for low furnace draft, the secondary air door will be opened more than high furnace draft.

The efficiency of the Dowtherm heater is dependent upon the amount of excess air in the combustion chamber. The heater is designed for 30% excess air over that required for combustion. The amount of excess air can be determined by running an Orsat analysis of the flue gas. The quantity is controlled by changing the setting of the secondary air door. The OHC foreman will specify when this test should be made.

a. Dowtherm Heater Firetron Control System: The furnace burner is controlled by a complex system of instruments that perform two functions: temperature control and emergency shutdown. Emergency shutdown can result from tube wall over-temperature, high Dowtherm pressure, no-flow of fuel gas, flame failure, or low Dowtherm flow.

Dowtherm outlet temperature is controlled by automatically throttling the gas flow to the burner. The control instrument is located at the main process control panel.

The shutdown devices and controls comprise the Firetron control system. The Firetron panel is located at the furnace. The Firetron controls insure that the furnace is started in the proper sequences and under the proper conditions. The two Maxon valves in the gas line to the burner are the key valves of the Firetron shutdown/startup system. The Maxon valves can not be opened unless a holding coil within the valve is energized, and the coil is not energized on startup unless the proper sequence is followed nor will it stay energized unless the operating conditions are correct. The Maxon valve closes when its coil is de-energized. Two Maxon valves in the gas line with an automatic pressure bleed-off between are necessary to conform with insurance regulations.

A discussion of each component of the burner controls is as follows:

1) Firetron startup and shutdown panel - This panel contains:

a) Power supply switch

b) Start switch and light

c) Reset start switch

d) Purge timer-allows 5 minutes purge after an emergency shutdown before the furnace can be restarted.

e) Control circuit - arranged so that furnace flame can be started only after correct procedure is followed.

2) Gas pressure control valve - This valve or regulator reduces the main line pressure to 20 psig.

a) Low pressure switch and alarm - immediately following the pressure control valve, this switch, by a hook-up to the Firetron control panel, causes a shutdown and sounds a panel alarm if the gas line pressure becomes too low.

3) Pilot line.

a) Solenoid valve - energized and opened by the start switch.

b) Pilot line plugcock.

4) Maxon valves and automatic bleed-off.

5) Pressure switch - this switch closes when there is sufficient gas pressure. It is the final signal in the Firetron system, and the furnace flame is ready to start when there is sufficient pressure to close this switch.

6) Temperature control valve - this automatic valve throttles the gas flow to the furnace according to the desired Dowtherm outlet temperature.

7) Factory Mutual main burner plugcock - while closed, this valve has air flowing through special ports. When opened, the ports are closed and the passage of air is blocked. The air closes a pressure switch that closes a relay in the Firetron circuit and allows the startup procedure to commence. When the gas line pressure switch (No. 5 above) is closed, the factory Mutual pressure switch is bypassed and the main burner plugcock can be opened without interrupting the procedure - thus producing flame.

8) Furnace burner assembly. Includes:

a) Main burner block and pilot burner.

b) Firetron scanner - this instrument senses the flame, transmits millivolt current to proper relay in Firetron circuit. Sensing element is a small photoelectric cell of "fire eye."

c) Spark plug - energized by the "start" button in Firetron panel, the sparkplug lights the pilot flame.

9) Tube wall temperatures - detected by a thermo-couple welded to each tube wall; high tube wall temperature causes shutdown when it exceeds the set-point on the tube wall temperature instrument in the Firetron panel. This prevents high temperature Dowtherm decomposition and subsequent overpressure and tube rupture. This would occur primarily with low Dowtherm flow.

10) Dowtherm outlet temperature thermocouple on the Dowtherm discharge line from the furnace measures the outlet temperature and transmit to a recorder-controller instrument on the main process control panel. The instrument pneumatically positions the gas flow valve to the furnace (No. 6 above.)

11) High pressure switch - located on Dowtherm discharge line from furnace. High pressure opens switch and causes shutdown through Firetron control circuit. The switch is set to open at a pressure just under the rupture disc - safety valve rating of 110 psig.

Detailed startup and shutdown procedures will be given in the appropriate sections. This heater will be intermittently operated. Routinely, the pilot light will be extinguished and the system will be put into operation only when needed. This precaution is necessary since the pilot flame could be dangerous in times of spills or combustible gas escape.

W. Steam Generation and Distribution and Condensate System

The steam produced in the Dowtherm coolers is exhausted into a steam header for use in the OHC plant and also in an Area "B" header. The pressure on the steam drum will be automatically controlled at 35 psig. The Area "B" header pressure will be at about 28 to 30 psig depending on the amount of pressure drop involved. The primary users of this low pressure steam will be the OHC plant reboiler and heaters, EDC plants stripper reboilers, and the VC plant stripper reboiler. Other users will mainly consist of utility drops in the EDC, MC, EC, HCl and VC plants. The expanded MC plant may use this low pressure steam on some of its process equipment. The amount of hot Dowtherm available for the generation of steam is proportional to the rates at which the reactors are being operated; thus the steam generation rate will vary with reactor rates. The header pressure will be maintained at a minimum of about 28 to 30 psig in the area of the EDC plant by make-up from the Area "B" 175 psig steam header. The make-up steam will be automatically injected through two control valves, operating with a split range controller, when the 30 psig header pressure falls below the set point.

A vent to the atmosphere is available on the steam system in the OHC plant. This vent will operate when the header pressure exceeds 35 psig. The vent consists of a steam silence which utilizes acoustical packing to reduce noise. The silencer vent will automatically take care of surges in the steam system.

Area "B" condensate is collected into a common header. Some of this condensate is metered into the OHC condensate storage tank at a rate sufficient to maintain a constant level in the tank. This condensate is used as feed to the steam drums. A small amount of condensate is also used for dilution at the recycle scrubbers and caustic tank.

Condensate from the storage tank is picked up by one of two condensate feed pumps. These pumps are capable of developing pressures in excess of 100 psig. The condensate entering the suction of these pumps is near its boiling point; if the pumps are operated against a blocked discharge, vaporization will quickly develop in the pump bowl. To avoid this, there is a restricting orifice in a recycle line back to the condensate storage tank. Valving through this restricting orifice should be open at all times when one or both condensate pumps are on.

When switching from one condensate pump to the spare, use the following procedure:

1. Open the suction valve to the pump not in service.

2. Start the pump and immediately start opening the discharge valve.

3. As soon as the discharge is open, then start closing the discharge valve to the other pump. When you get this valve closed, then immediately shut the pump down. Then close the suction valve to this pump.

The above procedure should be followed at all times. If the discharge valve is opened on a pump that is off, a high rate of backward rotation will result and might damage the motor or unscrew the pump impeller.

To prevent excessive corrosion and scaling in the steam generating system, the boiler feed water has to be chemically treated. The treating chemicals will be added to the boiler feed water in the condensate pump discharge line that feeds the steam drums.

The treating chemical batch will be made up in a 4' x 4' tank. The treating chemicals are soluble in steam condensate so the batch will be made up by dissolving the desired chemicals in reclaimed condensate. To assure that the chemicals rapidly go into solution, agitation by N_2 should take place as the chemicals are added.

The treating chemical mix will consist of flake caustic, sodium nitrite, Nalco 19 (Sodium Sulphite) and disodium phosphate. The following is a description of the concentration and function.

1. Flake Caustic: This is added to keep the hydrate alkalinity high enough to assure that the water softening reactions take place. The magnesium in the water reacts with the caustic to form magnesium hydroxide, $Mg(OH)_2$. The $Mg(OH)_2$ then reacts with the silicon dioxide, SiO_2, to form $Mg(OH)_2 \cdot SiO_2$. Both $Mg(OH)_2$ and $Mg(OH)_2 \cdot SiO$ are not objectional. The minimum level hydrate aklalinity will be 200 ppm.

2. Sodium Nitrite: This is added to prevent caustic cracking (embrittlement) of boiler steel. Caustic embrittlement is a condition where metal that is under stress is attacked by caustic in the boiler water. Low

concentrations of sodium nitrate tend to inhibit this type of corrosion. The sodium nitrite level will be maintained at approximately 30 ppm.

3. Nalco 19 (Sodium Sulphite): This is added to serve as a scavenger of the oxygen in the boiler water. Oxygen in a boiler system readily attacks the hot wet boiler steel. The following reaction shows the manner in which the sodium sulfite is used to "scavenge" the oxygen:

$$2\ Na_2\ SO_3 + O_2 \longrightarrow 2\ Na_2\ SO_4$$

The reaction product, sodium sulfate, is not objectionable. The sulfite level will be controlled at a minimum level of 20 ppm.

4. Disodium Phosphate: This is added to soften the boiler water. The calcium in the system combines with the phosphate to form $Ca_{10}(PO)_6(OH)_2$, which is not objectional. Proper treating to control hardness is very important. Should the boiler water not be maintained in a soft condition, scaling of the boiler tubes will quickly occur. The phosphate level will be controlled at a minimum of 20 ppm.

The exact levels of the various chemicals that will comprise the treating mixture has to be determined through experience. To get the desired control, the treating mixture will likely require adjustments from time to time. Tentatively, make a mixture per the table at the end of this section.

The treating chemicals are pushed into the system with a small positive displacement pump. There is a safety valve on the discharge of this pump that relieves back into the treating tank. This safety valve is set at 200 psig. Before starting the treating pump, always be sure that the discharge block valves are properly lined up in the open position. Extremely high pressures will be developed by this pump if it is operated against a closed discharge.

The stroke on the chemical addition pump is adjustable. To add more or less treating chemicals, accomplish this by adjusting the pump stroke.

To remove dissolved solids from the steam drums, a continuous blowdown will be taken from each drum. The blow-down will be adjusted by a small valve to keep the total dissolved solids in the steam drums at a level of 1500 to 2000 ppm.

Once per shift for each steam drum, the operator will run the following control test; hydrate alkalinity, sulfite level, phosphate level, total dissolved solids, and condensate pH. All these tests except condensate pH will be run on a sample of the blow-down from each steam drum. The condensate pH test will be on a sample pulled from the suction of the condensate pumps.

The main laboratory will run the following test weekly; total Fe, hydrate alkalinity, nitrate level, sulfite level, phosphate level, total dissolved solids, and condensate pH.

CONDENSATE TREATMENT BATCH

1 Full tank of condensate

2 Gallons (80% of a bucket) of flake caustic

1 Quart of sulphite - Nalco 19

1 Quart of sodium intrite

1 Quart of disodium phosphate

X. High Pressure Well Water Pumps

Well water is used as a coolant in the process side of the primary and secondary reactor condensers. These condensers can operate at pressures up to 46 psig and are at a high elevation. This requires that the well water be at extra high pressure. This is accomplished with one of two available well water booster pumps. One of these pumps must be operating at all times and problems with either pump should be handled on an emergency basis.

Y. Fluidization Air Blowers Flow Gauge X 300

During initial startup and prolonged reactor shutdowns, fluidization of the catalyst bed will be accomplished by blowing air into the windbox and nitrogen into the oxygen header. The fluidization air will be provided by two (one for each reactor) Gardner-Denver axial flow blowers. The blower discharges are tied together so either blower can be used on either reactor. Each blower is capable of delivering 1940 standard cubic feet per minute at 18 psig discharge pressure. The blowers are the rotary lobe-type positive displacement and are driven at 1770 rpm by a 200 horsepower electric motor coupled directly to the blower. The suction of each blower is equipped with a filter-silencer and the discharge has a snubber-silencer. The discharge also has a safety relief valve which opens if the pressure exceeds 20 psig. The blowers automatically shuts down if the discharge temperature exceeds 375°F.

The blowers are lubricated by a splash system. Each set of outboard bearings has an oil reservoir and the level in this reservoir should be routinely checked. Do not operate the blower unless oil shows in the sight glass. Oil is added through the breather-filter at the top of each bearing carrier.

Z. Vent Scrubber (Start-Up Scrubber)

A vent scrubber is available for scrubbing of large vents streams. The vents piped into this scrubber are:

1. Outlet gas from the primary reactor condensers.
2. Outlet gas from the refrigerated reactor condensers.

These vent streams will not exist under normal operation. The primary condenser vent will be used during reactor startups when a high inert load exists, thus causing excessive pressure drop in the condensers. The vent from the refrigerated condenser is part of the split range vent control on reactor pressure and will only operate during excessive vent flows.

The scrubber is constructed of Haveg 41 and is 42 inches in diameter and has 10 feet of 2 inch porcelain Intalox saddles. Well water is distributed across the packing by a ceramix weir riser distributor plate. A small flow of water should be left on at all times. The stack is provided with a steam snuff. Nitrogen is flow controlled into the inlet header and should be maintained at a low rate at all times.

AA. Refrigeration Unit

1. General: A 110 ton York refrigeration unit will be used to cool 526 gpm of $CaCl_2$ brine. The brine is pumped through the tube side of the shell-and-tube brine cooler and is cooled from 40°F to 34°F; it then passes to the shell side of the two reactor refrigerated condensers and the refrigerated vent condenser. The shell side of the brine cooler contains Freon-11, which is being vaporized as it receives heat from the brine. The low-pressure Freon is picked up by the rotating impeller wheel in the compressor and is discharged, at high pressure and temperature, into the condenser. Cooling tower water is pumped through the tube side of this shell-and-tube heat exchanger to remove the heat from the Freon by condensing it. The liquid refrigerant then flows through a float valve into the brine cooler to begin another cycle.

Since it is desirable to operate the system at low load conditions, some means of capacity control must be used to maintain a constant brine temperature leaving the cooler and to extend the operating range of the compressor to stable operation at low load conditions. Therefore, the compressor is equipped with adjustable prerotation vanes (PRV) located in the suction inlet to the impeller wheel. The position of these vanes is varied automatically by a piston operator on the outside of the compressor housing as a method of capacity control. Adjusting the position of these vanes provides compressor performance to match load conditions from full load with vanes wide open to minimum load with vanes nearly closed. An automatic hot gas by-pass is provided to return hot vapor from the compressor discharge to the suction. This will be in operation during startup (when the refrigeration load is low) or during sustained periods of operation with low refrigeration requirements.

The 200 hp motor is sized for 2300 volt operation. It turns at 3600 rpm while the compressor turns at 9000 rpm via a speed increaser which is mounted inside the compressor.

A purge compressor system is provided to remove inert gases from the refrigerant system.

2. Pneumatic Control System: The exit brine temperature is maintained by establishing a set point on the refrigeration unit temperature controller. This controller positions the prerotation vanes at the compressor suction to control the compressor suction pressure, which sets the F-11 boiling temperature. The output of this controller also positions the hot gas by-pass valve. At 15 psig controller output pressure the prerotation vanes are completely open. As the load decreases and the

output pressure drops to 9 psig, the hot gas by-pass valve begins to open. The vanes, however, do not close to their minimum opening until the controller output pressure reaches 7 psig. The hot gas by-pass continues to open until the controller signal is 3 psig; at this point the by-pass is completely open and the vanes are closed as much as possible.

A current limiting relay (CLR), installed in the controller output to the prerotation vane piston operator, will modulate the controller output signal in response to the current being drawn by the motor to prevent motor overload. If the motor current exceeds the setting of the maximum load adjustment, an air dump solenoid valve will bleed off part of the air signal being supplied to the prerotational vane operation, thus preventing the vanes from opening further. If the motor current continues to increase, the signal air pressure will decrease until the vane operator starts to close the vanes. When the motor current drops off to the setting governed by the maximum load adjustment, the vanes will stop closing. On a further load decrease the vanes will again be under the control of the temperature controller and will be allowed to open again within the limit of the setting of the maximum load adjustment.

The manual control valve can be used to manually put an air signal on the hot gas by-pass valve and the prerotation vane operator. Turning this valve knob counter-clockwise decreases the signal pressure to establish low-load operation conditions for both the by-pass valve and the prerotation vanes. Conversely, rotating the knob clockwise increases the signal air pressure to open the vanes and close the hot gas by-pass valve. A selector switch on the York panel board makes it possible to switch from manual to automatic control and vice versa. No set point adjustment is necessary when switching as the two automatically follow each other.

3. Electrical Control System: The following safety controls have been provided for the York refrigeration unit.

a. The low brine temperature (LBT) cutout causes the unit to shut down if the brine temperature should drop to 28°F.

b. The condenser high pressure cutout (HP) is an automatically reset safety control which opens the compressor motor control circuit if the condenser pressure exceeds 15 psig.

c. The evaporator low pressure cutout (LP) is an automatically reset safety control actuated by the brine cooler pressure. This cutout will shut the compressor motor down if the F-11 pressure drops to 24" Hg vacuum; this setting is sufficient due to the low freezing point of brine.

d. The oil pressure differential cutout (OP) is an automatically reset safety control actuated by the differential pressure between the compressor suction and the main oil pump discharge. The motor will be shut down if this differential drops at 27 psig; it cannot be restarted until the differential is 32 psig.

e. The high oil temperature cutout (HOT) is a safety control mounted on the oil cooler inlet line. When the oil temperature reaches 170°F, the motor will shut down.

f. The high discharge temperature cutout (HDT) causes the unit to shut down if the compressor discharge temperature exceeds 250°F.

4. Control Panel: The following controls are accessible from the front of the panel.

a. The compressor on-off switch is a pushbutton which manually energizes or de-energizes the compressor motor control circuit.

b. The auxiliary oil pump "Auto-Manual" switch controls the functions of the auxiliary oil pump. While in the "Auto" position, this pump runs for 8 seconds prior to compressor motor starting, about 200 seconds after the motor starts, and for 1 1/2 minutes following a motor shutdown. In the "Manual" position, the pump runs continuously, regardless of whether or not the compressor is running.

c. The purge unit on-off switch controls the purge unit.

d. The automatic-manual pneumatic control switch is used to switch from automatic to manual control and vice versa. No adjustment is necessary before making this switch.

e. The manual control valve is a manually operated air valve by which the signal air pressure to the PRV operator and the hot gas by-pass can be adjusted to vary the compressor load.

f. The automatic brine temperature controller is used to establish a set point for the brine temperature from the brine cooler. The signal air pressure to the PRV operator and the hot gas by-pass will automatically be varied to hold this temperature.

g. The following control air pressure gauges are provided on the board.

1) Signal air to PRV - Signal from solenoid to PRV operator.

2) Temperature controller leaving air - Signal from temperature controller to solenoid.

3) Control air from set point - Signal from automatic set point adjustment to temperature controller.

4) Signal air to current limiting relay - Signal from PRV operator to current limiting relay.

5) Signal air from current limiting relay - Signal from CLR to PRV operator. This should be the same as (4) in most cases. However, if too much current is being drawn by the compressor motor, this signal will be less than (4) due to some of the signal being bled to the atmosphere by the CLR.

6) Supply air - Main supply air pressure. This should be 20 psig. Other items appearing on the control board are the oil pressure differential, compressor discharge pressure, compressor suction pressure, motor amps, and several alarms.

5. Purge System: To assure satisfactory operation, it is important that the system be kept free of moisture-laden air and non-condensable gases. Air in the system usually collects in the condenser covering some of the condensing surface, causing the discharge pressure and temperature to rise, and can damage the unit. Moisture in the system may cause acid formations which are destructive to internal system parts.

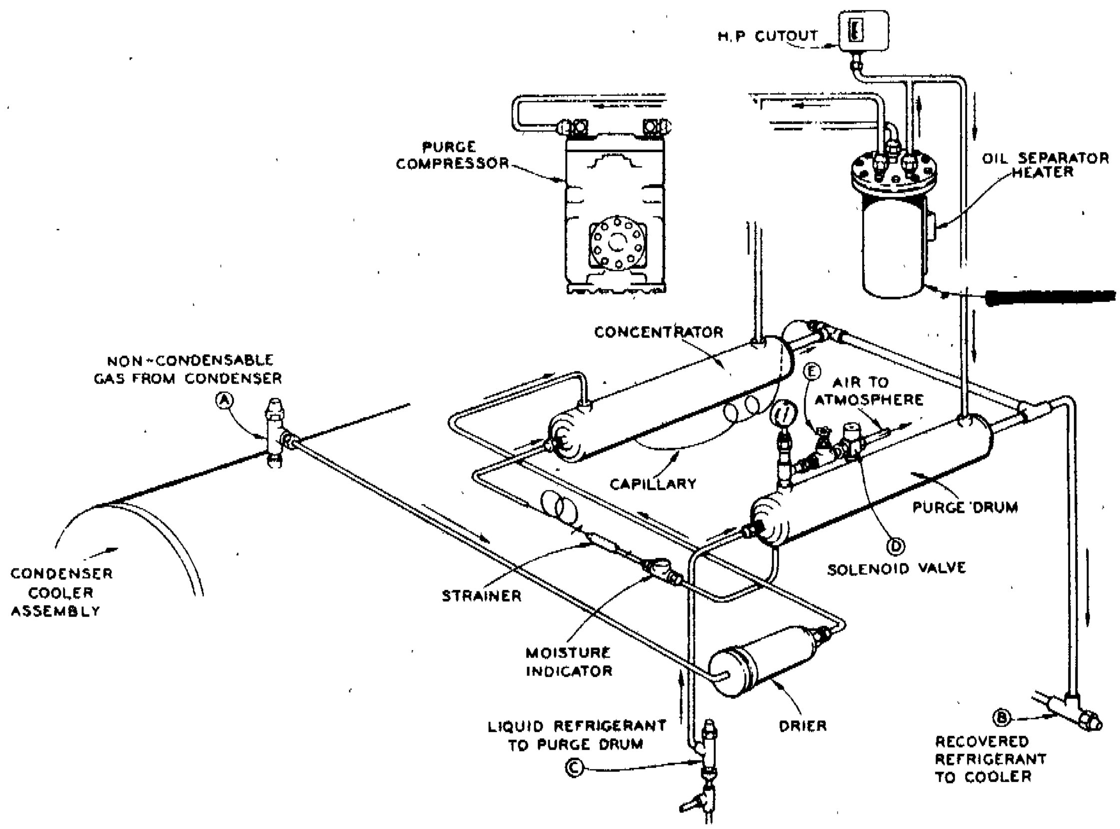
An air purge and refrigerant recovery unit is furnished and mounted at the front of the system. The purpose of this unit is to remove the mixture of non-condensable gases and refrigerant from the top of the condenser, expel the non-condensables to the atmosphere, and return the refrigerant to the system.

To avoid refrigerant loss, the purge unit should be operated only when the system is in operation.

To check a system for the presence of air, subtract the actual liquid temperature from the indicated temperature. If the indicated

FIG. 4-4

REFRIGERATION PURGE UNIT



temperature is more than 2^{o}F higher than the actual temperature, an excessive quantity of air in the system is indicated and the purge unit should be operated until the difference between the indicated temperature and the actual temperature is within 2^{o}F.

NOTE: A thermometer well is located on the side of the high pressure float chamber for the purpose of checking liquid refrigerant temperature.

To follow the actual operation of the purge unit, refer to the schematic arrangement shown in Figure 4-4.

NOTE: Before starting the purge unit initially or whenever the control circuit has been de-energized long enough to permit the oil separator to cool off, Valves (A), (B), and (C) should be kept closed for at least one hour after the oil separator heater has been energized. This permits the oil separator to warm up so that liquid refrigerant will not accumulate in the oil separator during the operation.

The mixture of refrigerant and non-condensable gas leaves the system condenser through Valve (A) and enters the purge unit concentrator shell through a drier.

In the concentrator, some of the refrigerant in the mixture is immediately condensed by the cold concentrator coil. This liquid refrigerant flows from the concentrator shell to the cooler. The compressor pulls the gas (which does not condense) from the concentrator shell and discharges this gas, at higher pressure, into the purge drum shell through the oil separator.

Liquid refrigerant from the float chamber flows through Valve (C) into the purge drum coil, where it evaporates as it cools the coil, and returns as a wet gas to the cooler with the recovered refrigerant through Valve (B). During operation, Valve (C) must be throttled until line downstream is cold to the touch.

The oil separator is equipped with an external heater (energized when the control center is energized) to evaporate any liquid refrigerant which may accumulate in the separator with the oil. The oil in the separator is drawn off to the purge compressor suction through a float valve in the bottom of the separator.

In the purge drum shell, the remaining refrigerant condenses and flows through the strainer and capillary tube to the concentrator coil and returns as a gas, to the cooler through Valve (B).

The non-condensable gas accumulates in the purge drum shell until the pressure within the purge drum reaches the setting of the high pressure cutout (70 psig for Refrigerant-11). At this time the high pressure cutout actuates to stop the purge compressor motor and energizes a solenoid valve to open and permit the non-condensables to flow to the atmosphere. As soon as the purge drum pressure is reduced to the cut-in setting of the high pressure cutout (20 psig for Refrigerant-11), the high pressure cutout will again actuate to de-energize and close the solenoid valve and start the compressor motor.

The inlet drier is installed to catch any moisture in the non-condensable gas before it enters the purge unit. When the system has been purged for the first time after the initial operating period, the drier cartridge should be changed. During normal operation the drier cartridge should be changed about every three months, or whenever the indicator shows saturation.

NOTE: When the purge compressor no longer cycles on its high pressure cutout, the air in the system has been reduced to a minimum and the purge unit may be stopped (after operating 1/2 hr. to circulate the oil) by moving the ON-OFF switch to OFF. After the purge compressor is stopped, close Valve (E) to prevent air from entering the system. If it becomes necessary to operate the purge unit for more than 1/2 hour daily, an air leak into the system in indicated and the system should be leak tested at the earliest convenient time.

6. Compressor Lubrication System: The lubrication system consists of the oil pumps, oil filter, oil cooler and all interconnecting oil piping and passages. There are eight main points within the motor-compressor which must be supplied with forced lubrication as follows:

a. Compressor Drive Shaft (Low Speed)

1) Shaft seal.

2) Front and rear main bearings - one on each side of driving gear.

3) Low speed thrust bearing.

b. Compressor Driven Shaft (High Speed)

1) Thrust bearing and thrust collar.

2) Pinion gear bearings.

3) Main bearing.

c. Speed Increasing Gears

1) Meshing surfaces of main and pinion gear teeth.

To provide the required amount of oil under the necessary pressure to properly lubricate these parts, three pumps are used:

a. The jet pump.

FIG. 4-5

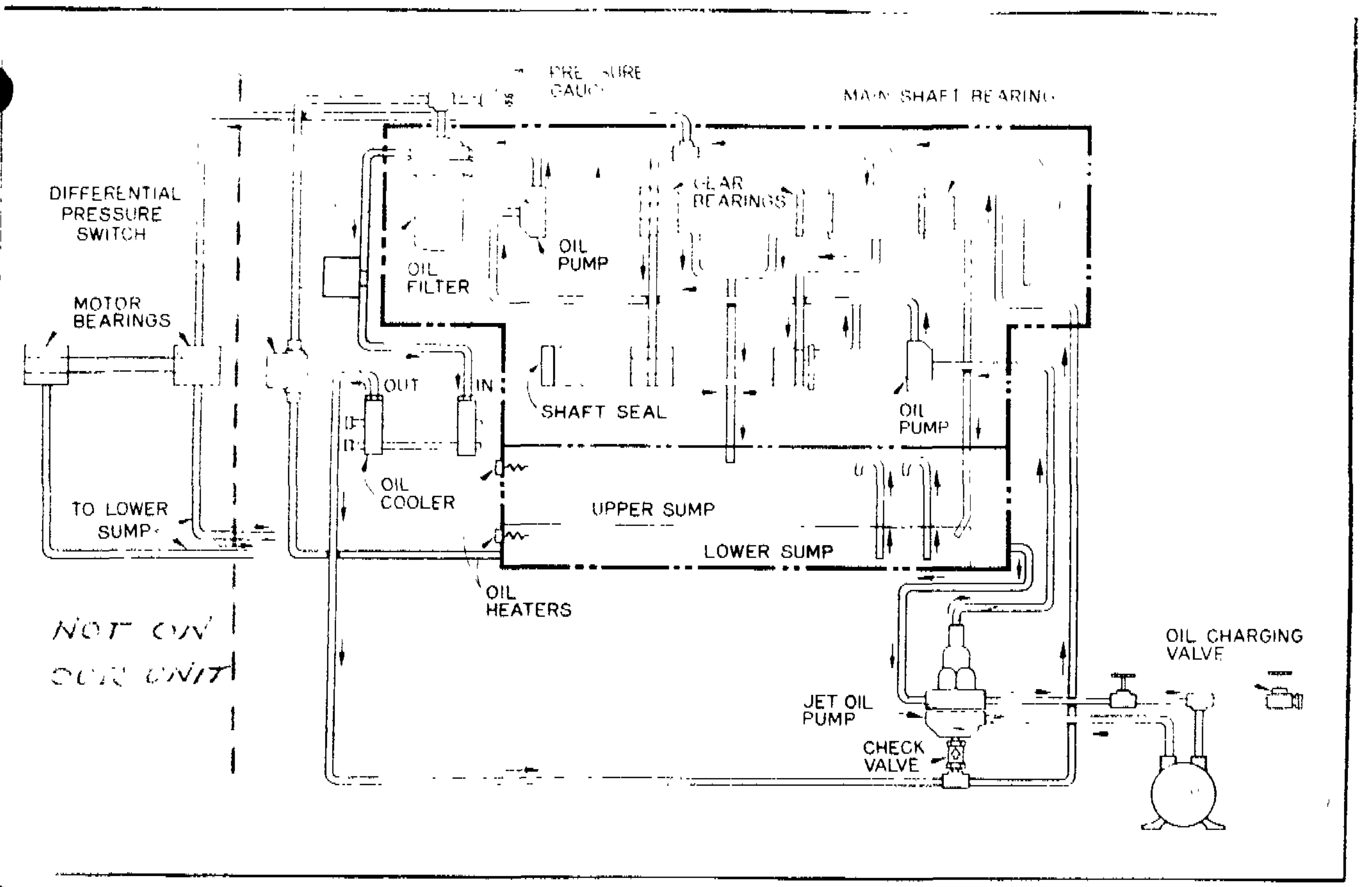
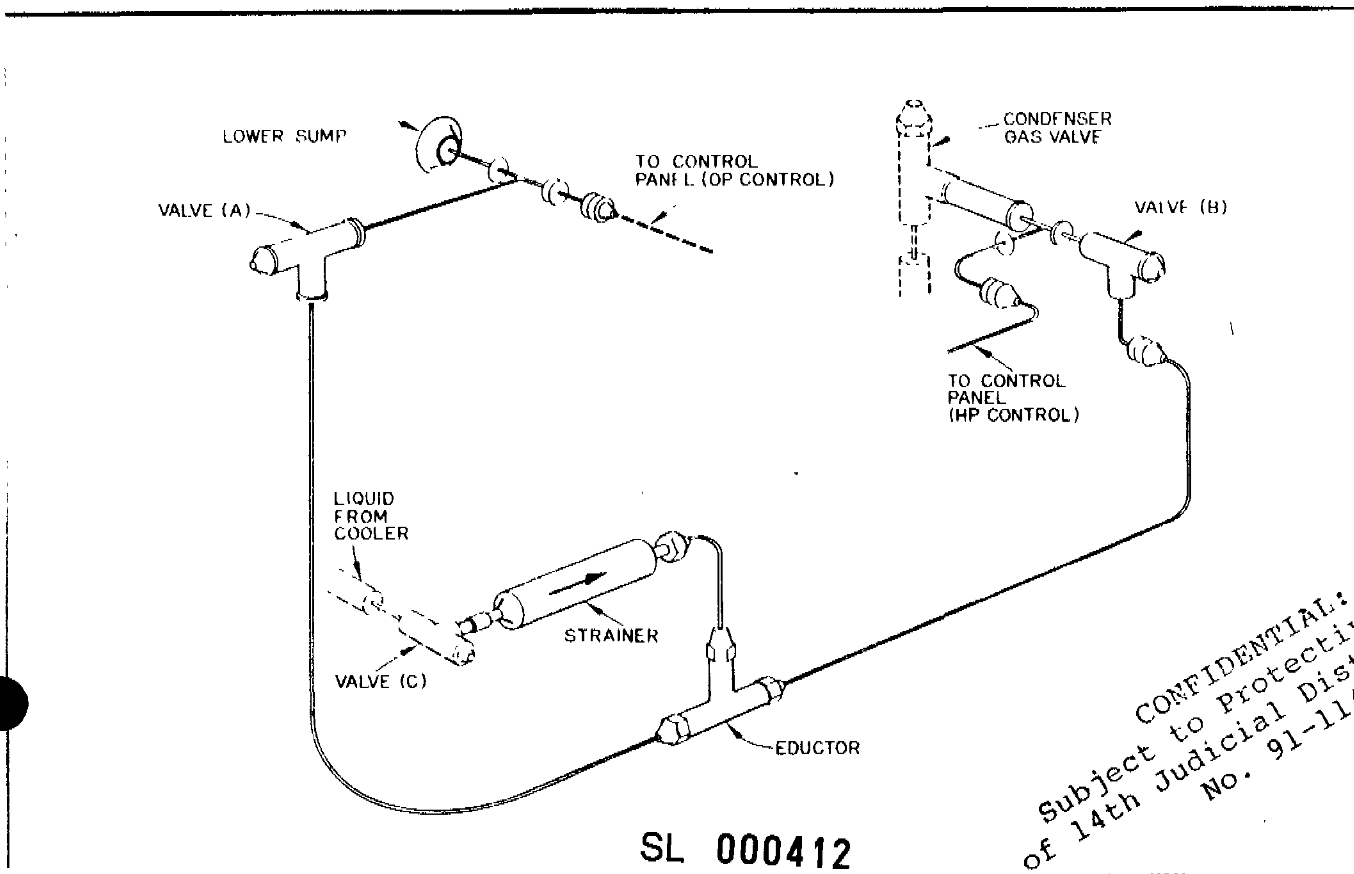


FIG. 4-6
OIL RETURN EDUCTOR



b. The low speed pump.

c. The high speed pump.

An auxiliary oil pump (AOP) provides added protection against system depression during startup. It also is an aid in charging oil and checking out the system.

The jet pump, as the name implies, has no moving parts and operates on the injector principle; the main pump is mounted on the low speed shaft and operates on the centrifugal principle; the high speed pump, also centrifugal, is mounted on the free end of the high speed shaft. Referring to the diagram of the oil circuit, Fig. 4-5, the flow of oil through these pumps and the entire system can be followed.

Upon depression of the ON-OFF switch at the control center, the auxiliary oil pump (AOP) should be immediately energized. After an 8 second delay to allow the system oil pressure to stabilize, and the (AOP) switch in the AUTO position the (AOP) runs for about 200 seconds and then automatically shuts down.

When the (AOP) is operating (during startup and shutdown) it takes suction from the main oil reservoir through the side connection on the rotor support, through the side connections of the jet oil pump. The oil discharges from the (AOP) through the bottom connection of the jet into the space below the flow diversion valve. This pressure forces the flow diversion valve against its upper seat which prevents oil from by-passing to the (AOP) suction so it flows through the jet nozzle to the compressor main oil pump suction.

The check valve between the jet pump and the oil cooler prevents oil passing back through the oil cooler. A factory set relief valve is installed in the bottom of the jet pump to relieve any excess pressure to the suction side of the pump.

When the (AOP) is idle and oil pressure is produced by the compressor main oil pump, the flow diversion valve drops to its lower seat, allowing oil from the oil reservoir to be induced to flow through the jet nozzle by the high pressure oil from the high speed pump.

The oil jet pump is located in the suction line to the main (low speed) oil pump. Oil under pressure flowing from the oil cooler through the jet nozzle, induces additional oil from the reservoir to flow to the main oil pump suction.

The main oil pump takes suction from the discharge of the jet pump and discharges to the suction of the high speed pump.

The high speed oil pump takes suction from the discharge of the low speed pump and discharges to the oil filter. The filter discharges to the oil cooler and from the oil cooler, high pressure oil feeds main bearings, seal, and jet nozzle. Main bearings discharge to main sump through gear cavity; rear bearing behind impeller discharges to lower sump and is returned to upper sump by gas pressure differential created by discharge pressure on seal behind impeller and gas vent from upper sump to impeller inlet. All bearings are flooded on standby.

The function of the oil filter is obvious. All the oil is filtered each time it passes through the system.

The oil cooler is required to remove heat picked up by the oil in the lubrication system. This heat is carried away from the oil cooler by cooling tower water.

7. Reservoil Oil Heater: During long idle periods, the oil in the compressor oil reservoir tends to absorb as much refrigerant as it can hold, depending upon the temperature of the oil and the pressure in the reservoir. As the oil temperature is lowered, the amount of refrigerant absorbed will be increased. If the quantity of refrigerant in the oil becomes excessive, violent oil foaming will result as the pressure within the system is lowered on starting. This foaming is caused by refrigerant gas boiling out of the oil as the pressure is lowered. If this foam reaches the oil pump suction, the bearing oil pressure will fluctuate with possible temporary loss of lubrication, causing the oil pressure switch to actuate and stop the system.

To maintain the lowest possible concentration of refrigerant in the oil, the compressor is equipped with 120 volt electric reservoir oil heaters.

These heaters are wired so that they are automatically energized when the compressor is idle for periods of more than 1 1/2 minutes and automatically de-energized at all times while the compressor is operating.

On shutdown of the system for any reason, the (AOP) is automatically energized and continues to run for 1 1/2 minutes. The system cannot restart during that time interval.

8. Oil Return Eductor: The oil éductor is provided to return oil which may be lost to the condenser. The eductor should not be operated continuously but only as required to maintain a safe operating oil level in the compressor.

To operate the oil eductor refer to Fig. 4-6.

a. With the system in normal operation, open valve (A) to permit oil return to the lower sump.

b. Open valve (B) to supply condenser gas to the eductor.

c. Open valve (C) to allow the mixture of refrigerant and oil to flow from the chiller and return to the lower sump.

The flow rate of the refrigerant and oil mixture can be regulated by throttling valve (B). The eductor is operating properly when the line to the lower sump is cold.

9. Auxiliary Oil Pump: For normal operation the (AOP) switch should be in the AUTO position at all times. This provides pump operation for both startup and shutdown. Manual pump operation should be used only for oil charging or to establish stable oil pressure before starting.

10. High Pressure Float Valve: Liquid refrigerant control is accomplished by means of a high pressure float valve located in the float chamber at the front of the cooler-condenser shell.

This float valve serves as a seal between the high and low pressure sides of the system by opening to permit the liquid refrigerant to flow from the condenser into the cooler and closing to prevent the passage of gas when condensation is at a minimum during light load conditions.

The float valve is equipped with a seal capped hand opening device which protrudes through the float chamber to manually open the float valve if level in the float chamber will fluctuate during operation, depending upon load conditions. However, during operation a liquid seal must be maintained to prevent condenser pressure from blowing into the cooler. If the float chamber fills up with liquid during operation, the float valve is sticking in the closed (or nearly closed) position. If this occurs, operate the float valve manually until the system can be shut down or until the valve is free. If the float continues to stick in the closed position, the float ball may be leaking.

11. Checking the Refrigerant Charge: The refrigerant charge should always be checked while the system is operating at normal full load design conditions. Since full load conditions are not always available, they may be temporarily obtained for check purposes as outlined below. This procedure is not an accurate method of checking charge but it will serve as a guide in checking the charge at partial loads.

a. Stop the compressor and permit the brine pumps to operate until the brine temperature rises 3 or 4 degrees above full load design temperature.

b. Start the compressor and when the brine temperature decreases to normal full load design conditions, check the charge by observing the compressor motor amperes, the discharge temperature, and the wetting of the top row of cooler tubes.

12. Refrigerant Relief Piping: A frangible carbon bursting disc assembly is located on the side of the compressor suction connection for the purpose of quickly relieving excess pressure of the refrigerant charge to the atmosphere as a safety precaution in case of an emergency, such as fire. The bursting disc is furnished in accordance with the ASME Code for unfired Pressure Vessels, and is set to relieve at 15 psig.

13. Recurring Restart Protection: For protection of the motor, the system cannot, due to a timer device, be restarted more than once every 30 minutes.

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TABLE 4-7

OPERATION ANALYSIS CHART

1. SYMPTOM - ABNORMALLY HIGH DISCHARGE PRESSURE

RESULTS	POSSIBLE CAUSE	REMEDY
Temperature difference between liquid refrigerant out and water off condenser higher than normal.	Air in condenser	Purge condenser of air with purge unit.
High discharge pressure	Condenser tubes dirty or scaled.	Clean condenser tubes. Check water conditioning.
	High condensing water temperature.	Reduce condensing water inlet temperature. (Check cooling tower and water circulation).
Temperature difference between condenser water on and water off higher than normal, with normal cooler pressure.	Insufficient condensing water supply	Increase the quantity of water through the condenser.
Temperature difference between condenser water on and water off lower than normal, with low suction pressure.	High pressure float valve fails to open.	Open float valve manually - inspect and repair as soon as possible.

2. SYMPTOM, ABNORMALLY LOW SUCTION PRESSURE

Temperature difference between secondary refrigerant leaving cooler and primary refrigerant in cooler greater than normal with high discharge temperature.	Insufficient charge of refrigerant.	Check for leaks and charge refrigerant into system.
Temperature difference between secondary refrigerant leaving cooler and primary refrigerant in the cooler greater than normal with normal discharge temperature.	Cooler tubes dirty or restricted.	Clean cooler tubes.
Temperature of secondary refrigerant too low with low motor amperes.	Insufficient load for system capacity.	Check prerotation vane motor operation and setting of low water temperature cutout.

3. SYMPTOM, HIGH COOLER PRESSURE

Low discharge temperature.	Liquid slugging. System overcharged with refrigerant.	Check the charge. Remove refrigerant if necessary.
High secondary refrigerant temperature.	Prerotation vanes fail to open.	Check the prerotation vane motor positioning circuit.
	System overloaded.	Be sure the vanes are wide open (without overloading the motor) until the load decreases.

4. SYMPTOM, FLUCTUATING OIL PRESSURE

Oil pressure cycling (fluctuation of over 10 psig with compressor running and vanes open).	Air leak on vacuum side of lubrication piping.	Check all external oil piping for leaks, especially the lines connected to the jet oil pump. Check the seal on (AOP).

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V. START-UP PROCEDURES

A. Reactor Catalyst Charging

The OHC catalyst has 7.5% copper and 5.3% potassium. The catalyst is delivered to our plant in 350 pound fiber drums with plastic liners. The drums are stored in the catalyst storage building immediately west of the OHC plant. The Per-Tri catalyst will also be stored in this building. Since these two catalysts have different properties, it is extremely important that no mix-up occurs. The OHC catalyst drums will be color coded with a green stripe around the side, and will be marked as Catalyst and Chemicals, Inc., C78-2 catalyst. Under no circumstances should catalyst be used which has not been positively identified as the OHC catalyst.

The OHC reactor must be operated with sufficient fluidizing medium at all times, including during initial catalyst charging. Thus, air and nitrogen will be used for fluidization when the catalyst charge is added. The fluidization air and nitrogen will be vented from the reactor system through the vent immediately following the primary condenser. The initial charge will be made through one of the 18 inch rupture disc openings; therefore, this opening will also vent some of the fluidizing gas.

The initial catalyst charge will be added with two large open-top catalyst charge hoppers. Catalyst will be charged to the reactor by gravity flow from one of the hoppers. The charge hopper will be lifted from the ground to the top of the reactor by a crane.

The following procedure should followed when the initial catalyst charge is made:

1. The catalyst to be charged should be inspected, sampled, and analyzed before addition to the reactor. Acceptable catalyst is free-flowing, dry, and free from lumps and debris. Analysis by the laboratory should be obtained by the foreman before charging.

2. The reactor, overhead piping and centrifix system should be completely dry so that the catalyst will not become wet, thus causing a corrosive and caking condition. The steam flow to the centrifix, catalyst catchpot, and tracing should be started so as to keep that part of the system dry.

3. Remove one of the rupture discs from the reactor head.

4. Open the valving on the vent flow to the startup vent scrubber from the outlet of the primary condenser. Start nitrogen and well water flow to the scrubber.

5. Open the valves on the fluidization air lines to the mixed feed header. Rotate the spectacle blind at the valve so that it allows flow through the lines. Start one of the fluidization air blowers and throttle the flow to 54,700 SCFH with the blower discharge valve.

6. Open the valve on the nitrogen addition line to the O_2 header at the point downstream of the oxygen flow control valve. Put the nitrogen addition flow controller on manual (this requires putting the O_2 header shutdown switch on bypass). Start nitrogen addition to the reactor at 7,800 SCFH.

7. Inspect the catalyst charge hopper and clean and dry as necessary. Transfer the catalyst from the drums to the hopper.

8. Have the catalyst lifted to the top of the reactor and secured in place by maintenance people.

9. All people in the surrounding area should obtain and put on goggles at this point. Some fine catalyst particles may carry overhead out of the reactor during the charging.

10. Open the bottom valve on the charge hopper and let the catalyst empty into the reactor. Have the charge hopper lowered to the ground by maintenance people and repeat the charging process until the catalyst level is even with the top of the Dowtherm tubes. This level can be observed through the rupture disc opening by visual inspection. During this visual inspection, a full face gas mask with <u>external</u> oxygen supply <u>must</u> be worn to protect the observer from catalyst carry-over and possible oxygen starvation (due to the nitrogen flow through the reactor).

11. During the charging operation, the fluidization air and nitrogen flows will tend to decrease due to the increased pressure drop caused by increasing bed height. The air and nitrogen flows <u>must</u> be held at the specified rates by the operator during the charging.

12. The amount of catalyst charged to the reactor should be carefully noted for record purposes.

13. After completion of catalyst charging, the rupture disc and sub-insulation should be replaced. Again, the full face mask should be worn during this reinstallation.

B. Dowtherm Charging

The reactor catalyst bed must be heated up to ~~450~~ 475°F before addition of the feeds. This reaction initiation temperature is achieved by circulating heated Dowtherm through the reactor tubes. It will be necessary to initially fill the system with Dowtherm and possibly after future maintenance on the reactor.

Since Dowtherm is flammable in the vapor state, appropriate precautions must be taken to insure safe operation. The Dowtherm system must be thoroughly purged with nitrogen before Dowtherm addition. The oxygen content should be established at a low figure (less than 1%) by checking the system at several points with an oxygen analyzer.

The Dowtherm will be pumped into the OHC Dowtherm system from the Per-Tri Dowtherm storage tank. The total volume of the OHC Dowtherm systems is X 9045 gallons; however, the system should be filled so that the Dowtherm surge tanks are only one-fourth full to allow for thermal expansion. This gives a total initial Dowtherm volume, at atmospheric temperature, of X 5870 gallons.

The Dowtherm make-up line from the Per-Tri storage tank ties into the line connecting the Dowtherm surge tank with the Dowtherm circulating pumps. The charging procedure is listed as follows:

1. Start pump at Dowtherm storage tank. The Per-Tri operator can start the pump, or should be informed when it is needed. He will have to check his valving arrangement.

2. Open the two valves into the bottom of the Dowtherm surge tank and the suction valve on the circulation pumps. Build a low level in the surge tank. Start the pumps; make sure that the seal flush flow is properly adjusted and the cooling tower water is on the flush cooler.

3. Open the bleed valve on the Dowtherm cooler and surge tank and bleed the inert gas to the atmosphere during filling. Also bleed off at the bleed valves on the Dowtherm inlet lines to the reactors. After the level in the surge tank reaches one-fourth, close the valve on the Dowtherm charging line and shut down the make-up pump at Per-Tri.

4. Some gas may remain in the hairpin loops in the reactor during filling. Sweep one tube bank at a time by stopping flow to the other banks. This should give enough pressure and velocity to sweep trapped gases out of the system.

5. After this sweeping, some Dowtherm addition may be necessary to bring surge tank level to one-fourth.

6. During reactor heat-up, dissolved gases may flash off, thus increasing the Dowtherm system pressure. If this occurs, slowly bleed the pressure off at the Dowtherm cooler.

C. Dowtherm Heating

Both OHC reactors could be heated simultaneously, but no controls are available to assure equal circulation to each reactor and the heat-up would be slow. Thus each reactor will be heated separately. The Dowtherm flow should be lined up so that the circulation pumps discharge to the Dowtherm heater, from the heater to the reactor, from the reactor through the temperature control valve (with the flow blocked to the cooler), and back to the pumps. The system is now ready for warm-up. The Dowtherm heater should be started up by the following checklist:

1. Start Dowtherm circulation.

2. Set the tube wall temperature instrument at 750°F.

3. Close the Factory Mutual main burner plugcock.

4. Actuate the Firetron panel by closing the power supply switch.

5. The gas pressure regulator valve should be put in service (if possible) and adjusted to hold 20-25 psig downstream of the valve. This valve may leak through too much to allow control during initial startup; if this is the case, throttle the flow with one of the block valves.

6. Check the atmosphere inside the furnace with an explosimeter to assure that no residual gases are left in the heater.

7. Light the pilot. This is achieved by opening the pilot light plugcock, then depressing the start switch on the Firetron panel. The start switch opens the solenoid valve in the pilot line, thus allowing gas flow, and energizes the spark plug which lights the pilot flame. The start switch comes on when the start switch is depressed and goes out when the Firetron scanner sees the pilot flame. The start switch may be released when the pilot light goes out.

8. Depress the Start reset button on the Firetron panel. This insures that all the relays and solenoids in the panel are in the "ready" position.

9. At this point in the sequence, the holding coils in the Maxon valves and the coil in the solenoid valve of the gas line vent bleed are energized and the Maxon valves may be opened. The Maxon valves are opened by the hand operated lever. The operating lever, through the holding action of the coil, engages the latching mechanism of the valve; as the lever is moved to the open position the valve is opened. The valve remains open until the coil is de-energized; it cannot be closed with the operating lever. An indicator on the Maxon valve operating mechanism will indicate whether the valve is open or closed.

10. After the Maxon valves are opened, the pressure in the burner feed line increases, actuating a pressure switch. This pressure switch bypasses the pressure switch that is actuated by the air passing through the main burner Factory Mutual plugcock. This action frees the main burner plugcock and it can be opened to establish the burner flame.

11. The burner flame is established by opening the main burner plugcock--the main flame is ignited by the pilot flame. This plugcock must be opened slowly and steadily.

12. During initial heater startup, it will be necessary to carefully control firing rate so as to properly dry out the heater refractory. This dryout is necessary in order to minimize the possibility of steam formation in the refractory and resultant spalling.

If the heater is being started for the first time, or after long (several months) periods of no use, adjust the burner flow to maintain a stack temperature of 400°F. Maintain this temperature for 6 hours. Then increase firing rate to provide a 50°F per hour increase in stack temperature until the stack temperature reaches 650°F. Hold for 4 hours.

For dryout after short periods of no use, the foreman should be consulted for specific times and temperatures.

13. After dryout, adjust the Dowtherm temperature set point (after putting the instrument on automatic) so as to maintain a 100°F differential between the tube wall and discharge Dowtherm temperature. This will require increasing the Dowtherm temperature set-point until the desired reactor temperature is achieved. Under no circumstances should the tube wall temperature exceed 750°F.

14. Trouble shooting list for furnace start-up:

a. Pilot light won't light.

1) Bad spark plug - remove and check.

2) Plugged or restricted gas line - dismantle and clean.

3) Burned out solenoid valve - valve makes audible click when actuated. If no click is heard, suspect bad solenoid, have replaced.

4) Pilot burner out of adjustment - adjust the air intake to give a steady flame that does not sputter or have yellow tips. Too much air intake causes a sputtering flame that is hard to ignite.

5) The pilot burner assembly could be broken in some manner. Inspect the burner for such defect.

6) Wiring trouble in the start circuit. This may have to be corrected by an electrician. One of the first things to check would be the fuse in the circuitry at the burner and the 110 V. circuit breaker behind the panel board. Some indications of this area:

a) Good spark plug won't spark.

b) Light won't come on when button is pushed.

c) Good solenoid valve doesn't open when button is pushed.

b. Pilot light goes out.

1) If the gas pressure is too low the flame may blow out. Check the gas pressure regulator and adjust if necessary.

2) The Firetron scanner may not be working properly and if not, the solenoid valve in the gas line will close when the start button is released. The scanner element has a small electric eye cell which can easily be replaced if necessary. Also after long periods of not being used, the receptacle that houses the scanner fills with rust or debris and obscures the flame from the eye. This item should always be checked on pre-start maintenance.

c. Maxon valves do not open.

1) Main burner Factory Mutual plugcock is in open position or air line from F.M. plugcock is plugged which acts the same as if valve were left open--check F.M. plugcock and air line through plugcock.

2) Press start reset button again.

3) Check to see if one of the emergency shutdown conditions exists.

4) Wiring difficulties exist in Firetron controls. Check fuses.

5) If one Maxon valve opens and the other doesn't, then the one that doesn't open is malfunctioning. Use care under these circumstances that the bleed valve between the two Maxon valves is not still open and discharging gas.

d. Main burner flame does not ignite.

1) Remember to open main burner plugcock slowly.

2) Burner air intake could be out of adjustment making flame difficult to start.

3) Burner gas orifices could be plugged with rust or other debris. This should always be included in the prestart maintenance check list.

4) Gas pressure could be too low.

a) Pressure bleed and vent between the two Maxon valves could be partially open which would prevent full pressure build-up.

b) Gas pressure regulator needs to be adjusted to give higher pressure.

e. High tube wall temperature.

1) Loss of circulation pump.

2) Incorrect line-up of valves blocking circulation.

3) Circulation pump not operating correctly.

If an emergency shutdown of the heater occurs, locate and correct the problem. The Firetron panel has a built in purge timer which requires that 5 minutes elapse between a shutdown and startup. Always thoroughly test the heater with an explosimeter.

During the heat-up of the reactor, the Dowtherm temperature, tube wall temperature, and stack temperature should be closely followed. Some adjustment may be required on the flow to the four passes so as to maintain an equal set of tube temperatures. Also, expansion of the Dowtherm and flashing-off of dissolved gases may cause increasing pressure on the Dowtherm system. Closely follow the Dowtherm pressure.

After the reactor bed reaches 450°F, shut down the main burner by closing the main burner gas plugcock valve. After burner shutdown, the heater may be isolated from the Dowtherm circulation. The main burner should always be shut down <u>before</u> the circulation is isolated from the heater.

D. Reactor Startup

N_2 on O_2 line's to not go through heater.

The actual startup of the OHC reactor with feed introduction must be based upon the basic criteria for the operation of the plant; that is, the system, at <u>all</u> points, must be operated with non-explosive mixtures. Further, this type of operation will require very thorough checks and rechecks during all phases of operation. As described previously, the criteria for determining whether the mixtures are safe is the oxygen content--the concentration of oxygen must be maintained at no more than 8-9%.

During the early stages of the reactor startup, the main vent stream will be through the MLS vent valve immediately downstream of the primary condenser. A small flow may go forward through the condensers and to the normal vent scrubber system. The reactor pressure controller should be placed on automatic control. The set point should be adjusted to the pressure desired on the system ([redacted] 5 psig). Since the oxygen analyzer and gas chromatograph both sample downstream of these vents, it will be necessary to obtain samples and analyze with the Fischer Gas Partitioner at this main vent point to determine oxygen content.

The detailed reactor startup procedure is as follows:

Blocked out Blocked out

1. The Chloral treatment system, drying still, and lights still should be in operation and leveled out. Startup of this system is described later in this section. Recycle system operating on N_2

2. The degasser should have a liquid level in it so as to provide a positive seal. All equipment downstream of the degasser should be nitrogen purged and ready for operation.

3. The reactor condensers should have cooling tower and refrigerant flow as required. The primary and secondary condensers should also have slow well water flows to the tubeside (flush water).

4. The reactor bed temperature should be at [redacted] 425°F.

5. The condensate tank should have already been filled from the main Area "B" condensate header. The steam drum can be filled with the OHC condensate pump.

6. The Dowtherm flow should then be lined up to go through the Dowtherm cooler. The Dowtherm heater should already be by-passed and shut down. The Dowtherm temperature set point should be set above the Dowtherm temperature so as to prevent cooling until desired. The steam pressure controller should be placed on automatic with a 35 psig set point. Also, put the condensate make-up level controller and feed pump in service.

7. At this point, the bed will be fluidized with fluidization air in the windbox and nitrogen in the oxygen header. The air must now be

completely purged from the reactor, condensers, and all associated lines and vessels. This is accomplished by replacing the air with nitrogen. The nitrogen must be slowly phased in as the air is phased out so as not to upset the fluid bed. The N_2 and air feed rates should be adjusted so as to maintain a constant total feed rate.

— Recycle system in, chromatogram + Hayes O_2

8. After completion of the phasing out of the air, all points in the system must be purged and tested until no oxygen (less than 0.5%) is detected. The startup cannot proceed until this step is thoroughly and completely executed. Water-filling of some of the system may be necessary to accomplish this.

Blow down centrifuge catch pot

9. The HCl system should be completely purged with N_2. The HCl feed can now be started. This is done by contacting the HCl lead operator (foreman, if on day shift) to get the HCl compressor started. The HCl personnel should also have been given adequate notice of startup plans and anticipated startup time. There are three compressors (one for each reactor and a spare). The HCl compressors can be started on total recycle. After they have been started and lined out, the line to OHC should be pressurized up to the Hi-Sil Filter. Flow should then be very slowly started through the filter so as to prevent slugging the filter and possible Hi-Sil carryover. HCl flow should then be slowly started through the HCl heater and into the reactor.

10. During the initial slow HCl feed rate, start steam flow to the HCl heater. Put the temperature controller on automatic and adjust the set point at 250°F after first manually increasing the temperature to 250°F. This temperature is absolutely necessary in order to insure temperatures at the distributor plate <u>above</u> the condensation temperature during startup.

11. Slowly increase the HCl flow, while decreasing the nitrogen flow a corresponding amount, until the HCl flow is 33,700 SCFH. This corresponds to 5 psig pressure operation, which is the minimum rate required for good distribution across the distributor plate. During the addition of HCl feed, the reactor top pressure should be raised slowly to 5 psig by manually throttling the vent flow off the primary condenser to the stack scrubber. The set point on the normal vent pressure controller should be set at 5 psig and allowed to do the actual pressure control automatically. The gases from this PCV pass to the vent scrubber. Put N_2 on stack + vent scrubber

12. Ethylene may now be introduced to the reactor. The entire feed system must be purged with nitrogen. The feed header should be pressurized up to the pressure control station located at the OHC plant. A slow flow should then be started to the reactor through the heater. The ethylene pressure controller should now be put in operation. Steam flow to the heater should be started and the temperature controller put on automatic and lined out at 250°F. This temperature must be maintained to prevent condensation at the feed point. The ethylene flow may now be slowly increased, with a corresponding decrease in nitrogen flow, until the ethylene flow is 35,200 SCFH. Add N_2 at 5600 SCFH This is equivalent to the sum of the ethylene and recycle flows at 5 psig rates. ~~At this point all the nitrogen flow should be stopped to the mixed feed header.~~

13. The final feed, oxygen, may now be introduced. The approximate final startup rate is 9200 SCFH; however, the initial oxygen flow will be about 1/2 of this value. 4600 SCFH The O_2 heater should also be put in service and lined out at 250°F outlet O_2 temperature. As the oxygen is introduced, reaction will start to take place and the reactor temperature should

Let temp line out. Note ΔT. Set temp controller on D.T to give 525°F 5-1407 full flow.

start to increase. ~~Maintain a slow oxygen feed rate increase until the reactor temperature reaches 525°F.~~ The vent stream must be checked during oxygen addition for oxygen content. The presence of any large oxygen content at this point should be thoroughly investigated before proceeding.

As oxygen is introduced to the reactor, the vent volume will start to decrease because of reaction to form EDC. As the EDC condenses the system pressure will decrease. The vent flow from the primary outlet should be manually decreased as soon as possible, but maintaining a constant reactor pressure of 5 [redacted].

14. Continue increasing the O_2 flow while checking the vent composition, up to 9200 SCFH. As soon as a measurable oxygen content is present in the vent, check for HCl utilization. At this point, the vent from the primary condenser should be completely closed. All of the vent gases are now passing through the condensing system. The oxygen flow should now be adjusted to give good HCl utilization, using the criteria described in the reactor operation section.

15. The recycle gas scrubber system must be put in operation before introducing reactor gases to the recycle system.

done before startup

a. Fill the $NaHCO_3$ surge tank with dilute cell liquor to about three-fourths level. The tank can be filled by blocking the discharge of the bicarb. circulation pumps and opening the valves into the scrubber. Dilute cell liquor can now be fed to the scrubber and will overflow to the $NaHCO_3$ surge tank. Note: Filling of the tank will be done before heat-up of the reactor. Bottled CO_2 can be added at this time to create a $NaHCO_3$ solution, thus avoiding removal of the CO_2 from the recycle stream immediately upon start-up.

b. After filling the tank, start the bicarb. pumps and establish circulation in the scrubber system. Put the flow controller on automated with about 7-8 gpm flow. (This flow is necessary to insure wetting of the packing.)

c. Put the pH meter, controller, and valve in service. Put the controller set point on a pH of 5.5.

d. Establish a slow makeup of caustic and condensate to the system.

e. Put the bicarb. surge tank level controller in service and put the set point at the tank level.

f. Start a N_2 purge (50% of the rotameter) to the C_2H_4 degassing tank. Check the steam snuff line to be sure steam is available, but keep flow blocked.

16. The recycle compressor and associated control system will be started up on nitrogen. The following pre-startup sequence will be followed:

a. Purge the recycle system using the nitrogen inlet located downstream of the normal reactor vent purge take-off. The valves in the recycle system should be opened to allow nitrogen flow through the scrubber, around the compressor through the safety relief valve bypass, through the surge drum and recycle superheater, up to the block valve at the mixed feed header inlet and out through the startup vent line to the vent scrubber. A purge stream should be taken from the bottom of the compressor entrainment separator and surge drum to sweep oxygen out of this system. The recirculation piping around the compressor should also be purged.

b. During the recycle purge with nitrogen, the oxygen analyzer and gas chromatograph should be put in service. The chromatograph analyzer requires several hours warmup time; therefore, advance notice should be given to the appropriate instrument personnel. The purge should be continued until the oxygen content is less than 1%. Possible dead spots, such as the entrainment separator and cooler line, should be checked with a portable oxygen analyzer. It will be necessary to raise the recycle system pressure to above 2 psig to get sufficient sample pressure for the on-stream analyzers. This may easily be done by restricting the flow to the vent scrubber.

17. Recycle compressor startup:

a. Check oil level in the compressor crankcase.

b. Start cooling water flow to the compressor head.

c. Start cooling water flow to the recycle cooler.

d. Put nitrogen pads on the distance pieces and adjust bleeds to give good pressure.

e. Put the recycle gas pressure transmitter in service and put the recycle pressure controller on manual with the PCV fully open.

f. Close the bypass around the safety relief valve.

g. Start the compressor. Check bearing and head temperatures. Also, note any excessive noise from the compressor.

h. Set the pressure controller set point at a low setting and put the instrument on automatic. The valve should be nearly fully open.

18. The recycle compressor system should now be operating essentially on recirculation, except for the nitrogen purge stream passing through. This operation should be continued until the system is completely

purged. The stream being vented to the scrubber should be checked at the drain valve at the flow control valve with a portable oxygen analyzer. This concentration should be less than 1% oxygen before proceeding.

19. Put the recycle superheater in service and manually raise the temperature to 250°F. Then put the controller on automatic.

20. The recycle pressure at the inlet to the recycle gas scrubber should be adjusted to match the pressure at the normal reactor vent point. The scrubber inlet pressure is about the same as the compressor suction pressure, which can be adjusted by raising (or lowering) the set point discharge pressure.

21. As soon as the pressure on each side of the valve separating the reactor gases from the recycle system are equal, the remotely operated valve should be opened. Slowly cut off the nitrogen purge; recycle gas will be automatically pulled in to maintain compressor suction pressure. The pressure control valve on the reactor vent line to the stack scrubber will gradually close to maintain reactor pressure. Part of the gases which were vented at the pressure control vent will now be vented through the recycle system to the vent scrubber. This flow should be greatly throttled by the recycle feed flow control valve.

22. Open the block valve (slowly) separating recycle gas from the mixing jet and close recycle vent valve simultaneously. Slowly increase the recycle flow to the reactor by further opening of the recycle feed FCV, while decreasing the ethylene feed rate a corresponding amount. The oxygen content in the system will now begin to increase due to the recycled oxygen. The oxygen level should be maintained at no more than 3% until all the recycle gas flow is going to the reactor; this may

require slightly increasing ethylene feed rate or lowering oxygen feed rate during the phasing in of recycle gas. Under no circumstances should additional recycle be added when the oxygen level is above the set limit. The flow rates and reactor pressure must be maintained at the values required for proper fluidization.

23. After completion of phasing-in of the recycle gas, the oxygen and ethylene flows should be adjusted as described in the "Reactor Operation" section. HCl utilization and ethylene conversion will be the primary considerations as long as the gas composition is acceptable from a safety standpoint.

24. Put a N_2 flow through the recycle purge system to the scrubber. Establish a 50% flow on the rotameter.

25. Place the oxygen flow controller on "Cascade" control after startup. During and after startup of the reactor, the condensing load on the reactor condensers will be changing. Water flow to the first two reactor condensers should be adjusted to give sufficient condensate for cooling of the graphite. The well water flush to the heads should also be adjusted as needed. The water flow to the tertiary condenser should be adjusted to give a cool enough gas to the refrigerated condenser to allow good control. This refrigerant flow should be on automatic temperature control, with the set point of the exit gas at $60^{o}F$. Well water flow to the compressor recycle cooler should be at a high rate at all times.

E. Lights Still Startup

The following startup sequence should be followed:

1. The lights still, reboiler, condenser, reflux drum, reflux drier, and all associated piping must be dried and then purged with nitrogen until the O_2 concentration is less than 1%.

2. The feed tank should also be purged with nitrogen and oxygen-checked. The nitrogen pad system should then be put in service. The feed tank should be filled to about 3/4 full of EDC.

3. A 3/4 level of EDC should be established in the reflux drum. The nitrogen pad and vent should be put in service on the reflux drum.

4. The reflux driers should now be filled with EDC so that later startup will go smoothly. This filling operation must be done slowly to prevent surging the calcium chloride bed and possible calcium chloride carryover. The reflux pump should be started and the inlet valve to the drier gradually opened. Then open the outlet valve from the drier. The outlet valve from the filter should remain closed. The inert gas should be very slowly bled out of the drier-filter by opening the small vent on top of the filter until EDC overflows. The sight-glass on the drier also will indicate when the drier is full. The pump should then be shut down.

5. A circulation of EDC through the lights still should now be established. The path of flow should be from the feed tank, through the feed pumps, to the still, and out the bottom of the still through the bottoms pump and bottoms cooler. This stream should then be recycled to the suction of the feed pumps by blocking off flow to the tanks and using the 2" recirculation line. The still feed rate should be set at or below the anticipated actual feed rate.

At this point, the reboiler level transmitter indicated level should be compared to the sight glass reading. If the two levels agree, the reboiler level controller should be put in service on automatic control.

6. Cooling water flows should now be established to the condenser, reflux cooler and bottoms cooler.

7. The still is now ready for heat-up. The steam condensate flow should be blocked from the condensate collection system and opened to the pad. After all residual steam condensate has been drained to the pad, very slowly start steam flow to the still (the flow control valve may be used if it does not leak through--otherwise, the bypass should be used initially). During the initial startup, or following reboiler maintenance, trapped air in the steam jacket may have to be bled off.

The still temperature profile should be observed during the heat-up; the steam should be flow-controlled to give a moderate rate of temperature rise. The reboiler level should be watched closely and any steps necessary to maintain the proper operating level must be taken (change steam flow, feed flow, etc.). The reboiler design calls for the liquid level in the column to be at the top tube sheet of the reboiler.

8. Overhead vapor flow will be indicated by a temperature rise at the top of the still. The reflux pumps should be started when the level starts to increase in the reflux drum. Flow should be established through the reflux cooler-drier system and the reflux drum level controller put in service. All of the overhead product should be recirculated back to the suction of the feed pumps through the 1 1/2" circulation line provided.

9. The steam flow, feed flow, and reflux flow should be adjusted to match the feed rate and content expected to be fed to the still. The condensate flow may be directed to the condensate collection system at any time after the reboiler is hot. Care should be taken in this switch-over, since the reboiler chest pressure may be initially below the condensate system pressure.

F. D-H Still Startup

The D-H Still may be started after the lights still has been put in operation and leveled out. The following startup sequence should be followed:

1. The entire D-H still (including reboiler, condenser, feed tank, reflux tank and all piping) must be nitrogen-purged until free of oxygen (less than 1%).

2. Establish on adequate feed tank level--this feed should be relatively dry, since during the initial phase of startup the reboiler will be in contact with the cold feed. Dry EDC may be brought back to the still through the rework header from the storage tanks.

3. The vent line from the condenser-reflux drum to the refrigerated vent condenser should be put in service.

4. EDC flow through the D-H still may now be established. The flow should be from the feed tank through the feed pumps to the still, out the bottom take-off, through the bypass around the drier, and through the rework line back to the feed tank. Care must be taken to make sure this section of the rework line is isolated from its other tie-ins. The feed rate should be set at or below the anticipated feed rate. The reboiler level controller should be put on automatic control with the set point at the level of the top of the tube sheet.

5. Start cooling water flow to the condenser. Put well water flow to the reflux drum seal loop.

6. The still heatup may now be started. Isolate the steam condensate flow from the main condensate collection system and open the flow to the pad.

Start steam flow to the reboiler at a very low rate until the temperature profile begin to come up. Keep increasing the steam flow until the boilup rate is at the design value of 50%. The necessary steam rate for 50% boilup of any feed rate is given in Fig. 10-6. The reflux flow is by gravity flow and is self adjusting; however, well water flow must be put into the seal loop off the reflux drum to insure a seal initially; this seal should be checked periodically.

The D-H bottoms drier should be put in service after the still is lined out. The flow should be very gradually introduced to the drier and filter while bleeding inerts off the filter. The sight glass on the drier top will indicate when the drier is full and EDC flow out the filter will then indicate that the filter and drier are ready for full service.

The D-H bottoms drier should be put in service after the still is lined out. The flow should be very gradually introduced to the drier and filter while bleeding inerts off the filter. The sight glass on the drier top will indicate when the drier is full and EDC flow out the filter will then indicate that the filter and drier are ready for full service.

G. Chloral Treatment System Startup

The following startup sequence should be used on the chloral treatment system:

1. Purge the chloral treatment tank, phase separator, and associated lines with nitrogen and check for oxygen.

2. Fill the caustic storage tank with a 50-50 mixture of cell liquor and condensate.

4. Start the flow of EDC from the intermediate crude tanks to the treatment tank. Let the tank overflow into the phase separator. Put the interface level controller on manual and line up the crude EDC flow from the phase separator back to the intermediate crude tanks through the rework line.

5. After establishing a circulating flow (the flow rate should equal the anticipated feed rate), the treatment tank agitator should be started. This agitator should <u>never</u> be operated without an overflow level in the tank.

6. Start caustic flow to the treatment tank. Adjust the caustic flow to give the specified (20%) excess of caustic. This will be equivalent to an 18:1 ratio of EDC to caustic. See Fig. 10-5.

7. After caustic addition, two phases will appear in the phase separator. The interface level should be adjusted to a 50% set point and the instrument put on automatic control.

8. Check for chloral content in the product. Do not proceed to the next step without a chloral-free product.

H. System Tie-In

After startup of the purification train and subsequent reactor start-up, the plant startup will be coordinated in the following sequence:

1. As the level in the intermediate crude tanks begins to rise, start forward feed to the DH feed tank. This is accomplished by opening the appropriate valves to the OH feed tank and gradually decreasing the recirculation flow from the chloral treatment system at the same time.

2. The forward flow from the D-H still is achieved in a similar manner. Decrease the D-H still recirculation rate and open the block

valves downstream of the rework take-off from the bottoms line to allow for a forward flow to the lights still feed tank. The feed temperature may change, so appropriate steam flow changes should be made.

3. Part of the D-H still product flow may go to the Per-Tri feed tanks. Start cooling water flow to the D-H still bottom cooler. Then direct flow to one of the Per-Tri feed tanks and shut off all recirculation flow around the D-H still.

4. The level in the lights still feed tank will now begin to rise. The lights still recirculation rate should be decreased to maintain a fairly constant feed tank level. The product valves should be arranged simultaneously to allow flow to the product tanks. Again, the feed temperature may change and steam changes may be made.

5. The overhead recirculation flow can also be cut off. At the same time, open the product line to the Per-Tri feed tanks.

I. York Refrigeration Unit

1. Compressor: The following steps should be carefully checked before starting the system for the first time or after a prolonged idle period:

a. Be sure the control center is energized and that the compressor oil heater has been hot for at least twelve hours.

b. Check the oil level of the compressor and the lubrication of the purge unit and the auxiliary pump motor.

c. Check the setting of the evaporator pressure cutout with a vacuum pump; it should be set to function as follows:

Refrigerant-11 - open at 24" Hg vacuum; close at minimum differential.

d. Check the setting of the condenser pressure cutout with air pressure; it should be set to open at 15 psig.

e. Open wide the water valves to the compressor oil cooler.

f. Be sure the prerotation vanes are closed. If they are not already closed, they should close immediately when the control center is energized. Move the pneumatic control auto-man. switch on the control center to the MANUAL position.

g. Move the (AOP) switch to the HAND position and run the auxiliary oil pump for a short time (not more than 30 seconds) to establish a stable oil pressure. The oil pressure gauge should register a pressure of approximately 35 to 38 psig above the system standby pressure. Then return the (AOP) switch to the AUTO position, and it should remain in the AUTO position for normal automatic operation.

h. Open the brine valves to and from the cooler, start the brine pumps, and vent the cooler.

i. Open the water valves to and from the condenser, and vent the condenser.

j. Start the compressor by turning the COMPRESSOR ON-OFF switch to on.

k. Manually open the vanes (a small amount at a time) as necessary to pull the system down to design temperatures. NOTE: A slight rapid oil pressure fluctuation may be noticed, (5 to 10 psig) with the unit and the (AOP) operating. This takes place only when the prerotation vanes are closed and is a normal occurrence caused by refrigerant separating from the oil in the (AOP) suction. This action should cease when the vanes are opened.

l. When the system is lined out, put the pneumatic controller on automatic.

m. Operate the purge unit as necessary to clear the system of any non-condensable gas (purge system).

2. Purge Unit: Since the purge unit compressor motor is energized through the control center, the control center must be energized before the purge unit can be started. The oil separator heater is always energized when the control center is energized.

To start the purge unit refer to Fig. 4-4 and proceed as follows:

a. Be sure the purge compressor suction and discharge service valves are wide open.

b. Open the following seal capped valves on the cooler-condenser shell:

1) Liquid refrigerant to purge drum (C) - open only a half turn.

2) Recovered refrigerant to cooler (B).

3) Non-condensable gas from condenser (A).

4) Air stop valve (E).

c. Move the ON-OFF switch on the control center to ON to start the purge compressor.

d. The purge compressor will cycle on and off, under control of its high pressure cutout, until the ON-OFF switch is manually moved to OFF.

e. Close stop valve (E) when the unit is down.

VI. OHC PLANT SHUTDOWN

Shutdown of the OHC plant will be necessary for major maintenance jobs and emergency situations. During routine shutdowns, the following sequence of major equipment shutdown may be followed: Reactors, chloral treatment, lights still and D-H still.

A. Reactor Shutdown

The major operating points to maintain during the reactor shutdowns are:

1. Non-explosive atmosphere in the vessels and lines.

2. Adequate gas velocities in the reactor for good fluidization.

3. Reactor pressure changes and feed changes should be slowly and evenly made to prevent excess catalyst carryover. The reactor or Teflon-lined pipe is not designed for full vacuum; use N_2 to avoid this condition.

The general scheme of reactor shutdown will be the replacement of all feeds with nitrogen to purge oxygen, HCl, and organics from the system. The following shutdown sequence is recommended:

1. Gradually decrease reactor rates to the minimum (2 psig) rate. During this rate cut, follow the standard procedure of reducing oxygen rate <u>first</u>. Then reduce C_2H_4, HCl, and recycle, respectively. Maintain a continuous monitor of the vent composition. Put the oxygen flow controller on "automatic" rather than "cascade" when lowering rates.

2. After reaching the minimum rate, replace the recycle feed to the reactor with ethylene. This is accomplished by decreasing the recycle flow rate and increasing the ethylene feed to the values shown in Table 10-1. When the recycle flow is almost down, close the block valve on recycle to the reactor.

3. The recycle compressor and associated equipment should now be isolated from the reactor and nitrogen-purged before compressor shutdown. First, open the flow control valve on the recycle vent to the vent scrubber at the compressor discharge. The reactor PCV should close back automatically to maintain the reactor pressure. Then remotely-operated flow control valve on the recycle gas flow to the recycle gas scrubber should be closed next. Nitrogen flow should now be introduced to the recycle system through the nitrogen flow control valve at the inlet of the gas scrubber. As the recycle system is isolated from the reactor system, the normal vent flow rate will increase as the pressure control valve automatically opens. Steam flow to the recycle heater should be stopped at this point.

The recycle system should then be purged by further opening of the recycle vent flow control valve, thus increasing the flow to the vent scrubber. This should be continued until the analyzers indicate no oxygen. Then shut down the compressor and block off the nitrogen and vent flows. The system should be left under several pounds positive pressure.

4. The oxygen flow should now be replaced with nitrogen in a corresponding amount. As this is done, it may become necessary to partially open the vent valve downstream of the primary condenser in order to keep the automatic pressure control valves in an operating range. It will also be necessary to add more water to the vent scrubber due to the increased HCl load. The nitrogen flow should be put in upstream of the oxygen heater and heated to 250°F before entering the reactor. This is necessary because residual EDC and water may still be present in the reactor.

5. As the oxygen flow is shut down, the reactor will stop evolving heat. As soon as the steam pressure control valve becomes almost closed, the Dowtherm cooler should be bypassed and steam PCV blocked off.

6. Ethylene flow may now be replaced with nitrogen in a corresponding amount.

7. Replace HCl flow with nitrogen in a corresponding amount. Purge for at least 30 minutes. At this point the three feed heaters may be shut down.

8. If the reactor is to be shut down for a sustained period of time, the fluidization air blowers should be started up and used to replace the nitrogen flows. The system must be completely free of ethylene and EDC. Then the nitrogen is phased out as a corresponding amount of air is phased in.

9. The Dowtherm circulation can be shut down at any time after stopping oxygen flow; however, this flow should be stopped as soon as possible if restartup is imminent, since the bed must be hot for startup. If the reactor is to be restarted soon, then start circulation through the Dowtherm heater.

B. Chloral Treatment System Shutdown

As the reactors are shut down, the flow to the intermediate crude tanks will stop. The chloral treatment system should then be shut down as follows:

1. Shut down the chloral treatment feed pumps.
2. Stop caustic feed pumps.
3. Stop treatment tank agitator.
4. Shut down crude EDC transfer pumps.
5. Leave N_2 purges on at low rates.

C. Lights Still Shutdown

The D-H still may be left operating while the lights still is shut down. The lights still should be shut down as follows:

1. Decrease feed, steam, and reflux rates until the feed rate is low.

2. Stop steam, feed, and reflux flows. Then shut down the bottoms and reflux pumps.

3. Leave nitrogen pad in service.

D. D-H Still Shutdown

The D-H still must be shutdown on dry EDC in order to insure that no water or HCl comes in contact with the reboiler or is carried into the product. The following sequence should be used.

1. Start up the VCl feed pumps (if not already on line) and open flow into the rework line. Open this flow into the suction of the D-H feed pumps. Close off flow from the D-H feed tank to the pumps.

2. Continue operation of the still until the still overhead temperature approaches the EDC boiling point (184^{o}F).

3. At this point, reduce steam and feed flows to a low rate and then shut down. Stop bottoms pump.

4. Block off rework line and stop VCl pump (if not being used for the liquid phase EDC plant).

5. Block off vent flow to vent condenser. The flow from the vent condenser should not be blocked off unless the N_2 purges on the intermediate crude tank are stopped. This should not occur under normal shutdown conditions.

E. Refrigeration Unit Shutdown

This unit may be shut down at any time after the reactor shutdown.

1. Turn the compressor ON-OFF switch to the off position.

2. The auxiliary oil pump is automatically energized and runs for 1 1/2 minutes. The system cannot be restarted during this time.

3. At this time, the reservoir oil heaters are automatically energized.

4. If the compressor is to be down for any length of time, shut off the cooling tower water to the Freon condenser. Also, shut off the water to the oil cooler. Shut off and isolate the purge unit.

VII EMERGENCY SHUTDOWN PROCEDURES

Several emergency situations could occur in the OHC plant. In general, the emergency shutdown system (push-button) should be used very sparingly. The corrective procedures to be followed will vary with the situation.

A. Loss of HCl Feed

One of the more probable emergency situations is the loss of an HCl supply compressor. Two immediate effects will occur in the OHC plant:

1. Gas velocity will decrease below the specified value required for fluidization.

2. The reactor off-gases will go into the explosive range.

To prevent the second condition, the reactor is equipped with an HCl to O_2 ratio controller. This instrument should automatically decrease oxygen flow to zero in the event of an HCl loss. An additional automatic instrument starts nitrogen flow to the oxygen header when the oxygen flow goes below 4000 SCFH. The following sequence should be followed in this situation:

a. Check to make sure the above described automatic controls have functioned.

b. Put nitrogen flow into the HCl header upstream of the heater.

c. Shut off the ethylene and recycle flows with an increase of a corresponding amount of nitrogen. The final nitrogen flow to the mixed feed header should be 54,700 SCFH with a reactor pressure of 0 psig.

d. It will be necessary to open the vent valves off the primary condensers to lower the reactor pressure.

e. If only one HCl compressor is lost, it may be possible to compensate by merely decreasing rates.

B. Loss of Recycle Flow

This is another of the more probable emergency situations. The loss of recycle will have similar effects as loss of HCl, but the problems should not be as severe; however, definite corrective action must take place.

1. Add ethylene flow equivalent to the lost recycle flow. This step should put the safe operating range.

2. Block off recycle tie-in to mixed feed header.

3. Find and correct the cause of the compressor kick-off or line up the spare compressor.

4. The recycle compressor should be restarted on nitrogen using the technique described in the reactor startup section.

C. Loss of Oxygen Flow

Loss of oxygen from the suppliers is a very remote possibility since we have dual suppliers. A malfunction of the pressure control valve on O_2 to OHC could cause temporary loss of flow. In any case, the nitrogen flow should automatically start to the oxygen header. The operator should check this flow and take any measures necessary to get oxygen flow restarted. Shut off the recycle flow immediately. Also, the reactor pressure may start increasing, making it necessary to open the vents off the primary condensers.

D. Loss of Ethylene

Loss of the ethylene feed is also a remote possibility; however, loss of this stream is a serious circumstance and corrective action must be immediately taken. Loss of fluidization velocity may occur and the EDC-rich reactor off-gases may go into the explosive range. <u>Oxygen flow must immediately be replaced with nitrogen</u>. Shut off the recycle flow. Nitrogen flow must be started to the ethylene feed header to maintain fluidization due to the loss of both ethylene and recycle. The reactor pressure should be lowered as fast as practical; the feeds should be correspondingly adjusted.

E. Loss of Nitrogen

Again, this loss is a very remote possibility. This, also, is a critical loss since it is the primary back-up stream for fluidization and purging. It should be remembered that ethylene can be used for both fluidization (in mixed feed header) and purging if the oxygen level is in the acceptable range. All nitrogen purges and sweeps should be shut off immediately, even though there are check valves in the nitrogen lines.

F. High Oxygen Levels

Both the O_2 analyzer and the gas chromatograph are equipped with high alarms on oxygen content in the recycle. This alarm will be set just above the normal operating range. In this way, the alarm will give the operator good advance notice before the system reaches the real danger zone. Under no circumstances should the oxygen content be let to exceed 9%.

The quickest response on oxygen content will be the result of raising of the ethylene flow. A decrease in the oxygen flow rate will also help, but the effects from this are not so drastic as the ethylene flow increase.

The results of the analyzers may not agree at some times. For safety sake, the analyzer indicating the highest oxygen will be believed until proven otherwise. Orsat or Fischer gas partitioner analyses should be run to determine which instrument is correct. Maintenance personnel should then be called in to correct the malfunctioning instrument.

High reactor temperatures with corresponding high vent rates will be grounds for cutting oxygen flow. The extent of the cut will depend on the exact situation.

G. Loss of Feed Heaters

The primary potential problem in the loss of one of the feed heaters is the possible condensation (and subsequent corrosion) in the reactor. The most probable cause of heater loss would be malfunction of the steam trap. In such a case, bypass the trap until it can be fixed. Malfunction of the temperature controller or steam valve can be temporarily compensated for by going on manual on the instrument or bypassing the control valve, respectively.

H. Loss of Cooling

Loss of cooling may occur from two sources; loss of cooling tower water pumps and loss of Dowtherm circulation pumps. Loss of either should immediately be counteracted by high well water flush rate to the primary coolers. The cooling water pumps are equipped with a water jet ejector operated off well water. This ejector will give very quick priming of the pumps.

Loss of the Dowtherm pumps may be from electrical problems or cavitation. If the Dowtherm flow cannot be established immediately, the oxygen flow to the reactor should be immediately replaced with nitrogen until Dowtherm circulation can be established. Also, shut off the recycle flow to prevent HCl from getting into the system. A low flow alarm on the Dowtherm circulation rate will be the first indication of trouble.

I. Leaks in the Reactor Gas System

Leaks from the gas system can be dangerous because of the possible combination of the flammable gas and static electricity generation. This situation is worsened by any entrained catalyst particles because they add to static electricity generation. In the event of a leak, the leaking section must be isolated and purged with nitrogen before repairing. This philosophy is true no matter where the leak is; for example, the reactor must be shut down (feeds off, not defluidized) if a leak develops in the overhead piping or condenser system. It may be possible to repair very small leaks temporarily by reducing reactor rates to give low pressure. This temporary approach must not be taken unless confirmed by the foreman.

J. Power Failure

One of the first considerations in the event of a power failure is to start a nitrogen flow to the mixed feed header to insure that good fluidization is maintained and to help cool the reactor down. The ethylene and HCl feeds will automatically shut down, as will the recycle system. The vent valves will open automatically, so the reactor pressure should drop to approximately 0 psig.

Probably the most serious hazard is created by the loss of Dowtherm and cooling tower water. The Dowtherm pumps should be restarted as soon as power is restored. This also applies to the cooling tower water pumps unless there is quite a delay before power is restored. In this case, the temperature shock would be too great on the heat exchangers, particularly the karbate ones. In any case, all heat supplied to the plant should be shut down automatically due to the fail-safe operation of the feed and steam valves.

K. Instrument Air Failure

The plant has been designed with all valves to fail in the "safe" position during loss of air (or power). The reactors should be checked to be sure they have adequate nitrogen for fluidization. Steam flows should be blocked to the still reboilers. The system should then be prepared for restartup when air pressure is regained.

L. Steam Failure

The main problem concerning steam is failure of the steam make-up automatic PCV's from the 175 psig header to the 30 psig header. If the 30 psig steam header should suddenly decrease, check the following:

1. Steam drum PCV's. If these fail, the steam drum safety relief valves will be blowing. Open by-pass to maintain 35 psig on steam drum.

2. Steam to steam silencer PCV. This may be stuck open. Close the block valve.

3. Make-up Steam PCV's. These may be stuck closed or cannot open fully. Open the by-pass valve to control the 30 psig header pressure.

VIII. SPECIAL PROCEDURES

A. Catalyst Charging

The best estimate from pilot plant experience is that each reactor will lose about 400 lbs. catalyst per day due to fines loss to the Centrifix. It will vary with rate because at higher pressure the denser reactor gas will have more buoyancy for the catalyst particle. One catalyst charge pot load per day should suffice until we learn more.

Catalyst will be added to the reactor from a portable charge pot. The procedure for charging the reactor is as follows:

1. Fill the pot with one drum of catalyst. Be sure that the correct catalyst is used. Secure the lid.

2. Hoist the pot to the upper deck and connect it to the reactor charge valve.

3. Pressurize the pot twice with nitrogen with a slow bleeddown to drive out air.

4. Pressurize the charge pot to about 20 psig above the reactor pressure (do not exceed the 60 psig relief point on the safety relief valves).

5. Open reactor valve first, then the valve on the bottom of the pot. Most of the catalyst will now flow into the reactor. Avoid continued blasting with nitrogen because this may affect reactor pressure control.

6. Record the amount of catalyst added.

7. Leave a dry N_2 pad on the charge pot when not in use.

8. Blow catalyst out of the hose and reactor valve inlet and wash up any spillage in the area.

B. Catalyst Dumping

The initial frequency for dumping the Centrifix-catchpot system will be twice per shift. This may be altered if deemed necessary after startup. The following dumping procedure should be followed:

1. Close the special ram-type valve on the bottom of the Centrifix.

2. Pressurize the catch-pot with N_2 and slowly bleed the pressure down. Repeat this as many times as required to clear the catchpot of all reactor gases.

3. Make sure that a good nitrogen purge has been flowing through the dump line to the waste drum and out to the small seal pot. All air must be removed before dumping the catch pot. Also, any blockage of the vent off the waste drum must be removed.

4. Slowly open the bottom valves on the catch pot (with the nitrogen purge cut off). Repeat the pressure and blowout steps if necessary. The blowdown must be done slowly so as not to rupture the waste drum.

5. After dumping, close the bottom valves on the catch pot, put a slow nitrogen purge into the dump line. Then open the special rod out valve on the bottom of the Centrifix.

6. Any catalyst spillage must be washed down.

PRELIMINARY: OHC ANALYTICAL SCHEDULE

SAMPLE POINT	DESCRIPTION	TYPE SAMPLE	SAMPLE CONTAINER	SAMPLE TAG NOMENCLATURE	SAMPLE POINT PRESS. / TEMP	SAMPLE FREQUENCY	ANALYSIS REPORT	TYPICAL ANALYSIS
S-1	COOLING TOWER WATER PUMP DISCHARGE #1	LIQUID	POLYETHYLENE BOTTLE	S-1 CTW	20 PSIG / 90°F	1 / SHIFT	pH, Cl^-, SiO_2 CONC. FACTOR	pH = 6.5, Cl^- = 450 PPM (AS NaCl) SiO_2 = 200 PPM, FACTOR = 4.5
S-2	WELL WATER COOLING TOWER MAKE-UP	LIQUID	POLYETHYLENE BOTTLE	S-2 W. W.	80 PSIG / 80°F	1 / SHIFT	Cl^-, SiO_2	Cl^- = 100 PPM (AS NaCl) SiO_2 = 50 PPM
S-3	CONDENSATE - FEED PUMP SUCTION	LIQUID	POLYETHYLENE BOTTLE	S-3 COND.	2 PSIG / 180°F	1 / SHIFT	pH, Fe	
S-4	REFRIGERATING BRINE - CIRC. PUMP SUCTION	LIQUID	POLYETHYLENE BOTTLE	S-4 BRINE	10 PSIG / 14°F	1 / SHIFT	pH, SP. GR.	pH = 7, SP. GR. = 1.24
S-5	STEAM DRUM BLOWDOWN No. 1 REACTOR	LIQUID	POLYETHYLENE BOTTLE	S-5 BLOWDOWN No. 1	35 PSIG / 140°F	1 / SHIFT	DISSOLVED SOLIDS, NITRATE, PHOSPHATE, SULFITE, HYDRATE ALKALINITY	DISS SOLIDS = 2000 PPM SODIUM NITRATE = 20 PPM PHOSPHATE = 20 PPM SODIUM SULFITE = 20 PPM ALKALINITY = 200 PPM
S-6	STEAM DRUM BLOWDOWN No. 2 REACTOR	LIQUID	POLYETHYLENE BOTTLE	S-6 BLOWDOWN No. 2	35 PSIG / 140°F	1 / SHIFT	SAME AS S-5	SAME AS S-5
S-7	DOWTHERM - No. 1 Rx CIRC. PUMP SUCTION	LIQUID	BROWN BOTTLE	S-7 No. 1 D.T.	80 PSIG / 300°F	1 / SHIFT	HCl	
S-8	DOWTHERM - No. 2 Rx CIRC. PUMP SUCTION	LIQUID	BROWN BOTTLE	S-8 No. 2 D.T.	80 PSIG / 300°F	1 / SHIFT	HCl	
S-9	No. 1 Rx CATALYST BLOWDOWN	GRANULAR SOLID	WIDE MOUTH BOTTLE	S-9 No. 1 CAT.	ATM / 500°F	1 / DAY	SCREEN ANALYSIS	

PITTSBURGH PLATE GLASS COMPANY
CHEMICAL DIVISION LAKE CHARLES, LOUISIANA

TITLE OHC PLANT - PROCESS SAMPLE SCHEDULE	DRAWN BY DDM	DATE 4-2-68	SCALE	DWG. NO.
	CHECKED BY	DATE	CHARGE	
	APPROVED BY	DATE	BILL OF MATERIAL	SHEET 1 OF 4

POINT		TYPE SAMPLE	SAMPLE CONTAINER	SAMPLE TAG NOMENCLATURE	SAMPLE POINT PRESS./TEMP.	SAMPLE FREQUENCY	ANALYSIS REPORT	TYPICAL ANALYSIS
S-10	NO. 2 RX. CATALYST BLOWDOWN	GRANULAR SOLID	WIDE MOUTH BOTTLE	S-10 NO. 2 CAT.	ATM. / 500 °F	1 / DAY	SCREEN ANALYSIS	
S-11	ACID FROM NO. 1 PHASE SEPARATOR	LIQUID	BROWN BOTTLE	S-11 NO. 1 ACID	ATM / 130 °F	2 / SHIFT	HCl	HCl = 5 %
S-12	ACID FROM NO. 2 PHASE SEPARATOR	LIQUID	BROWN BOTTLE	S-12 NO. 2 ACID	ATM / 130 °F	2 / SHIFT	HCl	HCl = 5 %
S-13	CONDENSED ORGANIC NO. 1 RX. PHASE SEPARATOR OUTLET	LIQUID	BROWN BOTTLE	S-13 NO. 1 ORG.	2 PSIG / 130 °F	1 / SHIFT	G.C. (INC. CHLORAL) H_2O	$CHCl_3$ 0.1 WT % EDC 97.64 WT % CCl_4 0.13 WT % CCl_3CHO 0.27 WT % TCE 1.25 WT % H_2O 0.39 WT %
S-14	CONDENSED ORGANIC NO. 2 RX PHASE SEPARATOR OUTLET	LIQUID	BROWN BOTTLE	S-14 NO. 1 ORG.	2 PSIG / 130 °F	1 / SHIFT	SAME AS S-13	SAME AS S-13
S-15	NO. 1 RX RECYCLE GAS TO RECYCLE SCRUBBER (1)	GAS	100 ml GAS BURETTE	S-15 NO. 1 VENT	3 PSIG / 60 °F	2 / SHIFT	OXYGEN, CO_2 BY ORSAT	OXYGEN = 5.7 % (VOL.) CO_2 = 37.66 % (VOL.)
S-16	NO. 2 RX. RECYCLE GAS TO RECYCLE SCRUBBER (1)	GAS	100 ml GAS BURETTE	S-16 NO. 2 VENT	3 PSIG / 60 °F	2 / SHIFT	OXYGEN, CO_2 BY ORSAT	SAME AS S-15
S-17	BICARB FROM NO. 1 RECYCLE GAS SCRUBBER	LIQUID	BROWN BOTTLE	S-17 NO. 1 BICARB	46 PSIG / 70 °F	1 / SHIFT	pH, DISSOLVED SOLIDS, ALKALINITY	pH - 6.0, DISS. SOLIDS = (2) ALKALINITY = (2)
S-18	BICARB FROM NO. 2 RECYCLE GAS SCRUBBER	LIQUID	BROWN BOTTLE	S-18 NO. 2 BICARB	46 PSIG / 70 °F	1 / SHIFT	SAME AS S-17	SAME AS S-17

(1) SAMPLE DOWNSTREAM OF VENT BACK PRESSURE CONTROL VALVE.

SL 000459

PITTSBURGH PLATE GLASS COMPANY
CHEMICAL DIVISION — LAKE CHARLES, LOUISIANA

TITLE OHC PLANT PROCESS SAMPLE SCHEDULE	DRAWN BY D.D.M.	DATE 4-2-68	SCALE	DWG. NO.
	CHECKED BY	DATE	CHARGE	
	APPROVED BY	DATE	BILL OF MATERIAL	SHEET 2 OF 4

Sample Point	Description	Type Sample	Sample Container	Sample Tag Nomenclature	Sample Point Press./Temp.	Sample Frequency	Analysis Report	Typical Analysis
S-19	Recycle Gas to No. 1 Reactor	Gas	100 ml Gas Burette	S-19 No. 1 Recycle	50 PSIG / 120°F ①	2 / Shift	O_2, CO_2, C_2H_4 by Orsat	O_2 = 6%, CO_2 = 25%, C_2H_4 = 50%
S-20	Recycle Gas to No. 2 Reactor	Gas	100 ml. Gas Burette	S-20 No. 2 Recycle	50 PSIG / 120°F ①	2 / Shift	Same as S-19	Same as S-19
S-21	Outlet from Vent Condenser Drier	Liquid	Brown Bottle	S-21 Vent Cond.	2 PSIG / 5[illegible]°F	1 / Shift	H_2O	H_2O < 50 PPM
S-22	Caustic - Suction of Caustic Feed Pump	Liquid	Brown Bottle	S-22 Caustic	2 PSIG / 80°F	1 / Shift	% NaOH	6% NaOH
S-23	Crude EDC after Chloral Treatment - Pump Suction	Liquid	Brown Bottle	S-23 Crude EDC	2 PSIG / 130°F	1 / Shift	G.C. (Include Chloral)	$CHCl_3$ 0.3 % wt. EDC 97.94 % wt. CCl_4 0.13 % wt. TCE 1.78 % wt. CCl_3CHO 0.000
S-24	Aqueous Effluent from Chloral Trmt Phase Sep.	Liquid	Brown Bottle	S-24 Chloral Effluent	2 PSIG / 130°F	1 / Day	Alkalinity	
S-25	D-H Still Feed Pump Suction	Liquid	Brown Bottle	S-25 D H Feed	10 PSIG / 120 F	1 / Shift	H_2O	H_2O = 0.25 % wt.
S-26	D-H Still Bottoms Pump Suction	Liquid	Brown Bottle	S-26 D H Bot	15 PSIG / 180°F	2 / Shift	H_2O G.C. ②	H_2O < 20 PPM
S-27	P-T Product Drier Outlet	Liquid	Brown Bottle	S-27 P-T Drier	50 PSIG / 180°F	1 / Shift	H_2O	H_2O < 20 PPM
S-28	D-H Still Bottoms Cooler Discharge	Liquid	Brown Bottle	S-28 Bot. Cooler	50 PSIG / 130°F	1 / Shift	H_2O	H_2O < 20 PPM

① This pressure will be reduced, for safety reasons, by a pressure regulator.

② Once / day

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SL 000460

PITTSBURGH PLATE GLASS COMPANY
CHEMICAL DIVISION — LAKE CHARLES, LOUISIANA

TITLE OHC Plant - Process Sample Schedule	DRAWN BY DJM	DATE 4-2-68	SCALE	DWG. NO.
	CHECKED BY	DATE	CHARGE	
	APPROVED BY	DATE	BILL OF MATERIAL	SHEET 3 OF 4

SAMPLE POINT	DESCRIPTION	TYPE SAMPLE	SAMPLE CONTAINER	SAMPLE TAG NOMENCLATURE	SAMPLE POINT PRESS. / TEMP.	SAMPLE FREQUENCY	ANALYSIS REPORT	TYPICAL ANALYSIS
S-29	LIGHTS STILL BOTTOMS PUMP SUCTION	LIQUID	BROWN BOTTLE	S-29 LIGHTS BOT.	20 PSIG/165 °F	1/SHIFT	G.C.	$CHCl_3$ = 0.01 % wt. CCl_4 = 0.01 % wt. EDC = 98.63 % wt TCE = 1.35 % wt.
S-30	LIGHTS STILL OVERHEAD PUMP SUCTION	LIQUID	BROWN BOTTLE	S-30 LIGHTS OVERHD.	2 PSIG/140 °F	1/SHIFT	H_2O G.C.	H_2O < 50 PPM $CHCl_3$ = EDC = CCl_4 =
S-31	LIGHTS STILL REFLUX	LIQUID	BROWN BOTTLE	S-31 LIGHTS REFLUX	50 PSIG/140 °F	1/SHIFT	H_2O	H_2O < 50 PPM
S-32	PER-TRI FEED TANK	LIQUID	BROWN BOTTLE	S-32 PER TRI FEED	2 PSIG/80 °F	AS NEEDED	H_2O, Fe, G.C.	H_2O < 50 PPM Fe nil $CHCl_3$ = EDC = CCl_4 =
S-33	VCl FEED TANK	LIQUID	BROWN BOTTLE	S-33 VCl FEED	2 PSIG/80 °F	AS NEEDED	H_2O, Fe	H_2O < 50 PPM Fe nil
S-34	FEED HCl	GAS	BURRETTE	S-34 HCl FEED	75/PSIG/150°F	1/SHIFT	% HCl	99 %+ HCl

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PITTSBURGH PLATE GLASS COMPANY CHEMICAL DIVISION LAKE CHARLES, LOUISIANA				
TITLE OHC PLANT PROCESS SAMPLE SCHEDULE	DRAWN BY DDM	DATE 4-2-68	SCALE	DWG. NO.
	CHECKED BY	DATE	CHARGE	
	APPROVED BY	DATE	BILL OF MATERIAL	SHEET 4 OF 4

TABLE 10-1
REACTOR FEEDS VS. REACTOR PRESSURE

Reactor Pressure PSIG	Oxygen feed Rate SCFH	Ethylene Feed Rate SCFH	HCl Feed Rate SCFH	Recycle Rate SCFH	Total Mixed Feed Rate SCFH
0	7,800	14,300	28,600	11,800	54,700
1	7,800	14,300	28,600	11,800	54,700
2	7,800	14,300	28,600	11,800	54,700
3	8,300	15,200	30,300	12,500	58,000
4	8,700	16,100	32,000	13,200	61,300
5	9,200	16,900	33,700	13,900	64,500
6	9,700	17,800	35,400	14,600	67,800
7	10,100	18,600	37,100	15,300	71,000
8	10,600	19,500	38,800	16,000	74,300
9	11,000	20,300	40,600	16,700	77,600
10	11,500	21,200	42,300	17,400	80,900
11	12,000	22,100	44,000	18,100	84,200
12	12,400	22,900	45,700	18,800	87,400
13	12,900	23,800	47,400	19,500	90,700
14	13,400	24,600	49,100	20,200	93,900
15	13,800	25,500	50,800	20,900	97,200
16	14,300	26,400	52,500	21,600	100,500
17	14,800	27,200	54,200	22,400	103,800
18	15,200	28,100	55,900	23,100	107,100
19	15,700	28,900	57,700	23,800	110,400
20	16,200	29,800	59,400	24,500	113,700
21	16,600	30,600	61,100	25,200	116,900
22	17,100	31,500	62,800	25,900	120,200
23	17,600	32,400	64,500	26,600	123,500

TABLE 10-1 (Cont'd)
REACTOR FEEDS VS. REACTOR PRESSURE

Reactor Pressure PSIG	Oxygen Feed Rate SCFH	Ethylene Feed Rate SCFH	HCl Feed Rate SCFH	Recycle Rate SCFH	Total Mixed Feed Rate SCFH
24	18,000	33,200	66,200	27,300	126,700
25	18,500	34,100	67,900	28,000	130,000
26	19,000	34,900	69,700	28,700	133,300
27	19,400	35,800	71,400	29,400	136,600
28	19,900	36,600	73,100	30,100	139,800
29	20,400	37,500	74,800	30,800	143,100
30	20,800	38,400	76,500	31,500	146,400
31	21,300	39,200	78,200	32,200	149,600
32	21,800	40,100	79,900	32,900	152,900
33	22,200	40,900	81,600	33,600	156,100
34	22,700	41,800	83,300	34,300	159,400
35	23,200	42,700	85,000	35,000	162,700
36	23,600	43,500	86,800	35,700	166,000
37	24,100	44,300	88,500	36,500	169,300
38	24,600	45,200	90,200	37,200	172,600
39	25,200	46,100	91,900	37,900	175,900
40	25,500	46,900	93,600	38,600	179,100
41	26,000	47,800	95,300	39,300	182,400
42	26,400	48,700	97,000	40,000	185,700
43	26,900	49,500	98,700	40,700	188,900
44	27,400	50,400	100,400	41,400	192,200
45	27,800	51,300	102,100	42,100	195,500
46	28,300	52,100	103,900	42,800	198,800

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TABLE 10-2
REACTOR FEEDS VS. REACTOR PRESSURE WITH NO RECYCLE FLOW

Reactor Pressure PSIG	Oxygen Feed Rate SCFH	Ethylene Feed Rate SCFH	N2	HCl Feed Rate SCFH	Total Mix Feed Rate SCFH
0	7,800	21400	4700	28,600	54,700
1	7,800	21400	4700	28,600	54,700
2	7,800	21400	4700	28,600	54,700
3	8,300	22700	5000	30,300	58,000
4	8,700	24000	5300	32,000	61,300
5	9,200	25200	5600	33,700	64,500
6	9,700	26600	5900	35,400	67,800
7	10,100	27800	6100	37,100	71,000
8	10,600	29100	6400	38,800	74,300
9	11,000	30300	6700	40,600	77,600
10	11,500	31600	7000	42,300	80,900
11	12,000	40,200		44,000	84,200
12	12,400	41,700		45,700	87,400
13	12,900	43,300		47,400	90,700
14	13,400	44,800		49,100	93,900
15	13,800	46,400		50,800	97,200
16	14,300	48,000		52,500	100,500
17	14,800	49,600		54,200	103,800
18	15,200	51,200		55,900	107,100
19	15,700	52,700		57,700	110,400
20	16,200	54,300		59,400	113,700
21	16,600	55,800		61,100	116,900
22	17,100	57,400		62,800	120,200
23	17,600	59,000		64,500	123,500
24	18,000	60,500		66,200	126,700
25	18,500	62,100		67,900	130,000

This table is based on replacing all recycle flow with ethylene.