

R. P. Lynch

EC Tar Buggy
60-722

PITTSBURGH PLATE GLASS COMPANY

CHEMICAL DIVISION

LAKE CHARLES, LOUISIANA

4

ETHYL CHLORIDE PLANT MANUAL

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

December 6, 1965

Manual # 1

SL 008836

TABLE OF CONTENTS

INTRODUCTION	1
GENERAL DESCRIPTION	2
SAFETY	4
Emergency Horns	4
Fire Protection	5
General	5
Dock Area	7
Control Building	8
Electrical Equipment	9
Relamping	9
Grounding	9
Tools	10
Procedures	11
Sampling Methods	11
Safety Permit	10a
General Fire & Explosion Rules	11
General Safety Rules	13
Special Safety Equipment	14
CHEMICALS IN THE AREA	17
PROCESS DESCRIPTION	35
1. Ethylene System	36
2. HCl System	37
3. EC Reactor System.....	37
A. Equipment	37
1. Reactor	38
2. Catalyst Chamber	38
3. Reactor Coolers	38
4. Reactor Dump tank	38
B. Reactor Shutdown System	38
C. Operation	39
D. Start Up	42
E. Reactor Dryer and Reactor Filter.....	43
4. Flasher Dopp Kettle	44
5. Bottoms Dopp Kettle	45
6. Flasher Condenser	46
7. Primary Vent Condenser.....	47
8. Secondary Vent Condenser	48
9. Vent Scrubber	49

CONFIDENTIAL:

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

SL 008837

10.	Primary Stripper Feed Drum	50
11.	Gland Seal Pump	52
12.	Primary Stripper Feed Dryers and Filters	52
13.	Primary Stripper and Reboiler	53
14.	Heavies Still Feed Drum	56
15.	Heavies Still	56
	A. Reboiler	56
	B. Condenser	57
	C. Hvs. Still Bottoms Cooler	57
	D. Hvs. Still Reflux Drum	57
16.	Secondary Stripper and Reboiler	58
17.	Product Surge Tank and Cooler	60
18.	Day Tank Area	61
	A. Day Tanks	61
	B. Vent Compressor	61
	C. Vent Condenser	61
	D. Transfer Pumps	61
	E. Rework Pump	61
19.	Dock Area	63
	A. Horton Spheres	63
	B. Excess Flow Valves	64
	C. Turbine Flow Meters	64
	D. Refrigeration Unit	64
	E. Brine Circulation Pumps	64
	F. Barge Loading Pumps	64
	G. Vent Compressor	64
	H. Vent Condenser	64
	I. Vent Condensate Receiver	64
	J. Vent Condensate Pump	65
20.	Cooling Tower	72
21.	Refrigeration Unit	73
INSTRUMENTS		76
PUMPS		81
LABORATORY PROCEDURES		82
1.	Ferrous and Ferric Chloride	82
2.	HCl in Liquid EC	84
3.	HCl in Gas Samples	85
4.	H ₂ O in EC	87
5.	Chromatograph Procedure for Liquid Sample	89
6.	Chromatograph Procedure for Gas Sample	90
ELECTRICAL SYSTEM		91
OPERATING GRAPHS		
1.	C ₂ H ₄ vs TPD	94
2.	HCl vs C ₂ H ₄	95
3.	FeCl ₃ vs C ₂ H ₄	96
4.	Product vs TPD	97
5.	Steam vs Rate, Primary Stripper	98
6.	Steam vs Rate, Secondary Stripper	99

SL 008838

CONFIDENTIAL:
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of 14th Judicial District Court
11-01-1145

PROCESS FLOWSHEET

MECHANICAL FLOWSHEET

ELECTRICAL ONE LINE DIAGRAMS

SL 008839

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of 14th Judicial District Court
No. 91-1145

INTRODUCTION

USES AND PURPOSE OF MANUAL

This operating manual has been assembled with the following purposes in mind:

1. To aid the operators in becoming familiar with the EC plant.
2. To serve as a ready source of reference for the operators throughout their tenure at the EC plant.
3. To provide a set of basic procedures so that everyone is working under the same instructions.
4. To serve as a place where data, facts, information, etc. on the unit are centrally located and readily available.

Like any new operating manual, it is expected that changes will be warranted in this write-up as operating experience is gained; please feel free to suggest these changes as it is intended that this manual be kept current and meaningful.

It should be remembered that this manual is the property of Pittsburgh Plate Glass, Chemical Division, Lake Charles Works and may be recalled at any time.

You are personally responsible for the safe keeping of this manual as well as the information therein. This is why the manual is numbered and charged to you.

SL 008840

-1-

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

GENERAL DESCRIPTION

The Ethyl Chloride process used at Lake Charles is by no means unique, but since certain phases of the process are technical improvements, you should not discuss the unit or its operations with personnel other than PPG employees.

HCl and Ethylene are reacted in the presence of a catalyst to form Ethyl Chloride (EC). The HCl for the EC plant is manufactured by one of two methods. One method is to burn Cl_2 and H_2 to produce HCl which is absorbed, stripped, dried, and compressed before coming to the plant. The other method is to thermally crack EDC to HCl and Vinyl Chloride. The HCl thus produced is separated from the other components and sent to the EC plant. The HCl for the EC plant will be produced by one or the other method and sometimes a combination of both methods. The ethylene is furnished through a pipeline by another company. The catalyst used is Ferric Chloride. It is delivered in 135# drums from other companies.

The rest of the plant consists of purification equipment. The spent catalyst is removed as tars in a Dopp Kettle network. Since the reactor is run with a stoichiometric excess of HCl this HCl must be removed and it is removed by stripping. The heavies formed in the reaction are removed by distillation. The essentially pure EC is sent to day storage to be analyzed before being transferred to the dock storage Hortonspheres.

The bulk of the EC produced is used in the manufacture of Tetraethyl lead (Antiknock agent in gasoline). The other uses for EC are a very small part of the market but it may be used in the manufacture of ethylcellulose, manufacture of a polymerization catalyst, a solvent, a dewaxing agent or as a local anesthetic.

As in all plants, safety is of the utmost importance. The safe

CONFIDENTIAL:

-2-

Subject to Protective Order
f 14th Judicial District Court
No. 91-1145

SL 008841

operation of this plant depends directly on you. EC is a volatile, highly flammable material. The material is a gas at temperatures greater than 54°F (at atmospheric pressure), and since this gas is heavier than air (2.2 times as heavy) any spills will result in a highly flammable material spreading over a large area. The process area is equipped with a sprinkler system for fire protection, and this system has a remote trip switch on the panel board. USE THIS TRIP in the case of spills. It will spray the area with water and prevent the spreading of flammable vapors. The water cannot hurt anything and it can prevent a dangerous situation.

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

SL 008843

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

SAFETY

EMERGENCY HORNS

There are two distinct emergency horns in the area: the plant or Area "B" evacuation horn and the EC operating area siren.

The Area "B" evacuation horn is a continuous warbling sound and may be activated from the EDC or EC-VC control rooms, or the guard house. The sounding of this horn will shut down all non-process equipment in Area "B" and will cause all non-essential personnel to leave the area. Area "B" includes all areas West of Columbia Southern Road. The Area "B" evacuation horn should be sounded in case of a major EC break, or if there is some reason to expect a major break, or if there is a possibility that EC vapors will escape the operating area. Whenever the evacuation horn is sounded the guards should be notified of the nature of the emergency if at all possible. If it is desired to evacuate the whole plant the guards will have to be notified and they will sound the plant evacuation. The guards are the only people able to sound the all-clear.

The operating area siren is to evacuate all non-essential personnel from the operating area and to have all arcing devices in the operating area shut down. This siren is controlled by a switch on the panel board or by a switch located near the bottoms Dopp kettle. The area siren should be used in case of a minor spill or a possible spill.

It is well to keep in mind the extreme flammability of EC. Should a spill occur, keep it from spreading if at all possible and eliminate all sparking devices in the area. One of the best means to prevent spreading is the use of the fire water system, use it and use it quickly. The arcing devices can be eliminated by sounding the siren or evacuation alarm. Evaluate the situation quickly and act quickly. It is much better to get someone wet or evacuate them than to have a fire or an explosion.

When you are in the operating area and the siren blows or you are in Area "B" and the warbler blows, see to it that all non-essential equipment is shut down (trucks, hysters, welders, etc.) and if you are non-operating personnel = get out of the area. All operating personnel should go to their respective control rooms, if possible.

FIRE PROTECTION

GENERAL

Fire protection in the EC area is provided in three forms, water, dry chemicals and CO₂. The dry chemical is the only effective way of fighting an EC fire. Water is good for knocking down the fumes and keeping vessels cool. The CO₂ cylinders are only good for small fires or small electrical fires. CO₂ cylinders are located in the switchgear room and are to be used on small electrical fires.

Anytime you are in doubt as to the type of extinguisher to use, use the dry chemical. It is good to avoid the use of the dry chemical on small electrical fires, as the chemical leaves a harmful residue. Throughout the plant are located 30# dry chemical units. On the perimeter of the operating area will be located a portable unit, this unit will be shared by EC and VC.

The water protection is a manual or automatic sprinkling device, tripped by a deluge valve. The EC process area, the cooling tower, the EC day tank area, and the Horton Sphere area are each protected with its 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 be manually tripped from the panel board or from the valve house. The cooling tower, day tank area, or Horton Sphere area have their manual trips located in each individual hose and valve house. Should

CONFIDENTIAL:

SL 008844

-5-

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

a fire break out in any of the areas the automatic system should trip. Do not be afraid to manually trip in any of the areas in case of a fire or spill in that area. Whenever any deluge valve is tripped an alarm will sound in the guard house, but the fire truck will not come unless the guards are notified or they can see the fire or smoke. Anytime a deluge valve is tripped, the valve must be reset before automatic protection is available again. The valve is reset by closing the water valve upstream of the deluge valve, removing the cover of the deluge valve and resetting the weight.

The automatic system is a thermo-pneumatic fire detection system. The thermo-pneumatic system is designed to detect an abnormal rate of temperature rise and, in such a situation, cause a Suprotex deluge valve to open. When the Suprotex valve opens the water is allowed to flow into sprinkler lines and from the nozzles, covering the area with a water density of 0.25 GPM/ft².

The HADs are the heat sensing elements and are located at all strategic points in the process. The HADs are empty shells connected to the release diaphragm of the Suprotex deluge valve by a manifold of 1/8" PVC coated copper tubing. The HADs and the tubing manifold are pressurized with 24 oz. air pressure. An abnormal temperature rise in one of the HADs, as would occur in the event of a fire, will create a pressure rise in the units and the tubing manifold. This pressure rise will cause a diaphragm operated release mechanism to release a suspended weight. This weight drops and disengages the latch holding the deluge valve closed, thereby starting the flow of water to the distribution system. The water is dispersed in the area by sprinkler heads, which are located so as to provide a spray over all exchangers, surge tanks, pumps, and skirts of columns. The manual trip lever is the handle located on the Suprotex deluge valve. When pulled, this handle releases the weight.

CONFIDENTIAL:

Subject to Protective Order
-6- of 14th Judicial District Court
No. 91-1145

A change in environment temperature will not trip the system because the Suprotex valve has a compensating vent valve in the diaphragm. This valve is sized so that a slow pressure build-up in the supervisory air (HAD system) system will bleed through the valve to the other side of the diaphragm thereby equalizing the pressure.

As may be seen from the description of the supervisory air system, an accidental break in the HADs or tubing manifold will not trip the deluge valve. Anytime the system has a break in it, all automatic fire protection for this system is lost. Anytime the supervisory air pressure gets below 16 oz. an alarm will sound. Check the alarm out immediately as you are in danger of losing your protection. The alarm will sound if any single circuit of the supervisory air system develops a leak. When the alarm sounds check the supply pressure and the overall supervisory air pressure. If both of these are normal then a particular circuit has a leak, but enough air is being bled into the system to maintain the required 24 oz. pressure. Automatic Sprinkler will have to be called to find and repair the leaking circuit. To silence the alarm the electrical power to the valve house will have to be shut off, the horn disconnected and power put back to the valve house. This power will come from the emergency circuit. Fire protection exists until the supervisory air is about 6 oz. or less.

DOCK AREA

In the dock area the Horton Spheres are not protected by water spray, but the pumps and the equipment platform are. The protection of the equipment is accomplished by HADs and a Suprotex valve just like the day tanks or production area. The bottom halves of the spheres and legs are protected with "ALBI" fireproof paint. This paint, when heated, will swell and insulate the sphere from the fire.

SL 008846

There are water turrets located on the North side of the tanks. In case of a fire these turrets should be used to keep a water stream on the spheres, to keep them cool. There is a foam nozzle located midway between the spheres on the North side. This nozzle may be used to blanket a layer of EC trapped in the dyked area. The foam is not very effective in fire fighting, but will retard the vaporization of the EC if there is no fire. The dyked area is designed to hold the contents of a full sphere. To drain rainwater from the dyked area, open the valves in the drain lines located on the South side of the dykes. Since the dyked area drains to the river the drain valves should never be left open without a man standing nearby. NEVER DRAIN EC TO THE RIVER. In case of a spill or fire in the area try to contain the EC in the dyked area, do not attempt to drain it to the river. If there is an EC spill remember that there are many sparking devices in the dock area, try to get everything downwind shut down and evacuated. If there is a fire keep the spheres cool with the water turrets.

CONTROL BUILDING

The control building is pressurized to prevent accumulation of any vapors inside. Since it is a pressurized building, non-explosion proof electrical equipment is used and smoking is permitted inside. The positive pressure is maintained by a fan which draws its intake air across an activated carbon filter. Should a major break occur, the filter would be unable to eliminate all contaminants, therefore the fan should be shut down. To prevent any vapors from entering the building, when the fan is off, instrument air bleeds should be opened. (Do not bleed the air system down by taking too much). To prevent dust from accumulating in the control room filter bags are in the suction of the air conditioner. For any problems with the air conditioner, notify the supervisor.

SL 008847

-8-

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

ELECTRICAL EQUIPMENT

The characteristic of EC is such that the operating area is classified as Class I, Group D by the National Electric Code. The methods 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.

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. To simplify this procedure, circuit breakers for lights in the process area and control laboratory have been grouped in the lighting panels. Globes and guards must be replaced after relamping.

GROUNDING

Grounding in the EC plant has been given special attention due to problems peculiar to the handling of hydrocarbons. Grounds for motors and other electrical devices are contained in the conduit supplying the device and connected to the device frame internally. Motor 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 or agitation, a system of jumpers for pipe flanges has been installed to provide metallic continuity of the piping systems that contain flammable materials. All vessels are grounded at 2 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 and the resulting spark.

The nearness of the radio transmitting station north of the process area presents a problem in that metal objects will act as receivers of radio frequency energy. It is not practically possible to prevent this effect. It is known that the energy picked up will be amplified if the height of the object is a multiple of the transmitter wave length. The height of the stills approach this condition, but the grounding system on the stills should eliminate personal hazard.

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. Particular attention should be paid to this problem if the crane height is greater than 90'.

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 EC plant.

TOOLS

Special spark-proof tools are not required unless there are EC vapors present, or a possibility that EC vapors will become present. Check with operations prior to starting each job to determine the type of tools needed.

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

ORGANIC AREA SAFETY PERMIT

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

Ethyl Chloride Unit.

To: John Jones Date: 17 Nov 65

Location: Dapp Kettle Equipment: Truck

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

To be
filled in
for truck
pass

Remarks: He will make 2 trips, will check before
the second trip.

Approved: Robert P Lynch

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

John Jones
Employee's Signature

WELDING OR BURNING PERMIT

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

pipe rack, west of product cooler
(Location)

It is good for: Date 17 Nov 65 Time 9 15 AM

To: Date 17 Nov 65 Time 1 00 PM

Special Comments: John will check with
operations before resuming, after
dinner.

Signed: Robert P Lynch
(Area Production Supervisor)

To be
filled in
for weld
Burning,
Blasting.

CONFIDENTIAL:

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

-10a-

SL 008850

PROCEDURES

SAMPLING METHODS

Sampling techniques used in this plant are different than those used in most other plants. EC is never purged to the atmosphere because of the fire hazard. Samples are caught in steel bombs and therefore, a visual check is impossible.

All bombs must be thoroughly cleaned, and then purged with nitrogen before any EC is introduced into the bomb. The bombs are equipped with a standleg so that a vapor space is always assured whenever the bomb is filled in the prescribed manner. The bomb will always have to be filled with the bomb in the vertical position. The bomb has a marked top and it should always be up. In order to obtain a true sample a high pressure liquid stream will be circulated through the bomb to some low pressure stream, i.e., a pump discharge to a pump suction. Be sure the low pressure stream is connected to the standleg connection. Do not run the circulation too long as the inert may be dissolved out and the bomb liquid filled. Any time a bomb is filled it should be checked (tilting or shaking) to be sure it has some vapor space in it. Failure to have a vapor space, or failure to have an inert gas in the sample bomb will result in someone having a real bomb in their hands.

GENERAL FIRE AND EXPLOSION RULES

1. Personnel entering the organics area will deposit lighters, matches, regular flashlights, etc., at the gate to this area. Safe flashlights have a label showing MSA approval for flammable areas.
2. Anyone entering an operating area should first check with operating personnel.
3. Smoking will be permitted only in the main control buildings

SL 008851

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

(except the laboratory), where permanent lighters are provided.

4. A vehicle permit signed by an operating supervisor will be required for any vehicle to go into the chained-off roads.
5. An equipment permit signed by the operating supervisor will be required before the following equipment can be carried into the EC area:
 - (a) Welding machine
 - (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 or chipping equipment
 - (h) Sandblasting equipment.
6. Only explosion-proof flashlights will be permitted.
7. In case of an emergency warning, (siren or plant howler), all vehicles or equipment that are in the area on permits will be shut off immediately and the occupants will evacuate the area on foot.
8. Do not dump ethyl chloride or other flammables into openings, such as sewers or other underground cavities, where harmful vapors could be evolved.
9. Do not leave samples or containers of flammable material sitting around to give off vapors.
10. Always put liquid EC or the other flammables into tanks through standlegs or through bottoms nozzles. Falling liquid can generate static electricity.
11. All steam-out nozzles or hose and purge equipment must be properly grounded to reduce the possibility of an arc from an accumulated static charge.

12. Do not permit air to enter any of the process equipment that contains a flammable.
13. Be sure all tank cars or tank trucks containing flammables are properly grounded before unloading begins. This includes a jumper around a non-conductive unloading base.
14. Fire protection equipment is different. Know it well! Report all damages immediately. Do not use the fire hoses for any other purpose.
15. It is intended that all personnel will obtain periodic re-fresher courses on fire-fighting techniques.

GENERAL SAFETY RULES

1. Pumps and Equipment -- any pump or pieces of equipment that is removed from the process stream must be thoroughly cleaned and inspected by a foreman before it is permitted to leave the area for the main shop or other work areas.
2. Regular plant utility hoses are not to be used for solvents, because they will become weakened and unsafe to use with pressure. Use the special hose provided .
3. The laboratory hood fans are to operate continuously. Do not attempt to run analyses without the fan being in service.
4. Use Full-Face, Chemox or Scott Air-Pak, masks for protection against organic vapors.
5. Do not permit chlorine to be heated above 300°F, as it will rapidly corrode most metals at higher temperatures.
6. Clothing that has been wet with EC should be removed immediately and the body thoroughly washed with soap and water. Severe frostbite can result from allowing EC to evaporate from the body

SL 008853

or clothing on the body.

7. Clearing 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.

Clearing Procedure:

- (a) The tank or vessel will be emptied of its contents and all valves will be closed and tagged.
 - (b) The vapor contents of the tank will be purged with an inert gas.
 - (c) Blinds will be inserted in all connecting lines.
 - (d) Purge the equipment with plenty of air.
 - (e) The equipment will then be checked with an explosion meter before work is begun. When a man is to enter the tank, it will first be checked for sufficient O₂. The apparatus for testing for sufficient oxygen is either a flame or a special metering device. These tests can be run only by a supervisor.
 - (f) Safety belts and safety lines will be required in top exit tanks.
8. If the emergency warning sounds, all personnel not needed for control purposes should walk upwind (or crosswind, if in fumes), to a safe location and remain there until the all-clear sounds. Those personnel required for operation should remain on the job, while taking safety precautions necessary to remain in the area.
 9. Tagging - Same as used throughout the plant. See General Section of Production Department Manual.

SPECIAL SAFETY EQUIPMENT

1. Stretchers (1) (a) Located inside control room
2. Chemox masks (3) (a) Two in control room
(b) One at main gate
3. Full Face Masks (10) All purpose cannister
(a) Three located in Control Room
(b) One located in day tank area
(c) Two in Shipping building
(d) Two at the main gate
(e) Two at the dock area

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

4. Scott Air Pak (6) (a) Two in control room
(b) One in supervisors office
(c) One in HCl area
(d) One in TE Catalyst bldg.
(e) One in EC Catalyst bldg.
5. Cylinder Type with connecting regulator, hose, and mask
(1) In EC control room

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

- (a) Scott Air-Pak -- 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. Note: the Scott-Air Paks in the EDC area only have a 15 minute life.
- (b) 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.
- (c) 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 areas and for escape

purposes.

(d) Cylinder Type With Connecting Regulator, Hose and Mask --

these units are good in any concentration of gas. They 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 rooms are pressurized, contaminating 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.

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

CHEMICALS IN THE AREA

In the course of discussion of the EC plant many abbreviations are used. Since everyone is not necessarily familiar with a majority of the chemicals used in Area "B", a quick review is listed.

CaCl ₂	--	Calcium Chloride
Cl ₂	--	Chlorine
FeCl ₃	--	Ferric Chloride
HCl	--	-gas- Hydrogen Chloride
		-liquid-(mixed with water) Hydrochloric Acid, Muriatic Acid
H ₂ SO ₄	--	Sulfuric Acid
NaOH	--	Caustic, Sodium Hydroxide, Cell Liquor is about 10% NaOH
N ₂	--	Nitrogen, Inert
O ₂	--	Oxygen
H ₂	--	Hydrogen
CH ₄	--	Methane, Natural Gas
C ₂ H ₄	--	Ethylene
C ₂ H ₆	--	Ethane
EC	--	Ethyl Chloride
EDC	--	Ethylene Dichloride (There are two EDCs - 1,1 and 1,2. 1,2-EDC is the most common one.)
MC	--	Methyl Chloroform, 1,1,1 - Trichloroethane
Per	--	Perchloroethylene, Tetrachloroethylene
TCE	--	1,1,2 - Trichloroethane
Tri	--	Trichloroethylene
VC	--	Vinyl Chloride, Chloroethylene
VDC	--	Vinylidene Chloride, 1,1 dichloroethylene

There are many more chemicals in the area, and if you need to know the names or abbreviations see the operating foreman.

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

SL 008857

NAME: Calcium Chloride

FORMULA: CaCl_2

MOLECULAR WEIGHT: 110.99

BOILING POINT: $> 1600^\circ\text{C}$

MELTING POINT: 772°C

SOLUBILITY IN WATER: 59.5 parts CaCl_2 /100 parts H_2O at 9°C
347 parts CaCl_2 /100 parts H_2O at 260°C

DENSITY: 2.512 g./cc. at 25°C

RELATIVE VAPOR DENSITY: Normally a solid

FLASH POINT: None

EXPLOSIVE LIMITS: None

HAZARDOUS PROPERTIES: Generally speaking, calcium compounds should be considered toxic only when they contain a toxic component (such as arsenic, etc.) or as calcium oxide or hydroxide.

The hydration of the anhydrous salt is exothermic (21.7 cal./mole to form the hexahydrate). Thus, when driers are being flushed out with water there is the possibility of heat and some splashing which may carry CaCl_2 solution into the operator's eyes. If CaCl_2 solution enters the eye(s), a sustained water flush will be most effective in removing the material.

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of 14th Judicial District Court
No. 91-1145

SL 008858

NAME: Caustic Soda (lye)

FORMULA: NaOH

MOLECULAR WEIGHT: 40.01

BOILING POINT: 2532°F

VAPOR PRESSURE: Normally a solid, but can be mixed with water to form a solution

FREEZING POINT: 604°F

HAZARDOUS PROPERTIES: When mixed with water, will react violently, splashing caustic solution on anyone nearby. Caustic soda in the form of solid, flake, or liquid attacks any tissue upon contact. The degree of injury depends upon the extent and duration of contact, temperature of the materials and concentration of the materials.

TREATMENT: Flush the contacted area immediately with an abundance of water. Remove any clothing or equipment that has been saturated with caustic. After thoroughly flushing the contacted area of the body with water, report to First Aid for further treatment. Eyes should be irrigated at once with plenty of warm water for at least 15 minutes anytime caustic comes in contact with the eye.

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of 14th Judicial District Court
No. 91-1145

SL 008859

NAME: Cell Liquor

FORMULA: Aqueous solution of NaOH

LIQUID DENSITY @ 77°F: 10.0#/gal., 1.1 gm/ml

HAZARDOUS PROPERTIES: Cell liquor is an aqueous solution of sodium hydroxide (caustic) and salt and as such should be handled with the caution given any caustic solution. This material has a markedly corrosive action upon all body tissues, and skin contact may result in moderate to severe chemical burns if the exposed surface is not washed immediately. Eye contact is quite painful and can result in impairment of vision.

TREATMENT: Speed in removing cell liquor from contact with the body is important to avoid injury. Removal of all contaminated clothing and thorough washing of the exposed surface is essential. If the eyes are involved they should be irrigated at once with plenty of warm water for 15 minutes. Call a physician.

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

SL 008860

NAME: Chlorine (Cl₂)

FORMULA: Cl₂

MOLECULAR WEIGHT: 70.91

BOILING POINT: -30°F

VAPOR PRESSURE @ 75°F: 92 psig

FREEZING POINT: -148°F

LIQUID DENSITY @ 77°F: 11.6#/gal., 1.391 gm/ml

RELATIVE VAPOR DENSITY: 2.45 (air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONC: 0.35 to 2 ppm

DETECTABLE ODOR CONC: 3.5 ppm

HAZARDOUS PROPERTIES: Liquid chlorine is very dangerous to the eyes as is chlorine gas. High concentrations of chlorine gas can cause pneumonitis and edema of the lungs. Lung irritation is one of the most serious effects of chlorine. Cl₂ is about 2½ times as heavy as air, therefore, it has a tendency to collect in the low spots or stay near the ground.

TREATMENT: Remove patient from toxic area and loosen all constrictive clothing about the neck. Oxygen should be administered in all cases to prevent cyanosis and relieve the pain of deep respiratory effort. Notify a physician.

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

NAME: Ethane

FORMULA: C_2H_6

MOLECULAR WEIGHT: 30.07

BOILING POINT: $-128^{\circ}F$

FREEZING POINT: $-277^{\circ}F$

RELATIVE VAPOR DENSITY (air = 1.0) 1.04

AUTOIGNITION: $950^{\circ}F$

EXPLOSIVE LIMITS: 3 - 11%

ODOR: Odorless

HAZARDOUS PROPERTIES: Ethane is a flammable gas. It is the main impurity in the ethylene, and may reach high concentrations in the vent streams. Ethane is a very dangerous explosion hazard upon exposure to heat or flame. It can react vigorously with oxidizing materials. Ethane is moderately toxic, but the slight effects disappear as soon as the patient is removed from the exposure. The main danger with C_2H_6 is asphyxiation. The ethane will displace the oxygen of the air causing the victim to suffocate.

TREATMENT: Remove the patient from the contaminated area, perform artificial respiration if breathing has stopped. Report to First Aid.

SL 008862

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

NAME: Ethyl Chloride

FORMULA: C_2H_5Cl

MOLECULAR WEIGHT: 64.52

BOILING POINT: $54^{\circ}F$ (Gas under normal conditions)

VAPOR PRESSURE @ $75^{\circ}F$: 1130 mm

FREEZING POINT: $-228^{\circ}F$

LIQUID DENSITY @ $68^{\circ}F$: 0.893

RELATIVE VAPOR DENSITY (air = 1.0): 2.22

FLASH POINT: Open Cup $-45^{\circ}F$ Closed Cup $-58^{\circ}F$

AUTOIGNITION: $966^{\circ}F$

EXPLOSIVE LIMITS: 3.6 - 12.0%

MAXIMUM ALLOWABLE CONC.: 1000 PPM

ODOR: Pungent

HAZARDOUS PROPERTIES: The liquid is harmful to the eyes, and if spilled on the skin will cause frostbite. The vapor gives some warning of its presence because it is irritating, but it is possible to tolerate exposure to it until one becomes unconscious. EC is the least toxic of all the chlorinated hydrocarbons. It can cause narcosis, but the effect are usually transient. Continued exposures may cause some kidney deterioration. Inhalation of EC vapor in concentrations of 1% (by volume) produces narcotic and anesthetic effects, concentrations of 4% or greater may produce deep or even fatal anesthesia. Although EC is not extremely poisonous, any exposure is very dangerous because of the extreme flammability of the material. When EC is burned phosgene and HCl are formed, so never approach burning EC without a gas mask. Any spill of EC will result in an explosive mixture being formed and because of the high density of the EC gas, the mixture will tend to stay near the ground. It will take very little to explode EC vapors; static electricity spark, friction spark or an arcing device are all dangerous when EC is spilled.

TREATMENT: Remove the injured person from the contaminated area, if breathing has stopped perform artificial respiration. In any case make sure the person injured is reported to First Aid immediately. If any liquid enters the eye, wash the eye continuously with a large amount of water for at least 15 minutes. If at the end of 15 minutes eye damage is still apparent continue washing until the doctor arrives or gives new instructions

SL 008863

CONFIDENTIAL:
Subject to Protective Order
-23- of 14th Judicial District Court
No. 91-1145

NAME: Ethylene

FORMULA: C_2H_4

MOLECULAR WEIGHT: 28.05

BOILING POINT: $-155^{\circ}F$

FREEZING POINT: $-273^{\circ}F$

RELATIVE VAPOR DENSITY (air = 1.0): 0.98

AUTOIGNITION: $1009^{\circ}F$

EXPLOSIVE LIMITS: 3 - 34%

ODOR: Sweet

HAZARDOUS PROPERTIES: Ethylene is a flammable gas. It is a very dangerous explosion hazard upon exposure to heat or flame. It can react vigorously with oxidizing materials. Ethylene is moderately toxic, but the slight effects disappear as soon as the patient is removed from the exposure. The main danger with C_2H_4 is asphyxiation. The ethylene will displace the oxygen of the air causing the victim to suffocate.

TREATMENT: (See Methane Treatment) Remove the patient from the area, perform artificial respiration if breathing has stopped. Report to First Aid.

SL 008864

-24-

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

NAME: Ethylene Dichloride (EDC)

FORMULA: $\text{CH}_2\text{ClCH}_2\text{Cl}$

MOLECULAR WEIGHT: 98.97

BOILING POINT: 182.3°F

VAPOR PRESSURE @ 75°F: 75 mm

FREEZING POINT: -31.8°F

LIQUID DENSITY @ 68°F: 10.45#/gal., 1.253 gm/mls

RELATIVE VAPOR DENSITY: 3.41 (air = 1.0)

FLASH POINT: 65°F Open Cup; 55°F Closed Cup

EXPLOSIVE LIMITS: 6.2 to 15.9% by vol. in air

MAXIMUM ALLOWABLE CONC: 75 to 100 ppm

DETECTABLE ODOR CON: Unknown

HAZARDOUS PROPERTIES: Ethylene dichloride is a flammable liquid and a dangerous fire hazard. It is toxic by inhalation, by prolonged or repeated contact with the skin or mucous membranes, and by ingestion. Excessive contact gives rise to symptoms such as headache, depression, mental confusion, fatigue, loss of appetite, nausea, vomiting, cough, loss of sense of balance, and visual disturbances. It has an anesthetic effect, and, in high concentrations, is immediately irritating to the eyes, skin, nose and throat. It can cause dermatitis upon prolonged or repeated contact with the skin. Ethylene dichloride can cause serious eye damage.

TREATMENT: Quick removal from exposure is important. Ethylene dichloride should be removed from the patient's person, his respiratory tract, skin, or gastrointestinal tract as quickly as possible. If breathing has ceased, start artificial respiration. If material gets in the eyes, wash promptly with copious quantities of water. If ingested, the patient should be made to vomit. Notify a physician.

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

SL 008865

NAME: Ferric Chloride

FORMULA: FeCl_3

MOLECULAR WEIGHT: 162.2

BOILING POINT: 590°F

VAPOR PRESSURE @ 75°F: nil

SUBLIMATION POINT: 572°F

SPECIFIC GRAVITY: 2.8

RELATIVE VAPOR DENSITY: Normally a solid

FLASH POINT: None

EXPLOSIVE LIMITS: None

HAZARDOUS PROPERTIES: Ferric chloride presents no particular problem in handling. However, in the anhydrous form it may be injurious to clothing, and all contamination of both skin and clothing should be washed off immediately. In the case of eye contact, flush immediately and thoroughly with water and then rinse with a weak solution of sodium bicarbonate or boric acid. A physician should always be consulted in such cases.

Ferric chloride will seriously stain clothing and skin when it is contacted so it is advisable to wear gloves and aprons when handling it.

Ferric chloride is extremely hygroscopic (absorbs water), and when this takes place, heat release occurs. Before opening a drum, the bleed valve should be opened to release any pressure that may have been generated in the drum. When taking material from a drum, remove the desired quantity as quickly as possible and immediately seal the drum to prevent it from absorbing moisture from the air.

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

SL 008866

NAME: Freon - 12 (dichlorodifluoromethane)

FORMULA: CCl_2F_2

MOLECULAR WEIGHT: 120.92

BOILING POINT: -29°C

MELTING POINT: -158°C

VAPOR PRESSURE: @ 16.1°C , 5 atmospheres

RELATIVE VAPOR DENSITY: 4.17 (air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONC.: 1000 ppm in air

DETECTABLE ODOR CONC.: Unknown

HAZARDOUS PROPERTIES: Dangerous: when heated to decomposition, it emits highly toxic fumes of phosgene and fluorides. Never allow any open flames to come in contact with F-12 or any vessels containing it.

SL 008867

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

NAME: Freon-22 (chlorodifloromethane)
FORMULA: ClHCF_2
MOLECULAR WEIGHT: 86.465
BOILING POINT: -40.8°C .
MELTING POINT: -146°C .
VAPOR PRESSURE: @ 24°C ., 7,600 mm. Hg.
RELATIVE VAPOR DENSITY: 3.87 (air = 1.0)
FLASH POINT: None
EXPLOSIVE LIMITS: None
MAXIMUM ALLOWABLE CONC.: Unknown
DETECTABLE ODOR CONC.: Unknown
HAZARDOUS PROPERTIES: Freon-22 is very dangerous when heated to decomposition. It emits highly toxic fumes of chlorides and fluorides. Never allow any open flames to come in contact with Freon-22 or any vessels containing it.

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

SL 008868

NAME: Hydrogen

FORMULA: H_2

MOLECULAR WEIGHT: 2.02

BOILING POINT: $-423^{\circ}F$

FREEZING POINT: $-434^{\circ}F$

RELATIVE VAPOR DENSITY (air = 1.0) 0.069

AUTOIGNITION TEMP: $1085^{\circ}F$

EXPLOSIVE LIMITS: 4-74%

HAZARDOUS PROPERTIES: Hydrogen is a flammable gas. As may be seen from the explosive range, any concentration is highly dangerous when exposed to heat or flame. Hydrogen gas itself is only slightly toxic, but will displace the O_2 in the air causing suffocation. Symptoms will be the same as methane.

TREATMENT: Remove the patient from the contaminated area, perform artificial respiration if breathing has stopped. Report to First Aid.

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Subject to Protective Order
f 14th Judicial District Court
No. 91-1145

SL 008869

NAME: Hydrogen Chloride

FORMULA: HCl

MOLECULAR WEIGHT: 36.47

BOILING POINT: -121°F

VAPOR PRESSURE @ 75°F: 36,000 mm

FREEZING POINT: -174°F

LIQUID DENSITY: Normally a gas

RELATIVE VAPOR DENSITY: 1.26 (air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CON: 10 ppm for 8 hour working day

DETECTABLE ODOR CONC: Unknown

HAZARDOUS PROPERTIES: Anhydrous hydrogen chloride is a gas which has a corrosive action upon the skin or mucous membranes. In this form, it will cause rapid and severe burns. It is particularly dangerous to the eyes. It is not flammable; however, the gas is highly soluble in water, forming hydrochloric acid, which attacks most metals with the evolution of explosive hydrogen.

TREATMENT: Immediate removal from the toxic area and thorough flushing of the patient's body and/or eyes with large quantities of water is of primary importance. Contaminated clothing should be removed from patient while he is being showered with water. It is essential that all affected body surfaces be washed with copious quantities of water for a sufficient time to remove all hydrochloric acid. No attempt should be made to neutralize the acid with alkaline solutions. Medical assistance should be summoned at the earliest possible moment.

SL 008870

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

NAME: Methane, Natural Gas

FORMULA: CH_4

MOLECULAR WEIGHT: 16.04

BOILING POINT: -258°F (gas under normal conditions)

FREEZING POINT: -296°F

LIQUID DENSITY @ 68°F : Flammable Gas

RELATIVE VAPOR DENSITY (air = 1.0): 0.54

EXPLOSIVE LIMITS: 3% to 29% by volume in air

MAXIMUM ALLOWABLE CONC: 3% to prevent fire, 33% for breathing

DETECTABLE ODOR CONC: Odorless: It is a mercaptan that is put in it to give it odor.

HAZARDOUS PROPERTIES: Methane is a simple asphyxiant gas. The gas has no toxic effect, but will deprive the lungs of oxygen when mixed with air. Harmful effects are noticed when the methane is greater than 33%. The first symptoms are rapid breathing and air hunger. Mental alertness is diminished and muscular coordination is impaired. Later judgment becomes faulty and all sensations are depressed. Emotional instability often results and fatigue occurs rapidly. As asphyxia progresses, there may be nausea and vomiting, prostration and loss of consciousness, and finally, convulsions, deep coma and death.

TREATMENT: Move the patient from the contaminated area. Give artificial respiration if required. Report to First Aid.

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

SL 008871

NAME: Nitrogen

FORMULA: N₂

MOLECULAR WEIGHT: 28.02

PHYSICAL APPEARANCE: Colorless, Odorless, Inert Gas

SPECIFIC GRAVITY: 1.0 with relation to air. (Air is 78% N₂)

HAZARDOUS PROPERTIES: Even though nitrogen is a very safe, inactive gas, it has some inherent dangers since it is used so universally throughout the plant. Its prime use is for padding and sweeping of equipment that has, or has had, EC in it. The hazard involved is that a vessel may have insufficient oxygen or that nitrogen is used to purge the vessel instead of air, before man-entry.

Therefore, in the EC plant, every vessel that is entered not only must be first checked for flammability, etc., it must also be checked for sufficient oxygen. Not only that, nitrogen lines, as well as toxic lines must be isolated from the vessel before entry and a clean air sweep provided.

TREATMENT: Remove person from the oxygen deficient area. Administer artificial respiration if necessary. (See Methane for symptoms).

SL 008872

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

NAME: Sulfuric Acid

FORMULA: H_2SO_4

MOLECULAR WEIGHT: 98.08

PHYSICAL APPEARANCE: Colorless to cloudy liquid

HAZARDOUS PROPERTIES: It is highly corrosive to most metals, particularly at concentrations below 75%. This reaction produces H_2 gas. At concentrations above 75%, H_2SO_4 may be handled in steel provided the temperature is never greater than 100°F.

It is very reactive with water or organic materials with heat being evolved.

Contact with the body produces a rapid destruction of the tissue. The acid will continue to destroy the skin until it is washed off. The destruction of the skin will result in a painful and a slow healing burn.

PROTECTIVE EQUIPMENT: Use rubber gloves and face shields whenever working with H_2SO_4 . Use a slicker suit whenever transferring acid, entering the shielded area around the HCl dryer, or where there is any danger of an acid spill.

TREATMENT: Flush the contacted area with an abundance of water. Remove any contaminated clothing or equipment. After thoroughly flushing the affected area, report to First Aid for further treatment. Treat for shock in case of severe burns. If any of the acid has been swallowed do not induce vomiting; if the patient is conscious, wash his mouth with water and feed him as much water as he can drink, get medical help as quickly as possible.

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

SL 008873

NAME: Vinyl Chloride (chloroethylene)

FORMULA: C_2H_3Cl

MOLECULAR WEIGHT: 62.50

BOILING POINT: 8°F

VAPOR PRESSURE @ 75°F: 2700 mm (Gas under normal conditions)

FREEZING POINT: -244°F

LIQUID DENSITY @ 68°F: 0.908

RELATIVE VAPOR DENSITY (air = 1.0): 2.15

FLASH POINT: Open Cup -107°F Closed Cup -162°F

EXPLOSIVE LIMITS: 4-22%

MAXIMUM ALLOWABLE CONC: 500 PPM for 8 hours

ODOR: Faintly sweet (smells like phenol when inhibited)

HAZARDOUS PROPERTIES: Vinyl Chloride is very much like Ethyl Chloride. It is not very poisonous, it is extremely flammable, and it has a narcotic effect. Continued exposures may cause some kidney deterioration. Inhalation of vapors greater than 500 PPM may produce a slight narcotic effect. At concentrations of 4% or greater VC vapors may produce a deep or fatal anesthesia.

The main danger with VC is the extreme flammability of the vapors. VC when burning will form phosgene and HCl, so never approach a fire without a gas mask.

Any spill of VC will result in an explosive mixture being formed and because of the high density of the VC gas, the mixture will tend to stay near the ground. It will take very little to explode the VC vapors, static electricity spark, friction spark or an arcing device are all dangerous when VC is spilled.

TREATMENT: Remove the injured person from the contaminated area; if breathing has stopped, perform artificial respiration, in any case, make sure the person injured is reported to First Aid immediately. If any liquid enters the eye, wash the eye continuously with a large amount of water for at least 15 minutes, then get the person to First Aid.

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of 14th Judicial District Court
No. 91-1145

SL 008874

PROCESS DESCRIPTION

The EC plant is somewhat similar to the EDC plant or the MC section of the Tri-Ethane^(R) plant. The reaction and catalyst removal is exactly like the MC plant. The stripping and purification is like the EDC plant except that a stripper has been substituted for the neutralizer.

Since EC is a gas under normal atmospheric conditions, the EC Process must be a pressure system. The pressure on the condensers will enable the condensation to be done by cooling tower water. The pressure differentials in the separation and purification areas are to aid and isolate the controls of each system. The reactor pressure effects the reaction rate. The reactor is designed to operate at 140 psig but an attempt will be made to operate at 90 psig. If successful, conversion can be made at the lower pressure, the reaction pressure will not be raised. If the pressure must be raised, the compressors will need spacer rings and the HCl stripper pressure in the HCl plant will have to be raised. The flows on the mechanical flow sheet are at 140 psig. Since the temperature and the liquid flows remain the same, only the gas flows are in error.

The process flow may be traced on the Process Flowsheet. The HCl and C₂H₄ are premixed in a sparger ring and injected into the bottom of the reactor. To remove heat of reaction, a liquid stream is removed from the top of the reactor ~~liquor~~, cooled and fed back into the bottom of the reactor. This stream has a small sidestream that goes through the Catalyst Addition Chamber for the addition of the FeCl₃ catalyst. The vent from the reactor, which is HCl, ~~H₂~~, C₂H₆, and EC, goes through an analyzer, the reactor PCV and to the Primary Vent Condenser. A liquid stream is drawn from the reactor and fed to the Flasher Dopp Kettle. In the Flasher the catalyst and tars are concentrated and sent to the Bottoms Dopp Kettle, where the remaining EC is boiled away and the tars sent to the Tar Buggy for disposal. The EC that is vaporized in the Flasher is condensed in the Flasher

Condenser. The inerts from the Flasher Condenser and the Primary Vent Condenser are sent to the Secondary Vent Condenser which is cooled with 0°F Freon. The inerts from the Secondary Vent Condenser go through a PCV that controls the pressure of the Flasher and Primary Stripper systems. From the PCV the inerts go to a scrubber or to an absorber in the HCl plant. All of the condensate from the three condensers is collected in the Primary Stripper Feed Drum. The crude EC liquid from the primary feed drum is fed through a dryer to the primary stripper where the HCl is removed. The overhead from the stripper goes to the Primary Vent Condenser. The stripper bottoms overflow to the Heavies Still Feed Drum. The liquid from the Heavies Still Feed Drum is fed to the Heavies Still where the heavies are removed. The still bottoms are cooled and sent to EDC for reclaiming. The overhead from the Still is condensed in the Heavies Still Condenser and collected in the Heavies Still Reflux Drum. The inerts from the Still Condenser go through a PCV to keep the Still pressure constant and then they go to the Primary Vent Condenser. The liquid from the Reflux Drum is sent to the Still as reflux and to the Secondary Stripper to remove any HCl that may have been formed in the Still. The overhead from the Secondary Stripper goes through a PCV and back to the Primary Vent Condenser. The Stripper bottoms overflow into the Product Surge Tank and from here are pumped through the Product Cooler to the Day Tank Area. The EC is stored in the Day Tanks until the analysis is complete and then it is pumped to the Horton Spheres in the Dock Area.

1. Ethylene System

The ethylene for the EC Reactor comes from the ethylene metering station that is located North of the EDC-MC control room. At the metering station the C_2H_4 enters at 600 psig, it is reduced to 300 psig and heated to 100°F, it then is metered through a mass flowmeter then reduced to 165 psig. At this

point, the C_2H_4 comes through the fence to PPG where it goes through PPG's mass flowmeter and is ready for distribution to EDC, MC and EC. The C_2H_4 to EC flows through 4" line that is located downstream of the meter. There is a block valve in the line to EC. The 4" line goes underground at the PPG metering station and comes out of the ground at the NE corner of the EC process rack. If the reactor must be run at a pressure that is too high for the C_2H_4 to enter the reactor bottom, the whole PPG metering station pressure may be raised. The C_2H_4 line is split into 2 meter runs along the north side of the process platform. Whenever it is planned to operate the reactor at less than 60 TPD, use the top 3" line. From the metering station the ethylene line goes across the platform to the ethylene FCV and from the valve, through a seal loop, check valve and into the sparger ring. The C_2H_4 FCV has a solenoid on it that will close it in the event of a reactor shutdown.

The C_2H_4 fed depends on the desired rate, see fig. 1.

2. HCl System

The HCl, regardless of whether it comes from the HCl or from the VC plant, will come to the EC plant from the HCl compressors. The HCl line is a 4" line that comes down the pipe rack, through the middle of the EC process rack, has a double meter run (the top 3" line for reactor rates of less than 60 TPD), goes through a FCV, through a seal loop and through a check valve into the sparger ring. The HCl FCV has a solenoid that will close it in the event of a reactor shutdown. The pressure of the HCl will depend on the reactor pressure and can vary from 125 psig to 165 psig.

During the initial startup of the plant 4% excess HCl will be fed to the reactor. See the HCl feed graph, fig. 2.

SL 008877

3. EC REACTOR SYSTEM

A. Equipment

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

1. Reactor 64A-59-8 - The EC reactor is a steel vessel 9' ID x 31'8" seam to seam. The design pressure is 225 psi to full vacuum. The capacity of the reactor is 13,042 gallons with a liquid height of 26½'.
 (SRV) The safety valve is a 3L4; the sparger design consists
 (Sparger) of a 6" distribution ring around the reactor about 2' from the bottom. From this ring four 3" spargers stick 4'2" into the reactor and have 51 5/16" ID holes drilled into the bottom side. These holes are spaced further apart towards the middle of the reactor to give a uniform concentration of the inlet gases throughout the reactor.
 (Recirculation) The recirculation liquid enters the bottom of the reactor through a T that has horizontal elbows on the two discharge ends to give the liquid a tangential discharge and to aid in distribution.
2. Catalyst Chamber 64A-60-595 - The catalyst addition chamber is an 18" OD x 22" seam to seam tank. The top is an 8" 300# Tube Turn quick-opening closure. The "O" ring is a 8½ x 9 x ¼ Viton ring. Design pressure is 200 psig at 135°F.
3. Reactor Coolers 64A-71-777 & 778 - The reactor coolers are 34" OD x 16' tubes. The shell side is two pass; the tube side is four pass. The design pressure of the tube side is 250 psi at 130° F. and the shell side is 150 psi at 300° F. There are 880 3/4" OD x 14 BWG seamless steel tubes. The exchangers are to remove 8,864,000 BTU/Hr. by cooling 1920 GPM of reactor liquor from 130° F. to 105° F. The 709 GPM of cooling tower water on the shell side is to be heated from 90° F. to 115° F.
4. Reactor Dump Tank 64A-60-594 - The Reactor Dump Tank is 10' ID x 23'8". The capacity of the tank is 15,120 gallons with no outage. The design pressure is 225 psi at 200° F. to full vacuum. The SRV is a 3K4.

B. Reactor Shutdown System

The EC Reactor has an automatic shutdown on the feed streams to the reactor. The purpose of this system is to prevent the feed streams from back-feeding one another or to shut down the feed streams in case the reaction ceases. Each feed stream has a solenoid on the FCV that will shut if the HCl flow is lost, or if the C₂H₄ flow is lost, or if the reactor overpressurizes (goes above 165 psig), or if the emergency shutdown button is pushed. The emergency system is shown on Dwg. 64A-7700. The shutdown system may be completely bypassed by

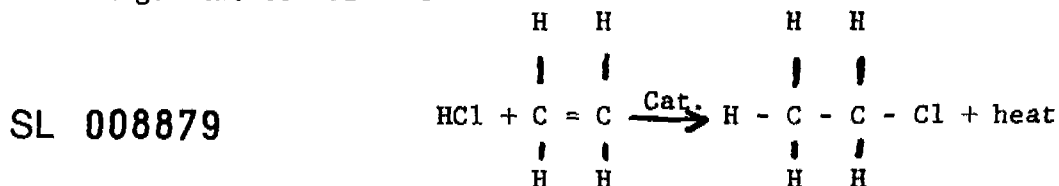
turning on the bypass switch that is located on the shutdown system box behind the panel board.

Should the shutdown system activate, it will be 5 minutes before the FCV may be opened again. The shutdown system is ready for operation when the light comes on about 4 seconds after the start button is pushed. It is well to remember that the light is the indicator of the cycle of the shutdown system. Anytime the light is off, the shutdown system will close the solenoid on the FCVs.

(Start-up) To start up the reactor push the start button and hold it in until the light comes on (about 4 sec.). At this time you now have 5 minutes to get flows through the HCl and C₂H₄ valves; if at the end of 5 minutes, both are not flowing then the system will shut down and you will have to wait 5 minutes more before starting up again. If start-up is successful, the timer will run to the shutdown position and stop (5 minutes). The timer will rest at this point until the shutdown system is activated, i.e., no HCl flow, no C₂H₄ flow, high (165 psig) reactor pressure, or the stop button is pushed, then the timer restarts, locks out start-up for 5 minutes, then it shuts down again until the start button is pushed.

C. Operation

The reaction between HCl and C₂H₄ proceeds at a slow rate unless catalyst is present. That is why the two gases may be premixed in the sparger ring. The reaction is



Since the reaction is rapid with a catalyst present, it is dangerous to have the gases mixed where catalysis could take place. If HCl and C₂H₄ are allowed to leave the reactor vent together they could contact FeCl₃ in the piping, start reacting, liberating heat, which makes more reaction, which makes more heat, and this would continue until the pressure and temperature would be

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

sufficient to rupture a line or a vessel. To prevent this from happening two things are done. One is the feeding of HCl in excess of that required for reaction. The unreacted HCl prevents any C_2H_4 from getting in the vent by reacting with all of the C_2H_4 . To begin with, the reactor will run with 4% excess HCl. This will probably be trimmed back after better operating techniques are learned. The second thing done is the installation of an analyzer in the vent stream from the reactor. By using the analyzer the exact HCl excess required will be determined.

As the gases are mixed with reactor liquor carrying catalyst, the reaction to EC takes place liberating a large amount of heat. To remove this heat, liquor is removed from the top of the reactor and pumped through two exchangers and back into the bottom of the reactor.

(Temp) The reaction temperature of 125°F gives the optimum reaction and therefore care should be taken to keep the reactor temperature at this figure. The temperature is controlled by the amount of heat removed in the reactor coolers.

(Recirculation) If the reactor liquor flow is throttled, too much cooling will take place, and the tars and catalyst will deposit on the tubes requiring unnecessary cleaning of the coolers. For this reason, control the reactor temperature by throttling the cooling tower water discharge from each cooler. Always keep reactor liquor flowing through both exchangers, but if one cooler can remove all the heat, stop the CTW flow to the other. The velocity of the reactor liquor through one exchanger would be high enough to cause erosion. The reactor liquor coming out of the exchangers should be 105°F or warmer.

(Pressure) The reactor pressure will be an unknown until some plant experience is gained. The reaction takes place readily at 140 psig and reluctantly at 90 psig. The plant will start up at 90 psig. This means that the temperature will have to be very closely controlled. The reactor pressure is controlled by the PCV on the reactor vent stream. The PCV is located on the second deck, South

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of the Flasher Condenser. The impulse point for the PCV is on the reactor vent line above the top platform on the reactor.

(Catalyst) The catalyst used for this reaction is FeCl_3 . The Ferric Chloride comes in 135# drums. The ultimate usage of FeCl_3 when the plant is up to design rates is 60#/hr. For this reason the catalyst chamber is designed to hold 1 drum of catalyst. The chamber is designed to have a slow flow up through the catalyst and out the side of the chamber. The slow flow will dissolve the catalyst and only the soluble catalyst will be added to the reactor. The chamber is in parallel with the heat exchangers, so if a larger flow through the chamber is desired, the flow through the exchangers will have to be throttled. The need for catalyst will be indicated by an increase in the vent flow and a decrease in reactor temperature. The catalyst addition frequency will be every 2 hours during the initial start-up. After everything is lined out, larger additions will be made to determine the optimum addition rate. This means during low reactor rates that less than a drum of catalyst will be added at a time. See the FeCl_3 addition graph, fig. 3. To add catalyst close the 4" ball valves upstream and downstream of the catalyst chamber. Then pressurize the chamber to 200 psig with N_2 from the N_2 bottles. Open the 1" valve on the Kettle bottom and bleed the EC into the 4" line. When the pressure of the chamber approaches the line pressure, start bleeding Nitrogen into the chamber. When all of the EC is out of the chamber (you can tell by listening to the N_2 bubbling into the 4" line) close the bottom drain valve and open the 1" purge line. Sweep the chamber with nitrogen until all EC vapors are out (2 to 3 minutes). While adding catalyst a small flow of N_2 should be kept going through the chamber so that no air enters the chamber. After adding the catalyst be sure all air is purged out before closing the chamber back up and putting it into service. Note: the purge line is kept free of explosive mixtures by purging continuously with N_2 . The Nitrogen purge flow is indicated by the rotameter

located near the North side of the catalyst chamber. Check this flow after each catalyst addition. The product is removed from the reactor through a level control valve. The normal draw off point will be a nozzle 22'4" from the bottom tangent line.

(Level) The operating level should be kept around 28'. Loss of recirculation will occur if the level gets below 24'. If the level gets below 22'4" (bottom nozzle on sight glass) no liquor can be withdrawn through the product line. If product is to be removed from the reactor when the level is less than 22'4" then the product stream will have to be fed from the recirculation system. By closing the normal draw off valve on the reactor and opening the 2" valve on the recirculation system, the product may be taken from the recirculation stream. When the level is low the lower recirculation suction point will have to be used.

D. Start-up

Normal start-up of the EC reactor is fairly straight forward. With the reactor full of liquor and the proper catalyst in the liquor (about 3900 PPM FeCl_3) the reaction will take place as soon as the proper temperature and pressure is reached.

Start the reactor liquor circulating through the coolers. On one cooler cut the CTW off completely and on the other allow just a small flow of CTW to flow through the inlet valve. Just after the inlet valve is a 1" steam valve: open this valve. The steam and water flow should be varied so the water temperature into the cooler is never warmer than 120°F.

When the reactor liquor temperature reaches 105°F, push the start button and start feeding HCl. When the reactor pressure is up to 90 psig (or the operating pressure), start the C_2H_4 flow. When the reactor starts warming up, the steam flow to the cooler will have to be decreased and the CTW flow increased. Control the CTW flow by the discharge valve after the reactor has

started heating up. After the reactor is warmed up to 125°F the HCl flows and the C_2H_4 flows may be brought up to rates. These adjustments should be made in small increments, with the HCl always increased first. Any decrease in rates is made by decreasing the C_2H_4 first.

Anytime the reactor liquor is below the recirculation valve for start-up the normal method of recirculation can not be used. There is a valve located 7½' from the bottom of the reactor that is tied into the recirculation system. If the reactor is started up using this valve no product can be removed until the normal circulation system is used.

If the reactor has no level, then crude EC will have to be added to the reactor. The moisture in this liquid may be removed by circulating the reactor liquor through the reactor dryer. The moisture should be less than 1000 PPM before any catalyst is added. Enough catalyst should be added to bring the $FeCl_3$ content to 3900 PPM. The new charge is then started up in the normal manner.

E. Reactor Dryer and Filter

1. Reactor Dryer (64A-73-56) - The Reactor Dryer is 30" OD by 6'0" tan. to tan. The capacity of the dryer is 990# of $CaCl_2$. The design pressure of the dryer is 225 psig to full vacuum @ 200°F. The SRV is a 1E2. The bottom support is a 13 ga. type 304 SS mesh cloth located on a steel grate. (The grate is split for removal.) The head is designed to hold up to a 24" wide fibreglass roll. The floating support is a 1" expanded metal.
2. Reactor Filter (64A-72-103) - The Reactor Filter is 9-9/16"OD by 42" high. The filter holds 18 10" filter elements. The elements are triple stacked. The housing is 316 SS and the gaskets are teflon. The original elements are C75A105 designed to stop 75 micron. The element size is fairly independent so replacement may be made with any 10" element.
3. Operation - The dryer and filter will be operated only during start up. This is because the dryer would become clogged very quickly with Ferric Chloride. The dryer is designed for a flow rate of about 70 gal./min. The filter will handle 36 to 180 gal./min. The disposal method for the spent dryer will be determined by the condition of the EC in the dryer. After all the liquid is drained, the material should be swept with nitrogen until no EC is left. The nitrogen purge may be sent to the vent by tying into the vent line. There is a valve located in the vent line near the dryer for this purpose.

4. FLASHER DOPP KETTLE

A. Equipment - The Flasher Kettle is a scraped side, jacketed vessel that is 84" in diameter and 96" on the straight side. The capacity of the kettle when filled to the top of the jacket (78" from the tangent line) is 2000 gallons. The gross volume is 2600 gallons. The jacket area is 180 ft.². The duty of the kettle is 2,300,000 Btu/hr. The kettle design pressure is 85 psig to full vacuum. The jacket is designed for 25 psig.

(Agitator) The driver for the agitator is 15 hp and 1150 RPM. The agitator speed is 20 RPM. The agitator has a tripod step bearing on the bottom end.

(SRV) The safety relief valve for the kettle is a 3L4. The jacket has no relief valve because the 25 psig steam system is relieved.

(Gas Outlet) The vapor outlet is designed so a dish and doughnut may be installed if foaming becomes a problem.

The Flasher Dopp, agitator, and driver equipment numbers are 64A-60-602, 64A-61-86, and 64A-50-1613 respectively.

B. OPERATION

The feed to the flasher is a liquid drawoff from the reactor. The liquid feeds through the LCV located on the West side of the kettle platform and into the kettle. Any rework material can be fed to the kettle by feeding through the rework line which joins the kettle feed line downstream of the Reactor LCV. The Flasher Kettle is designed to concentrate the spent catalyst and the tars about 10 times. The steam flow to the jacket is controlled by the level of liquor in the kettle. The concentrated liquor is removed from the bottom of the kettle through a steam traced line to the Bottoms Dopp Kettle. The feed to the Bottoms Kettle is a gravity drain and is controlled by

throttling the valve at the Bottoms Dopp. There is a Strahman Valve located on the bottom of the Flasher that is to be used if the normal drain is plugged. There is a thermocouple located in the bottom of the kettle and one located in the vapor line. The bottom temperature will be correlated to the tars in the kettle, and this temperature will be used to control the blowdown to the Bottoms Kettle.

C. Start-Up - The kettle starts up fairly easy. When an operating level is reached in the kettle, steam should be slowly opened to the jacket. The agitator is never shutdown unless the kettle is completely empty. Steam flow can be controlled by level or put on manual, as with a constant steam flow the level will decrease until an equilibrium level is reached. With the steam on manual, be sure the bottom temperature does not get too high before the equilibrium level is reached.

5. BOTTOMS DOPP KETTLE

A. Equipment

The Bottoms Dopp Kettle is a scraped side, jacketed vessel that is 36" in diameter and 36" on the straight side. The capacity of the kettle when filled to the top of the jacket is 150 gallons and the gross volume is 185 gallons. The heat load of the kettle is 210,000 Btu/hr. The kettle design pressure is 85 psig to full vacuum. The jacket is designed for 25 psig.

(Agitator) The driver for the agitator is 5 hp and 1750 RPM. The agitator speed is 37 RPM.

(SRV) The safety relief valve is a 1½ H3. The jacket is not relieved. The Bottoms Dopp, agitator, and driver equipment numbers are 64A-60-603, 64A-61-87 and 64A-50-1614 respectively.

B. OPERATION The feed to the Bottoms Kettle is gravity fed from the bottom of the Flasher kettle. The flow to the kettle is throttled by the globe valve at the inlet to the kettle. The feed to the kettle is determined by the concentration being done in the Flasher. The vapors from the kettle are fed to the Flasher or to the Flasher vapor line. Normal feed is to the Flasher. The steam to the jacket is controlled by a field mounted PCV. This valve is controlled by adjusting the bolt on the compression spring. Turn the bolt clockwise to increase steam pressure, and always reset the lock-nut when finished adjusting. When tars are to be drained from the kettle, stop the feed to the kettle and concentrate the tars so as little EC is lost as possible. Then hook up the tar-buggy to the drain connections and to the vent line and then slowly open the Strahman valve on the bottom of the kettle. When the kettle is empty, put the kettle back in service. Leave the tar buggy connected to the vent line as long as it is in the area, but open and drain the drain line between the kettle and the buggy. The thermocouple for the Bottoms Kettle is located in the vapor outlet. Tar concentration will be determined by experience.

The material in the Reactor Dump tank may be fed directly to the Bottoms Kettle.

C. START-UP There are no special problems with the kettle operating or starting up. Never shut down the agitator unless the Kettle is completely empty. Observe the tar thickness to control steam.

6. FLASHER CONDENSER

A. EQUIPMENT 64A-71-780 - The Flasher Condenser is 18" OD with 14' tubes. The shell side is single pass with side to side segmental baffles at 16" intervals. The tube side is 4 pass. The design

pressure of the tube side and the shell side is 150 psig. There are 244 3/4" OD x 14 BWG seamless steel tubes. The exchanger is designed to remove 2,190,000 Btu/hr by condensing 14,336 #/hr. of EC at 139°F. The cooling tower water is to be heated from 90°F to 110°F.

B. OPERATION The Flasher Condenser condenses the vapors that are boiled off in the Flasher Kettle. These vapors should be essentially pure EC. The vapors from the Bottoms Kettle may be fed directly to the Flasher Condenser, also. The cooling tower water should be throttled with the discharge valve from the exchanger to give a temperature of 110°F. The first 1/2" tap under the exchanger on the condensate line is a sample point. The condensate from this exchanger should run about 110°F, and it is gravity fed to the Primary Stripper Feed Drum. The inerts from the exchanger leaves at about 110°F and are fed to the Secondary Vent Condenser. The inerts are the dissolved HCl, C₂H₆, H₂ and the non-condensed EC.

7. PRIMARY VENT CONDENSER

A. Equipment 64A-71-779 The Primary Vent Condenser is 10" OD with 16' tubes. The shell side is single pass with side to side segmental baffles at 16" intervals. The tube side is 4 pass. The design pressure of the tube side and the shell side is 150 psig. There are 72 3/4"OD x 14 BWG seamless steel tubes. The exchanger is designed to remove 610,000 Btu/hr by condensing 3,816#/hr of mostly EC and cooling 1809#/hr of inerts to 90°F. The well water is to be heated from 78°F to 90°F.

B. Operation The Primary Vent Condenser condenses and cools the vapors from the Reactor, Primary Stripper, and the Secondary Stripper. The vapors enter the exchanger at 122°F and are cooled to 90°F.

CONFIDENTIAL:

-47- Subject to Protective Order
of 14th Judicial District Court
No. 91-1145

SL 008887

Cooling this stream from 110°F to 90°F nets a savings of 15 tons of refrigeration, so all attempts should be made to keep the inerts stream as cool as possible. The well water discharge from this condenser is fed to the cooling tower as make-up. Should the water required for this exchanger be more than cooling tower make-up, throttle the 3" gate valve that is located above the addition basin. By throttling this valve the excess water is spilled to the sewer. The discharge water temperature should be kept around 90°F, by throttling the discharge valve. A close watch should be kept on the tubes of this exchanger. Any time the plant is down the tubes should be examined until some history has been built on well water cooling. If severe corrosion is observed then the exchanger may be put on cooling tower water. (Stub-outs are provided on the cooling tower headers). The inerts from this exchanger (mostly HCl and EC) are sent to the Secondary Vent Condenser. The condensate gravity feeds to the Primary Stripper Feed Drum. The first ½" tap under the exchanger on the condensate line is a sample point.

8. SECONDARY VENT CONDENSER

- A. Equipment 64A-71-786 The Secondary Vent Condenser is 14"OD with a 16' U-bundle. The shell side is designed for pool boiling with a deentraining section while the tube side is 2 pass. The design pressure of the shell side is 300 psi and of the tube side 150 psi. There are 58 3/4 x 14BWG seamless steel tubes. The exchanger is designed to remove 365,000 Btu/hr by condensing 1819#/hr of essentially EC and cooling 909#/hr of inerts to 2°F. The cooling is done by boiling Freon 22 in the shell side, where the pressure is 24 psig.
- B. Operation The inerts from the Primary Vent Condenser and from the Flasher Condenser are fed to the Secondary Vent Condenser. In

CONFIDENTIAL:

Subject to Protective Order
-48- of 14th Judicial District Court
No. 91-1145

this condenser the last of the salvageable EC is reclaimed and sent to the Primary Stripper Feed Drum. The vents are fed into the tubes of the condenser. The non-condensables and the condensate come out of the bottom of the head and are fed to a vapor-separator. The condensate is fed from the bottom of the vapor separator to the Primary Stripper Feed Drum. Sampling of the condensate may be done on the first valve under the vapor separator. The valve located in the vapor separator bottom is for draining. The inerts are fed from the top of the vapor separator through a PCV. This PCV controls the pressure on the Flasher Dopp, Bottoms Dopp, the Primary Stripper and the Primary Stripper Feed Drum. From the PCV the inerts may go through the valve on the same level as the PCV to the EC scrubber, or the inerts may be sent through the valve at the PCV bypass height to #2 Absorber in the HCl Plant. The temperature of the freon in the condenser shell is controlled by the reciprocating compressors. The normal pressure will be 24 psig, which is about 2°F. Full operating controls are discussed in the refrigeration machine discussion. The vent system (Primary and Secondary Condensers) may be bypassed. The vents from the Primary and Secondary Strippers as well as the Still vent may be sent to the PCV direct. The result would be a loss of EC.

9. VENT SCRUBBER

A. Equipment 64A-67-20 The vent scrubber is a Haveg column 2'ID by 10' high. The material of construction is Haveg 41 composite and all gaskets are Teflon envelope with asbestos filler. The column is designed for 10 psig pressure at 200°F. The column is packed with 6' of 1½" Intalox saddles. The support plate is a chemical porcelain U. S. Stoneware figure 808 gas injection plate, split into 3 pieces.

CONFIDENTIAL:

Subject to Protective Order
-49- of 14th Judicial District Court

SL 008889

The two liquid inlets are sparged into the middle of the column. The column will neutralize 5,700#/hr of HCl with 95 GPM of water.

The outlet temperature should be 180°F.

B. Operation The vent scrubber is designed to handle a sudden loss of reaction. With the advent of the Wet HCl system it became feasible to absorb and reclaim the excess HCl, so for the great part of the time no HCl will be sent to the scrubber. When the scrubber is in use, the Cooling Tower blowdown may be used for the majority of the neutralization. Enough well water should be added to keep the outlet liquid temperature less than 180°F. When the tower is not being used enough water should be added to keep a seal on the bottom as EC and N₂ are continuously purged through the tower and to the vent stack. All purging of evacuated vessels, catalyst addition chamber, or Dryers will be sent through the Scrubber, out the top and to the vent stack.

The vent stack is a 3" Haveg line that runs from the top of the vent scrubber to the top of the Hvs Still. The top 5' of the line is constructed of nickel pipe and 9" from the top of this is a nickel mesh flame arrestor. The vent line has a steam snuffer at the top to extinguish any fire. During electrical storms, it is expected that anytime flammables are vented, the stack vent will be ignited. Whenever the vent is burning, go to the Northwest corner of the platform and open the valve in the 1" line that takes off the steam to the Hvs Still Reboiler line just before the control station. This 1" line will inject steam into the vent and smother the fire. When the fire is extinguished, shut the steam back off.

10. PRIMARY STRIPPER FEED DRUM

A. Equipment 64A-60-596 The Primary Stripper Feed Drum is 5'0" in

diameter and 15'6" tangent to tangent. The capacity of the Drum is 2430 gallons with no outage. The design pressure is 100 psi and full vacuum at 175°F. The three liquid inlet lines have anchored standlegs. The SRV is a 2J3.

B. Operation The Primary Stripper Feed Drum was designed for 30 min. retention $\frac{1}{2}$ full to full. The equalization of this tank feeds back to the inlet to the Secondary Vent Condenser. Close attention should be paid to this line. If the vent is too cold, too much HCl will be condensed and will be boiling back off in the drum. Also, if the Vent Condenser becomes plugged, it is possible for vapors to come down the equalization line through the Drum, up the Vapor Separator condensate line and out the vent. A loop was avoided in the condensate line because corrosion products will collect and plug the loop. Liquid is removed from the bottom of the drum and pumped to the Primary Stripper through a LCV, a CaCl_2 dryer, and a filter to the top of the Primary Stripper.

The Primary Stripper Feed pump is the sample point for the Primary Stripper Feed Drum. The high pressure point is in the discharge line at the "T" and the low pressure point is on the suction header.

The feed to the Primary Stripper is normally level controlled. To flow control this column would require close attention, as there is very little surge time in the feed drum. When the operation dictates that the feed should be flow controlled, it is possible to switch to the flow controller. Behind the instruments there is a 3 way valve arrangement that is marked so that the control mode may be switched from flow to level by opening and shutting the correct valves.

Should the contents of the Primary Stripper Feed drum get abnormally high in moisture, the contents of the drum may be recirculated through the dryers and back to the drum by making use of the 2" line that is located in the filter discharge manifold. This may be done with or without shutting down the stripper and may be done with either flow or level control. Probably flow control would give the smoothest operation.

11. GLAND SEAL PUMP

A. Equipment 64A-55-848 The Gland Seal Pump is a Lapp Pulsafeeder CPS-1 designed for 2 GPH at 150 psig. The pump will pump up to 6.6 GPH at 450 psig. The diaphragm is Teflon as are the valve and cap gasket. The valve seat is 316 SS.

B. Operation The Gland Seal Pump is to furnish a clean liquid for the seals on the Reactor Recirculation pumps. The Gland Seal Pump takes its suction from the suction line of the Primary Stripper Feed Pumps. The pump then pumps the material through a rotameter, through a back pressure valve and to the seal of the operating pump. The backpressure valve should be set to keep the gland pump discharge 5 psi above the discharge pressure of the Reactor Recirculation Pumps. The flow to the seal should be set for 2 GPH. All flow adjustments are made by varying the stroke length on the pump. Do not throttle the discharge of this pump, even though it has a built in relief valve.

12. PRIMARY STRIPPER FEED DRYERS AND FILTERS

A. Equipment

1. Dryer 64A-73-57 & 58 - The Stripper Feed Dryers are 30"OD by 6'0" tangent to tangent. The capacity of the dryers is 990# of CaCl_2 . The design pressure of the dryer is 150 psi to full vacuum at 200°F. The SRV is a 1E2. The bottom support is 13 GA type 304SS mesh cloth located on a steel grate. (The grate is split for removal.) The Head is designed to hold up to a 24" wide fibreglass roll. The floating support is 1" expanded metal.
2. Filter 64A-72-101 & 102 - The filters are 9-9/16"OD by 32"

The filter holds 12 10" filter elements. The elements are double stacked. The housing is stainless steel, the gaskets are Teflon. The original elements are C75Al05 designed to stop 75 micron. The element size is fairly independent so replacement may be made with any 10" element.

B. Operation The dryers and filters are arranged so that either filter may be used with either dryer. The dryers should dry over 215,000# of fairly wet EC. Under normal conditions the dryers will last for months. High CaCl_2 use indicates trouble.

To empty the spent dryer the EC may be pushed from one dryer to the next with nitrogen. Be sure and check pressures before tying in the nitrogen. When pushing material out the bottom of the dryer be sure and observe the material through the bulls-eye. Any cruddy or water phase will come out first and should be disposed of as it will harm the good dryer. After all of the liquid is drained, then the material should be swept with nitrogen until no EC is left. The nitrogen purge may be sent to the vent by tying into the vent line. There is a valve located in the vent line near the dryer area.

13. PRIMARY STRIPPER AND REBOILER

A. Equipment

1. Still 64A-67-17 The Primary Stripper is a vessel 22" OD by 37'8" tangent to tangent. The skirt is 14'10". The column has 2 12' packed sections. The packing used is 1" Intalox saddles. The column internals are as follows: the top section has a hold down plate and a distribution plate at the top and a gas injection support plate and a redistribution plate at the bottom, the bottom section has a gas injection support plate at the bottom. The SRV is a 1½H3.
2. Reboiler 64A-71-781 The Primary Still Reboiler is a 8-5/8" OD shell with 55 3/4" 13BWG tubes that are 8'0" long. Both the shell and tube are designed for 150 psi at 400°F. The exchange has an expansion joint. The reboiler should vaporize 5,071#/hr of EC by condensing 793#/hr of steam at 5 psig.

B. Operation The Primary Stripper is to remove the soluble HCl from the forward EC stream. The HCl and any other lights are taken overhead from the column to the Primary Vent Condenser. The still operates at the system pressure of 50 psig. The feed to the column is in essence

both a feed and a reflux and is fed to the column at the top of the top section. The steam to the reboiler is flow controlled. The actual flow rate will be determined by HCl analysis of the Hvs Still feed. Figure 5 shows a preliminary correlation and should be used until a better graph is obtained. Any increase in steam flow will increase the overheads which will increase the level in the Primary Stripper Feed Drum which in turn, increases the feed. The net result is an increase in the bottom temperature of the stripper which will result in less HCl in the bottom stream. The purified EC is overflowed from the bottom of the still to the Heavies Still Feed Drum. The overflow system is designed to keep a constant head on the reboiler liquid and to prevent any flooding of the reboiler. This type of arrangement makes the column easy to operate but hard to start-up and shut-down. In the case of this column, small amounts of material high in HCl content may be spilled into the Hvs Still Feed Drum without doing any harm, but the net result of continuous bad material being overflowed is a high vapor loading of the Hvs Still and condenser, a high load on the Secondary Stripper, a higher operating cost and a possible out of spec. product.

There are three thermosyphon reboilers in this plant and a brief review of the principles of this type of reboilers is merited. The principle of the thermosyphon reboiler is to obtain a rapid circulation through the exchanger tubes by vaporizing part of the liquid in the tubes and allowing the vapors to leave the top of the exchanger and enter the side of the column below the first tray or support plate. (This is the same principle that may be observed in a percolator coffee pot. The vaporized material lifts the liquid up the tube.) The vaporized material is replaced by the liquid which comes down from the

first tray in the still through a liquid leg into the bottom of the exchanger. This vaporization creates a natural circulation of liquid (thermosyphon) through the reboiler, thus eliminating any need for pumps. The rapid circulation rate through the exchanger accomplishes two things: First, a high rate of heat transfer to the liquid due to the high thermosyphon liquid velocity is obtained. Second, this high liquid velocity lessens the chance of the formation of scale or tars on the exchanger walls and thereby either eliminates the need for cleaning or greatly extends the time between cleanings.

Three very important items must be remembered when starting up and operating a thermosyphon reboiler: First, never let a reboiler run dry or operate with a low level. This allows some or all of the tubes to become dry and heat up to the temperature of the steam. This excessively high temperature can cause baking of tars onto the tubes, decomposition of the material in the reboiler, and formation of more tars which can plug the reboiler tubes. Second, when starting up a cold reboiler, drain all accumulated steam condensate out of the shell of the exchanger and begin feeding steam slowly. The trap should be bypassed until the condenser is warmed up. Any condensate in the shell will cause a steam hammer ~~and hot steam hitting the tube walls quickly will flash the reboiler dry~~. Third, never run a reboiler with a high level. If the liquid level gets above the vapor line, the thermosyphon action will stop and result in a drastically reduced heat transfer. Whenever a reboiler becomes flooded the steam should be shut off and the liquid level drained to an operating level. A flooded thermosyphon will develop a high steam pressure due to the loss of circulation which results in a high temperature difference and probable coking.

C. Start-up The Primary Stripper can never be started up with a flooded reboiler as long as the overflow line is left open. To start up the column, start feed to it, when the reboiler shows an operating level slowly start steam to the reboiler; enough feed should be maintained to keep the liquid level in the column just below the overflow line. When shutting down the column, stop the steam and then stop the feed. This will allow the column to dump, but keeps the reboiler full.

14. HEAVIES STILL FEED DRUM

A. Equipment 64A-60-597 The Heavies Still Feed Drum is a 9' OD by 21' tangent to tangent tank. The capacity of the tank is 10,880 gallons with no outage. The design pressure is 100 psi to full vacuum at 225°F. The liquid overflow line from the Primary Stripper is the only standleg in the tank. The SRV is a 3L4.

B. Operation The Heavies Still Feed Drum is designed for 2 hours retention half full to full. This drum is the only surge capacity in the plant and its purpose is to give the Hvs Still a constant feed. The Hvs Still Feed stream comes from the bottom of the drum, through the Hvs Still Feed pumps, a FCV, and to the Hvs Still feedpoints.

15. HEAVIES STILL

A. Equipment

1. Heavies Still 64A-67-18 The Hvs Still is a 44" OD by 50' tangent to tangent. The skirt is 16'6". The column is designed for 100 psi to full vacuum at 300°F. The column has 36 Koch Flexitrays each with 103 type "A" caps. The trays are on 12" intervals and are made of carbon steel with the bolts 410 SS and the nuts 304 SS. The valve caps are 410 SS with some 14 ga and some 16 ga. The manways are removable from either side. The feed nozzles are located on trays #20, #22, #24, or #26, reflux is on tray #36. The pot contains 6.58 gallons/inch. The SRV is a 4P6.

2. Heavies Still Reboiler 64A-71-782 The Heavies Still Reboiler is a 20" OD shell with 349 3/4" 13BWG tubes that are 10' long. The shell is designed for 150 psi at 500°F and the tubes are designed for 150 psi at 250°F. The exchanger has an expansion joint. The reboiler should vaporize 327,000#/hr of EC by condensing 5,740#/hr of steam at 25 psig adding 5,369,000 Btu/hr.

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of 14th Judicial District Court

3. Heavies Still Condenser 64A-71-784 The Hvs Still Condenser is a 20" OD shell with 308 3/4" OD 14BWG tubes that are 16' long. The shell side is single pass with side to side segmental baffles at 24" intervals and the tube side is 4 pass. The design pressure of the shell side is 150 psi at 300°F and the tube side 150 psi at 150°F. The exchanger is designed to remove 5,250,000 Btu/hr by condensing 36,714#/hr of EC at 150°F with 210,000#/hr of cooling tower water. The CTW is to be heated from 90°F to 115°F.

4. Heavies Still Bottom Cooler 64A-71-509 The Heavies Still Bottoms cooler is a 8" diameter shell with 48 3/4" 16 BWG tubes that are 8' long. The shell is designed for 150 psi at 250°F and the tubes are designed for 150 psi at 125°F. The exchanger is 1 shell pass with two tube passes. The shell baffles are on a 24 inch pitch. This exchanger is a reclaimed exchanger with a mixture of tubes in it. About 1/3 of the tubes are plugged.

5. Heavies Still Reflux Drum 64A-60-598 The Hvs Still Reflux Drum is a 5' OD by 13½ tangent to tangent tank. The capacity of the drum is 2,130 gallons with no outage. The design pressure is 100 psi to full vacuum at 225°F. The liquid drain from the condenser is the only standleg. The SRV is a 2J3.

B. Operation The Hvs Still is to remove any heavies that might be formed in the reactor or brought in with the HCl feed. The product EC plus any lights is taken overhead to the Hvs Still Condenser. The pressure on the column is maintained at 60 psig by a PCV on the inert discharge of the condenser. If no inerts are present then N₂ will bleed into the condenser until the pressure is maintained. This is a likely spot to cause the vent condenser to overload by leaking N₂ through the valve and carrying EC to the vent condenser. The heavies are collected in the bottom of the tower and periodically blown to the EDC plant disposal. Originally the bottoms will be piped to flasher system at EDC. When the exact bottoms concentration is known the final storage of bottoms will be determined. The bottoms should never be allowed to get any higher than the top of the sight glass nor any lower than the bottom of the sight glass. If the blowdown is taken from the side of the column rather than the bottom of the reboiler, too much bottoms cannot be removed. The blowdown will be taken from the bottom of the reboiler until all moisture problems are eliminated.

The column is designed to run at a reflux ratio of 1 to 1.

The feed should enter on tray #22 under normal conditions. The reboiler is to be handled like all thermosyphons, as explained on the Primary Stripper Reboiler. Sample points are located every 4 trays to withdraw a liquid sample. Thermocouples are located every other tray in the downcomer. The normal mode of control will be steam flow controlled by the bottom temperature. In order for the column to be temperature controlled, some bottoms will have to be built up and a proper thermocouple for control located. Until the bottoms are built up the column will operate on manual.

The level in the reflux drum controls the feed to the Secondary Stripper, the reflux to the Hvs Still is flow controlled. One pump pumps both the reflux and the Stripper feed. Any HCl that is formed in the still by cracking will go with the product stream or be vented back to the Secondary Vent Condenser.

16. SECONDARY STRIPPER AND REBOILER

A. Equipment

1. Still 64A-67-19 The Secondary Stripper is a vessel 22" ID by 37'8" tangent to tangent. The skirt is 12'10". The column has 2 12' packed sections. The packing used is 1" Intalox saddles. The column internals are as follows: The top section has a hold down plate and a distribution plate at the top and a gas injection support plate and a redistribution plate at the bottom. The bottom section has a gas injection support plate at the bottom. The SRV is a 1½H3.

2. Reboiler 64A-71-783 The Primary Still Reboiler is a 8-5/8" OD shell with 55 3/4" 13 BWG tubes that are 8'0" long. Both the shell and tubes are designed for 150 psi with the tube side at 250°F and the shell side at 500°F. The exchanger has an expansion joint. The reboiler should vaporize 4,136#/hr of EC by condensing 633#/hr of steam at 5 psig.

B. Operation The Secondary Still is to remove all traces of HCl that are in the product stream. Some HCl will come from cracking in the Hvs Still. The Secondary Stripper is designed to remove HCl down to 1 PPM, it is expected that the bottoms will run between 1 and 10 PPM.

The HCl and any other lights will go overhead from the still through a PCV to the Primary Vent Condenser. The PCV is to control the stripper pressure at 60 psig. This higher pressure will isolate it from the rest of the system and prevent surging. There is no attempt to purge any inerts into the system. As with the Primary Stripper the feed is both a feed and a reflux and is fed to the top of the first packed section. The steam to the reboiler is flow controlled and the actual flow rate will be determined by HCl analysis of the product stream. Figure 6 shows a preliminary correlation and should be used until a better graph is obtained. If too much steam is used the result will be an increase in the operating costs of the stills, however, if too little steam is used the product will be out of spec. The column is designed to handle some rework along with the normal feed. The rework plus the feed should run less than a total of 70 GPM.

The product grade EC is overflowed from the bottom of the still to the Product Surge Tank. The overflow system is designed to keep a constant head on the reboiler liquid and to prevent any flooding of the reboiler. This type of arrangement makes the column easy to operate but hard to start-up and shut-down.

C. Start-Up To start up the Secondary Stripper the Product Surge Tank must be recycled back to the Hvs Still Reflux drum. Material should be fed to the Stripper until the reboiler has a level, then the feed cut way back. Slowly heat up the reboiler, but always keep the level in the bottom of the stripper overflowing. Continue to increase the steam until the Product Surge Tank contents show an acceptable HCl content, then send the stream to the day tank. To shut down the Stripper the product stream should be sent to the Heavies Still Reflux Drum and then the steam to the reboiler stopped.

CONFIDENTIAL:

-59- Subject to Protective Order
of 14th Judicial District Court
No. 91-1145

SL 008899

Because of limited surge volume available in the back end of the plant, feed to the still should be stopped before shutting down the stripper.

17. PRODUCT SURGE TANK

A. Equipment

1. Product Surge Tank 64A-60-599 The Product Surge Tank is a 4' OD by 10½' tangent to tangent vessel. The capacity of the tank is 1040 gallons with no outage. The design pressure is 100 psi to full vacuum at 225°F. The liquid overflow line from the Secondary Stripper is the only standleg in the tank. The SRV is a 1½H3.

2. Product Cooler 64A-71-785 The product cooler is a Brown Fintube multitube exchanger. The exchanger is Model 06-1B000-320. There are 7 fintubes in each bundle. Each tube is 7/8 OD with a 0.083" wall and a length of 20'. The design pressure is 500 psi at 650°F for both the shell and tube. The surface area is 261.1 ft². The exchanger is to cool 15,221#/hr (35.2GPM) of EC from 139°F to 110°F by heating 21.6 GPM of CTW from 90°F to 115°F.

B. Operation The Product Surge tank is level controlled. The product stream is pumped through a level control valve, through the Product Cooler and to the Day Tank Area. Under the Product Cooler there is a valve with an extension handle on it. This valve is the last valve before the day tank valves, and should be closed whenever any material is being reworked to prevent the reworked material from contaminating the line.

The rework line (line #507) begins in the storage area at the Rework Pump and goes all the way through the area to the feed line to the Flasher Dopp. From this line the material may be fed to the Flasher Dopp or to the reactor. The rework line has ties to the suction and discharge of the Primary Stripper Feed Pumps, the Hvs Still Feed Pumps, the Hvs Still Reflux Pumps, and a tie from the discharge of the Product Cooler. By the use of this rework line material can be moved to any part of the plant from any part of the plant.

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

18. DAY TANK AREA SYSTEM

A. Equipment

1. Day Tanks 64A-60-604, -605, -606, -607 Each Day tank is 12' OD by 54'8" seam to seam. The capacity of each tank is 52,270 gallons with no outage. The design pressure is 75 psi and full vacuum at 225°F. The SRV is a 6R8. The production line and the 3" recirculation lines are the only standlegs.

2. Vent Compressor 64A-56-145 The vent compressor is a Corken Model D-290-107 with no aluminum parts. The compressor operates at 850 RPM and compresses 12.7 CFM at 90°F from 40 psig to 200 psig. This compression requires a 7½ hp, 1850 RPM motor. The SRV is a Farris model 2740 set at 250 psig.

3. Storage Vent Condenser 64A-71-789 The Storage Vent condenser is 6" OD with 10' U-tubes. The shell side is single pass with side to side segmental baffles at 6" intervals. The tube side is 4 pass. The design pressure of the shell side is 200 psi and the tube side 150 psi, both at -15°F. There are 6 3/4" OD x 14BWG seamless steel U tubes. The exchanger is designed to give 15°F inerts leaving at 200 psi. The SRV is a 1D2.

4. Transfer Pumps 64A-55-849, -850 The transfer pumps are designed to pump 250 gallons apiece at 125' of head. The pumps are Gould's 3196 2 x 3-6 with a 5-3/4" impeller.

5. Rework Pump 64A-55-851 The rework pump is designed to pump 40 GPM at 200' of head. The pump is a Gould's 3196 1 x 2-8 with a 7" impeller.

B. Operation The day tanks are designed to hold approximately 1 days production. Actually with a 10% outage the tanks will hold about 22 hours of production at design rates. The tanks are designed for a common equalization system, and for this reason care should be taken to see that a tank is never isolated. If for any reason a tank is isolated, a close watch on the tank should be maintained to prevent either over or under pressurization. Each tank is equipped with a Varec and a level transmitter. If trouble with the Varec is suspected there are taps on the level transmitter system for a temporary sight glass installation. There are two withdrawal lines on each tank. One line goes to the rework pump which will pump EC back to the process area. The other line goes to the transfer pumps where the EC may be pumped back to any of the tanks or to the tank car loading facilities or to the Horton Spheres. EC may be pumped back from the Horton Spheres, or pressurized from a tank car, bypassing the pumps into the pump suction

CONFIDENTIAL:

Subject to Protective Order
of 14th Judicial District Court

line and from this line into any tank. The transfer pump suction line is insulated to prevent EC from vaporizing in the suction line when the pumps are not in use. Some trouble may be experienced with the rework pump in getting it to start pumping. When this happens it is best to try to fill the suction line with cool EC. The material in the dock line will normally float against one of the spheres. ANYTIME THE LINE IS CLOSED GOING INTO THE SPHERES THE BLOCK VALVE IN THE BYPASS AROUND THE PUMPS AND THE SUCTION BLOCK VALVE ON ONE TANK SHOULD BE OPENED TO GIVE EXPANDING EC A PLACE TO GO. Under normal conditions both suction lines on the bottom of each tank will have the valve closed.

Due to fluctuating levels in the tanks and sudden temperature changes, it was necessary to install a vent system for the tanks. The vent system will bleed in nitrogen when the pressure is low and will purge N_2 and EC when the pressure is high. To prevent a loss of a large amount of EC a compressor and a refrigerated condenser were installed. Whenever the pressure in the Day Tank vent system goes below 15 psig, nitrogen will be bled into the system. Whenever the pressure goes above ³⁵~~40~~ psig (this pressure will depend upon what is acceptable to our customers), a valve will open in the suction line to the compressor and the compressor will start-up. The compressor discharges to the vent condenser where ¹⁷⁵~~200~~ psig is maintained by a PCV in the inert gas discharge of the condenser. Brine from the HCl plant at 5° F. is circulated through the tubes of the condenser. The inert discharge should run about 15° F. The condensate goes through a float chamber, a LCV, and to #3 or #4 Day Tank. The float chamber and LCV prevent vapors from the tanks from

entering the condenser and artificially loading the condenser. The vent compressor has a distance piece which is swept with N₂ from one side with the inerts and any leakage leaving the other side and going to the vent. There is a nitrogen PCV that will bleed N₂ into the vent condenser whenever the pressure in the condenser is less than ¹⁰~~5~~ psig. The compressor may be bypassed, if necessary, with a resultant loss of EC. The tank car loading area has a line to the suction of the compressor. Material should be pulled from the tank car and put through the vent system only after analysis has shown the material to be in spec. Any off-spec material should either bypass the vent system, or if it is put through the vent system, the condensate should not be collected in the day tank. Anytime off-spec material is being sent through the vent system, be sure and isolate each tank from the system to prevent further contamination.

Anytime a tank is to be opened up, the compressor will be used to pull a vacuum on the tank. After a vacuum has been pulled, the tank is to be purged with nitrogen; and since there should be little EC in the N₂, this purge may be sent straight to the vent condenser. (The venting pressure will have to be lowered to a pressure less than the N₂ pressure.) When the vessel purge shows no flammables, then the tank should be isolated and air purged through it if it is to be entered.

19. DOCK AREA SYSTEM

A. Equipment

1. Horton Spheres 64A-60-610, -620 Each Horton Sphere is 43½' in diameter. The capacity of each tank is 323,000 gallons with no outage (at 85% full 275,000 gallons). The design pressure of the spheres is 75 psi at ambient temperatures. The vacuum rating is 6.39 psi vacuum. There are no standlegs. The SRV is a 8T10.

CONFIDENTIAL:

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

2. Atwood-Morrill Excess Flow Valves These valves are in the barge loading line to prevent the contents of the sphere from being dumped into the lake in case of a rupture during loading. The valves will allow up to 1000 GPM to flow before closing.

3. Turbine Flow Meters These are Foxboro Turbine Flow Meters, Model 81-F5A1-4, nominal 4" size, range is 18 to 1050 GPM.

4. York Refrigeration Unit The York compressor is a 10-ton unit designed to cool 60 GPM of brine from 5° F. to 0° F. The unit includes:

A. Compressor, 64A-56-128, York Model FX 3049-5BE, 3 3/4" x 3", 4 cylinder V/W Refrigerant 22 compressor operating at 1170 RPM. This unit has an automatic 50% capacity reducer, a forced feed lubrication system, an oil filter, an oil heater, suction strainer, internal relief valve and the other normal accessories.

B. The condenser, 64A-71-541, is a York Model 805 4-pass horizontal Shell and tube type with copper tubes and tube sheets, steel shell, cast iron water heads and relief valve. The total area is 101 ft.². The exchanger is 8 5/8" diameter with 37 5/8" tubes, 5 1/2' long. The tubes are extruded fin copper tubes. The shell side is designed for 300 psi; the tube side for 150 psi.

C. The Brine Cooler, 64A-70-49, is a model C1208, 4-pass horizontal shell and tube flooded type furnished with 16 gauge extruded finned steel tubes, steel shell, cast iron water heads, bullseye sight glass, relief valve, float controller and an area of 190 ft.². The exchanger is 12" diameter, with 48 3/4" extruded fin, seamless steel tubes. The shell is designed for 225 psi at 300° F. and the tubes for 150 psi at 120° F.

5. Brine Circulation Pumps - 64A-55-654 & 655 The brine circulating pumps are designed for each pump to pump 57 GPM at 78' of head. The pumps are Durco DCHD1R 1 1/2 x 2-9 with 8 1/2" impellers.

6. Barge Loading Pumps - 64A-55-858 & 859 The loading pumps are designed for each pump to pump 600 GPM at 100' of head. The pumps are Goulds 3196, size 3 x 4-11 with 10 11/16" impellers.

7. Vent Compressor - 64A-56-147 The dock area vent compressor is a Corken Model D-490-107 with no aluminum parts. The compressor operates at 850 RPM and compresses 27.3 CFM from 40 psig and 90° F. to 200 psig. This compressor requires a 20 HP, 1850 RPM motor. The SRV is a 3/4" Farris 2740 set at 250 psig.

8. Dock Storage Vent Condenser - 64A-71-930 The dock storage vent condenser is 8 5/8" OD with 8' U-tubes. The shell side is single pass with side to side segmental baffles at 6" intervals. The tube side is 2-pass. The design pressure of the shell is 300 psi at -20° F. and the tube side is 150 psi at -20° F. There are 20 3/4" OD x 16 BWG seamless steel U-tubes. The exchanger is designed to give 15° F. inerts leaving at 200 psi. The SRV is a 1DZ.

9. Vent Condensate Receiver - 64A-60-609 The vent condensate tank is 36" ID by 7'10" tan to tan. The capacity of the tank is 425 gallons with no outage. The design pressure of the tank is 275 psi to full vacuum at -20° F. The SRV is a 1 1/2" F2. The vessel has one standleg which is the condensate line. The sight glass is a frost-free.

CONFIDENTIAL:

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

10. Vent Condensate Pump - 64A-55-639 The Vent Condensate Pump is designed to pump 10 GPM at 109' of head at 3450 RPM. The pump will run at 1750 RPM. The pump is an Ingersoll-Rand 1CRVM with a 5½" impeller.

B. Operation Each Horton Sphere is designed to hold one barge-load of material. Both barge tanks are to be loaded out of one sphere. From the sphere to the suction of each pump is a common line. Each barge tank is fed through an individual line from each pump.

Since the EC spheres cannot be taped before each shipment, an accurate means of determining the amount of EC shipped was needed. It was felt that Verac gauges on the spheres would serve if they were double checked. To check the Verac a flow meter was installed in the loading lines. The flow meters will also serve to keep the barge tanks filled equally. The rate of flow can be read as total gallons through each turbine meter. At the pump discharge there is an equalizing line between the two pump discharge lines. Once the EC is past this point, it flows in separate lines through the turbine meters to the barge tanks. When it is desired to pump EC back to the plant, it may be pumped from the discharge equalizing line through the 3" line into the transfer line. With the lines closed going into the spheres the EC will come back to the area. When it is desired to recirculate a sphere or transfer from one sphere to another, the EC is pumped through the equalizing line into the transfer line, with the appropriate sphere opened and the Day tanks and the other sphere closed. This line is also used for expansion liquor also. When the barge is finished being loaded, the block valves at the loading arm and at the sphere bottom are closed. ~~The 2" intake line (line 635) is to be opened.~~ This allows any thermo-expansion of the liquid in the barge lines to ~~come back~~ back to the sphere.

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

SL 008905

From the turbine meters the EC flows underground to the EC loading dock. At the dock the EC flows through an excess flow valve, through the loading arm and into the barge tank. The excess flow valve is an Atwood-Morrill and should allow up to 1000 GPM to flow through before closing. This valve works on the principle of comparing the flow through the valve against the downstream pressure. When the pressure difference becomes greater than the designed ΔP , the valve will close. The valve is opened by switching the downstream pressure tap to the upstream side by means of a manifold. (The manifold is a 3-way valve activated by a foot pedal.)

When the barge is being filled, the displaced gases will return to the top of the sphere the liquid is being removed from. For this reason the chemical analysis of a barge vapor space must be known before it is equalized back to the tanks. If the vapors are contaminated they could contaminate a sphere of good material, as well as get impurities into the sphere to contaminate pure EC that is put into the sphere later on. For this reason the sphere that is not being loaded from should be isolated. DO NOT FORGET TO PUT THE SPHERE BACK INTO THE VENT SYSTEM AFTER LOADING. If analysis shows impurities in the barge vapors, the vapor may be sent direct to the vent recovery system. The vent compressor may be used to pull the vapors from the barge, but will not pull the vapors fast enough to load the barge at the same time.

When it is desired to transfer material from a barge to the sphere, only one loading line can be used and that is the line from

SL 008906

-66-

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

the western loading arm (line 645). Material transferred back through this line does not flow through the turbine meter. The north meter run will be blocked, forcing the returned EC to flow through the 6" takeoff at the meter run downstream block valve. From the takeoff the returned EC will flow through two block valves and into the 12" pump suction line and then to whichever sphere is open. In between the two block valves on this return line is a bleed. Whenever the line is not being used, the block valves will be closed and the bleed open. This is to assure that all EC flowing through the turbine meter enters the barge.

The vent system on the spheres is the same as the system on the day tanks. The system will bleed in nitrogen when the pressure is low and purge nitrogen and EC when the pressure is high. To prevent a loss of a large amount of EC a compressor and a refrigerated condenser were installed. Whenever the pressure in the spheres goes below 15 psig, nitrogen will bleed into the system. Whenever the pressure goes above 30 psig (this pressure is dependent on acceptability to the customer) a valve will open in the compressor suction line and the compressor will start up. The compressor discharges through a water cooler to the vent condenser where 200 psig is maintained by a PCV in the inert gas discharge of the condenser. Brine from the refrigeration unit is circulated through the tubes of the condenser. The inert gas discharge should run about 15° F. The condensate is gravity fed to the Vent Condensate Receiver. Since the receiver vapors can return to the condenser, the material in the receiver will be near 0° F. and any samples of this material will expand when warmed to ambient temperature, so be extremely

careful in collecting a sample. When the Vent Condensate Receiver is nearly full, a sample of the material will be analyzed; and if it is in spec, the material will be pumped to a sphere. If the condensate is out of spec, it will have to be disposed of, probably back to the Bottoms Dopp Kettle. Whenever the pressure on the vent condenser goes below 5 psig, nitrogen will be bled into the condenser. The inerts from the PCV go to a vent stack to be vented to the atmosphere. The vent compressor has a distance piece which is swept with nitrogen, and this nitrogen along with any EC is vented from the vent stack. The vent compressor may be bypassed, with a much higher loss of EC resulting. When one is full and the second sphere is nearly full, the transfer rate from a day tank might have to be slowed down to about 200 GPM. The compressor will handle gases displaced by a liquid at 200 GPM. If it is imperative that the normal transfer rate be held, then someone will have to bypass the compressor and lower the setting on the vent condenser inert PCV from 200 psig to 45 psig. This will result in EC being vented, and appropriate steps should be taken to see that the EC doesn't carry into an area where a spark might start a fire.

The refrigeration unit is a 10-ton unit built by York. This is the same unit that was in the old HCl plant. The unit has a 50% unloader, and a 100% shutdown. The unit when operating on automatic will 50% shut down when the brine temperature is 0° F. and will shut down when the brine temperature is -8° F. When the unit is on manual, the 100% unloader is bypassed, but another low temperature cutout will operate when the brine temperature is -15° F. When the unit shuts down due to low temperature, it will start back up by itself.

To start the York unit, put water on the Freon condenser, start brine to recirculate (be sure the brine has no load on it until the temperature is down around 5° F). Operate compressor at 50% capacity or throttle suction valve to prevent motor overload during pulldown.

Place HOA switch in auto position. This will allow compressor to operate and maintain correct brine outlet temperature. Compressor will start and stop automatically.

The compressor will operate at 100% capacity cooling brine from 5° F. to 0° F. and on drop in brine temperature near setting desired, the compressor will operate at 50% capacity. This reduction in capacity will prevent excessive short cycling. Should temperature continue to drop to setting of operating thermostat in automatic circuit, then compressor will automatically stop. As brine temperature warms to thermostat high setting, compressor will again start.

To shut system down, place HOA switch in off position. Stop brine pump.

Compressor operation is protected with High and Low pressure cutout in R-22 system. In case of high pressure cutout (caused from lack of condenser water, dirty condenser tubes - closed valves); the system can or should be started after correction is made. This is accomplished by a reset button.

High pressure cutout setting is 240 psig.
Low pressure cutout setting is 12 psig and cut in at 20 psig.

In case of oil failure, the compressor will stop and can only be restarted by reset button in cover. (Min. 5 minutes) The cause

of oil failure should be checked to see that system oil is sufficient for operation in compressor base and oil receiver. Oil pressure cutout setting at 20 psi pressure differential between oil pressure and suction pressure and cut in at 30 psi differential.

A third thermostat is also provided with a setting lower than the other two thermostats for protection of tubes in brine cooler. This setting of ⁰-15 F. is above the freezing point of the brine mixture used. This is an emergency thermostat only and will allow the machine to restart when temperature increases to a safe setting of the thermostat.

Solenoid valve on oil receiver will open when compressor starts and will close when compressor stops. A hand throttling valve located in the oil line is to be throttled so as to allow oil (mixed with Freon) to flow from the cooler to the oil receiver in a small quantity to allow 2,000 watt oil heater to go on and off periodically. Approximately 7 minutes on and off is satisfactory.

Oil heater in compressor base is on when compressor is shut down and off when compressor is in operation. The oil heater in oil receiver is on and off continuously to boil Freon off and leave only oil for compressor operation. This circuit should be closed at all times. Pilot light is lit when heater is on.

Refrigerant level in the cooler is controlled with H.P. float drainer. Float will open when liquid Freon is condensed in condenser and allow refrigerant to flow to the cooler. System should be charged for operating charge only, so that refrigerant level in the cooler under full load conditions is high enough to wet top row of tubes. Observe level through cooler sight glasses. Excessive

charge of Freon will cause slop over on machine and cause serious damage.

System should be completely evacuated of all moisture before charging with R-22. See printed Service Instructions for evacuating procedure. Services of York Service Engineer should be called upon in getting system ready for charge of R-12 and start up.

With all of the equipment at the dock, there is only one alarm. This alarm is sphere high pressure, and it is located on the EC panel board. Whenever anything goes wrong with the brine or refrigerated unit, the ultimate result will be a high pressure, but this might not show up for a long time. For this reason the round made at the dock area should include a close check of all equipment.

The valving in the dock area is extremely important and should be left in only one way when no one is in the dock area. The valving is as follows:

1. The 12" suction lines to the pumps are closed except when in use.
2. The vent system is open from both tanks to the vent stack, except for the PCV's. The route is through the compressor and the vent condenser.
3. The barge loading lines are closed at the loading arm and allowed to equalize back to the spheres through the thermo-expansion equalizing line.
4. The transfer line is always open to one sphere except when loading a barge.

Last but not least, whenever pulling the sphere pressure down with the compressor, never pull down to a vacuum.

SL 008911

-71-

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

20. COOLING TOWER

A. Equipment 20A-71-790 The cooling tower is a Fluor Model 1F60E-144-2424 C4P counterflow with a 6' parabolic fan stack, redwood distribution pipes, polypropylene nozzles, polypropylene ("Poly-Grid) fill, vibration cut-out switch, sheathed with 8 oz. corrugated fiberglass reinforced polyester, adjustable pitch SS fan with 4, 14' blades to run at 229 RPM, 112 hp Thomas Flexible Coupling and a FW45A speed reducer. The tower is one cell 24' x 24' and 35' high constructed of heart redwood, and treated to 0.75#/ft. retention with Erdalith. The tower will cool 3,000 GPM of water from 115° F. to 90° F. The fan motor will operate at 1750 or 825 RPM requiring 40/10 hp.

B. Operation Excessive vibration will shut the cooling tower down. Anytime it shuts itself down, open the vibration switch box (by the fan motor) and see if it is tripped. Other than this, there is very little else that can go wrong with the tower. Rocks and dirt can plug the distribution section.

The water in the basin should not be allowed to be cooler than 75° F. During winter months the fan can be slowed down and if the basin water is still too cold, the louvers on the air intake may be arranged to allow only a small amount of air to enter the tower. During hard freezes the north side of the tower should be blocked off to prevent icing. The cooling tower water treatment and blow-down will depend on the chemicals used and the hardness of #8 well. The control levels will be given by the operating foreman.

The make-up water for the tower comes from a water-cooled condenser in the EC plant. Normally all water flowing through the condenser will be sent to the tower. When this water flowrate causes the basin to overflow, then some of the cooling water should be diverted to the sewer. This is done at the chemical addition basin on the east side of the cooling tower. When the cooling tower make-up requires more water than the condenser requires, then a float control valve will add the extra water automatically. The

CONFIDENTIAL:

SL 008912

-72-

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

21. REFRIGERATION UNIT

A. Equipment

1. Compressors - 64A-56-143 & 146 The EC refrigeration compressors are Carrier 5H80, reciprocating complete with automatic cylinder unloaders. The compressors will operate at 1160 RPM with a discharge pressure of 228 psig and a suction pressure of 24 psig. The refrigerant used will be F-22.

2. Freon Condenser - 64A-71-775 Carrier 5H100 shell and tube with copper tubes, steel tube sheets, 14" OD shell, 97 3/4" long with 6 water passes. The condenser is designed to remove 650,000 Btu/hr. by heating 110 GPM of CTW from 90° F. to 103° F.

3. Motors are 40 hp. 1200 RPM

4. Liquid level control valve.

B. Operation Like all compressors there are two cardinal rules to observe. 1. Never start the compressor with the discharge valve closed. 2. Never run the compressor with liquid in the suction line.

The EC refrigeration requirements were such that one unit would not do the job. By running two units in parallel, and using Freon 22, a full 35 tons of refrigeration could be achieved. The suction of the compressors is 24 psig and 0° F., while the discharge is 229 psig.

Whenever both units are in operation the oil equalizer as well as the gas equalizer must be open. If these plug or are closed, all of the oil will accumulate in one compressor, causing damage to the other. Each compressor is equipped with an unloading device, activated by the suction pressure, which allows the compressor cylinders to be cut out one at a time to reduce the capacity of the unit stepwise. This system is used to avoid excessive "on-off cycling" of the compressor when operating at low capacities. The capacity control valve is located on the compressor end cover at the opposite

end from the compressor drive. When this valve is turned counter-clockwise as far as it will go, all eight cylinders are in operation and the compressor is fully loaded. By turning the valve clockwise, the cylinders are unloaded one at a time until only two cylinders are left loaded. This can lower the capacity of each compressor by one-fourth.

Safety control devices on the compressor include an oil failure switch which shuts off the compressor motor if the differential between the oil pressure and the compressor section pressure drops below 35 psi. This switch has a line delay device which takes approximately 1 minute to cut the motor power after the oil pressure drops. This is a thermal switch which must be given 3 minutes to cool before it will reset. The compressor also has a high discharge pressure cut-off which cuts off the compressor driver if the compressor head pressure goes above 260 psig. This cut-off resets at 200 psig. A low suction pressure cut-off is also used to stop the compressor if the suction pressure drops below 15. This resets at 30 psig.

Other auxiliaries on the compressors are an oil filter, a crankcase heater and a suction strainer. The crankcase heater comes on automatically when the compressor drive is turned off. This keeps the compressor oil warm so that the Freon will not dissolve in it while the unit is down and cool. The heater is turned off automatically when the compressor drive is turned on. The suction strainer is located in the suction line. During the initial start-up of the machine, the strainer will be equipped with a felt sock to filter out scale and particles present in new piping. This sock should be changed after the first 50 hours of operation. If it is clean, leave it out; if not replace it for another 50 hours.

The compressor heads are cooled by circulating a stream of well water through them. The well water should be throttled to give the discharge water a temperature of about 100° F.

The compressed Freon leaves the compressor as a gas and enters the Freon condenser where it is condensed. The liquid leaves the bottom of the condenser, flows through a level control valve, through a filter dryer and into the bottom of the Secondary Vent Condenser. The LCV is controlled by a small float chamber located under the condenser. The Secondary Vent Condenser is the Freon surge tank and enough Freon should be kept in the system to keep all of the tubes in the Secondary Vent Condenser covered. From the bottom of the Secondary Vent Condenser a 3/8" line returns to the compressor suction line. This is the oil return line. The oil will settle at the bottom of the Secondary Vent Condenser and must be returned to the compressors. The oil return line must never be left open when the compressors are down. For this reason, there is a solenoid in the line that will shut whenever neither compressor is running. Do not depend on this solenoid. Always close the block valve in this line just before stopping the compressor. When the compressors or compressor is running, the block valve should be open about 1/4 of a turn. Enough steam should be on the line to keep the oil about ambient temperature out of the heated area. If the oil level in the compressors continues to fall, then open up the throttling valve in the oil line a little more. It is well to remember that Freon can flow down this line as well as oil, if the valve is opened too much; and if this Freon is not vaporized, it will fall into the cylinders and cause the heads of the compressor to be blown off.

INSTRUMENTATION

General One of the major requirements in a plant like the EC plant is smooth, uninterrupted control of the plant streams in order to obtain specification grade product. A distillation column cannot be operated with varying flows. Columns like the Heavies Still can take hours to level out after an upset, and rapid changes in operating conditions can cause such upsets with resulting poor quality product. To obtain the desired type of control, the EC plant has been equipped with automatic instruments wherever practical.

A thorough job of instrumentation has been done on this plant, but certain points must be understood to get full and efficient use of the instruments. The instruments are the operator's tools, and how he uses them determines how efficiently the plant will operate and how good will be the quality of the product produced.

All important control points in the EC plant are transmitted to the operating room where they are continuously recorded or indicated. The first indication of trouble within the plant will generally be from these instruments. These should be continuously checked for indications of abnormalities. Most of the records and/or indicators will be read and logged on the data sheets.

The instruments serve as your safety guard. All points in the plant which can cause serious trouble or operating difficulties have been equipped with alarm devices to give warning when abnormalities occur. If an alarm sounds for any reason, it should be checked immediately. DO NOT IGNORE THE WARNING SYSTEM. It is there for your protection as well as for protection of the equipment.

Alarm System When an abnormality occurs, it will be alarmed by a horn blowing and by a flashing light on the panel board. Under normal operation, the warning lights will show green as long as a piece of equipment is in operation. If a high flow, high pressure, high level, etc., occurs, the horn will blow and the warning light will flash red. If a low flow, low pressure, or low level occurs, the horn will blow and the warning light will flash amber. Pumps are also connected to warning lights. As long as a pump is in operation, the light on the panel board will show green. If for any reason a pump is shut down or stops, the light will flash red. In almost all motors in the area, the alarm horn will blow in addition to the flashing light. The motors that are not connected to the horn are on intermittent service and not continuously operating units.

The horn will continue to blow and the light flash until an acknowledgement is made. This is done by pressing the acknowledgement button located at the bottom center of the panel board. When this button is pressed, the horn will stop and the light will stop flashing. But, until the trouble is corrected, the light will show either red or amber, depending upon the nature of the trouble. When the correction is made, it will change back to green.

Checking Alarms A failure in the alarm system could result in serious trouble. For this reason, the alarm system should be checked periodically to be sure it is functioning properly. To check it, pull out on the acknowledgement button and turn it to the high alarm. Check all warning lights to see if they show red. If they do not, there is either a burned-out bulb or a defect in the circuit - either of which should be corrected immediately. Repeat this procedure for both the

normal and low alarm position. Make this check at least once per shift.

Safe operation depends upon this sytem's functioning properly.

Recorder-Controller Operation The recorder-controller instruments are Taylor Transcope Recording Receivers and are provided with manual and automatic operation. Under normal conditions, these will be operated on automatic control, but during start-up or shutdown, or possibly in the case of trouble, the manual control can be used. When on manual control, the process valve can be operated from the panel board by regulating the output pressure to the control valve with the set point adjusting knob on the lower right hand corner of the instrument facing.

The automatic valves go from full open to full close (or vice versa in the case of an air pressure closing valve) at an output air pressure variation of 15 to 3 psig. This pressure is indicated on the scale at the top of the controller (on the instrument facing at the panel board.) The approximate opening of the valve is indicated by this scale, i.e., 9 psig output is half open, 6 psig and 12 psig are 1/4 and 3/4 open or vice versa, depending on valve action. The output pressure will remain at the value set by the set point adjusting knob while the instrument is on manual control, and the valve opening will not vary.

When the instrument is on automatic control, the set point is adjusted to the desired control point by the set point adjusting knob. The instrument will then attempt to regulate the process to this control point by opening or closing the automatic valve. At proper control, the set point and the indicator pen will remain opposite each other. The opening or the closing of the automatic valve to maintain this condition will be shown by the variation in output air pressure.

The recorder-controllers can be readily switched from manual to automatic, or from automatic to manual control, but care must be exercised when doing this. If the indicated control point and the instrument set point do not correspond when the switch is made, a snap action of the automatic valve will occur. This will serve to upset the system as well as being hard on the instruments. The following procedures should be followed when making the transfer to avoid "bumping" the system during the change-over.

Manual to Automatic

1. Set the transfer lever at the "Seal" position. This isolates the control valve from the controller air circuits and seals the pressure that is on the control valve at that time.
2. Adjust the set point adjusting knob until the set pointer indicates the same output pressure as the output gauge.
3. Set the transfer lever to the "Automatic" position. The process is now on automatic control.

Automatic to Manual

1. Note the output pressure and then set the transfer lever at the "Seal" position.
2. Adjust the set pointer with the set point adjusting knob until it corresponds to the same pressure as noted in step one.
3. Set the transfer lever to the manual position. The process is now on manual control.

Set Point Changes The number one rule for operation of this type of plant is smooth steady control. The purpose of automatic instruments is to provide this control and they should be handled with this in mind.

The instrument set point can be set at the desired control point, and the instrument will regulate the control valve until this point corresponds to what is actually occurring in the process. By raising or lowering the set point, the control point is changed accordingly.

It must be remembered though, that when a set point change is made, the instrument will attempt to correct for this change immediately. If a large change is made, this could result in complete opening or closing of the automatic valve which could be severe enough to upset the system. Use small stepwise adjustments and give the system time to "catch-up" before making the next step. This will result in a smooth change and little cycling of the process.

SL 008920

-80-

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No. 91-1145

PUMPS

Pump	Manufacturer	Size	Impeller	Capacity	Head	Motor
Vent Condensate	Ingersoll-Rand	1 CRUN	5 1/2	10 GPM	109 @3450	1 HP
Reactor Recirc.	Goulds 3189	8x10-11	10"	1928 GPM	75'	40 HP
Primary Stripper Feed	Goulds 3196	1x2-6	5 3/8"	46 GPM	115'	5 HP
Hvs. Still Feed	Goulds 3196	1x2-6	5 3/4"	43 GPM	136'	5 HP
Hvs. Still Re-flux	Goulds 3196	1 1/2 x 3-6	5 1/8"	90 GPM	105'	5 HP
Product Pump	Goulds 3196	1x2-6	4 1/2"	38 GPM	80'	3 HP
Gland Flush	Lapp	CPS-1	--	2 GPH	150 psig	1/3 HP
Transfer	Goulds 3196	2x3-6	5 3/4"	250 GPM	125'	15 HP
Rework	Goulds 3196	1x2-8	7"	40 GPM	200'	10 HP
Cooling Tower	Goulds 3406	6x8-14	14 1/8"	2000 GPM	150'	100 HP
Sanitary	Hydr-O-Matic	3x3 Self-Priming	7 5/32"	100 GPM	50'	5 HP
Barge Ldg.	Goulds 3196	3x4-11	10 11/16"	600 GPM	100'	20 HP
Dock Brine Recirc.	Durco	DCHD1R 1 1/2 x 2-9	8 1/2"	57 GPM	78'	5 HP

ANALYTICAL PROCEDURE FOR ETHYL CHLORIDE OPERATION
METHOD EC-LC-1

1. TEST: Ferrous and Ferric Iron as FeCl_3
2. SAMPLE: Ethyl Chloride Reactor Crude
3. SAFETY PRECAUTIONS: Chlorinated hydrocarbons are toxic and breathing of vapors and repeated contact of liquid with skin should be avoided. FLAMMABLE!
4. APPARATUS & REAGENTS:
 - a. Bausch & Lomb, Model Spectronic 20, 25 Ml curvettes
 - b. 1.0 Ml pipette
 - c. 100 Ml volumetric flasks
 - d. 50.0 Ml volumetric flasks (2)
 - e. 2.0 Ml pipette
 - f. 5.0 Ml pipetters (3)
 - g. Ethanol, 95%
 - h. Hydroxylamine hydrochloride, 10%
 - i. Ortho-Phenanthroline, 0.3%
 - j. Potassium acid phthalate, 4%
5. PROCEDURE - GENERAL:
 - a. Chill a 1.0 ml pipette in dry ice chest.
 - b. Pipette 1.0 ml of chilled liquid ethyl chloride into approximately 50.0 mls. of 95% ethyl alcohol in a 100 ml volumetric flask.
 - c. Make up to volume with 95% ethyl alcohol.

PROCEDURE - FERROUS IRON

- a. Pipette 2.0 ml of above dilution into a 50 ml volumetric flask.
- b. Add 5.0 mls of 4% potassium acid phthalate.
- c. Dilute to volume with 95% ethyl alcohol.

PROCEDURE - TOTAL IRON:

- a. Pipette 2.0 ml of dilution into a 50 ml volumetric flask.
- b. Add 5.0 ml of 4% potassium acid phthalate, 5.0 ml of 10% hydrochloride and 5.0 ml ortho-phenanthroline.
- c. Dilute to volume with 95% ethyl alcohol.
- d. Measure absorbance of resulting solutions within 30 minutes at 512 mu using a distilled water blank.

CALCULATIONS:

Each absorbance unit is equivalent to 353×10^{-6} GMS of total iron as FeCl_3 .

FERROUS IRON AS FeCl_3 :

$$\% \text{FeCl}_3 = \frac{\text{Absorbance} \times 353 \times 10^{-6} \times 100}{\text{Wt. of Sample}}$$

$$\% \text{FeCl}_3 = \frac{\text{Absorbance} \times 0.0353}{.00\text{ml} \times 0.917} = \text{Absorbance} \times 19.3 = \% \text{FeCl}_3$$

TOTAL IRON AS FeCl_3 :

$$\% \text{FeCl}_3 = \frac{\text{Absorbance} \times 353 \times 10^{-6} \times 100}{\text{Wt. of Sample}}$$

$$\% \text{FeCl}_3 = \frac{\text{Absorbance} \times 0.0353}{0.002 \text{ ml} \times 0.917} = \text{Absorbance} \times 19.3 = \% \text{FeCl}_3$$

FERRIS IRON AS FeCl_3 :

$$\% \text{FeCl}_3 = \% \text{total} - \% \text{Ferrous}$$

ANALYTICAL PROCEDURE FOR ETHYL CHLORIDE OPERATION
METHOD EC-LC-2

1. TEST: HCl Determination in Liquid Ethyl Chloride
2. SAMPLE:
 - a. Primary Stripper Product
 - b. Heavies Still Overhead
 - c. Secondary Stripper Bottoms
3. SAFETY PRECAUTIONS: Chlorinated hydrocarbons are toxic and breathing of vapors and repeated contact of liquid with skin should be avoided. FLAMMABLE!
4. APPARATUS & REAGENTS:
 - a. Mag-Mix
 - b. Teflon covered stirring bar
 - c. 2.0 ml pipette
 - d. 250 ml beaker
 - e. Sodium Hydroxide, 0.10N
 - f. Phenolphthalein indicator
5. PROCEDURE:
 1. Chill a 2.0 ml pipette and approximately 150 mls. distilled water in a 250 ml beaker in dry ice chest.
 2. Pipette 2.0 ml of chilled liquid ethyl chloride into the chilled H₂O while stirring with Mag-Mix.
 3. Stir approximately 2.0 minutes.
 4. Titrate to a Phenolphthalein end point with 0.10 N NaOH.
 5. Record titer.

CALCULATION:

$$\% \text{ HCl} = \frac{T \times N \times 0.03647 \times 100}{\text{Wt. of Sample}}$$

$$\% \text{ HCl} = \frac{T \times 0.3647}{2.0 \times 0.903} = T \times 0.20$$

$$\text{ppm HCl} = \frac{T \times N \times 0.03647 \times 10^6}{\text{Wt. of sample}} = \frac{T \times 36470}{2.0 \times 0.903}$$

$$\text{ppm HCl} = T \times 2000$$

READ % HCl OR ppm HCl FROM CHART

SL 008924

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No. 91-1145

ANALYTICAL PROCEDURE FOR ETHYL CHLORIDE OPERATION

METHOD EC-LC-3

1. TEST: HCl Determination in Vent Gas
2. SAMPLE: a. Reactor Vent Gas
b. Plant Vent Gas
3. SAFETY PRECAUTIONS: There will be some EC gases in the vent and should be treated as a toxic, flammable material.
4. APPARATUS & REAGENTS:
 - a. Gas burette, 100 ml
 - b. Leveling bottle, 250 ml
 - c. Orsat measuring burette
 - d. Confining solution
5. PROCEDURE:
 - a. Sampling
 - (1) Place approximately 200 mls of confining solution in leveling bottle. Change confining solution once/week.
 - (2) Fill connecting tube from leveling bottle to gas burette by elevating leveling bottle to allow a small amount of confining solution from leveling bottle to flow into gas burette.
 - (3) Connect gas burette to sample line and purge with sample approximately 5.0 minutes.
 - (4) Close sample valve and burette stopcocks so as to leave a slight pressure in gas burette. Disconnect from sample point.
 - (5) Allow gas burette to come to room temperature and bleed off pressure by quickly rotating top stopcock. Continue until pressure is equalized.
 - (6) Open bottom stopcock of burette and allow confining solution to flow into gas burette and absorb HCl. DO NOT SHAKE BURETTE.
 - (7) Close bottom stopcock of gas burette and invert burette to facilitate absorption.
 - (8) Right burette, open bottom stopcock and allow additional confining solution to flow into burette.

- (9) Connect gas burette to Orsat measuring burette and transfer unabsorbed gases to measuring burette.
- (10) Disconnect gas burette and allow confining solution to flow back into leveling bottle.

b. HCl Determination

- (1) Measure volume of residual gases and record as % HCl.
- (2) Save residual gases for analysis by gas chromatograph.

ANALYTICAL PROCEDURE FOR ETHYL CHLORIDE OPERATION

METHOD EC-LC-4

1. TEST: Karl Fischer Moisture Determination
2. SAMPLE: a. Primary Stripper Product
b. Secondary Stripper Bottoms
3. SAFETY PRECAUTIONS: Flammable and toxic liquids and vapors. Avoid contact with skin and breathing of vapors.
4. APPARATUS & REAGENTS:
 - a. Karl Fischer Titrator
 - b. Karl Fischer Reagent
 - c. Karl Fischer Grade Methanol
 - d. Pyridine, Reagent Grade
5. PROCEDURE:
 - a. Turn switch on. Drain titration vessel and refill to red mark with dry methanol. (If sample contains free HCl, add 10 mls of pyridine)
 - b. Add concentrated Karl Fischer reagent until, by visual observation, the end point is approached. Make final adjustment with standardized Karl Fischer reagent from burette until the end point is indicated by a meter reading of 10. DO NOT GO BELOW 10.
 - c. Just before sample is added to the titration vessel, check to see if meter is holding between 10-12. If not, solution must be readjusted to the end point before adding the sample.
 - d. Pipette 20 mls of chilled liquid ethyl chloride with a chilled pipette and drain into titration vessel.
 - e. Stopper titration vessel and start titration with full burette. Deliver Karl Fischer reagent at approximately 1 drop/second. Titrate to the end point as indicated by a meter reading of 10. End point is reached when meter holds between 10-12 for 20 seconds. If meter drifts above 12, continue titration until end point holds 20 seconds.
6. CALCULATIONS:

$$\text{ppm H}_2\text{O} = \frac{\text{Final KF titer} \times N \times 1,000,000}{\text{Grams Sample}}$$

$$\text{ppm H}_2\text{O} = \frac{\text{Final KF Titer} \times K}{\text{Grams Sample}}$$

Where N = Normality as obtained by titrating 20 mls of a methanol standard as described in the above procedure. Methanol stand will be labeled as "Grams H₂O/20 mls."

$$N = \frac{\text{Grams H}_2\text{O}/20 \text{ mls}}{\text{KF titer for this standard}}$$

$$\text{And } K = N \times 1,000,000$$

$$\text{ppm H}_2\text{O} = \text{KF titer} \times K \times 0.0005$$

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No. 91-1145

G. C. PROCEDURE FOR ETHYL CHLORIDEMETHOD NO. G. C. - 12 - EtCl-1

Sample: Ethyl Chloride Product
Instrument: Wilkens G. C. Model 1520
Column Packing: 20% Diisodecyl Phthalate on Gas Chrom P, 70/80 Mesh
Column Size: 5.0 Meter x 1/4" Stainless Steel Tubing
Column Temperature: Start: 70°C Finish: 175°C
Heating Rate: 6°/min., 3 min. after sample injection.
Recorder: Minneapolis-Honeywell Sensitivity: - 0.05 to 1.05 m.v.
Carrier Gas: Helium Outlet Flow: 80 ml./min. Inlet Pressure: 75 psig
Cell Temperature: 225°C Current: 250 m.a.
Injection Port: "A" (Phthalate) Injection Port Temperature: 150°C
Sample Size: 0.005 ml. Sample Phase: Liquid Sample Inject: Syringe¹
Peak Measurement: Peak Height x Peak Width at 1/2 Height
Calculation of Results: Internal Normalization

<u>Component</u>	<u>Retention Time, Min. from Air Peak</u>	<u>Calibration Factor, Wt. % Basis - EDC = 1.000</u>
Ethyl Chloride	1.8	0.90

1. Syringe must be chilled in order to retain liquid C_2H_5Cl in syringe.

G. C. PROCEDURE FOR ETHYL CHLORIDEMETHOD NO. G. C. - 12 - EtCl-2

Sample: Ethyl Chloride Reactor Vent Gas
Instrument: Wilkens G. C. Model 1520
Column Packing: Deactigel (Silica Gel)
Column Size: 5.0 Meter x 1/4" Copper Tubing
Column Temperature: Start: 70°C Finish: 175°C
Heating Rate: 10°/min. following sample inject.
Recorder: Minneapolis-Honeywell 15 Sensitivity: - 0.05 to 1.05 m.v.
Carrier Gas: Helium Outlet Flow: 80 ml./min. Inlet Pressure: 75 psig
Cell Temperature: 225°C Current: 250 m.a.
Injection Port: "B" (Deactigel) Injection Port Temperature: 150°C
Sample Size: 1.0 ml. Sample Phase: Gas Sample Inject: Gas Valve
Peak Measurement: Peak Height x Peak Width at 1/2 Height
Calculation of Results: Peak Area x Calibration Factor : Sample Size

Component	Retention Time	Calibration Factors	
	Min. from Injection	(C ₂ H ₄ = 1.000)	(Sample Size = 0.25 ml.)
Hydrogen (H ₂)	0.7	51.540 ¹	By Difference
Nitrogen (N ₂)	1.0	0.855	0.000833 ²
Methane (CH ₄)	1.4	1.092	0.001065
Ethane (C ₂ H ₆)	4.5	0.653	0.000637
Ethylene (C ₂ H ₄)	8.0	1.000	0.000975
Ethyl Chloride (C ₂ H ₅ Cl)	30.0	0.420	0.000410

1. When H₂ is not present or can be measured.
2. When H₂ is present but cannot be measured.

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 of 14th Judicial District Court
 No. 91-1145

ELECTRICAL SYSTEM

The electrical ties in the EC plant are fairly straight forward. Dwgs. 62A-7100, 64A-7100-01-02 show the ties and typical circuitry necessary for each motor.

The only thing unique about the EC electrical system is the fact that the 13.8 KV source may come from either EDC or Per-Tri. Because of this, many different arrangements may be used in feeding the organics area; see Dwg. 20A-901-1.

Normally the power for EC-HC1-VC will come from Per-Tri through breaker 0-408 and disconnect 0-503. The Per-Tri power comes from powerhouse bus #1. Air Circuit Breaker 0-309 in the EDC switch room will normally be closed, with disconnect 0-502 open. The cable is hot but protected. Never keep the cable hot by opening 0-309 and closing 0-502, as any fault in the cable will cause the EC-HC1-VC plants to come down by opening 0-408. Until the H-138 feeder cable from the powerhouse to EDC is increased in size, it will carry only EDC, Per-Tri, EC-HC1 but not VC. Before any tie that would connect EC-HC1-VC, when VC is operating, to the EDC cable the area supervision will have to determine the relative loads of the plants. In case of an emergency, leave the units down until the proper decision can be made by the area foreman.

Emergency ties may be made by the shift engineers until the VC plant is running. Anytime a tie is made, even though it is an emergency, be sure and take your time and do all things in proper sequence. Any omissions from safe practices are forbidden.

When the EC-HC1 bus loses power, the shift engineer should proceed immediately to the EC-HC1 switchroom and take a quick look at the 13.8 system,

SL 008931

-91-

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of 14th Judicial District Court
No. 91-1145

odor, and a visual check of the general area will suffice. If ozone or damage is present, then finish shutting the plants down. If the trouble appears to be elsewhere:

1. Call Powerhouse Switchboard Operator (417) and tell him you are switching load from PH Bus #1 to PH Bus #4.
2. Open disconnect 0-503 (feed from Per-Tri).
3. Close disconnect 0-502 (feed from EDC).

If power is back on the bus and stays on, then start back up the motors.

If power is not back, then nothing can be done. Per-Tri CANNOT be tied to the EDC bus without considerable delay as disconnect 0-404 is interlocked with the powerhouse OCB, i.e., the OCB in the powerhouse will have to be opened and the key removed and used to open disconnect 0-404. The same thing is true with the EDC disconnect 0-301.

Normal switching of the power source for EC-HC1-VC will be done by Maintenance with the cooperation of Operations. Although Operations will never make this switch, it is well that they know how and serve as a double-check. The procedure is as follows:

1. Close 0-309 (in EDC) momentarily (be sure 0-502 is open); if it doesn't drop out, then the cable from EDC to EC has no fault. Be sure 0-309 is open before proceeding.
2. Close 0-502 (in EC).
3. Call Powerhouse Switchboard Operator (417). Notify him that load is going to be switched from PH Bus #1 to PH Bus #4. The operator is to check and see that the synchronous tie breakers are closed. Then the powerhouse operator is to equalize the voltages on the two buses. The voltage meters that must be equal are the ones on the EDC and the EC buses in the respective switchgear rooms.
4. After voltages are equal, close 0-309 (in EDC).
5. Open 0-408 (in Per-Tri).
6. Open disconnect 0-503 in EC.

SL 008932

-92-

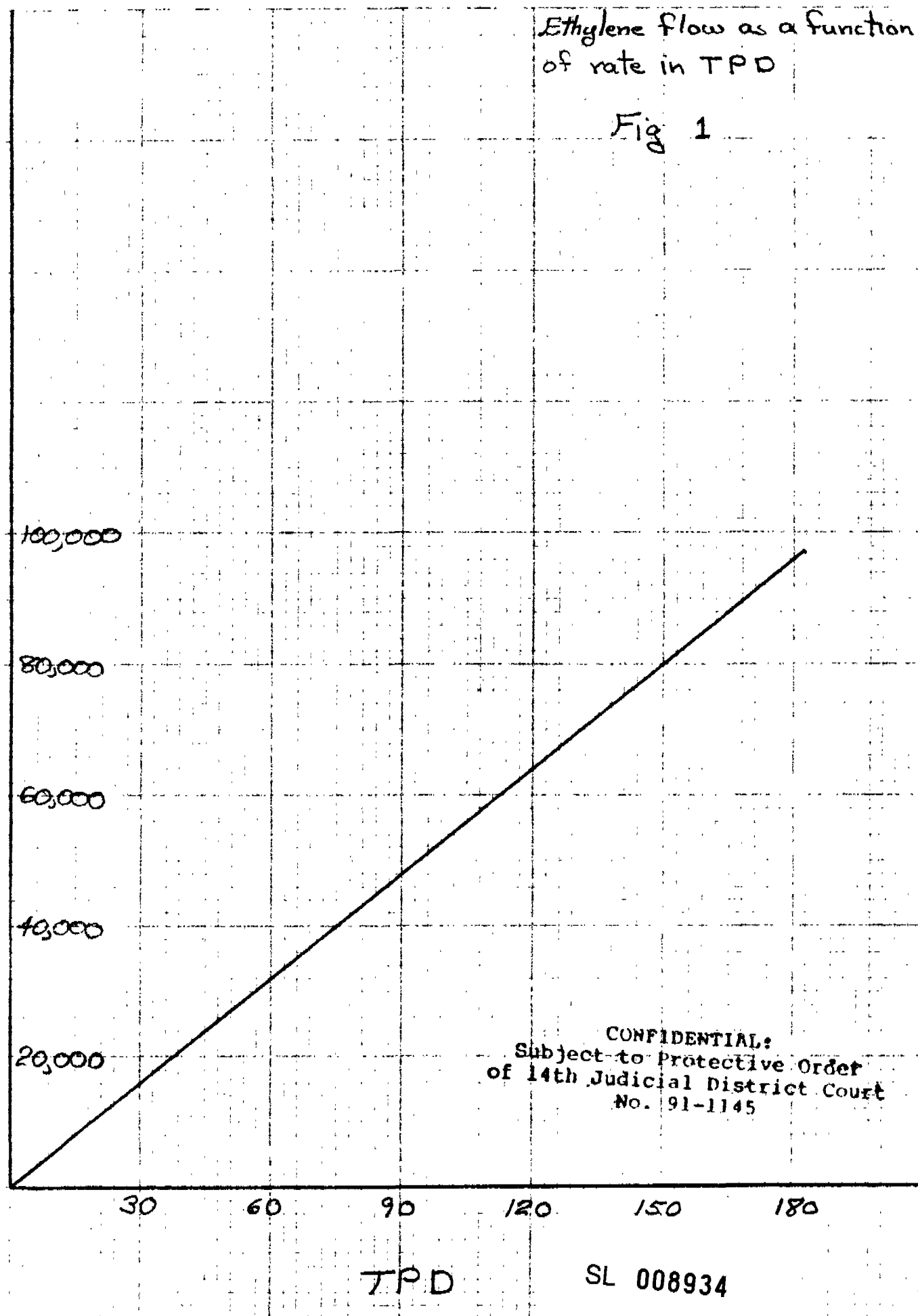
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No. 91-1145

There are many other combinations of operating and switching that may be done. Each procedure is similar to the one given above. It is best to remember that all of this switching is done with 13.8 KV present and therefore any ties made into a faulted bus will cause some damage. Anytime a tie is made into a bus that might have a fault, the consequences of the arcing damage must be weighed against the dangers of the plant having only emergency power.

Ethylene Flow as a function
of rate in TPD

Fig 1

Ethylene
SCFH



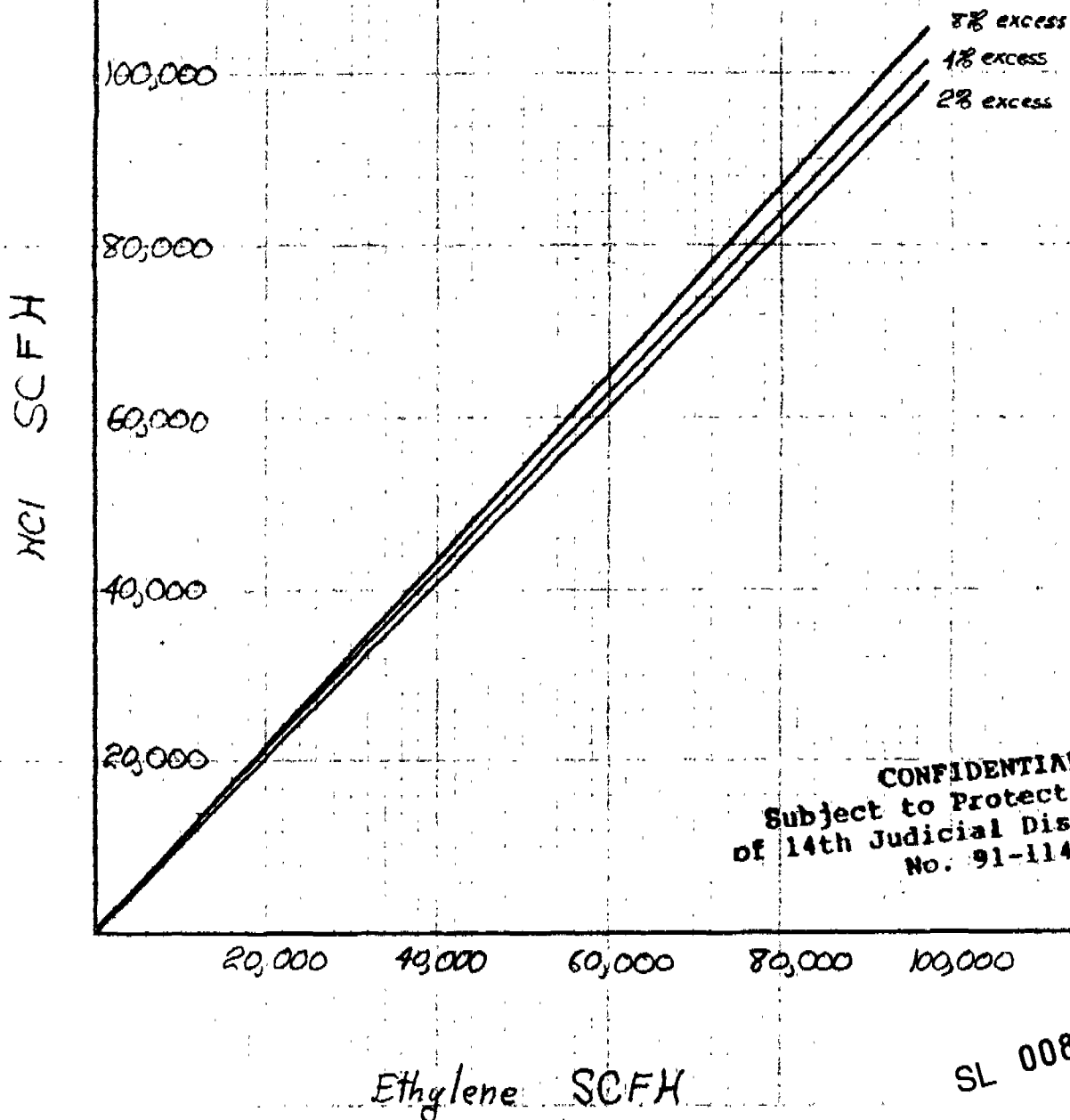
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No. 91-1145

TPD

SL 008934

HCl flow as a function
of ethylene flow

Fig. 2



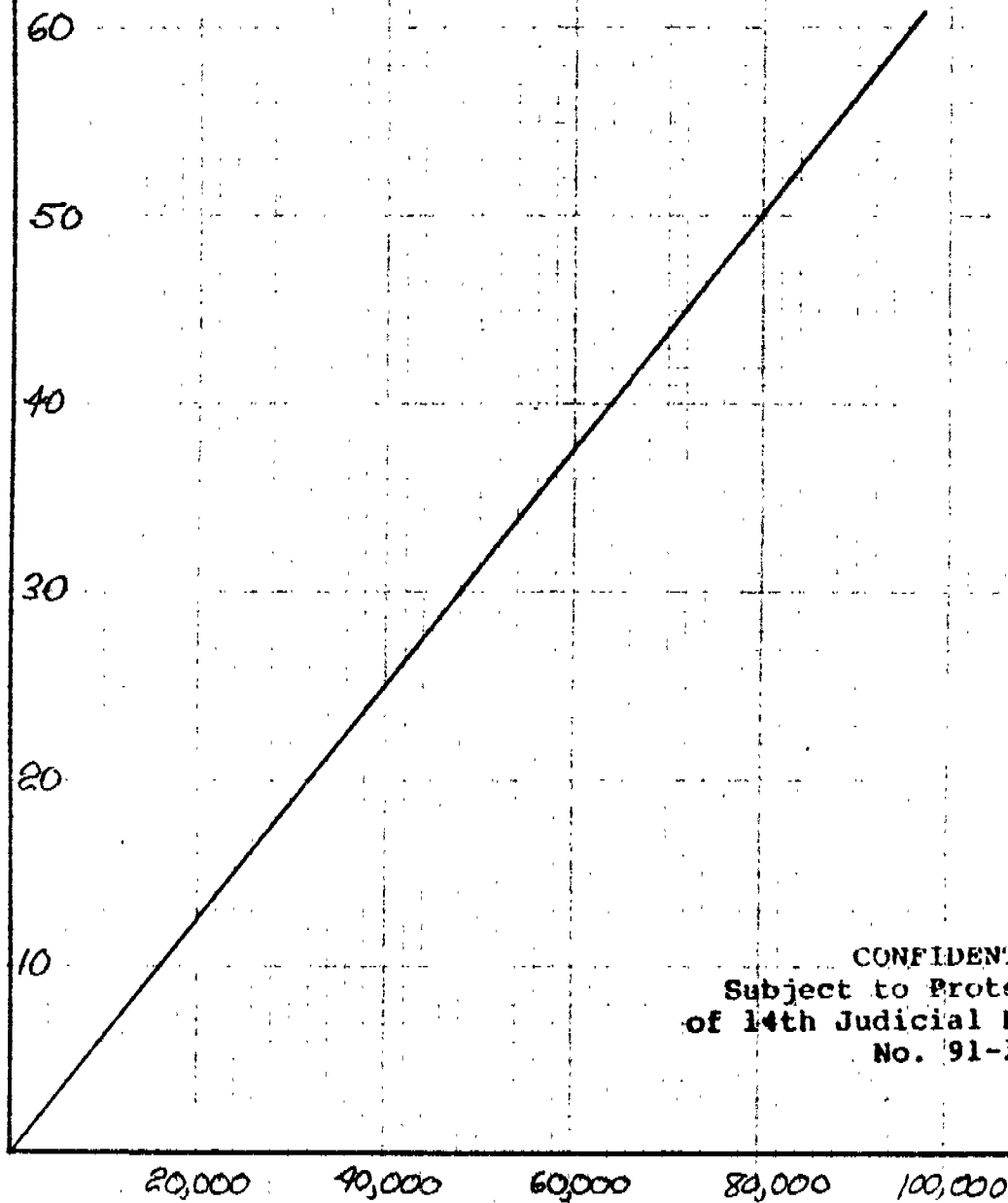
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FeCl_3 addition as a function
of ethylene flow

Fig 3

FeCl_3 pounds per hour



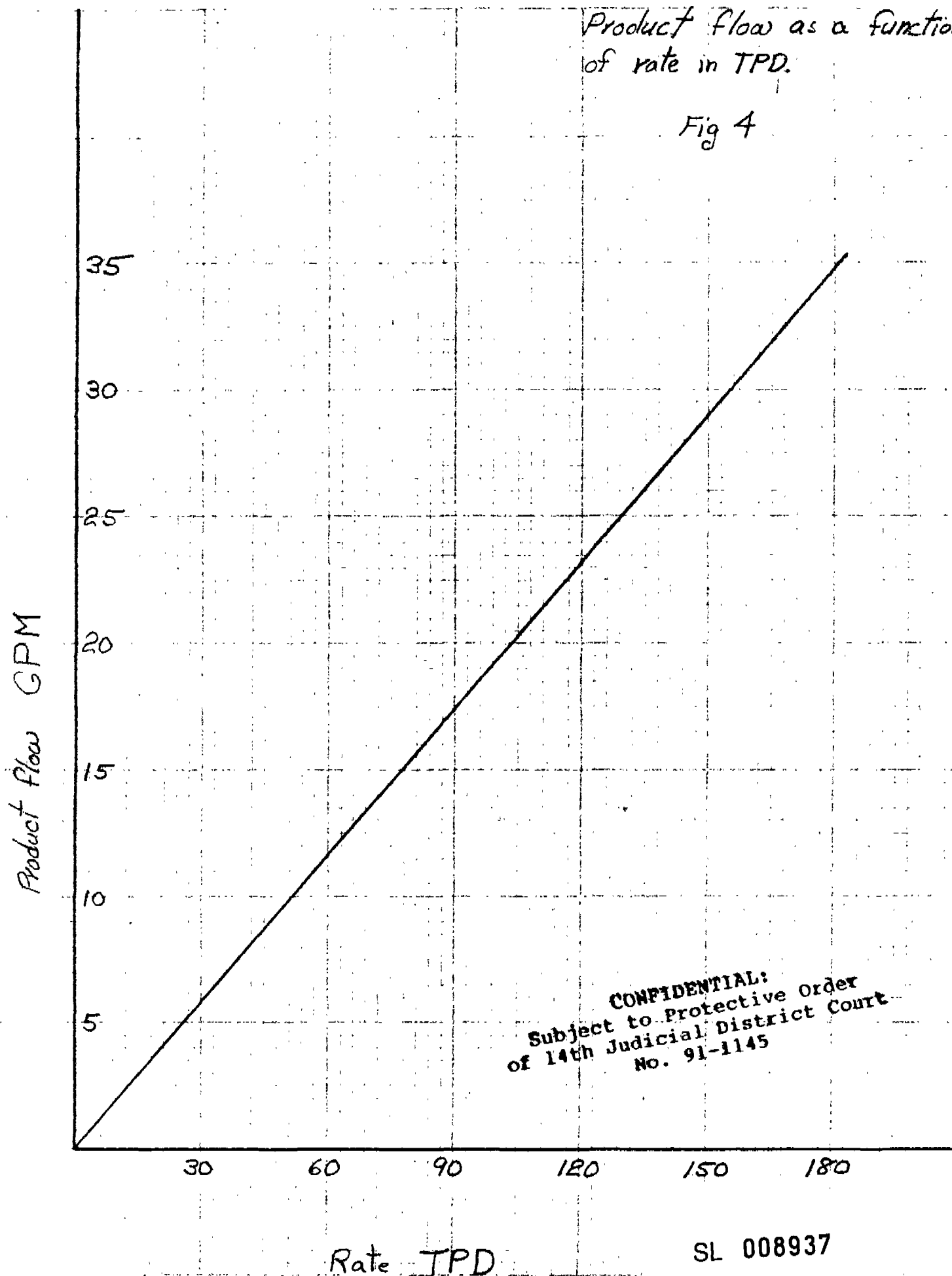
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No. 91-1145

ethylene SCFH

SL 008936

Product flow as a function
of rate in TPD.

Fig 4



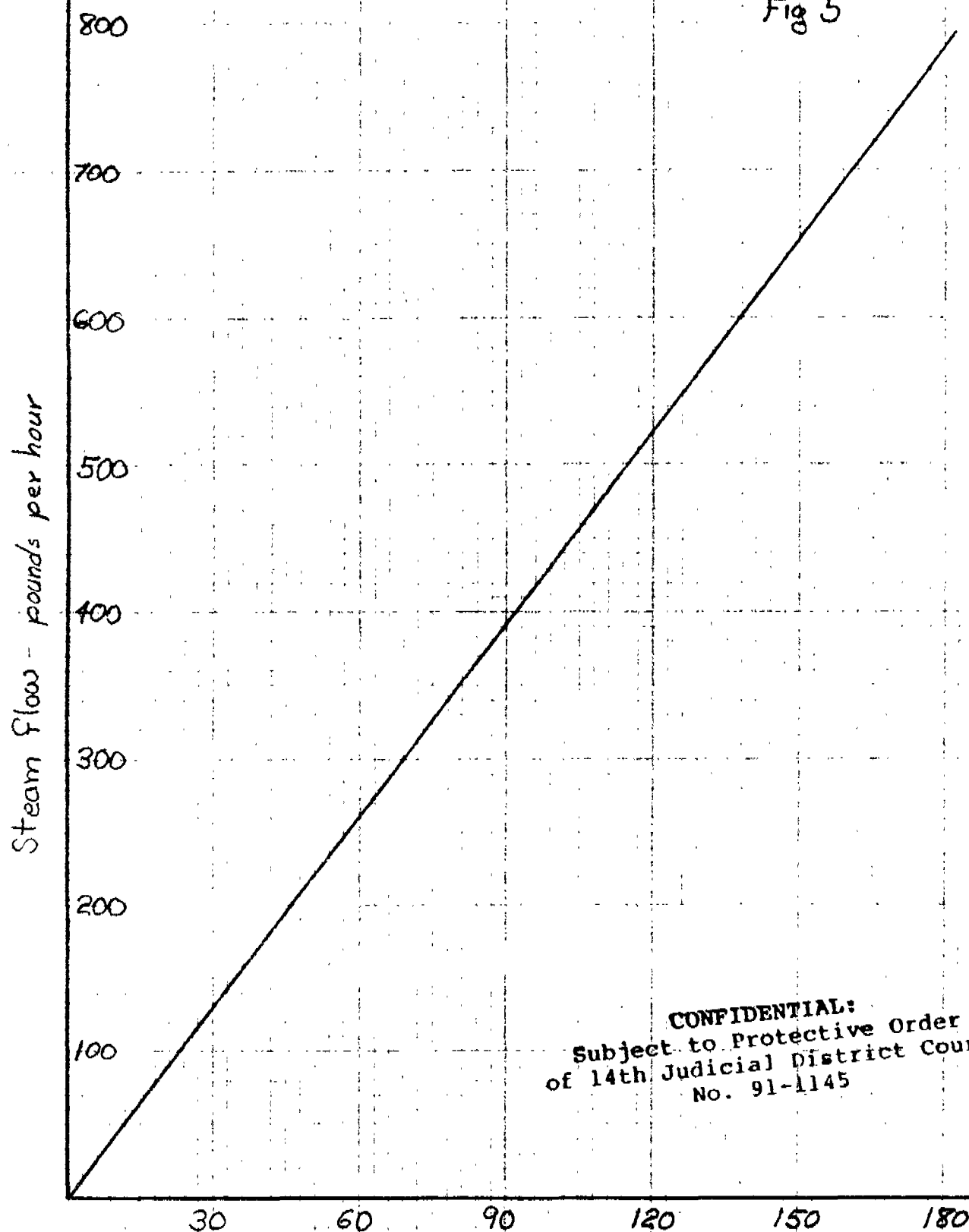
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No. 91-1145

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Rate TPD

Steam flow to the Primary
Stripper as a function of rate

Fig 5



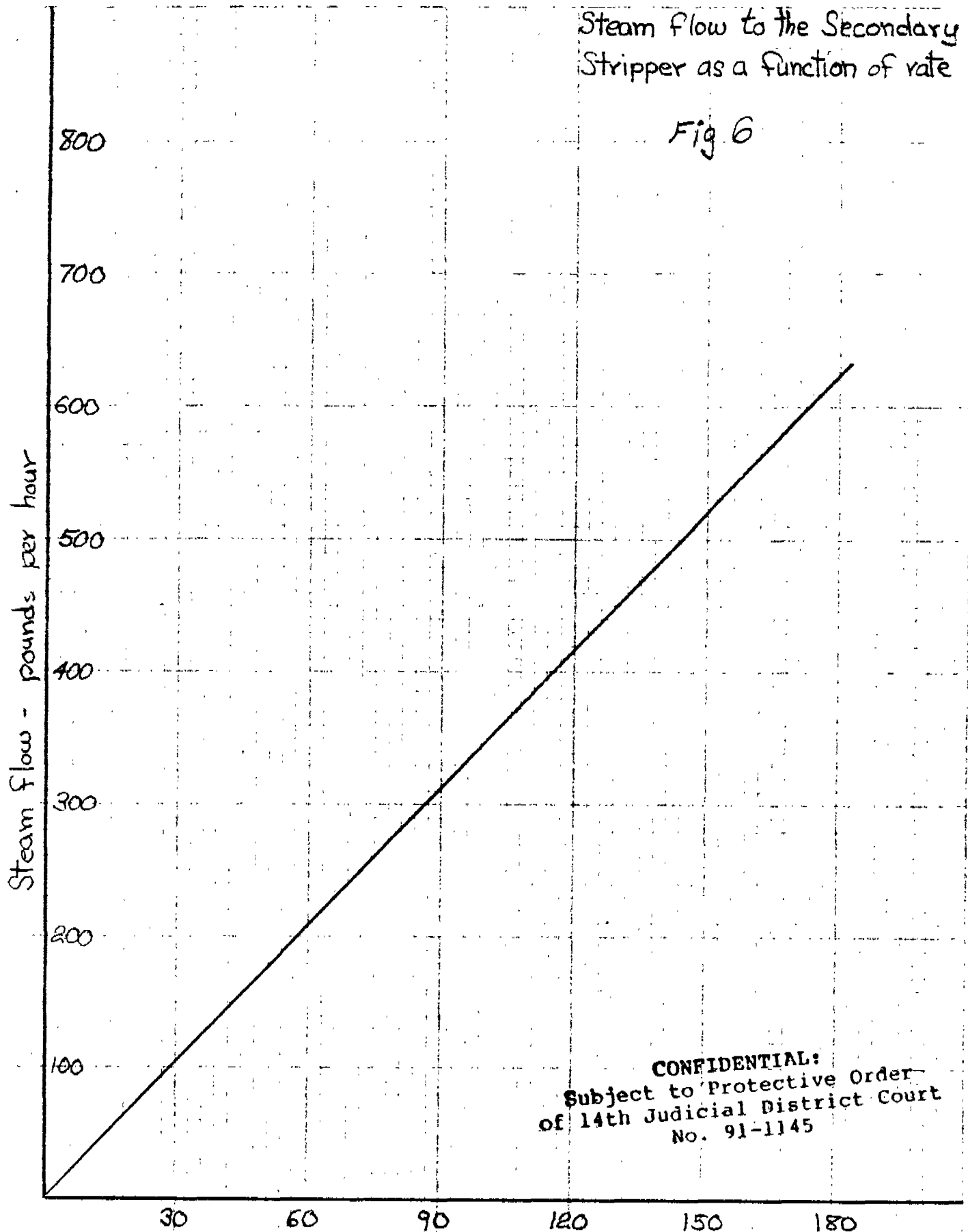
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No. 91-1145

Rate TPD

SL 008938

Steam flow to the Secondary
Stripper as a function of rate

Fig 6

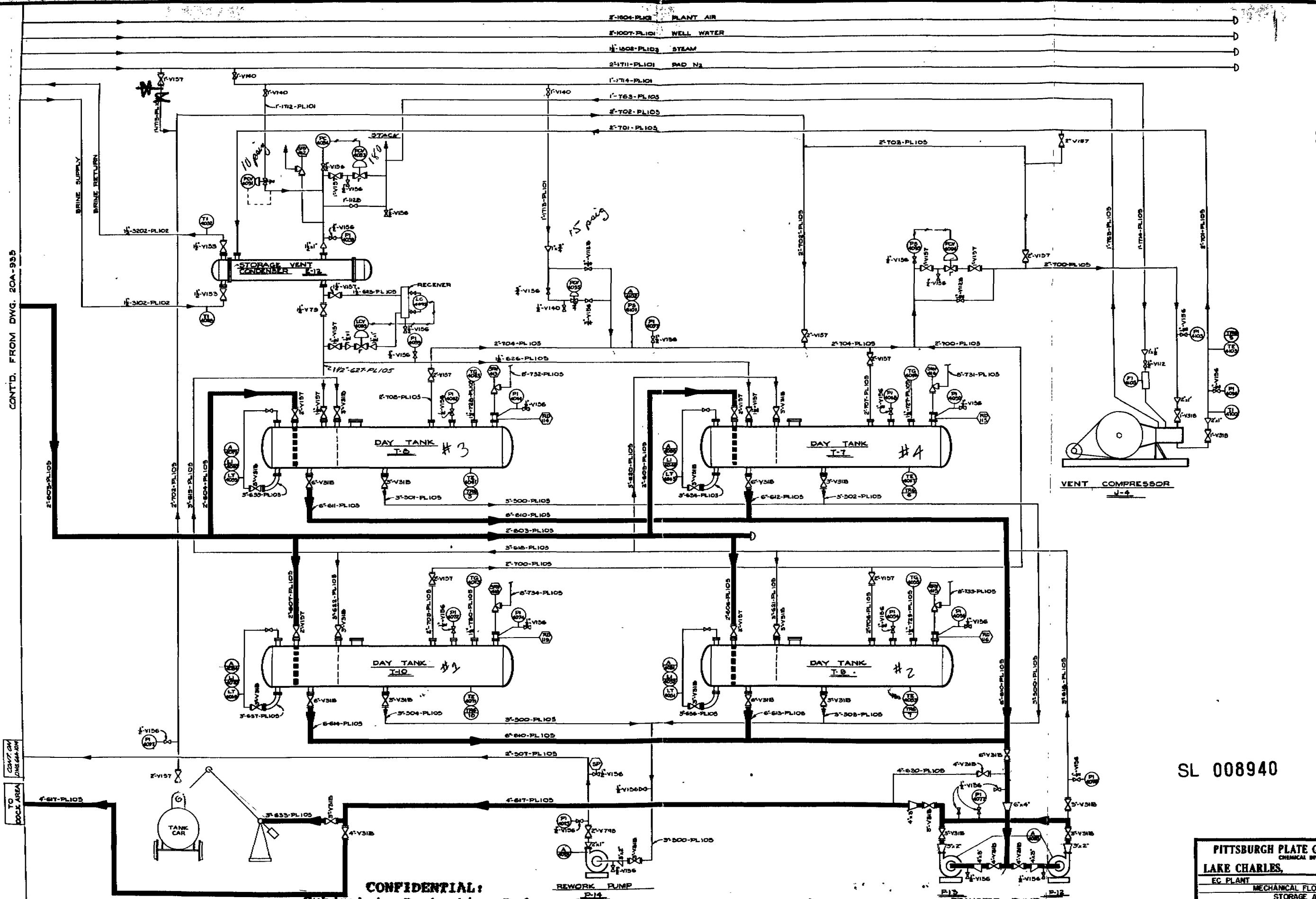


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No. 91-1145

Rate TPD

SL 008939

CONT'D. FROM DWG. 20A-935



REVISIONS

NO.	REV.	DATE
1	GEN.	1-4-66
2	REV.	M.L.

ALL PRINTS DATED BEFORE LAST REVISION ARE CANCELLED AND RECALLED

E-12
J-4
P-12
P-13
P-14
T-7
T-8
T-9
T-10

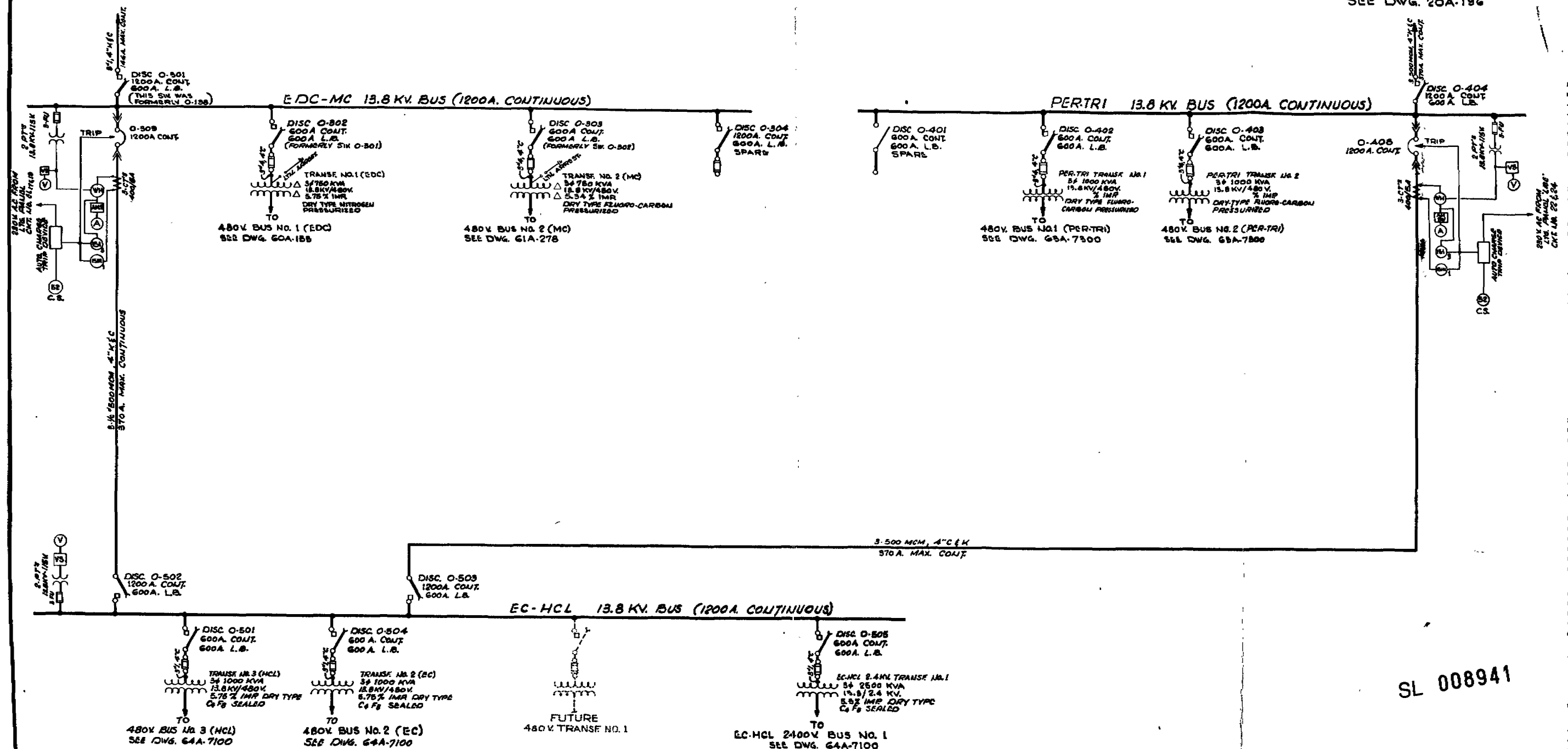
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PITTSBURGH PLATE GLASS COMPANY	
LAKE CHARLES, LOUISIANA	
EC PLANT	FLOW SHEET
MECHANICAL FLOW SHEETS	
STORAGE AREA	
SHEET 6 OF 6	
DRAWN BY L. D. DERRICK DATE 11-24-65 SCALE NONE	
CHECKED BY J. H. DATE 11-24-65 CHANGE	
APPROVED BY [Signature] DATE 3-6-66 SUB.	
BILL OF MATERIAL	DWG. NO. 64A-1015-1

TO 13.8 KV. BUS NO. 4 (H-106)
SEE DWG. 20A-196

TO 13.8 KV. BUS NO. 1 (H-104)
SEE DWG. 20A-196



SL 008941

REFERENCE DRAWINGS

- 20A-196 13.8 KV. ONE LINE DIAGRAM
- 60A-153 EDC PLANT ONE LINE DIAGRAM
- 61A-278 TRI-ETHANE PLANT ONE LINE DIAGRAM
- 62A-200 HCL PLANT ONE LINE DIAGRAM
- 63A-7300 PER-TRI PLANT ONE LINE DIAGRAM
- 64A-7100 EC-HCL PLANT ONE LINE DIAGRAM

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No. 91-1145

PITTSBURGH PLATE GLASS COMPANY	
CHEMICAL DIVISION	
LAKE CHARLES, LOUISIANA	
20A	ELECTRICAL
ORGANICS AREA COMPOSITE	
ONE LINE DIAGRAM	
DRAWN BY MC DANIEL	DATE 7-6-68 SCALE 1/8" = 1'
CHECKED BY R. J. J.	DATE 10-22-65 CHANGE P. 67
APPROVED BY	DATE 10-27-61
BILL OF MATERIAL	
DWG. NO. 20A-901-1	

REVISIONS	
THIS DRAWING	SUPERSEDES (V) 1006
ORIGINAL DWG.	20A-901 & ADDS
EC-HCL BUS	
674-952	10-26-65
11/4/64	S.D.M.
PER DWG.	20A-1087

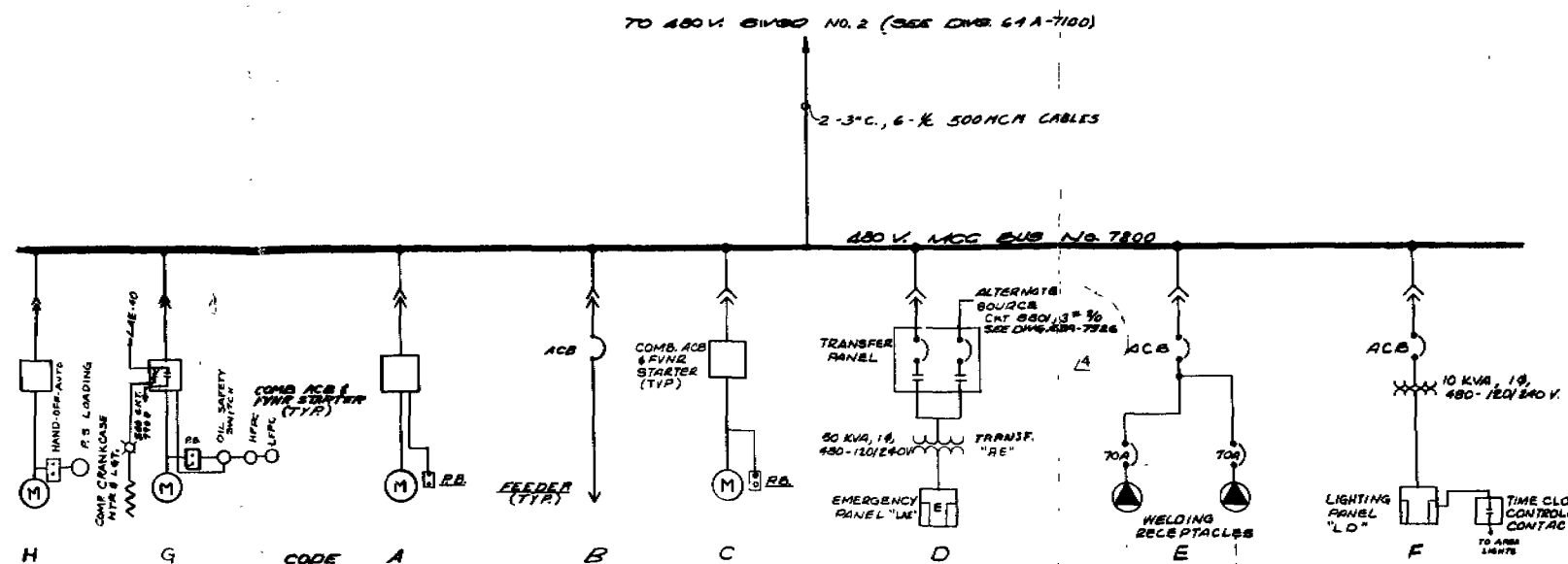
PITTSBURGH PLATE GLASS COMPANY
CHEMICAL DIVISION
LAKE CHARLES, LOUISIANA

EC - HCL PLANT ELECTRICAL
ONE LINE DIAGRAM
480 VOLT, 2 & 13.8 KV SYSTEM

DRAWN BY: *OSTERLUND* DATE: *6/11/68 LOCAL NONE*
CHECKED BY: *TM* DATE: *6/11/68 CHARGE*
APPROVED BY: *TH* DATE: *5/18/68 SUB.*

BILL OF MATERIAL DWG. NO. 54A-7100.1

BUS 7800				
INCOMING TERMINALS	7804	7809	7815	FUTURE
7801	7805	7810	100 AMP TRANSFER PANEL	FUTURE
	7806	7811		FUTURE
7802		7812		FUTURE
		7813		FUTURE
	7807 ACB		7816 FUTURE	FUTURE
7803	7808	7814	7817	FUTURE



SPECIFICATIONS:

- 1-MOTOR CONTROL CENTER SHALL BE FABRICATED PER GENERAL ELECTRIC'S SPECIFICATIONS FOR 7800 LINE MOTOR CONTROL CENTERS. VERTICAL SECTIONS ARE TO BE 20" HIGH, 20" WIDE & 13" DEEP FOR PLUG IN STARTERS ON ONE SIDE ONLY.
- 2-WIRING IS TO BE NEMA CLASS 1, TYPE B.
- 3-ENCLOSURE TO BE NEMA TYPE 1.
- 4-POWER SUPPLY, 480 VOLT, 3 PHASE, 60 CYCLE.
- 5-CONTROL POWER: 120 VOLT, 60 CYCLE, USE A SEPARATE CONTROL TRANSFORMER FOR EACH STARTER
- 6-PROVIDE A 600 AMP GROUND BUS WEIGHT 1/2" DIAMETER HOLES FOR EACH VERTICAL SECTION OF MOTOR CONTROL CENTER.
- 7-MAIN HORIZONTAL BUS TO BE RATED 600 AMPS & VERTICAL BUS TO BE RATED 300 AMPS. ALL BUS WORK TO BE SILVER PLATED & SUITABLY BRACED TO WITHSTAND A MAXIMUM OF 50,000 RMS AMPERES SHORT CIRCUIT CURRENT.
- 8-INCOMING LINE CABLES WILL BE TOP FED WITH NO 500 MCM CABLES PER PHASE.
- 9-OUTGOING CABLES FROM STARTERS ETC. WILL LEAVE THE CONTROL CENTERS FROM THE TOP.
- 10-PHYSICAL ARRANGEMENTS OF STARTERS AND BREAKERS ARE TO BE AS SHOWN ON THESE DRAWINGS. STARTERS SHALL BE OF THE CIRCUIT BREAKER COMBINATION TYPE.
- 11-OVERLOAD BIMETALLIC RELAYS (EXTERNAL HAND REST TYPE AMBIENT COMPENSATED) ARE TO BE PROVIDED IN ALL THREE PHASES. HEATER ELEMENTS ARE TO BE PURCHASED & INSTALLED BY OWNER AT LATER DATE.
- 12-ONE EXTRA NORMALLY OPEN AUXILIARY CONTACT IS TO BE PROVIDED IN EACH MOTOR STARTER CONTACTOR.
- 13-LAMACOID NAMEPLATES, LETTER HEIGHT OF CIRCUIT NUMBER TO BE 1/2". LETTER HEIGHT OF CIRCUIT DESCRIPTION TO BE 1/4". LETTER HEIGHT OF BUS NUMBER TO BE 1/4". LETTERS TO BE WHITE ON BLACK BACKGROUND.
- 14-VENDORS NAMEPLATE TO BE LOCATED TOP AND CENTER OF MOTOR CONTROL CENTERS VIA LAMACOID NAMEPLATE SHOWING THE BUS NUMBER DIRECTLY BELOW.
- 15-INSTALL LAMACOID NAMEPLATES DESIGNATING INCOMING FEEDER SPACES & ALL CHUBBLE DOORS.
- 16-MOLDED CASE BREAKERS FOR FEEDER USE ONLY ARE 30000 AMP. INTERRUPTING (100 A. FRAME ARE TYPE F - 225 A. FRAME ARE TYPE THFN). BREAKERS USED IN COMBINATION MOTOR STARTERS SHALL BE 15,000 AMP. INTERRUPTING (100 A. FRAME ARE TYPE THFN - 225 AMP. FRAME ARE TYPE TFD).

SEE DATA BELOW FOR DETAILED INFORMATION

CODE	CID	UNIT	STARTED	HEATED	EXTR.	H.P. 600 KVA	NAME PLATE	CONS.	DESCRIPTION	CONDUIT & MISC SIZE	NAME PLATE ENGRAVING	CODE	CID	UNIT	STARTED	HEATED	EXTR.	H.P. 600 KVA	NAME PLATE	CONS.	DESCRIPTION	CONDUIT & MISC SIZE	NAME PLATE ENGRAVING
	No.	NO.	NO.	No.	TRIP	TRIP	TRIP	TRIP					No.	NO.	NO.	No.	TRIP	TRIP	TRIP	TRIP			
C	7801	2	3	CR 123-FLS 80	100A	40	0	0	PUMP NO. P-3 MOTOR NO. M-3	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7801 REACTOR RECIRC. PUMP #2												
G	7802	2	3	CR 123-FLS 80	100A	40	35	35	COMPRESSOR NO. 16 MOTOR NO. M-31	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7802 EC REF. UNIT COMP. NO. 2												
	7803	1	2								CKT. # 7803 SPARE STARTER												
C	7804	1	1	CR 123-CT 780	20A	5	3.5	3.5	PUMP NO. P-4 MOTOR NO. M-4	TRAY, 1" C, 1/2" 1/2"	CKT. # 7804 PRIM. STRIPPER FEED PUMP #1												
C	7805	1	1	CR 123-CT 780	20A	5	4	4	PUMP NO. P-6 MOTOR NO. M-6	TRAY, 1" C, 1/2" 1/2"	CKT. # 7805 HVS STILL FEED PUMP #1												
A	7806	2 1/2	4	CR 123-F 140	175A	100	95	95	PUMP NO. P-16 MOTOR NO. M-16	TRAY, 3" C, 1/2" 5" 1" 1/2"	CKT. # 7806 COOLING TOWER PUMP #2												
F	7807	1	-		30A	10	2.5	2.5	10 KVA TRANSFORMER	TRAY, 4" C, 3" 1/2" 4" 1" 1/2"	CKT. # 7807 EC STORAGE AREA LIGHTING												
C	7808	1	2	CR 123-CT 780	50A	15	12	12	PUMP NO. P-13 MOTOR NO. M-13	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7808 EC TRANSFER PUMP #2												
C	7809	1	1	CR 123-CT 780	20A	5	5	5	PUMP NO. P-3 MOTOR NO. M-3	TRAY, 1" C, 1/2" 1/2"	CKT. # 7809 HVS STILL REFLUX PUMP #1												
C	7810	1	1	CR 123-CT 780	15A	1/2	1/2	1/2	PUMP NO. P-24 MOTOR NO. M-22	TRAY, 1" C, 1/2" 1/2"	CKT. # 7810 GLAND FLUSH PUMP #1												
M	7811	1	1	CR 123-CT 780	30A	1/2	7	7	VENT COMPRESSOR #2 MOTOR NO. M-23	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7811 STAG. VENT COMPRESSOR												
E	7812	1			125A	0	0	0		2- 2" 3" 1/2" 1" 1/2"	CKT. # 7812 WELDING EC & HCL AREA												
C	7813	1	1	CR 123-CT 780	15A	3	2.2	2.2	PUMP NO. P-10 MOTOR NO. M-10	TRAY, 1" C, 1/2" 1/2"	CKT. # 7813 PRODUCT PUMP #1												
C	7814	1	2	CR 123-CT 780	50A	15	12	12	AGITATOR NO. A-1 MOTOR NO. M-50	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7814 FLASHER DOPP AGITATOR												
D	7815	1 1/2	2	CR 123-CT 780	150A	30	30	30	TRANSFERS TO RT. SOURCE ON LOSS OF POLY. ON TRC	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7815 EMERG. POWER TRANSFER PANEL												
	7816	1	1		0	0	0	0	FUTURE SPACE ONLY		CKT. # 7816 FUTURE												
C	7817	1	1	CR 123-CT 780	40A	10	1	1	PUMP NO. P-14 MOTOR NO. M-14	TRAY, 1 1/2" C, 1/2" 4" 3" 1/2"	CKT. # 7817 EC REWORK PUMP												

LOAD SUMMARY DATA				KVA RM H.P.	AMPS *
ACTUAL CONSUMED LOAD (MEAN OF 60 SEC AVG WITH PLANT AT 100% PRODUCTION)				217.53	271
TOTAL CONNECTED NAMEPLATE				305.83	361
RUNNING NAMEPLATE				265.83	333
SPARE NAMEPLATE				40	50

LOAD SUMMARY DATA
ACTUAL CONNECTED LOAD (BASED ON EST. AVG. WITH PLANT AT 100% PRODUCTION)

KVA @ 480V
217.53

AMPS*
271

TOTAL CONNECTED NAMEPLATE

305.83

361

RUNNING NAMEPLATE

265.83

333

SPARE NAMEPLATE

40

50

8 AMPS @ 480V @ 1.05

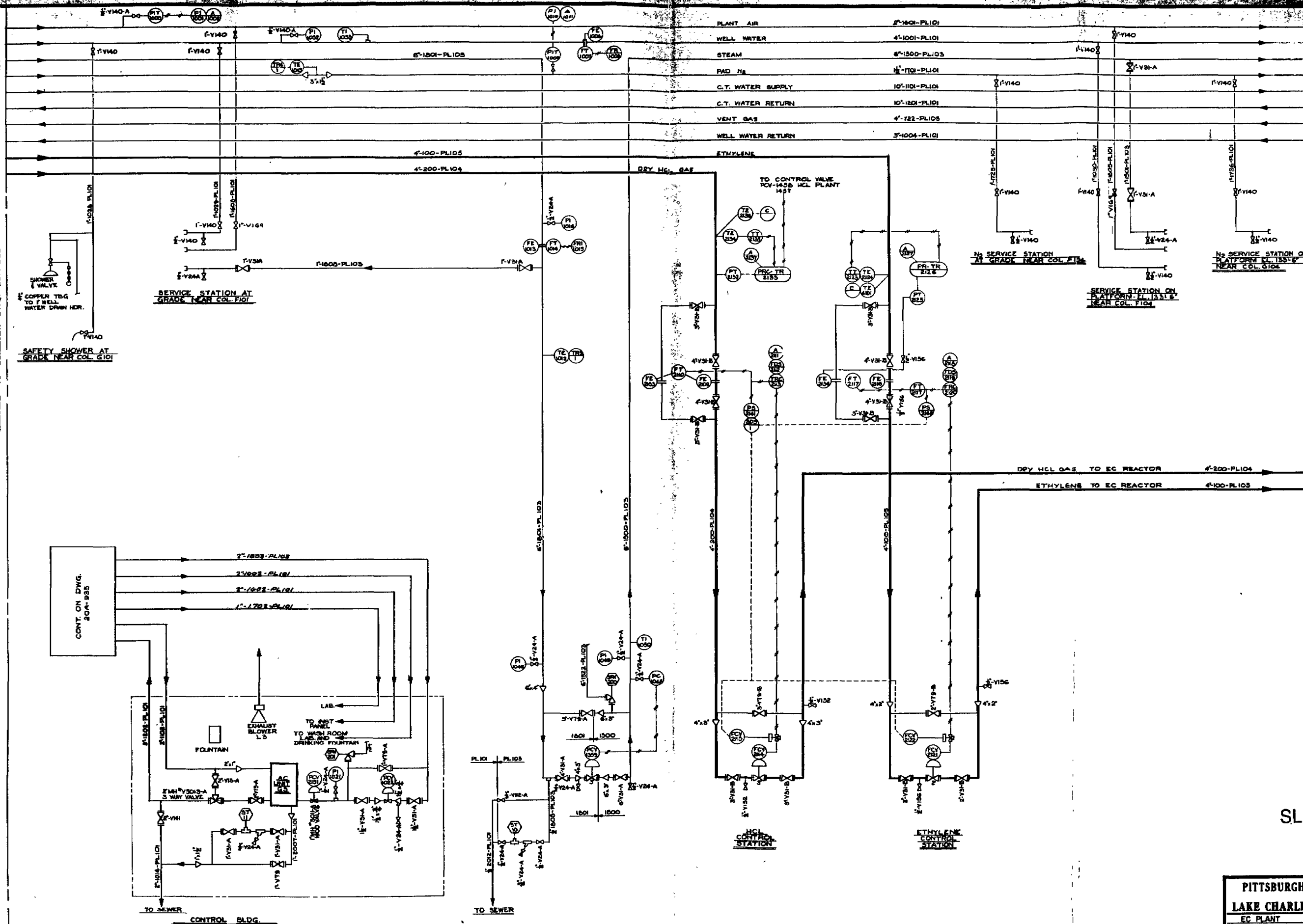
SL 008943

Ford, Bacon & Davis
Construction Corporation
NEW YORK, N.Y.
MEMPHIS, TENN.
DATE 5-28-65

PITTSBURGH PLATE GLASS COMPANY
LAKE CHARLES, LOUISIANA
EC PLANT ELECTRICAL
480VOLT MOTOR CONTROL CENTER
ONE LINE DIAGRAM & FRONT LAYOUT
BUS NO. 7800
DRAWN BY: J. STERN
CHECKED BY: J. STERN
APPROVED BY: J. STERN
DWG. NO. 64A-7101

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

CONT. ON DWG. 20A-935



REVISIONS	
1-5-66	GEN. REV. M.L.

CONT. ON DWG. 64A-101 SMT. NO. 2 OF 6

SL 008945

CONFIDENTIAL:
Subj ct to Prot ctive Ord r
of 14th Judicial Di strict Court
No. 91-1145

PITTSBURGH PLATE GLASS COMPANY	
CHEMICAL DIVISION	
LAKE CHARLES, LOUISIANA	FLOW SHEET
EC PLANT MECHANICAL FLOW SHEETS	
SHEET 1 OF 6	
DRAWN BY L.D. DERRICK	DATE 11-24-60 SCALE NONE
CHECKED BY M.H.	DATE 11-24-60
APPROVED BY	DATE 3-17-61
DWG. NO. 64A-101	

