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

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PPG INDUSTRIES

CHEMICAL DIVISION

LAKE CHARLES, LOUISIANA

NUMBER ONE ETHYLENE DICHLORIDE PLANT

OPERATING MANUAL

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Book No. 2

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PAGE NUMBERING SYSTEM

This manual is designed to permit revision and additions without disturbing the order of page numbers or the figure designations of illustrations and tables other than in the particular section affected by the changes. This is accomplished by using a combination of two numbers, the first being the section number while the second represents a page, illustration or table in the section. Thus, page 10-5 represents Page 5, Section X; Figure 3.5 is Figure 5 of Section III; and Table 8.5 is Table 5 of Section VIII.

I. COMPOUND ABBREVIATIONS

Abbreviation	Name	Formula	Atm. B. P., ^{o}F
N_2	Nitrogen (Light)	N_2	-345.75
CO_2	Carbon Dioxide (Light)	CO_2	-109.30
O_2	Oxygen (Light)	O_2	-297.40
CO	Carbon Monoxide (Light)	CO	-313.60
H_2	Hydrogen (Light)	H_2	-422.86
CH_4	Methane (Light)	CH_4	-258.52
C_2H_4	Ethylene (Light)	C_2H_4	-154.68
C_2H_6	Ethane (Light)	C_2H_6	-127.53
HCl	Hydrogen Chloride (Light)	HCl	-121.09
Cl_2	Chlorine (Light)	Cl_2	-29.29
EDC	1,2-Dichloroethane (Ethylene Dichloride)	$C_2H_4Cl_2$	182.25
1,1,2-TCE	1,1,2-Trichloroethane (Heavy)	$C_2H_3Cl_3$	236.79
Unsym TeCE	1,1,1,2-Tetrachloroethane (Heavy)	$C_2H_2Cl_4$	264.2-266.0
Sym TeCE	1,1,2,2-Tetrachloroethane (Heavy)	$C_2H_2Cl_4$	295.16
Tars or HvCl	Any Heavy Chlorinated Hydrocarbons	-	-

Note: "Heavy" indicates compounds having higher boiling points than EDC.

"Light" indicates compounds having lower boiling points than EDC.

II. INTRODUCTION

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

1. To be a training guide for the operators in learning the EDC plant operations.

2. To serve as a ready reference for operating personnel during their stay at the EDC plant.

3. To provide a standard approach for operating the plant from shift to shift.

4. To serve as a place where current data, facts, information, operating procedures, etc., related to the EDC plant are compiled.

As operating experience is gained, it is expected that some of the Standard Operating Procedures (SOP's) outlined in this manual will be altered. Please feel free to suggest changes that will aid in keeping this manual up-to-date.

This manual is the property of the Chemical Division of PPG Industries and may be recalled at any time. You are personally responsible for the safe keeping of this particular manual which has been numbered and charged to you.

III. GENERAL DESCRIPTION OF EDC PROCESS

A. Uses of EDC

The major uses of EDC generally fall into these categories:

1. It serves as a raw material to produce vinyl chloride.

2. It is a raw material for production of chlorinated organic compounds such as Tri-Ethane, perchloroethylene and trichloroethylene.

3. It is used as an additive in compounding tetraethyllead fluid.

4. It is a degreasing solvent, extraction solvent, dewaxing solvent, and fumigant agent.

The major use of EDC is as a raw material in producing vinyl chloride monomer from which the enormous line of vinyl plastics is made.

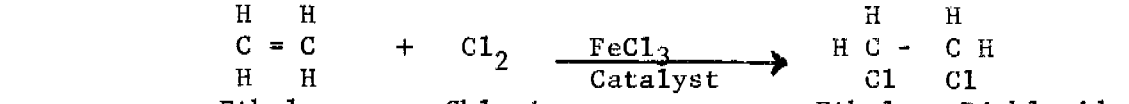
A large portion of the EDC produced by PPG in Lake Charles is captively used as a raw material for producing other chlorinated organics.

EDC is used in compounding tetraethyllead fluid (TEF). The commercially important TEC blend consists of various amounts of tetraethyllead, ethylene dichloride, ethylene dibromide, kerosene, dye, and an inhibitor. EDC has not gained much favor as a degreaser or dry cleaning solvent because of its flammability. However, it is used in vapor phase degreasing of metal parts. Other incidental uses of EDC include extraction solvents for fats and oils, caffeine, cottonseed oils, etc. It has been employed in dewaxing of oil products, deoiling of petroleum waxes, and as a component in fumigant compounding.

B. Process Description:

Process flow sheets for the operations to be described in this section are given on pages 3-6 and 3-7.

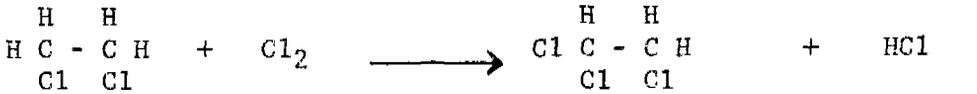
1. Reactor: Ethylene dichloride is produced by the reaction of one mole of chlorine with a mole of ethylene in a volume of liquid ethylene dichloride containing ferric or iron chloride ($FeCl_3$) as a catalyst. This is an addition reaction; i.e., chlorine adds chemically to ethylene to produce EDC as shown in the following equation:



Mol. Wt.	28.052	70.914	98.966

All of the ethylene and chlorine is utilized to produce the product and essentially no by-products are formed.

Unfortunately, some undesirable secondary reactions do occur. The more prevalent of these is the substitution reaction in which EDC and chlorine react to produce trichloroethane and hydrogen chloride, as follows:



	EDC	Chlorine	1,1,2-Trichloroethane (TCE)	Hydrogen Chloride
Mol. Wt.	98.966	70.914	133.415	36.465

The TCE can undergo further substitutive chlorination to produce tetrachloroethanes (TeCE):

	TCE	Chlorine	Sym. TeCE	Hydrogen Chloride
Mol. Wt.	133.415	70.914	167.864	36.465

Actually the $FeCl_3$ is not a true catalyst in this operation. In reality, it is used to depress or inhibit the secondary substitution reactions.

The light products from secondary reactions, the impurities in the incoming gas streams, and unreacted ethylene and chlorine make up the vent gases from the unit.

The reaction of ethylene and chlorine is exothermic, that is, it gives off heat. Theoretically, the heat liberated from this reaction is more than six times the heat required to vaporize one mole of ethylene dichloride. The heat of reaction at 25 C is 50.4 Kcal/gm. mole while the latent heat of evaporation of EDC at its boiling point is 7.7 Kcal/gm. mole. It is necessary to cool the liquid EDC in the reactor, otherwise, the reactor would boil dry. Cooling is accomplished by condensing the vaporized EDC and returning a stream of liquid EDC to the reactor to maintain the level. In such an operation, known as a boiling reactor, the cooling is accomplished at the condenser using cooling water.

In the pilot plant for every 4.5 moles of EDC vaporized, 3.5 moles of cooled EDC were returned to the reactor. This is defined as the reflux ratio of 3.5/1. Theoretically, in this plant, with smaller heat losses for every 6.5 moles of EDC vaporized and condensed, 5.5 will be returned to the reactor (reflux ratio 5.5/1).

The vaporized ethylene dichloride plus the by-products and impurities leave the top of the reactors in the gaseous form, and go to the reactor condensers. The condensed liquid flows down through a stand leg to the reactor gas separator. The major part of this stream is refluxed back to the reactors to keep the levels constant. The reflux flows are automatically controlled by level controls on the reactors. The condensed liquid that is not required for reactor level control goes to one of two places; to the stripper as feed or to the Per/Tri plant as feed.

The vapors from the reactor condensers, the reactor gas separator, the stripper condensers and the stripper gas separator are vented through the separator condensers. The liquid from the separator condensers flows back to the reactor gas separator through a stand leg.

The vent gas from the separator condensers goes to the vent condenser to recover the last traces of EDC. The condensed liquid is returned to the reactor gas separator.

The noncondensable gases from the vent condenser flow to the stack seal where they are scurbbed with water, then discharged to the atmosphere through the vent pipe mounted on the side of the still. The water scrubbing removes most of the HCl that is formed as a by-product.

The pressure drop through the vent system dictates the operating pressure on the reactor; atmospheric plus the pressure drop through this system. In a given size system, the more vent gases that flow the higher the back pressure on the reactors.

2. Stripper: The EDC liquor from the reactor gas separator that is not fed back to the reactors for level control or to the Per-Tri plant as feed flows as feed to the stripper. This feed rate is regulated by the level control in the reactor gas separator.

The primary function of the stripper is to separate the low boiling components (lights) from the EDC. These light components are primarily HCl, some unreacted ethylene and impurities that enter the reactor in the feed streams.

The liquid flow from the reactor gas separator enters the top of the packed stripper. The liquid flows down through the packing counter-current to the upward flow of hot vapors containing light products and EDC. The stripping action removes the low boiling components from the EDC before it reaches the bottom of the stripper. Product is removed through an overflow and stand leg into the EDC process crude tank.

To obtain the most efficient operation of the stripper, a certain amount of EDC is refluxed. In the pilot plant operation, 1.5 moles of EDC was vaporized overhead for every mole of product that was taken from the bottom of the stripper. The EDC is vaporized by applying steam to a reboiler on the side of the stripper. The steam flow to the reboiler is regulated to produce the desired reflux ratio.

The light components plus the vaporized EDC leave the top of the stripper and pass to the stripper condensers. The condensed EDC flows through a stand leg into the stripper gas separator. This stream constitutes the stripper reflux. The reflux flow to the stripper is controlled by the level control in the stripper gas separator and is regulated by the quantity of steam that is fed to the stripper reboiler. This column is essentially on total reflux of EDC; HCl and lights being the only materials vented overhead.

The noncondensable from the stripper condensers are vented to the separator condensers where they are mixed with the noncondensables from the reactor system.

3. EDC Still: The liquid in the process crude tank is EDC with a purity of 97 to 99%; however, it still contains the heavier chlorinated organic compounds. These impurities are separated from the finished product in the distillation column or what is commonly called a still.

The liquid from the crude tank is pumped to the feed tray of the still. This flow is set to maintain a constant desired feed rate to the still.

The Number one and Number two EDC plants are tied together such that EDC from either plant crude tank can be fed to the other plant still.

The liquid feed flows downward through the column counter-current to ethylene dichloride vapors. The heavy chlorinated organic compounds with high boiling points flow down the column to the reboiler. Here they accumulate and are periodically withdrawn and sent to the still bottoms storage tank.

Liquid in the bottom of the still is vaporized in a thermosiphon reboiler. This produces the upward flow of EDC vapors which become increasingly pure toward the top of the still. These vapors leave the top of the still and are condensed. The condensed liquid drains to the still reflux drum through a stand leg. Any noncondensables are vented through the stack seal. The pressure drop through the stack system determines the operating pressure of the still over and above the slight nitrogen and pressure imposed on the condenser.

To obtain the maximum efficiency and separation in the still, a part of the condensed overhead product is circulated back to the top of the still as reflux. The ratio of EDC returned to the still as reflux over the EDC removed as product is termed the still reflux ratio. This reflux ratio should be approximately 1:1. The reflux flow is set at a constant desired rate.

The excess condensed product from the still reflux drum is pumped through the product cooler to storage. The product flow rate is controlled by the still reflux drum liquid level.

The still bottoms are pumped periodically to the still bottom storage tank.

The product storage tanks are padded with nitrogen to eliminate the mixing of EDC vapors with air to form an explosive mixture. The pad is maintained at about 3 inches water pressure at the top of the tanks. The pad system also prevents water contamination of the stored EDC by wet air.

4. Flasher: This system is designed to remove tar and carbon solids from the EDC still bottoms so that these bottoms (primarily TCE) may be used as feed to the Per-Tri plant. A thermosiphon reboiler will vaporize the lighter boiling compounds. The overhead stream, consisting primarily of TCE and EDC, will be condensed in the flasher condenser and will enter the flasher condensate tank. From this tank the bottoms stream is pumped to the bottoms storage tank, this tank is the reactor dump tank. From the dump tank the bottoms are sent to the Per-Tri plant.

IV. DETAILED EQUIPMENT DESCRIPTION

A. Process Equipment

1. Ethylene Feed System: Gaseous ethylene at 600 to 900 psig is supplied by a 6-inch pipeline from the Petroleum Chemicals, Inc., plant approximately 5 miles west southwest of our plant. At a junction point near Louisiana Supply is the 4-inch take-off serving PCI's metering station, situated in the northwest corner of the EDC area. No PPG facilities are to be located within 75 feet of the metering station without prior written consent from PCI. Maintenance of the station and the line feeding it is PCI's responsibility. In case of trouble in the station notify the Shift Engineer. The station is provided by PPG with a 1-inch instrument air line, 2-inch steam line and necessary power for instruments and lighting.

Upon entering the station, the ethylene flows through a filter charged with activated alumina to remove scale and oil.

From the filter the ethylene passes through one or both of two tube-type steam heated exchangers which are arranged in series. The exchangers provide 70°F gas and eliminate icing in the first stage of pressure reduction. The ethylene temperature at the exchanger discharge is controlled and monitored; steam flow to the exchanger is controlled by the temperature. The temperature recorder is located in the sheltered meter house. PPG has agreed to change the temperature chart daily; it is to be sent to PCI via our Record Department.

From the exchangers the ethylene goes to the first stage reducing station which brings the pressure down to 300 psig.

The ethylene then proceeds through two G.E. mass flow meters arranged in series. These meters are digital integrators which read directly in pounds. While either or both meters can be bypassed, normally both are in service. When the temperature chart is changed these integrators are to be read and their values recorded and forwarded to the Record Department for transmittal to PCI.

Leaving the mass flow metering installation, the ethylene enters the second stage reducing station which lowers the pressure to 165 psig. It is then metered by a G.E. mass flow meter owned by PPG. This integrator reading is to be recorded when the ethylene temperature charts are changed.

The mass flow meters are measuring the total ethylene consumption of Production Area B, not just the ethylene consumed by the EDC units.

Leaving the PPG metering station the ethylene flows through a 6-inch line to the EDC pressure reducing station. The ethylene flows to the two EDC units are split upstream of the station. Separate pressure reducing station are used for the units. The pressure is lowered to 45 psig and is continuously recorded and controlled in the control room. High and low pressure alarms are provided on both the upstream and downstream sides of the reducing station.

The pressure reducing station consist of two pressure control valves arranged for parallel flow. The smaller of the valves will regulate the downstream pressure and the larger valve will be closed at the lower flow rates. The larger valve will open and control the downstream pressure when the flow rate becomes larger than the small valve can handle; the small valve is completely open at this time.

The ethylene stream splits downstream of the pressure reducing station. From this point the feed systems to the two reactors are separate. Each stream passes through a recording flow controller and a loop seal before entering the reactors through the spargers. The temperature of the ethylene at the metering orifice is continuously recorded along with the control pressure on a strip chart in the control room. This chart and the ethylene flow chart should be torn daily and sent to the Record Department for ethylene consumption calculations.

2. Chlorine Feed System: The No. 1 EDC plant gets its chlorine feed from the liquefaction area through two systems. One feed system supplies liquid chlorine while the other supplies chlorine cell gas. The plant feed will be either all liquid chlorine or a combination of liquid chlorine and cell gas.

Liquid chlorine is piped to Production Area B from the No. 7 and 8 chlorine scale tanks in the liquefaction area. The liquefaction lead operator is responsible for switching the chlorine feed from one scale tank to another to assure a continuous supply. When a switch is made a slight change in supply pressure may be experienced. The liquefaction department will notify the EDC plant when a switch is going to be made.

The liquid chlorine is pumped to the Production B area. The supply pressure is regulated by manually adjusting a recirculation valve at the pump. The liquefaction lead operator should be notified if a low or high supply pressure is experienced. A supply pressure of 120 to 130 psig is desirable at the EDC plant.

The other production units in Area B take liquid chlorine from the same supply system and should be notified of any intended large change in liquid chlorine consumption.

The liquid chlorine comes from the scale tanks to the chlorine vaporizers located on the southeast corner of the EDC plant slab. The chlorine vaporizers are arranged so that they can be operated individually, in series, or in parallel depending on the rate of vaporizing required.

A liquid chlorine stream from the vaporizers flows to the Tri-Ethane plant where it is vaporized and fed to the Tri-Ethane and HCl plants. The purpose of sending the chlorine to be used in these two plants through the EDC vaporizers is to lower its oxygen content, which makes for a better reaction between chlorine and EDC in the Tri-Ethane plant.

One major precaution which should be taken when operating the chlorine vaporizer is to be sure that the liquid chlorine is not turned on when there is no steam on the vaporizer. This would allow the chlorine feed system to fill with liquid chlorine which when introduced into the reactor would create a violent reaction and possibly do damage to the reactor or connecting equipment. Always turn steam on to the vaporizer first and allow the tubes to heat up some before turning on the chlorine supply

If steam failure occurs and the cause is not apparent or readily corrected, the liquid chlorine should be turned off. Possibilities of loss of steam to the vaporizer are:

a. Failure of the main steam supply. This would then make it necessary to shut down the whole plant.

b. Failure of the steam trap on the vaporizer. The bypass around the trap can be opened and the unit operated this way until the trap is repaired.

c. Failure of the automatic steam pressure control valve. If this should occur, it is possible to operate with manual control of the steam pressure by means of the by-pass around the automatic valve. This will require considerable amount of attention when operating in this manner.

The chlorine vaporizer will operate at the pressure imposed upon it by the chlorine feed system. From the vaporizers the vaporized chlorine can follow one of two routes to the chlorine surge drum. When only liquid chlorine feed is being used, the vaporized chlorine passed through a pressure control valve and directly into the surge drum. The surge drum pressure is maintained at 35 psig by the pressure control valve.

If a combination of liquid chlorine and cell gas is being fed to the EDC plant the two streams are mixed at the surge drum. It is necessary to measure the individual streams before they are mixed. A system consisting of a pressure control station, a small surge drum and flow controller recorder station is used for measurement of the vaporized chlorine stream. The chlorine leaves the vaporizer and is pressure reduced to 45 psig before entering the small surge drum. From the small surge drum it passes through the flow recorder-controller station to the large feed surge drum which operates at 35 psig.

The chlorine cell gas comes to Area B directly from the discharge of compressors in the liquefaction area. The EDC and wet HCl plants use cell gas.

The cell gas line pressure will be approximately 42 psig at the EDC plant but will vary some depending on the Area B consumption. For this reason the HCl plant should be notified before making changes in the rate of cell gas consumption.

Large variations in the amount of cell gas used in Area B will affect the liquefaction area operation. If the cell gas is not consumed as gas it has to be liquefied. A large cut in cell gas consumption in Area B would increase the liquefaction load and perhaps require start-up of additional liquefaction equipment. Therefore, the liquefaction area should be notified as soon as a consumption rate change is anticipated. The change should be made slowly if possible.

After the cell gas leaves the main header it passes through one of two systems which contains a pressure control valve and flow recorder. The two systems have different capacities and the one to be used is determined by the rate of cell gas consumption.

The cell gas passes next to the feed surge drum where it is mixed with the vaporized chlorine. The cell gas pressure control valve controls the surge drum pressure at 35 psig.

The chlorine flows through individual lines from the feed surge drum to each reactor. Each flow, regulated by an automatic flow recorder controller goes through a seal loop and a sparger system before entering the reactor.

Strip charts showing the temperature, pressure and flow of the cell gas, vaporized chlorine and feed streams will be torn daily and sent to the Record Department for calculation of chlorine consumptions.

3. Chlorine Vaporizers, 60A-37-729: The chlorine vaporizers were manufactured by the Richard M. Armstrong Company. They are size "H" vertical vaporizers with an approximate capacity of 11,500 pounds of chlorine per hour. The vaporizers are 20" in diameter by 7.5' and have bayonet type tube heaters. The No. 1 vaporizer has approximately 1/3 of its tubes plugged to reduce its capacity for use at low production rates. The vaporizers are steam heated with low pressure steam. They are equipped with a steam pressure control valve which reduces the steam supply to the desired vaporizer chart pressure.

The vaporizers are protected against high pressure by a pressure relief valve set for 225 psig.

4. Small Chlorine Surge Drum, 60A-60-422: The small surge drum used in the vaporized chlorine measurement system is a steel vertical cylindrical tank with dished heads. The vessel is protected against high pressure by a three inch rupture disc which will rupture if the pressure reaches 125 psig. A pressure relief valve set to relieve at 115 psig is mounted above the rupture disc.

5. Large Chlorine Surge Drum, 60A-60-465: The large surge drum is a steel vertical cylindrical tank with dished heads. The vessel is protected against high pressure by a 3" rupture disc which will rupture at 125 psig. A pressure relief valve set for 115 psig is mounted above the rupture disc.

6. EDC Reactors, 60A-59-7, 60A-60-344: There are two reactors in the No. 1 EDC plant. The combined capacity of the reactors is 429 TPD.

The No. 1 reactor is a nickel-clad vessel that measures 8'0" I.D. by 45'0" tangent to tangent and sits on a 6' support skirt. The reactor is essentially an empty vessel, but contains a sparger feed distribution system, a boiling bed and a 10" nickel demistor

The sparger system is constructed of 4" nickel pipe. Each feed stream has four spargers which extend 3'11" into the reactor and has a 3/16" slot 2'9" long in its bottom. The ethylene spargers are located 1'6" below the chlorine spargers. The feed gas streams are fed to their respective spargers by means of a manifold that circles the reactor.

Located above the chlorine sparger system is a boiling bed support basket containing two layers of 3" ceramic spiral rings. The boiling bed distributes and breaks up gas and vapor bubbles so that the reactor will boil evenly.

The vapor leaving the reactor has a tendency to entrain with its liquid (containing $FeCl_3$ and heavies). In order to remove this unwanted liquid from the vapor, a 10" nickel demistor is located in the top of the reactor with imprigement baffles just below it.

The No. 2 reactor is a verticle nickel-clad vessel that is 5'6" I.D. by 37' from bend line to bend line. It sits on a 6' support skirt.

The feed distribution or sparger system is constructed from 4" schedule 40 Ni pipe. Each pipe extends 2'8" into the reactor. The bottom of each pipe contains a 1/4" slot that is 2' long. Each feed system consists of four spargers fed by a manifold which circles the reactor. The chlorine spargers are located 1'6" above the ethylene spargers.

The reactor is equipped with a boiling bed located just above the feed spargers and a 4" Ni demistor section which is located 3' from the top of the vessel.

Each reactor is protected against high pressure by a rupture disc which will rupture at 50 psig pressure.

The reactors are equipped with liquid level controllers which maintain a constant operating level. The No. 1 reactor normally is operated with a liquid level of approximately 24' while the No. 2 reactor is operated at approximately 18'. If it were not for the liquid level control system, the reactors would soon boil dry. It was mentioned previously that the reaction of chlorine and ethylene is exothermic. The heat generated in the formation of one mole of EDC is sufficient to vaporize over 6 moles of liquid EDC from the reactor. Therefore, it is necessary that the liquid EDC reflux stream be returned to the reactor to maintain the liquid level.

There are three ways to determine the liquid level in the reactors. First there are the sight glasses mounted on the side of the reactors. These glasses are used for checking the liquid level control when the need arises. Normally these sight glasses will be closed off and drained. Light shining on the sight glass containing EDC will cause further reaction of the material to form undesirable heavies.

A second way to determine the reactor level is to read the indicated pressure near the bottom of the reactor. Each foot of reactor liquor will impose about 0.5 psi on the pressure guage. Knowing this pressure and the pressure in the vapor space of the reactor the liquid level can be calculated using the following equation.

$$\text{Reactor Level (Ft. Above Press. Tap)} = \frac{\text{Bottom Press., psig - Vap. Space Press., psig}}{0.497 \text{ psi/ft.}}$$

The other system for determining the reactor level is the level control instrument. This instrument provides a record of the reactor levels at the panel board. Incorporated into the controller is a high and low level alarm to notify the operator of trouble in the reactors.

The normal operating temperature of the reactors will be between 208°F at the bottom and 194°F at the top. Should the temperature increase to 225°F due to an uncontrolled reaction, high ack pressure, insufficient reflux, etc., the reactor should be shut down until the difficulty can be resolved.

The reactors will be operated with an excess of ethylene at all times. This is about the most important thing to remember about the control of the reactor. If it is desired to increase the production rate of the plant, the ethylene flow is increased first. Then the chlorine flow is increased to the desired rate. The reverse procedure is followed in cutting the production rate. The chlorine flow is now decreased first; then the ethylene flow. These procedures will provide the desired slight excess of ethylene that will be vented through the stack seal at all times.

A violent explosion can occur if chlorine and ethylene are mixed in the reactor vapor space. This is the reason for operating the reactor with an excess of ethylene. When an excess of ethylene is being used no free chlorine will get into the vapor space. Should the ethylene flow drop below the chlorine flow, however, chlorine gas will accumulate in the vapor space. If the lower explosive limit of chlorine and ethylene is reached an explosion may occur. Never change from excess ethylene to excess chlorine.

If a deficiency of ethylene is noted and an explosion has not occurred, start a nitrogen purge through the system and add sufficient ethylene for the excess.

There will always be some HCl in the vapors coming off of the EDC reactors. The HCl is due mainly to the substitution reaction of EDC and chlorine to give TCE and HCl.

A certain amount of heavy chlorinated compounds called tars will be formed in the reactor. These do not vaporize overhead and will accumulate in the reactor liquor. Periodically, some of the reactor liquor will be drained from the bottom of the reactor into the clean-up kettle. Here the EDC will be vaporized and returned to the No. 2 reactor vapor space. The tars will be drained from the kettle into drums and discarded.

7. Reactor Condensers, 60A-71-466 and 730: There are two horizontally mounted reactor condensers. The reactor vapor enters the shell side of the condensers midway at the top. Cooling tower water is on the tube side of each exchanger. The shells and tubes are constructed of nickel.

These exchangers are designed to cool the reactor vapors from 190°F to 167°F and condense a corresponding amount of EDC.

8. Reactor Gas Separator, 60A-60-345: The reactor gas separator is a horizontal cylindrical tank with ellipical heads. The tank dimensions are 5'-6" I.D. by 15'-0" tangent to tangent. The material of construction is nickel clad steel.

The tank is equipped with a sight glass and a field mounted liquid level controller. If the tank level goes high or low an alarm sounds at the panel board.

The separator is protected against high pressure by a rupture disc which will rupture at 50 psig.

9. HCl Stripper, 60A-60-333: The stripper is fed reactor crude from the reactor gas separator by one of two reactor pumps. The flow forward to the stripper is controlled by the reactor gas separator level controller. The flow is measured and recorded at the panel board.

The stripper is a Ni-clad vertical vessel 4'-9" in diameter by 28' and sits on a 16' support skirt. The column is packed with 18' of 1 1/2 inch Intalox saddles. The packing support is located 5'6" from the bottom of the column and has two rows of 3" x 3" partition rings on it to support the 1 1/2" saddles. A distributor plate is located above the packing to give complete distribution of the liquid feed and reflux.

A thermosiphon reboiler circulates liquor from the bottom of the stripper. Some of the liquid is vaporized in the reboiler tubes and passes up through the column to be condensed and refluxed to the stripper. The stripper boil-up rate is regulated by controlling the flow of steam to the reboiler with the steam flow recorder controller located at the panel board. The stripper reboiler level is indicated by a sight gauge located on the side of the stripper.

The purpose of the stripper is to strip the discharged HCl and other light components from the crude EDC. These light materials go off the top of the column with the vaporized EDC and gas through the stripper condenser where most of the EDC is condensed. The HCl and light components and some EDC that does not condense go to the separator condenser where they are combined with the reactor vent stream.

The stripper is protected against surges in pressure by a rupture disc which will rupture at 50 psig.

10. Stripper Condenser, 60A-71-469 and 723: There are two horizontally mounted stripper condensers. The stripper overhead vapors pass through the single pass shell side of the condenser. Cooling tower water is on the four passes tube side. Each exchanger contains 106-1" tubes 14' long. The tubes and shell are of nickel construction.

Valves in the cooling water return lines are adjusted to give the proper cooling water discharge temperature.

11. Stripper Gas Separator, 60A-60-334: The EDC condensed in the stripper condensers passes to the stripper gas separator. The nickel vessel is a horizontal cylindrical tank with ellipitical heads and measures 4' I.D. by 10' tangent to tangent.

The stripper gas separator has a field mounted level controller which maintains a constant level by controlling the stripper reflux flow rate. If the separator level drops low or rises too high an alarm sounds at the panel board. A sight gauge is mounted on the gas separator and is used for checking and setting the level controller.

The separator is protected against high pressure by a 6" rupture disc which will rupture if the pressure reaches 50 psig.

12. Stripper Reflux Drier, 60A-73-18: The stripper reflux stream is pumped through a $CaCl_2$ packed drier to insure against water contamination in the stripper system. If water gets into the stripper system it will come overhead with the stripper vapor stream and be removed in the reflux drier.

The reflux drier is a vertical cylindrical vessel with a dished bottom. The top is fitted with a blind flange cover.

A support grid is fastened at the bottom of the drier. A layer of glass wool will be placed over the grid before the drier is charged with calcium chloride desiccant. To prevent the carry over of large desiccant particles, a grid is located in the top section of the drier and is fastened to the blind flange cover.

When the drier has been in use for some time a heavy liquid phase of $CaCl_2$ in water will form in the bottom. While there is still solid desiccant in the drier the reflux will continue to be dried. However, as the solid desiccant is used up the treated EDC water content will rise. When the predetermined maximum water concentration is reached the drier will have to be recharged. Liquid $CaCl_2$ brine can be drained off the bottom of the drier. The remaining mass of wetted $CaCl_2$ can then be washed out of the drier with water. Water remaining in the drier after wash-out should be swept out with nitrogen.

The reflux drier will be by-passed while being recharged.

13. Process Crude Tank, 60A-60-335: The carbon steel crude tank receives the stripper bottoms stream through a stand leg. The tank is a horizontal vessel with dished heads and is 8.5' by 17' tangent to tangent.

The crude tank is equipped with a level recorder at the panel board and a sight glass which is used to check and set the recorder. It is protected against high pressure by a 6" rupture disc which will rupture if the pressure reaches 50 psig.

14. Distillation Column, 60A-70-38: The EDC still is designed to remove the high boiling impurities from the liquid EDC and to limit the loss of EDC in the heavies purge stream. The column will produce an EDC overhead product of 99.9% purity. EDC loss to the bottoms stream will be limited to less than 1.0%.

The column is made of carbon steel and is 5' 8 1/4" I.D. by 88' 4" tangent to tangent. A cylindrical skirt 12' 5" tall supports the still. Four manways are provided for access to the inside of the column.

The still is fitted with two thermosiphon reboilers. It is expected that the heavies which will collect and be purged from the bottom of the column will have a tendency to "crack" to form carbonaceous substances or coke. This material will build up deposits on the tubes of the reboiler in service and reduce the heat transfer capability of the reboiler. Valves are provided so either of the two reboilers can be valved off from the column for removal and cleaning.

Phosplex-L is added to the still reboiler to prevent cracking and thus reduce tube fouling. Free iron in the reboiler increases cracking. The Phosplex-L ties up the iron and renders it inactive as a cracking catalyst. It is added continuously from an elevated drum through a rotometer.

The composition of the liquor in the still reboiler will vary proportionately with its temperature and will remain constant if the temperature is held constant. The steam flow rate to the still reboiler is controlled by a temperature controller which controls the temperature in the reboiler of the column. The temperature control valve may have to be adjusted from time to time by the operators to compensate for changes in the bottom or overhead composition. Need for these changes will be dictated by analyses of the product and bottoms stream.

The reboilers are single pass on both sides and constructed of carbon steel.

The column contains 54 trays fitted with 161 valve caps per tray. The trays are numbered from bottom to top. All trays are on an 18" spacing except the 27th and 28th trays. They are on a 24" spacing for the manways. The valve units on the trays are the V-1 Ballast unit as manufactured by Fritz W. Glitsch and Sons, Dallas, Texas. The trays are made of 12-gauge carbon steel, and the V-1 valves are made of type 410 stainless steel.

The trays all have straight segment downcomers made of 1/4" plate that is welded in place. The downcomer section of each tray has an area of 1.231 ft^2. Each downcomer is 16 1/2" tall, and thus, the overflow area onto each tray is 1 1/2". The downcomer from the No. 1 tray is the only exception. This downcomer is 62" long to provide a seal into the bottoms liquid of the column. All downcomers are extended 3" above their respective trays to provide a 3" overflow weir. The weirs are notched 1/2" on all trays above the 27th tray (lowest feed tray) but below this tray they are straight. The overflow weir is 40" long.

The trays are clamped into place on the tray support rings. Each tray is fitted with a 24" manway which is removable from either top or bottom. The trays are packed with Garlock 900 and AAA grade asbestos tape and wicking.

Feed to the column can be introduced on the 27th, 31st, 35th or 39th tray.

The column is fitted with 14 sample valves and 14 thermocouple wells. The sample points are paired with the thermocouple wells on the 2nd, 4th and 8th trays and thereafter on every 4th tray. The points sample the liquid for a particular tray as it is flowing through the downcomer to the tray below.

Feed to the column is set at a rate which is determined by the plant production rate. The feed rate may be changed by the plant operators to take care of level variations in the process crude tank.

The liquid feed starts down through the column from the feed point. In the course of its flow across each tray it comes in contact with the vapors generated by the reboiler. These vapors strip the EDC from the liquid and rectify the EDC toward the top of the column. The net results is a build-up of pure EDC in the top of the column and a concentration of feed impurities such as TCE in the bottom.

The vapors leaving the top of the column are condensed in the still condenser and the condensate collected in the still reflux drum. A predetermined portion of the liquids in the still reflux drum is returned to the top tray of the column via the reflux flow control valve. The amount of liquid reflux to be returned to the column is set by the operator. Under normal loading conditions the reflux will be approximately one-half of the liquid flow from the still reflux drum.

The liquid in the still reflux drum that is not returned to the column is drawn out of the column as product. The plant product is pumped to the product transfer tanks for storage or to the Tri-Ethane plant as feed. The amount of EDC going to the Tri-Ethane plant is controlled by the Tri-Ethane operator. The product flow to storage is automatically controlled by the product level control valve, which gets its control signal from the reflux drum.

A nitrogen gas line is tied in to the still condenser to provide a constant pressure of 1 1/2" water on the column. This is necessary to prevent pressure, and consequently temperature, fluctuations in the column. The padding system should always be in service when the still is being operated. The gas supply comes from the pressure regulator located at the transfer tanks.

From the above description it should be evident that the operators of the still must thoroughly know and understand the column before they can successfully control its operation.

15. Still Condenser, 60A-71-471,733: Vapor from the still passes to the horizontally-mounted steel heat exchangers where complete condensation takes place. The EDC vapors pass through the one pass shell side of the exchanger with cooling tower water on the four pass tube side. Valves in the return cooling water lines are adjusted to give the desired cooling water discharge temperature.

The condensers are padded with nitrogen and a vent line is provided for bleeding inerts from the still system to the vent stack.

16. Still Reflux Drum, 60A-60-337: The still reflux drum receives condensed EDC from the still condenser through a stand leg.

This vessel is a horizontal cylindrical tank with dished heads and measures 4.0' by 10.0' tangent to tangent. It is constructed of carbon steel. The reflux drum is equipped with a sight gauge and a field mounted level controller. The level controller controls the product flow rate to storage such that the reflux drum level is constant. High and low level alarms are provided on the panel board to indicate any imbalance in the EDC product system.

The vessel is protected against high pressure by a 4" rupture disc which will rupture if the pressure reaches 50 psig.

B. Equipment on Plant Exit Streams

1. EDC Product to Storage:

a. Product Coolers, 60A-71-473,735

The carbon steel product coolers are mounted horizontally and have a one pass shell side and one pass tube side. EDC is on the shell side and cooling tower water is on the tube side. The cooling water flow rate is manually controlled with valves in the return lines to maintain the desired discharge temperature.

b. Product Neutralizers

The product neutralizers are steel chambers 18" x 9' high located at the northeast end of the process pad. The chambers are packed with anhydrous flake caustic. The purpose of the neutralizers is to hold the HCl and iron concentration within the specification limits. When the still

reflux HCl concentration exceeds a predetermined value the product will be passed through the neutralizer. The efficiency of the neutralizer can be checked by analyzing the still reflux stream and the product stream. The only difference between these two samples is that the product stream has passed through the product neutralizer.

It will be necessary to recharge the neutralizer periodically. One neutralizer will be used while the other is being recharged.

c. Product Storage Tanks:

Refer to Section VIII for a discussion of product storage and transfer.

2. Vent Gas Disposal: The final vent stream from the EDC plant is composed primarily of inerts which enter the plant as impurities with the feeds, unreacted ethylene and chlorine, HCl, and methane. The several processing steps applied to this vent gas are covered below.

a. Separator Condensers, 60A-71-731,467

Vapors from the reactor condensers, reactor gas separator, stripper condensers and stripper gas separator are vented through the separator condensers. The condenser condensate flows back to the reactor gas separator through a stand leg.

The No. 1 condenser is a horizontally mounted nickel condenser with one pass on both the shell and tube sides. The vent gas is on the shell side of the exchanger with cooling tower water on the tube side. The exchanger has 68 one inch nickel tubes, 6' long. The shell is constructed of nickel and has a 13 1/4" O.D.

The No. 2 separator condenser is a horizontally mounted nickel exchanger with one pass on both the shell and tube sides. Vent gas is on the shell side of the exchanger with cooling tower water on the tube side. The exchanger has 125 one inch nickel tubes, 6' long. The nickel shell is 18" O.D.

b. Methane Addition System

The oxygen content of the chlorine feed passes through the reactor unreacted and can form an explosive gas mixture with the combustibles in the vent stream. To prevent an explosive vent gas mixture methane is added downstream of the separator condenser to adjust the concentration of combustible materials to a value above its upper explosive limit.

The concentration of oxygen in the vent stream is measured and recorded by the chromatograph. An oxygen content of less than 10% in the vent stream has been found to be safe under normal operating conditions and will not allow an explosive gas mixture to be formed during a plant upset. Therefore the amount of methane added to the vent stream is regulated to maintain an oxygen content of less than 10%. The methane flow is controlled by a control valve which is manually adjusted at the control panel.

The flow of methane is measured and recorded on a strip chart at the control panel. This strip chart should be torn daily and sent to the Record Department along with the methane temperature and pressure readings for methane consumption calculations.

c. Vent Condenser, 60A-71-468.

The vent gases from the separator condensers pass to the vent condenser where refrigeration is used to recover potential EDC losses. The condensate from the condenser flows back to the reactor gas separator.

The vent gases are cooled in the two pass tube side by Freon-12 on the one pass shell side. The exchanger has 86 one inch nickel tubes, 12' long.

The vent stream leaves the condenser through a vapor separator which knocks out and collects any EDC liquid that might be entrained in the vent stream. The vapor separator is a nickel vessel 14" O.D. x 20" high. It contains a 5" layer of wire mesh in the top.

d. Seal Pot and Stack

The vent stream is then scrubbed with water at the stack seal pot. The seal pot is a 2' diameter by 8' high Haveg tank where the vent stream passes countercurrent to a spray of water. The water washes the HCl out of the vent stream to prevent venting it to the atmosphere. The HCl containing water is sewered. The scrubbing water is the blow-down stream from the cooling tower and will be run at a set rate regardless of the plant production rate. The remaining vent stream passes up the exhaust stack to the atmosphere. The 4" diameter Chemtite exhaust stack is attached to the side of the distillation column for support.

The piping to and from the seal pot is constructed of Chemtite pipe. This was used for corrosion protection since the water at the stack seal contains HCl. The last few feet of the exhaust stack is made of Hastalloy. Since the material of which the final vent stream is composed is highly flammable, it is possible that the vent could catch fire from static electricity or lightning. The last section of Hastalloy pipe is to prevent the Chemtite pipe from being damaged from the heat produced if this should happen.

At the junction of the Chemtite and Hastalloy pipe a flame arrestor is installed. This serves as a block to prevent a flame from traveling down the exhaust stack if a fire were to start.

A steam line runs to the top of the exhaust stack and discharges at the stack opening. If the vent stream ever catches fire, it can be be extinguished by discharging steam into the exhaust stack. A manual steam valve is located on the north end of the catwalk above the pipe galley and can be opened if a fire should ever occur.

e. Refrigeration

The system employed to cool the vent stream is a direct expansion refrigeration system using Freon-12. The compressor is a Carrier Corporation Model 5H40 operating at 1160 rpm. The compressor is protected by the following safety devices:

1) The oil failure switch shuts off the compressor motor if the compressor oil pressure drops below 18 psi greater than the compressor suction pressure. It can be manually reset when the oil pressure goes above 11 psi greater than the compressor suction pressure. The pressure switch is connected to a thermal switch which it energizes if the oil pressure drops below the specified amount. The thermal switch takes approximately one minute to cut off the compressor. After this switch has been activated, three minutes must be allowed for it to cool before it can be manually reset and the compressor started.

2) The high pressure cut-off. This switch cuts off the compressor if the head pressure goes above 151 psig and resets at 113 psig.

3) Low pressure cut-off. This switch cuts off the compressor if the suction pressure drops below 20" vacuum and resets at 10" vacuum.

The 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 and thereby reduce the refrigeration capacity of the machine stepwise. This is necessary to be able to operate the refrigeration system satisfactorily at reduced ethylene dichloride production rates. The manual unloading valve is located on the bell cover in front of the compressor. When this valve is turned counter clockwise as far as it will go the compressor is full loaded. By turning it clockwise the compressor will unload three cylinders, one at a time, reducing the refrigeration capacity by one-fourth as each cylinder is unloaded.

The compressor is also equipped with an oil filter and a crackcase heater. The crankcase heater is used when the compressor is not running to prevent the accumulation of refrigerant in the crankcase oil when the compressor is put in operation. The oil filter has a throwaway element which should be changed periodically.

Manual discharge and suction shut-off valves are located on the compressor. The compressor should never be started up or operated with the discharge valve closed. This can result in serious damage to the compressor.

A suction strainer is provided in the low pressure Freon stream. This strainer is located at the rear of the compressor.

The compressor heads are cooled by a circulating stream of cooling water. Manually adjust the flow of water until its exit temperature is approximately 100°F.

The low pressure Freon passes from the vent condenser through a double pipe heat exchanger to the Freon compressor. The low pressure Freon cools the liquid Freon to remove the super-heat. This also insures complete vaporization of any entrained liquid Freon that may be in the low pressure gas stream. Liquid Freon returning to the compressor can cause considerable damage to the machine.

The high pressure Freon condenses in the water cooled condenser and is then drained to the vent condenser through the double pipe exchanger and a liquid seal valve. The purpose of this valve is to keep a liquid seal on the Freon condenser to prevent gaseous Freon from entering the vent condenser without condensing. The Freon is condensed on the shell side of the Freon condenser with cooling water on the tube side. The Freon is then vaporized on the shell side of the vent condenser by the vent condenser load.

The vaporized Freon passes from the vent condenser and returns to the suction of the compressor via the double pipe exchanger as the low pressure stream.

3. Flasher, 60A-70-47: Feed for the flasher comes from the bottoms storage tanks. This flow is controlled by a flow control valve and the rate may be read from a field-mounted indicator. Steam to the flasher reboiler is controlled by the level in the flasher.

The bottoms vaporized by the reboiler are condensed by the flasher condenser and collected in the flasher condensate tank. When the condensate tank becomes full the liquid is pumped to the reactor dump tank for storage until it can be sent to the Per-Tri plant.

The heavy impurities in the flasher feed stream concentrate in the flasher bottom and are drained periodically for disposal.

4. Clean-Up Kettle, 60A-60-331: The clean-up kettle is a jacketed 500 gal. steel kettle. EDC that is to be cleaned up is pumped from the reactors or the dump tanks to the kettle and evaporated by means of steam in the kettle jacket. The vapors from the kettle go off the top and are piped to the No. 2 reactor vapor space. Here they combine with the reactor vapors and are condensed in the reactor condenser. The heavies retained in the kettle are drained to drums and discarded. Steam to the kettle is automatically pressure controlled. The rest of the system is manually controlled.

Provisions are also made for using the clean-up kettle for catalyst mixing. This will be discussed in Section IX.

C. Utilities

The utilities for the EDC plant include cooling tower water, instrument air, steam, and the electrical system.

1. Cooling Tower Water: Cooling tower water for the No. 1 and No. 2 EDC plant and the MC plant is supplied by four connected units.

The No. 1 cooling tower was manufactured by the Water Cooling Equipment Company. The tower is constructed of creosate-treated redwood. This unit is a two-cell, forced draft tower in which the air is drawn from the bottom through a section packed with redwood slats. The draft is created by fans located at the top of each tower cell. The tower is designed to handle a recirculation rate of 1700 gpm at 90° to 115°F temperature rise.

The No. 2 cooling tower was manufactured by the Foster Wheeler Corporation. The tower is constructed of redwood and is a two-cell, forced shaft tower. The tower is designed to handle a recirculation rate of 5,000 gpm at 90° to 115°F temperature rise.

The No. 3 and No. 4 cooling towers were manufactured by the DeFlon-Anderson Company. The towers are one-cell, forced draft towers constructed of redwood. These towers are sheathed with Lascolite fiberglass panels. Each tower is designed to handle a recirculation rate of 2,500 gpm at 90° to 115°F temperature rise.

The water enters a distribution tray on top of the towers and showers down through the packed section countercurrent to the flow of air. In this fashion, the air is saturated with water vapor. The evaporation necessary to provide the water vapor cools the water until the exit temperature approaches the air wet bulb temperature. This temperature of the exit water will be below the ambient air temperature, but is dependent upon the humidity. Design temperature is 90°F.

The cooled water is pumped from the tower basin through the process coolers by the cooling tower pumps and back to the distribution tray on top of the tower.

A purge stream is required on the cooling tower water to prevent the accumulation of minerals in the water. In the case of this location, the chloride content of the make-up water is the controlling factor. This should be controlled at approximately 4-5 times the make-up chloride content by the purge rate. This purge stream is discharged through the vent stack seal of the No. 1 EDC plant and must remain on as long as the cooling tower is in operation.

To make up for the purge stream and the evaporation (windage) losses, well water is continuously added to the tower basin. The flow of well water is level controlled to maintain the proper level in the tower basins.

To insure the proper life of the tower and to prevent corrosion and scaling of the exchanger walls, the tower water is continuously treated. The types of treatment and how they are controlled are as follows:

a. pH Control

A continuous recording pH meter allows the operator to regulate the addition of sulfuric acid to the tower to keep the water slightly acid. An acid pH is required for two reasons; one is to guard the redwood from damage which occurs in an alkaline system; two is to keep the mineral solubility high enough to prevent scaling on the exchanger walls.

b. Chromate - Phosphate Addition

A control analysis for chromate-phosphate concentration will be run periodically and the addition regulated to give the desired concentration. These materials are used as corrosion inhibitors. They provide the metallic surfaces exposed to the cooling water with a protective film to keep down excessive corrosion.

c. Betz 107 Addition

Betz 107 is a specially blended organic anti-foulant and corrosion inhibitor which is used in combination with the chromate-phosphate blend (Betz P-40).

d. Chlorine Addition

Chlorine is added to the cooling tower water to prevent the formation of algea and slimes in the tower. This is particularly important in the summer since there these can form in sufficient quantities to plug up the tower distribution trays and even the piping if an algaecide is not present.

The cooling tower water is shock chlorinated three times per week. The residual chlorine level is raised to 0.75 ppm during these treatments. The chlorine is added directly from a ton chlorine cylinder.

A water treatment firm (Betz) will tell us what treatment is necessary and what concentration should be maintained. They will check the tower operation periodically (once per month) and make necessary recommendations.

Betz 107 and P-40 and sulfuric acid are added continuously to the cooling tower water by gravity flow into water aspirators that discharge into the cooling tower basin. The rate of addition is controlled by adjusting the discharge valve on the individual storage tanks.

2. Instrument Air System:

a. Compressors

The compressors for the system were manufactured by the Joy Manufacturing Company. They are Model WG09S compressors. They are water cooled, single stage, double acting, heavy duty, vertical compressors--designed to operate at a maximum pressure of 100 psig. Number 1, 2 and 3 compressors

have 10" x 9" cylinders. Number 4 and 5 compressors have 8" x 7" cylinders.

Number 1, 2 and 3 compressors are driven by Westinghouse, 60 HP, 1800 RPM, 440 volt, 3 phase induction motors. Number 4 and 5 compressors are driven by General Electric, 40 HP, 1770 RPM, 440 volt, 3 phase induction motors.

Since these compressors are essentially positive displacement machines there must be provisions to allow for variation in their output. These machines are operated as constant speed compressors and they have been provided with an electro-pneumatic constant speed unloading system for load variation. This system consists of an automatic pressure switch, a manual transfer switch, and a three way valve. The pressure switch senses the discharge pressure, and if the pressure goes above the desired maximum level (as would occur when the compressor output is more than the air being consumed), the pressure switch changes the three way valve and allows the compressor cylinders to unload (the compressor valves are blocked open so no compression can occur). The compressor will continue to operate in this manner until the discharge pressure lowers (90 psig) and then the pressure switch will automatically load the cylinders and compress up to 100 psig.

The compressor can be manually unloaded by putting the transfer switch, located on the side of the compressor, in the "OFF" position. The compressor will run with no load until the transfer switch is placed in the "ON" position. This switch is used when starting up the compressor to allow it to be started under no load. Note: The compressor should always be started up and shut down with the transfer switch in the "OFF" position.

A condensate trap is also located on the side of the compressor for collection of water. This should be drained periodically.

b. Aftercoolers

The discharge of the compressors is piped to four pipeline shell-and-tube type aftercoolers. These units are piped for countercurrent flow with compressed air on the tube side and cooling water on the shell side.

The aftercoolers discharge into moisture separators. These separators reduce the air velocity and reverse the air flow, thus separating free moisture and oil. These liquids are trapped and isolated in a bottom reservior. They are automatically removed from this reservior via a condensate trap installation.

c. Driers

The air from the separators flows to two sets of air driers which were manufactured by the C. M. Kemp Manufacturing Company. The number one drier consists of two towers connected in parallel. Each tower contains 335 pounds of silica gel and has a capacity of 500 SCFM of air. The unit is

fully automatic and electrically reactivated. The reversal cycle is 4 hours; the heating period for reactivation is 3 1/2 hours. The number two unit consists of two towers connected in parallel. Each tower contains 170 pounds of silica gel and has a capacity of 300 SCFM of air. This unit is steam reactivated and fully automatic. The reversal cycle is 6 hours; the heating period for reactivation is 4 hours.

d. Distribution

The air from the air driers is piped to a receiver tank which is located just outside the south side of the compressor building. This receiver is designed to smooth out surges and pulsations in the air supply and to provide a reservoir of air for periods of peak demand.

From the instrument air receiver, the air flows directly into the instrument air distribution header.

3. Steam Supply: The 12" steam line which comes across the road and into Area "B" brings 400 psig steam to the plant. An 8" line leaves the 12" main header and enters the EDC plant. The steam passes through a pressure reducing station and is metered. Here the pressure is reduced to 175 psig by a self-contained control valve before going to the individual vessels where it is controlled to meet the specific pressure or flow needs.

4. Electrical System:

a. General

Power for the organics area is presently being supplied by two 13.8 KV feeders. Feeder H-138 fed from power house bus No. 4 feeds directly into the EDC-Tri Ethane 13.8 KV bus. Feeder H-104 fed from power house but No. 1 feeds the EC-HCl-VC 13.8 KV bus. Feeder H-104 can feed the EDC-Tri Ethane Area through tie switches and breakers. The feeder is switched by oil circuit breaker No. 138 which is controlled only at the Powerhouse switchboard. After following an underground and overhead route, the 13.8 KV feeder is terminated at the interrupter switch located within the south cubicle of 13.8 KV metalclad switchgear in the Control Building. The door on this cubicle cannot be opened unless breaker No. 138 is open and in the lowered position thereby releasing the key which opens the interrupter switch cubicle door. Closing this interrupter switch connects the 13.8 KV feeder to the bus contained within the metalclad switchgear. The north cubicle of 13.8 KV switchgear contains a fused interrupter switch, the load side of which is connected to the primary of the 750 KVA transformer. Voltage is stepped down from 13.8 KV to 480 volts in the 750 KVA transformer. The transformer secondary is connected to the terminals of an air circuit breaker in the 480 volt switchgear which in turn supplies the 480 volt bus located within this switchgear. Three (3) circuit breakers connected to this bus supply power to two motor control centers which contain motor starters and circuit breakers for control of individual loads.

b. 13.8 KV Metalclad Switchgear

Unit No. 1 contains a manually operated 3-phase load interrupter switch rated 1200 amperes continuous and interrupting. Also included are fuses which are sized to protect the 750 KVA transformer. The door to this cubicle cannot be opened unless the switch is in the open position. In passing, it should be pointed out that any three phase fused switch or circuit presents the possibility of single-phasing due to only one fuse blowing thus leaving two lines connected to the load. The motors are protected by the motor starters for this condition. If a single fuse blows and the remaining two fuses happen to be on the lines which supply lighting, then lighting would be normal but motors would be dropping off the line. The simplest check for this situation is to determine if current is flowing in all three phases of the transformer secondary by reading the ammeter located on the 480 volt switchgear. Current in only two phases would indicate a single blown fuse, and the transformer should be taken off the line.

c. 750 KVA transformer

The transformer supplying power for the operating equipment is rated 750 KVA, 13,800/480 volts, and is a sealed dry-type transformer. The unit is filled with dry nitrogen which serves as an insulating and cooling medium. Lightning arrestors provide surge voltage protection. The transformer is equipped with alarm and indicating devices for pressure and temperature. Pressure should not exceed 10 1/2 psi under any condition since permanent tank distortion may result from higher pressures. A red alarm on the instrument control panel indicates excessive pressure which can be corrected only by reducing load on the transformer. An amber alarm indicates excessive vacuum, but it is very unlikely that this situation will occur. Normal pressure for the transformer when de-energized is between 1 and 1.5 psi, the pressure increasing with load to approximately 7 psi at rated load. Although it is not desirable to do so, the transformer can be operated with no pressure.

A hottest spot indicator-relay is provided having alarm contacts which operate at 210°C and 225°C. An amber alarm will indicate that the transformer has reached 210°C. This is the rated temperature. The transformer should not be loaded beyond this point. A second alarm at 225°C provides additional protection. Lower the temperature by decreasing the load.

d. 480 Volt Switchgear

The 480 volt switchgear is metal enclosed and equipped with draw-out type manual air circuit breakers. The unit is equipped with a 1200 amp. main breaker which is connected to the transformer secondary terminals. When closed this circuit breaker energizes the bus within the unit. Power is distributed by three 600 amp. draw-out type manual air circuit breakers to three motor control centers which are located in the Control Building and one in the Air Compressor Building. Metering for 480 volt power is provided in this unit consisting of an ammeter with selector switch, a voltmeter with selector switch, and a watthour meter. A ground detector device is included to indicate grounds on the 480 volt system. A single phase to ground condition will sound an alarm at the control board. This situation should be remedied as soon as reasonably possible.

e. Motor Control Centers

Three motor control centers are installed, one in the Air Compressor Building and two in the Control Building. Process pump motors on dual or spared pump installations are arranged so that the two motors involved are fed from different motor control centers. All important motors are provided with shutdown alarms which annunciate at the control board. Each motor starter is equipped with a control transformer which reduces the voltage at the pushbutton stations to 110 volts. Three over-load relays are provided to improve motor overload protection.

f. Instrument Power and Lighting

Instrument power and emergency lighting are supplied by a 10 KVA, 480/120 volt, single phase transformer which receives power from an automatic transfer panel. In case of power failure on the bus normally feeding the 10 KVA transformer, relays will automatically switch to a feeder which originates at an emergency power sub-station at the Per-Tri plant.

Normal lighting is supplied by the EDC No. 1 lighting transformer. A lighting panel is provided on the north wall of the Control Building starter room.

Yard lighting is accomplished by the use of mercury vapor floodlights. These lights are controlled by circuit breaker (Circuit No. 6623) which must be operated manually.

Lighting panels have been provided on the north wall of the starter room. The east panel contains circuits for general lighting while the west panel contains emergency and critical lighting. The emergency panel is fed from a distribution panel on the south wall of the starter roon, which is connected to the 10 KVA transformer previously mentioned.

g. Installation Methods and Equipment Classification

The characteristics of EDC are such that it is classified in Group D, Class I by the National Electrical Code. Ethylene, however, is classified in Group C, Class I. The method and materials of installation are those recommended by Factory Insurance Association. In general, the installation is Class I, Group D, Division 2. Motors are TEFC, lighting is vapor-tight, and all arcing devices are explosion proof with seal-offs. Since the Control Building is pressurized and removed from the process area, general purpose equipment is used with the exception of the laboratory which requires a Class I, Group D, Division 1 installation. Seal-offs are provided in all conduits leaving the Control Building on the process side as required for conduits entering a hazardous area from a non-hazardous area. Seals are also provided for conduits leaving the Control Building but not entering a hazardous area. However, this is to prevent flow of air due to pressurization of the Control Building.

h. Relamping

Although vapor-tight fixtures are used in the process area, it is felt that some precaution should be used when relamping. The procedure will be to first determine 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.

i. Grounding

Grounding in the EDC plant has been given special attention due to problems peculiar to the handling of hydrocarbons and the location of the process area. 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 followed up to see that the ground has been connected.

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 system. All vessels are grounded at two points. The result is a system of lines, tanks, and vessels operating at ground potential. This will not prevent the generation of static charges but should provide adequate leakage to ground to prevent the accumulation of dangerous charges.

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 still approaches this condition. The grounding system on the still should eliminate personal hazard and to some degree, act as a shield to equipment south of the still.

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

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

D. Instrumentation

1. General: One of the major requirements in a plant like the EDC plant is smooth, uninterrupted control of the plant streams in order to obtain specification grade product. For instance, a distillation column can not be operated with varying flows. A column like the EDC still can take hours to level out after an upset and rapid changes in operating conditions can cause poor quality product during the upset period. To obtain the desired type of control, the EDC plant has been equipped with automatic instruments whenever possible.

Even though a thorough job of instrumentation has been done on the EDC plant, certain items must be understood to get full and efficient use of the instruments. The instruments are the operators tools and how he uses them determines how efficiently the plant will operate and the quality of the product.

All important control points in the EDC plant are transmitted to the control room where they are indicated or recorded. The first sign of trouble within the plant will generally be from the instruments. These should be continually checked for indications of abnormalities. Most of the recorders or indicators will be read and the data recorded on the log sheets.

The instruments serve also as a 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 the protection of the equipment.

2. 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 operations, the warning lights will show green. If a high flow, pressure, level, etc. occurs, the horn will blow and the warning light will flash red. If a low flow, pressure or 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 remain green. If for any reason the pump stops the horn will blow and an amber light will flash.

The horn will continue to blow and the light flash until an acknowledgement is made. This is done by pressing the acknowledgement button located on 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 on the nature of the trouble. When the correction is made the light will change back to green. A list of the alarm points and their settings are given in Table 4.2, page 4-31.

3. Checking Alarm: A failure in the alarm system could result is 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 acknowledge button and turn it to the high alarm. Check all warning lights to see that 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. Then turn the button to low alarm and check the warning lights again. Make this check once per shift. Safe operation depends upon a properly functioning alarm system.

4. Instrument Operation: The recorder-controller instruments are provided with manual and automatic operation. Under normal conditions, these will be operated on automatic control, but during start-up or shut-down, or possibly in the case of trouble, the manual control can be used. When on manual control, the valve can be operated from the panel board by regulating the output to the control valve. The output will remain at the valve set by the manual 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 and closing the automatic valve.

The automatic control valves go from full open to full closed (or vice versa in the case of an air pressure opening valve) at an output pressure variation of 3 to 15 psig.

On the pneumatic controller the output pressure is indicated on the scale at the top. The approximate opening of the valve is indicated on 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 the valve action. At proper control, the set point and the indicator pen will remain opposite each other. The opening and closing of the automatic valve to maintain this condition will be shown by the variation in output air pressure.

5. Bumpless Transfer: The controllers can be readily switched from manual to automatic, or automatic to manual control, but care must be exercised when doing this. If the controllers are not switched correctly, a snap action of the automatic valve will occur. This will upset the system as well as be hard on the instruments.

The following procedure should be followed when making the transfer on pneumatic controllers:

a. Manual to Automatic

(1) Set the transfer lever to 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.

b. Automatic to Manual

(1) Note the output pressure and then set the transfer lever to 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.

6. 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 will be set at the desired control point. The instrument will then 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 and cause a severe enough change in the process variable to upset the system. In some cases the rapid change may not have any adverse affects, but in others this could be a definite upset. How rapid a change can be made depends strictly upon the operation that is being performed. For example, the feed to the stripper can withstand considerable variation without particularly bothering the rest of the system. But, the feed to the distillation column should be changed step-wise and the set point gradually worked up to the desired valve so that the rest of the column controls have sufficient time to regulate to the change. This is also true for changes in ethylene and chlorine feed to the reactor.

7. Pressure Gauges with Protector Bowls: Some pressure gauges in the EDC plant have been equipped with protector bowls. These bowls are liquid-filled and the liquid is separated from the process stream with a flexible diaphragm. The purpose of these bowls is to protect the gauge from corrosion by the process streams and still give an accurate pressure indication. If one of these gauges fails to function for any reason, do not replace just the gauge. The gauge and protector bowl must be removed as a unit and a new unit installed since they are factory sealed full of silicone oil.

8. Instrument Trouble Shooting: Instrumentation can and does contribute its share of problems, especially during plant start-ups. Unless instruments are given special care when being checked out and put into service, process operations may be seriously handicapped.

During initial operations, plant personnel should become familiar with the characteristics of the instruments in the plant. Operators should learn to recognize the normal state of their instruments and check them by supplementary observation of physical conditions in the process. Comparing centralized recorder-controller instrument readings with spot observations will show whether all is well.

The operator should neither trust or distrust his instruments. When something appears to be out of line, do not draw conclusions without having supporting data. Watch trends in process variables and thus be better prepared to recognize a true deviation in the process.

Instruments should be calibrated against standards if there are questions. This is particularly important when instrumentation is vulnerable to straying or where process upsets have disrupted instrument accuracy.

In starting up a plant, it is normal practice to use manual controls until the flows and operating conditions are properly leveled out. There is often a tendency to attempt automatic control before it has been established that suitable operation of the process is possible. On the other hand, delaying the switch to automatic control can also cause problems. Close cooperation between the operating personnel should be maintained in switching from manual to automatic.

Some typical checks for instrument troubleshooting during start-up are summarized in Table 4.3. Operating personnel can significantly reduce downtime caused by instrument problems, if they follow such a check list.

E. Pressurizing and air conditioning system for EDC Control Building

1. General: The EDC Control Building, with the exception of the Control Laboratory, is completely air conditioned and pressurized by equipment located on the second floor in the northwest corner. Air conditioning was required since the building had to be pressurized so that other than explosion-proof equipment could be installed in the building. The Control Laboratory is not air conditioned since there cannot be any open connection between the laboratory and the rest of the building. The equipment is designed to maintain a constant temperature and to prevent the relative humidity from going above 70% while keeping the pressure in the building at approximately 1/4" water. Refer to Figure 4.2 , page 4-34 . Diagrams of both the pneumatic and electrical controls are shown.

IN CASE OF FAILURE OF THE PRESSURIZING AND/OR AIR CONDITIONING SYSTEM NOTIFY THE SUPERVISOR AT ONCE. DO NOT OPEN DOORS AND WINDOWS FOR VENTILATION AS THE POSSIBLE DRAWING IN OF GASES COULD RESULT IN AN EXPLOSION OR FIRE.

2. Pressurizing System: The pressurizing system consists of an air intake on the roof which connects through a suction duct to an Ilg BC-135 Blower. The blower discharges air through a motorized damper into a plenum which contains filters and directs the air toward the suction of the packaged air conditioning unit. This blower is rated at 2520 cfm or 1580 cfm at 1/4" water depending on the speed at which it is set.

The blower motor is 3/4 hp, 440 volts with the start-stop controls located at the motor control center. An emergency stop button is located in the Control Room on the first floor and a warning light is located on the alarm panel of the main panel board to give indication of failure of operation of the motor.

The pressurizing controls are Minneapolis-Honeywell and any maintenance on them is to be done by the Minneapolis-Honeywell Service Mechanic ONLY. In case the controls malfunction, call the supervisor. Operators should not attempt to correct the difficulty.

The pressurizing controls operate the damper on the blower discharge to allow only sufficient air to enter the building to balance the losses and maintain a 1/4" water pressure within the building.

Personnel working in the building will notice an outrush of air each time a door is opened and if this is not noticed the pressurizing system should be checked. All windows should be kept shut and doors should be open only for passage.

A solenoid valve on the damper control is wired into the blower motor control circuit so that the damper will remain closed when the blower is off. This will prevent the pulling in of outside air at ground level through the stack effect of the outside air intake on the roof.

Operation of Pressurizing System:

To Start

a. Start fan at motor control center.
b. Check blower outlet in front of unit to be sure unit is operating.

To Stop

Stop fan at motor control center or at emergency stop on first floor.

Controls

Static pressure regulator should be set at 1/4" water by Minneapolis-Honeywell Service Mechanic.

Maintenance

a. Fan and Blower: Service Department. As required for PM.
b. Pneumatic Controls: Minneapolis-Honeywell. Four month check.
c. Filters: Air Conduit. Monthly clean or replace.

Comments

a. Keep all windows shut and open doors only for passage.
b. Failure of blower motor is indicated on alarm panel on panel board.

3. Air Conditioning System: The central air conditioning system is composed of an 8-ton Chrysler Airtemp unit which has its suction at the unit on the second floor and discharges through a series of ductwork to the various rooms on the first floor. Air return from the first floor is through grills in the false ceiling and the second floor. The unit uses well water in its condenser.

The package unit is equipped with electric heating coils in addition to the cooling equipment.

The Chrysler Airtemp package unit is covered in the service contract with Air Conduit Company and their mechanics only should work on the unit. They will check the unit and clean or change filters once a month. In case of malfunction of the unit, contact the supervisor.

The air conditioning control system is composed of two parts; one which controls temperature and one which controls the relative humidity. Temperature control is maintained by the action of the thermostat located on the center wall on the ground floor. This thermostat resets the control point of the submaster Grad-U-Stat located in the supply air duct which energizes the cooling circuit through a pneumatic-electric relay or energizes four stages of electric heat in sequence through pneumatic-electric relays to reset the supply air temperature. The humidity is controlled by the humidistat, located adjacent to the thermostat, which energizes the cooling circuit through a pneumatic-electric relay. The cooling of the air removes moisture on the coils and if cold air is not required the submaster Grad-U-Stat will energize the heating system to warm the air.

Operation of Air conditioning system

To Start

a. Turn well water valve on to unit.
b. Energize circuit 6724 at motor control center.
c. Energize Circuits 6724-1, 6724-2 and control transformer at auxiliary control panel adjacent unit. Unit will not operate with control transformer off.
d. Start unit by turning rotary switch on front of unit to "Cooling" position.
e. Turn thermostat rotary switch on front of unit clockwise to highest position. It should be left in this position at all times.

To Stop

a. Stop unit by turning rotary switch on front of unit to "OFF" position.
b. De-energize circuits at auxiliary control panel or motor control center as required.

Controls

a. Set thermostat on first floor at 78°F. (Plus or minus 2° depending on exterior weather conditions.)
b. Set humidistat on first floor at 70% R.H. Do not set at lower values.

Maintenance

a. Electrical and Plumbing: Service Department. As required.
b. Package Unit: Air Conduit Company. Monthly check.
c. Pneumatic Controls: Minneapolis-Honeywell. Four month check.

Comment

This unit does not have a low pressure water cutoff and should be shut down whenever the well water is shut off. If not, unit will overload and shut down anyway but damage could be done.

TABLE 4.1
SAFETY RELIEF DEVICES

Equipment	Type Relief	Size Inches	Relief Setting psig
Cl_2 Vaporizers	Relief Valves		225
Small Cl_2 Surge Drum	Rupture Disc	3	125
	Relief Valve	3	115
Large Cl_2 Surge Drum	Rupture Disc	3	125
	Relief Valve	3	115
No. 1 Dump Tank	Rupture Disc	8	50
No. 2 Dump Tank	Rupture Disc	6	50
No. 1 Reactor	Rupture Disc	10	50
No. 2 Reactor	Rupture Disc	6	50
Clean-Up Kettle	Rupture Disc	3	50
Reactor Gas Separator	Rupture Disc	6	50
Stripper	Rupture Disc	4	50
Stripper Gas Separator	Rupture Disc	4	50
Crude Tank	Rupture Disc	6	50
Still	Rupture Disc	6	50
Still Reflux Drum	Rupture Disc	4	50
Flasher	Rupture Disc	2	50
Flasher Accumulator	Rupture Disc	2	50

TABLE 4.2

ALARM SETTINGS

Service	Variable	Setting	
		High	Low
Chlorine	Liquid Pressure	150 psig	110 psig
Chlorine	Feed Pressure	40 psig	30 psig
Ethylene	165# Pressure	175 psig	155 psig
Ethylene	Feed Pressure	50 psig	40 psig
Reactor #1	Level	26 ft.	22 ft.
Reactor #2	Level	20 ft.	16 ft.
Reactor	Pressure	8 psig	---
Reactor Gas Separator	Level	4.5 ft.	2 ft.
Stripper Gas Separator	Level	3 ft.	1 ft.
Crude Tank	Level	7 ft.	2 ft.
Still Reflux Drum	Level	3 ft.	1 ft.
Storage Tanks	Level	25'-6"	4'-6"
Cooling Tower	pH	---	6
Steam to Stripper	Flow	---	1,225#/Hr.

TABLE 4.3

TIPS ON INSTRUMENT TROUBLESHOOTING

I. Recorders and Recorder-Controllers

Trouble (A): Pen Dead at Zero

Cause: (1) Check isolating valves at process impulse line; see that equalizing valve is shut. (2) Look for plugged impulse line. (3) In case of pneumatic transmitter, check air supply to transmitter. (4) Look for broken or loose lines. (5) Primary sensory device not installed. (6) Be sure process step is actually in operation.

Trouble (B): Recorders with two or more pens record at the same point on the chart, but should be at different points.

Cause: (1) Pens may not pass owing to mechanical interference. (2) Make sure instrument leads are clear and properly connected.

Trouble (C): Chart not moving

Cause: (1) Chart loose on hub. (2) Chart loose on chart rewind roll. (3) Clock not running; power not on drive.

Trouble (D): Chart readings apparently not correct or not consistent with local gauges.

Cause: (1) Trouble in gauge movement. (2) Gauge may not have been read properly. (3) Check agreement of chart with transmitter range. (4) Check agreement of output pressure from transmitter with pen.

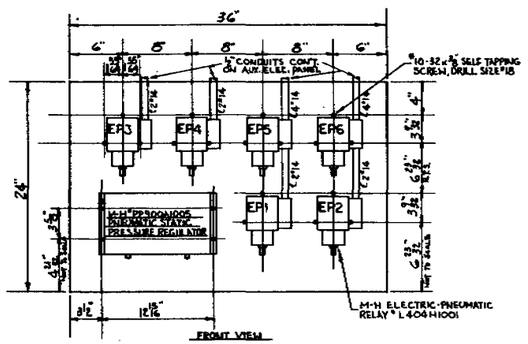
II. Control Valves

Trouble (A): Valves do not respond to output change.

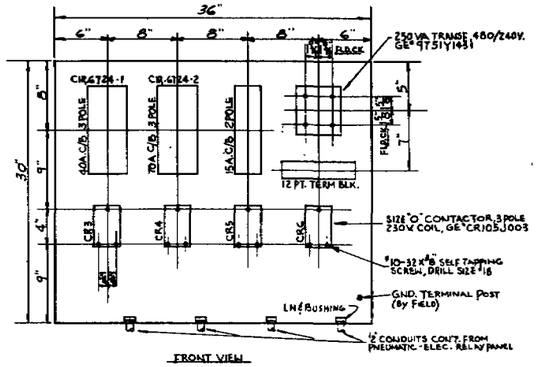
Cause: (1) If valve has a valve positioner, check its air supply. (2) Check for leaking air lines. (3) Valve stem may be stuck.

Trouble (B): Valve changes position, but brings about no change in process flow.

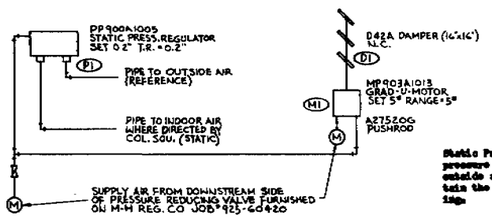
Cause: (1) Valve may be plugged. (2) Block valves in process line may be closed.



PNEUMATIC-ELECTRIC RELAY PANEL



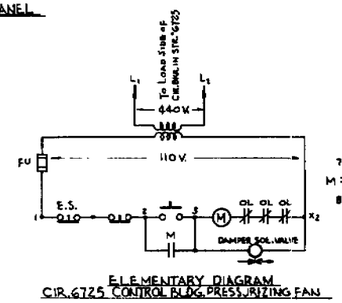
AUXILIARY ELECTRICAL CONTROL PANEL



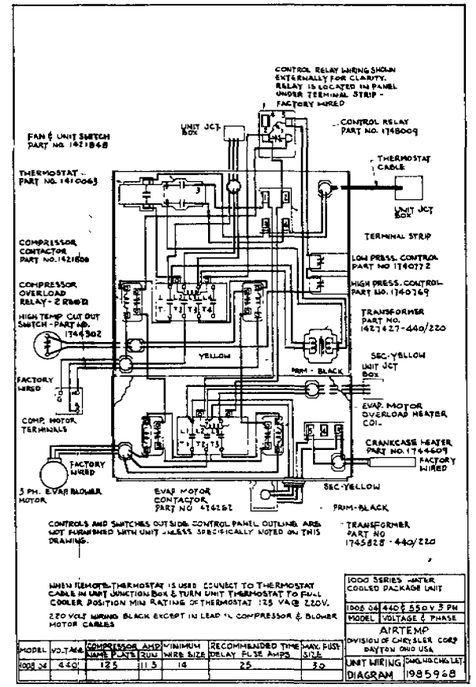
STATIC PRESSURE CONTROL

SEQUENCE OF OPERATION

Static Pressure Regulator P1 senses outside air pressure and building pressure and modulates outside air damper D1 through Motor M1 to maintain the desired static pressure in the building.

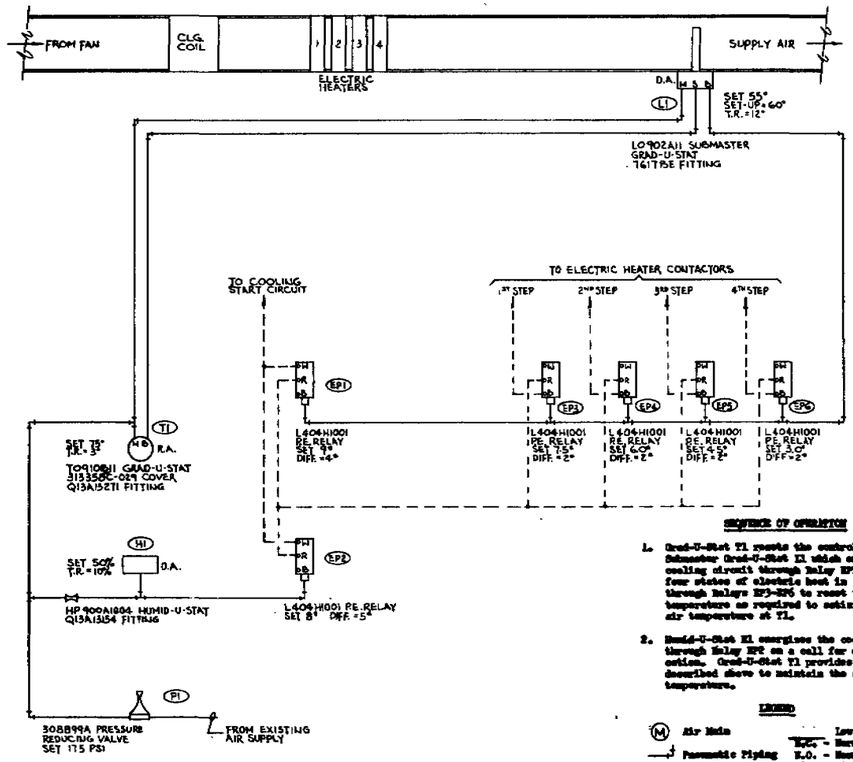


ELEMENTARY DIAGRAM
CIR. 6725 CONTROL BLDG. PRESS. /RIZING FAN



MODEL NO.	VOLTAGE	COMPRESSOR AMP NAME PLATE	RUN	MINIMUM WIRE SIZE	RECOMMENDED TIME DELAY FUSE AMPS	MAX. FUSE SIZE
1008.04	440	12.5	11.5	14	25	30

VENDORS WIRING DIAGRAM - 8 TON AIRTEMP UNIT
NOTE: WIRING DIAGRAM SHOWN FOR UNIT AS RECEIVED FROM VENDOR & BEFORE THE ADDITION OF HEATING & HUMIDITY CONTROLS



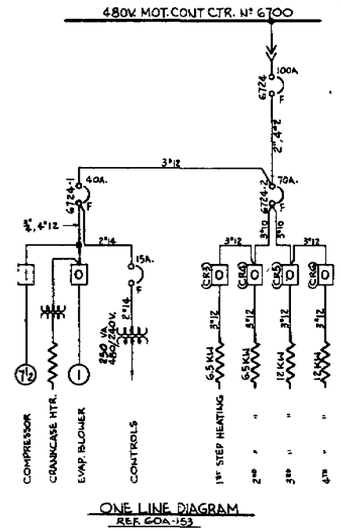
AIR CONDITIONING UNIT CONTROL

SEQUENCE OF OPERATION

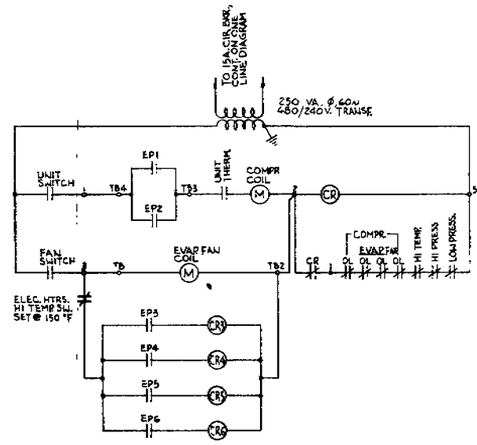
1. Grad-U-Stat T1 reacts the control point of Submaster Grad-U-Stat L1 which energizes the cooling circuit through Relay EP1 or energizes four stages of electric heat in sequence through Relays EP3-EP6 to reset the supply air temperature as required to satisfy the space air temperature at T1.
2. Humid-U-Stat H1 energizes the cooling circuit through Relay EP2 on a call for de-humidification. Grad-U-Stat T1 provides reheat as described above to maintain the space air temperature.

LEGEND

M Air Main	Low Voltage
Pneumatic Piping	N.C. - Normally Closed
Air Gap	N.O. - Normally Open
Line Voltage	D.A. - Direct Acting
	R.A. - Reverse Acting
	T.R. - Throttling Range



ONE LINE DIAGRAM
REF. 60A-153



ELEMENTARY DIAGRAM
CIR. G724-1/2 8 TON AIRTEMP UNIT

REF DWG.
60A-100 EDC PLANT MASTER DWG. LIST
60A-328 CONTROL BLDG. AIR COND., HEATING, & PRESS. EQUIP

NOTES
1. REMOVE CONTROL TRANSF. PART № 1427427 FURNISHED BY VENDOR AS A PART OF THE ORIGINAL CONTROLS OF THE 8 TON AIRTEMP UNIT. REVISE CONTROL WIRING PER ELEMENTARY DIAGRAM

COLUMBIA-SOUTHERN CHEMICAL CORP.
SUBSIDIARY OF PITTSBURGH PLATE GLASS CO.
LAKE CHARLES, LOUISIANA

DEPT. NO. 60A
DEPT. TITLE EDC PLANT
CONTROL BLDG. AIR CONDITIONING UNIT
PNEUMATIC & ELECTRICAL CONTROL DIAGRAMS

DRAWN BY [illegible] DATE 5-17-60 SCALE
CHECKED BY [illegible] DATE 5-20-60 CHARGE P-341
APPROVED BY [illegible] DATE 5-20-60 SUB. 602.632

BILL OF MATERIAL NO. 60A-328 DWG. NO. 60A-329-1

REVISIONS

ISSUED	5-22-60
FOR	Permit
BULLETIN NO.	6491
⚠ ADDED DAMPER SOL VALVE TO CIR. 6725 B/M 60A-164 2 CHARGE P-341-682	6-22-60
ITEM	6-22-60
F.O.R.	Permit
BULLETIN NO.	6577

PAGE 4-35
FIGURE 4.5

d. Pressurize the chlorine feed line as per chlorine system start-up procedure.

e. Bring the ethylene flow up to the desired flow rate (refer to Figure 14.1, page 14-2) while simultaneously following stepwise with the chlorine flow increases.

f. Bring the chlorine flow up to the desired flow rate; refer to Figure 14.1, page 14-2.

g. The reactor level will rise until the reactor temperature reaches approximately 185° to 190°F and the vapors start to distill over at the top.

h. The reactor gas separator level will now begin to rise as EDC is made in the reactor.

i. At the sign of the level increase in the reactor gas separator, start the reactor pump and put the reactor level control in service.

j. The stripper should be up to temperature and about lined out on total reflux by this time with the top temperature close to 185° to 190°F. Start the feed from the reactor gas separator to the stripper.

k. As soon as the proper operating level is obtained in the stripper gas separator, stop recirculating EDC from the crude tank back to the stripper.

l. Set the stripper reboiler steam flow to give the desired reflux ratio. (One pound of 175# steam will vaporize approximately 6.3 pounds of EDC.)

m. When the desired level is obtained in the crude tank, start the still feed at a flow rate about equivalent to the stripper feed rate or production rate. The still feed rate is adjusted by the operator to hold a constant level in the crude tank.

n. The level will begin to rise in the still reflux drum. When the drum level reaches the half-way mark open the product line to the product cooler and on to storage.

o. The EDC still bottoms can be pumped to storage periodically.

B. Detailed Equipment Start-up

1. Reboiler Start-up and Operation: Three thermosiphon reboilers are used in the EDC plant; one is on the HCl stripper, two are on the distillation column. The principle of the thermosiphon reboiler is to obtain a rapid circulation through the exchanger tubes by vaporizing 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. The vaporized material is replaced by 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 (thermosiphon) through the reboiler, thus eliminating any need for pumps. The rapid circulation rate through the exchanger accomplishes two purposes; first, a high rate of heat transfer to the liquid is effected due to the high thermosiphon liquid velocity obtained; secondly, this high liquid velocity makes the formation of scale or tars on the exchanger walls more difficult and thereby either eliminates the need for cleaning the exchanger walls or greatly extends the period of operation between cleanings over what would be experienced in a static kettle-type reboiler.

Three very important items must be remembered when starting up and operating a thermosiphon 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 the tars on to the tube, decomposition of the material in the reboiler, or even formation of more tars which can plug the reboiler tubes. The liquid level in the column should always be operated above the top tube sheet of the reboiler to provide for proper circulation rates.

Secondly, when starting up a cold reboiler, drain all accumulated steam condensate out of the shell of the exchanger and start the steam feed slowly. Gradually increase the steam until the reboiler comes up to temperature. Leave the steam condensate valve at the bottom of the reboiler open a slight amount and purge wet steam until the tubes have a chance to warm up. This procedure prevents bumping which occurs when live steam contacts cold condensate and it also prevents slugging of the liquid phase in the reboiler tubes. Either of these conditions can damage a reboiler if they become violent enough.

Thirdly, never run a reboiler with a high level, especially when the liquid in the reboiler blocks or partially blocks the vapor outlet of the reboiler. This will stop the thermosiphon and drastically reduce the heat transfer. If this happens, the still must be shut down, the reboiler level lowered to the proper level and the still started up again to re-establish circulation.

If a reboiler is failing to supply the design heat flux, and fouling is not involved, check the steam supply and/or the condensate trap. The trap may be inoperable and cause water to back up into the reboiler. When steam is at the proper pressure and the condensate trap is working, but there is still no heat transfer, chances are the tubes are fouled. In the case of the EDC still, there are two reboilers. One is a spare. The dirty exchanger should be cleaned up so as to be ready for service as soon as possible.

2. Start-up and Operation of Process Pumps: All process pumps in the EDC plant are centrifugal pumps with external mechanical seals. The mechanical seals by their nature, present some items which should be noted when starting up or operating these pumps.

The seal rings for these seals are made of ceramic. For this reason, the seal should be protected from mechanical or thermal shock or the ceramic ring could crack. Be careful of an extreme temperature shock such as might occur if water was sprayed on a hot seal while washing down the area. Mechanical shock to either a running or an out of service seal could be damaging.

The seals are equipped with a back flush line which comes from the discharge of the pump. The purpose of this line is to provide a liquid flush past the face of the seal to keep foreign matter from collecting under it and damaging the surface. If the flow through this line is at a high enough pressure, though, it can actually cause the seal to leak. For this reason the pumps should always be operated with the valve in the seal flush line only about 1/2 open. If a seal starts to leak, close down on this valve a little more. If the leak is caused by excessive seal fluid pressure, restricting the valve should stop the leak. If this does not affect the leak, then it is caused by a defective seal.

The correct procedure for starting up a centrifugal pump is to open the suction line and start the pump with the discharge valve closed. When the pump is running, slowly open the discharge valve to keep from slugging liquid through the piping. When starting a pump against a shut-off valve the discharge pressure would be great enough to cause the mechanical seal to leak if the back-flush line were open too wide. For this reason, always have the flush valve almost closed when starting the pump. After the pump is running with an open discharge, open the flush valve about half way.

A centrifugal pump can run with a closed discharge valve for a short period of time with no harm. But, if it is allowed to operate this way for an extended length of time, the pump will eventually heat up sufficiently to vaporize the ethylene dichloride. This creates gas bubbles at the pump suction and essentially allows the pump to run dry or cavitate which can damage the seal. A centrifugal pump should never be operated dry (no liquid in the lines). Since there is no fluid to lubricate the seal, it can be seriously damaged.

3. Utilities:

a. Electrical System

To provide 3 phase, 440 volt and 1 phase 115/230 volt power for the No. 1 EDC plant, the switching sequence is as follows: (Assuming that all breakers and switches are open and clear of hold tags, and all equipment is in operating condition.)

(1) Call the powerhouse switchboard operator and request him to close the 13.8 KV OCB No. H-138.

(2) Close the manually operated 13.8 KV disconnect No. 0-138 after the powerhouse acknowledges that OCB H-138 is closed.

(3) Close manually operated 13.8 KV fused disconnect No. 0-301. The 750 KVA 13.8 KV/440 volt transformer is now energized.

(4) Check for 3 phase, 440 volt power at the 440 volt metalclad switchgear. All three phases should read the same voltage; if not, it is possible that a 13.8 KV fuse in the switch cubicle No. 0-301 is blown or there may be trouble in the volt meter circuit. Call the service department for an electrician to clear the problem before proceeding further. Should the voltage check properly, you may proceed.

(5) Check the transformer pressure. The gauge should indicate between 1 and 7 psig. The reading will vary with load but should never exceed 10 1/2 psig.

(6) Check the transformer for proper temperature. The temperature should not exceed 210°C.

(7) Close the 440 v. A.C.B. Note: Be alert at this time for a ground alarm indication. If ground conditions are indicated, log the last switch which operated before the alarm sounded, silence the alarm and proceed with switching. When switching is complete, notify the service department as soon as possible of the ground condition.

(8) Close the metalclad 440 v. A.C.B.'s Numbers 6500, 6600 and 6700. Voltage is now available at the motor control center buses. Individual units of equipment may now be started as desired by closing the starter breaker and the start pushbutton; usually located near the drive motor.

(9) After all switching is complete and the station is on the line, check the volt and ampere meters to determine if the system is normal.

b. Instrument Air System

The following procedure should be followed when starting up the instrument air system.

(1) Line up system piping. Be sure the 1/2" air pressure impulse line is open to the compressor. Drain the receiver tank. Be sure to close the drain valve after having drained the receiver tank.

(2) Start the air driers. The driers will be automatically switched and reactivated.

(3) Open cooling water to the aftercoolers.

(4) Close the liquid discharge from the separators. At least for initial start-up, leave the separator discharges closed until a water level appears in the sight glass. When a liquid level appears in the sight glass open the separator discharges. Be sure the by-passes to the liquid traps are closed. The traps must operate with water in it. After each shut-down, the traps should normally retain water thus eliminating Step 4.

(5) Open cooling water to the compressor jacket.

(6) Unload the compressor by turning the transfer switch to the "OFF" position. Make sure there is an oil level in the bullseye.

(7) Start the compressor motor. Check the oil pressure (25-40 psig).

(8) Load the compressor by turning the transfer switch to the "ON" position. Watch to see that the compressor unloads at 100 psig and loads at 90 psig (approximately).

(9) Start dry air purge (14 SCFM) to the drier being re-activated.

Periodic checks that should be made on the compressor operation are listed below:

(1) Check oil level and pressure.

(2) Check the compressor belt tension by observation only.

(3) Check the flow of cooling water to the compressors and aftercoolers by feel for proper temperature (slightly warm).

(4) Check the separator liquid level. No level should appear normally.

(5) Check the drier operation. The drier being reactivated should be hot and have no pressure on it. The drier on stream will be cool and have pressure on it (100 psig).

(6) Check air temperature (80-100°F) and pressure (100 psig).

(7) Drain the moisture trap on the electro-pneumatic control system by opening the pet-cock momentarily.

c. Steam Supply

The following procedure should be followed when starting up the steam supply:

(1) If 400 psig steam is not on to the EDC plant open the main supply valve located at the south end of the plant. Open the by-pass around the steam trap at the 175 psig reducing station and allow steam to bleed through the line until it has warmed up.

(2) Check to see that all steam users in the plant are off.

(3) Crack the valve on the steam line to the vent stack to allow a bleed through the steam header.

(4) Open manual block valves on either side of the 175 psig reducing station.

(5) Slowly pressurize the steam header by raising the set point on the steam controller. Allow time for the line to warm up.

(6) Set the steam controller to give 175 psig steam. Use the gauge on the down-stream side of the control valve to adjust the pressure.

(7) Continue to bleed steam through the header until it is put in use.

(8) Set individual pressure reducer stations and flow control stations as required.

(9) Check all steam traps periodically to insure against wet steam.

d. Cooling Tower System

The following procedure should be used when starting up the cooling tower:

(1) Turn on well water make-up and fill the tower basin to the required level.

(2) Provide a full supply of treatment chemicals.

(3) Turn on the pH meter and check its operation.

(4) Start the tower fans.

(5) Open up cooling water lines through the reactor condenser, separator condenser, stripper condenser, still condenser, and product cooler.

(6) Start up the cooling tower pumps. Start the pumps with the discharge valve closed and open the discharge slowly.

(7) Start treatment chemical addition to the tower.

(8) Check for proper return flow and distribution at the top of the tower.

(9) Check fans and pumps for proper operation after they have been running for a few minutes.

Periodic checks that should be made on the cooling tower operation are listed below:

(1) Check the pH.

(2) Check the automatic pH meter by sampling the cooling water and testing with the laboratory pH meter.

(3) Check treatment chemical addition.

(4) Run test for treatment chemical concentrations and adjust chemical addition flows accordingly.

(5) Check operation of fans and pumps for vibration or hot bearings.

4. Refrigeration System: The following procedure should be followed in starting up the refrigeration system for the vent condenser:

a. Check oil level in crank case (1/2 sight glass).

b. Turn on cooling water to Freon condenser.

c. Turn on cooling water to compressor head.

d. Open Freon discharge valves through system. Open compressor suction valve only a slight amount.

e. Start the compressor and open the suction valve slowly; listen to make sure no liquid is being fed to the suction. Liquid in the compressor suction will cause knocking.

f. Open oil drain line about one turn.

Periodic checks that should be made on the refrigeration system operation are listed below:

a. Be sure liquid Freon is not being fed to the suction of the compressor.

b. Check the suction and discharge pressures.

c. Check the oil level.

d. Check the compressor cooling water discharge. Should be approximately 100°F.

e. Check the oil pressure (40 psi above suction pressure).

f. Check the water temperature on the Freon condenser.

Detailed instruction for maintaining this system are included in the Carrier operating manual.

5. Stack Seal: To put the stack seal on line, turn on the water flow to the top of the vent scrubber. Cooling tower blowdown and cooling water from the Freon condenser are used on the stack seal. Its flow is controlled manually and indicated on a rotometer.

6. Ethylene Supply System:

Start-up Procedure

a. Check the controller by-pass valves to be sure they are closed.

b. Open ethylene supply to the feed pressure control station.

c. Set pressure control valve set point at 45 psig.

d. Open by-pass around the pressure control valve and pressurize the ethylene line up to the flow control valve to 45 psig.

e. Ethylene is ready to be fed to the reactor. The remaining steps in the ethylene system start-up procedure are described under reactor start-up.

Check Points

a. Be sure by-pass valves are closed around the pressure and flow control valves.

b. Be sure all instrument impulse lines are open in the system.

7. Chlorine Supply System:

a. Liquid Chlorine

(1) Check all chlorine lines and be sure that all by-pass and block valves are closed.

(2) Line up the liquid chlorine from tank 7 or 8 in liquefaction to the last manual valve before the chlorine vaporizers in the EDC plant. If MC or HCl is already in operation, do not close valve on the liquid chlorine line to MC.

(3) Check the chlorine pressure and flow control valves for proper operation. Someone should observe the movements of these valves in the field. After checking, close the valves at the panel board.

(4) Start steam to the vaporizer slowly. Warm up the vaporizer by by-passing the steam trap until live steam starts passing through the trap by-pass. Check the steam pressure reducing station operation.

(5) Slowly open the liquid chlorine line to the vaporizer.

(6) After chlorine has had time to warm up, slowly pressurize the large chlorine surge drum to 35 psig using the pressure control valve by-pass. If cell gas is going to be used the small chlorine surge drum system will be pressurized to 45 psig.

(7) The remainder of the start-up described is discussed under reactor start-up.

(8) The other vaporizer can be brought up by slowly opening the vapor line, slowly turning on the steam to warm the vaporizer and then opening the liquid chlorine block valve.

(9) When the vaporizers are on line the MC chlorine feed can be switched to pass through the vaporizer and thus have its oxygen content reduced. Close the block valve in the liquid line supplying MC directly.

Check Points

(1) Be sure all by-pass valves are closed.

(2) Steam to vaporizer.

Note: Never open liquid chlorine to a cold vaporizer nor close the liquid and vapor lines off with steam on the vaporizer. Do not trap liquid chlorine between closed block valves.

b. Cell Gas

(1) Check out the cell gas line to see that chlorine is available to the EDC plant. Notify the liquefaction operator of intended use of cell gas.

(2) Open instrument impulse lines on orifice run that is to be used.

(3) Set the cell gas pressure control valve at 35 psig and slowly open the upstream and downstream block valves around the pressure control valve.

Note: Some liquid chlorine will always be used in the No. 1 EDC plant. Therefore the plant will be operating when the cell gas feed system is put in service.

8. Reactor: The following procedure should be used when starting up the reactor: (Refer to Figure 14.1, page 14.2 for ethylene and chlorine flow rates.)

a. The reactor level should be 24 or 18 feet respectively for the No. 1 and No. 2 reactors before start-up. Adjust the level with material from the reactor gas separator if necessary.

b. Check reactor for a vacuum and break it with nitrogen if one exists.

c. Be sure all instrument impulse lines are open.

d. Check to be sure cooling water is on the reactor and separator condensers. Have the refrigeration unit ready for start-up.

e. Line up the reactor vent piping through the stack seal and vent stack. Be sure sufficient scrubbing water is going to the stack seal and methane is available at the methane flow control valve.

f. Be sure the dump tank and rework kettles are isolated from the reactor system.

g. Open manual valves between the reactor level control valve and the reactor pump. Open the manual valve between the reactor pump and the reactor gas separator leaving only the pump discharge closed.

h. Open the manual valves in the chlorine and ethylene sparger headers.

i. The reactor is now ready for start-up. Open the block valves around the ethylene flow control valve and check to see if the valve leaks through. Open the block valves around the chlorine flow control valve and check it for leak through.

j. Start the ethylene flow first by opening up the ethylene flow control valve with remote manual control. Do not open valve too fast. Bring the ethylene flow rate stepwise up to the desired set point and switch to automatic control. Follow the increase in ethylene flow with stepwise increases in chlorine flow.

k. Start the chlorine flow by opening the chlorine flow control valve with remote manual control. Do not start chlorine flow until the ethylene flow has been started. Bring the flow rate up to the desired set point and switch to automatic control.

l. The reactor is now in operation except for the reflux flow. When the reactor temperature comes up and the level starts dropping, start refrigeration unit and adjust the reactor level controller set-point to the proper setting.

m. Shut off the nitrogen purge if it is in service.

n. Start the reactor reflux pump and begin recycle to the reactor.

o. The reactor is now on automatic control. As soon as the proper operating level is obtained in the reactor gas separator start feed from the separator to the stripper.

9. HCl Stripper: Refer to Figure 14.2, page 14-3 for the curves that will be used to determine the stripper reboiler steam flow (which determines the stripper reflux ratio).

Start-up Procedure

a. Check for cooling water flow to the stripper condenser and for proper steam pressure up to the reboiler steam flow control valve.

b. Check to be sure the reboiler is full of liquid. The reboiler should be filled to the stripper overflow take-off line. If the level is low pump EDC to the stripper from the reactor gas separator or stripper gas separator to obtain the proper level.

c. Open manual valves between the stripper gas separator and stripper reflux pump.

d. Open manual valves through the stripper reflux drier and around the stripper gas separator level control valve. Leave the stripper reflux pump discharge valve closed.

e. Open manual valves in crude tank recycle line and ready the still feed pump for operation.

f. Start the recycle flow from the crude tank back through the stripper feed line.

g. Turn on steam to the stripper reboiler. Start up the reboiler according to the start-up procedure previously discussed.

h. Bring steam flow up to the control point gradually. The column and a large mass of packing must be heated up, so it will take some time before the full steam flow can be utilized. The desired steam flow will depend upon the plant production rate and stripper reflux ratio.

i. When the stripper vapor line comes up to approximately 183^{o}F and a level rise occurs in the stripper gas separator, start the stripper reflux pump.

j. The stripper is now operating on recycle. The stripper should be operated in this manner until feed is available from the reactor gas separator.

k. When the proper operating level is reached in the reactor gas separator, start feed from the separator to the stripper. Once feed from the reactor gas separator is started the EDC recycle from the crude tank can be discontinued.

Check Points

a. Check the steam rate to the reboiler.

b. Check the stripper reflux pump for proper operation.

c. Check the reboiler level. Never start-up or operate with a low reboiler level.

d. Check the vent system. Make sure the non-condensables are venting and not accumulating and building up pressure on the system.

e. Check to see that enough cooling water is on each exchanger.

10. Distillation Column: Refer to Figure 14.3, page 14.4, for the graph from which the proper reflux rate can be selected.

Start-up Procedure

a. Make certain cooling water is on the still condenser and product cooler, and that steam pressure is available to the still reboiler steam valve.

b. Open the proper feed tray valve.

c. Open the manual valves on each side of the still reflux control valve and back through the reflux pump to the reflux drum. Leave the reflux pump discharge valve closed.

d. Open the manual valves in the still feed line between the crude tank and the still. Leave the discharge valve on the still feed pump closed.

e. Check the still reboiler level. If the bottoms withdrawal valve was closed when the column was last shut down, the level should be above the sight glass. Lower the level to the top of the sight glass. If the reboiler level is low it will have to be corrected with material from the crude tank.

f. Start Phopflex-L addition to the reboiler.

g. Turn on the steam to the still reboiler according to the reboiler start-up procedure. As the reboiler temperature starts rising the still pressure will come up. When a slight positive pressure is obtained, open the still vent line to bleed down my inerts. Close off the nitrogen pad line.

h. As the reboiler temperature comes up, the level will drop. If the level goes below normal operating level, add feed from the crude tank. Watch the reboiler level closely, as it can easily change during start-up.

i. Slowly raise the steam flow until it is approximately at the desired operating valve. The steam flow is automatically controlled by the temperature of the column bottom trays. On initial start-up the amount of reflux obtained can be used as a guide to the proper steam flow setting.

j. As the still vapor temperature comes up to 183°F and a level rise is indicated in the still reflux drum, start the still reflux flow. Begin the flow at a low rate and increase it to hold a constant level in the reflux drum. The block valves around the level control valve should be closed. Increase the reflux until the proper flow for a 1:1 reflux ratio is obtained. Base the reflux ratio on the desired still output. Regulate the reboiler steam flow to hold a constant level in the reflux drum at this rate.

k. Close the still vent line after the temperatures are established and open the nitrogen pad to the still condenser.

l. The still is now on total reflux with no feed withdrawal. When operating temperatures are essentially reached, put the steam controller on automatic control with the set-point at the temperature established by previous operations.

m. Start feed to the still from the crude tank. Slowly increase the feed until it is equivalent to the output from the stripper.

n. As level builds up in the reflux drum start product withdrawal.

o. Make bottoms withdrawals from the reboiler to hold a proper operating level.

Check Points

a. Make certain cooling water is on the condenser and cooler.

b. When the still is in operation the nitrogen pad system should be in service.

c. Check the reboiler level.

Trouble Shooting

There can be many operating difficulties associated with distillation columns, particularly when they enter service for the first time. Such problems include flooding, entrainment and similar occurrences that interfere with proper fractionation. How can the operator recognize and correct these situations? Temperatures, pressures, and product purity are the diagnostic tools for the difficulties described.

You must have vapor pressure-temperature data on the materials being fractionated when you are operating distillation columns. Pressures (read on process gages) can serve as a temporary guide to performance when key temperature control is inoperable or not dependable. Grabbing samples from various trays for check analyses is another conventional technique for determining performance.

Operators should check the temperature profile across the column to be certain that it is consistent with the anticipated pattern. Excessively high temperatures in upper parts of the column may indicate vapor bypassing, due to excessive velocities or inadequate liquid tray-loading. Insufficient liquid on trays can be caused by inadequate weir height or missing or leaky valves.

Note that inconsistent temperatures may not always indicate process upsets. Checking thermocouples or seeing that thermowells are properly installed can sometimes eliminate what at first appears to be a process problem. Be certain that thermocouple immersion liquids are provided where required and see that insulation is sufficient to stop heat leaks around thermocouples or thermometer bulbs.

Column flooding is most frequently confirmed by differential pressure readings. When much higher than normal Δp's prevail, particularly with fluctuation or surging, it is very likely that there is flooding. Typical techniques for "dumping" flooded columns are: withdrawing or curtailing feed, dropping reboil heat, or temporarily increasing pressure.

Usual cause of flooding is excessive liquid downflow at high vapor loading. Plugged trays can also cause this condition. Too cold a feed can also result in flooding and poor column operation.

11. Rework Kettle: The rework kettle will be operated only periodically.

Start-up Procedure

a. Close manual valves around the kettle so that it is isolated from all other process equipment.

b. Turn on steam to the kettle jacket. Set the steam pressure control valve at the desired pressure (5-10 psig).

c. Make certain the condensate trap is working. Bleed condensate and inerts from the steam jacket.

d. When the kettle temperature rises open the vapor line from the kettle to the reactor vapor space.

e. Using the dump tank pump, slowly add organic to the kettle.

f. The EDC and lights will vaporize and be condensed in the reactor condenser. This slight increase in the plant production rate will be handled without any problem by the automatic instruments.

g. When the kettle level drops and the vapor temperature from the kettle rises above 195°F drain the tars to drums for disposal. Close the vapor line before draining. Make use of the two sight glasses (one lighted) on top of the kettle to monitor the progress of each batch.

h. When the kettle has been drained it is ready for more feed. Be careful during the draining operation because painful burns could result if operating personnel get splashed with the hot tars. Avoid breathing the vapors from the hot tars.

12. Bottoms Purification: The following procedure should be used when starting up the flasher.

a. Check to make sure cooling water is on the flasher condenser and steam is available to the flasher reboiler steam valve.

b. Be sure the reboiler is full of liquid. Adjust the liquid if necessary.

c. Turn on steam to the flasher reboiler. Start up reboiler according to reboiler start-up procedure.

d. Using the N_2 jet pull a vacuum on the system.

e. As the reboiler temperature comes up, the level will drop. Maintain a constant level by opening the feed valve. Watch the liquid level closely as there is no recirculation flow.

f. Slowly raise the feed flow to the desired rate.

g. As the condensate tank fills the vacuum that was pulled on the system will decrease. The reboiler temperature will increase due to the increase in system pressure.

h. When the condensate tank is full pump its contents to the reactor dump tank for storage. A N_2 pad will be used to transfer the flasher product from the dump tanks to the Per-Tri plant.

Note: As the condensate tank is pumped down the vacuum will be re-established on the system. A loss of vacuum indicates a leak which should be located and repaired to prevent air mixing with the hot organic vapors in the flasher system.

i. The flasher must be drained periodically to remove the heavy impurities which concentrates in the flasher bottoms. After draining the flasher will be restarted. Phosplex-L should be added to the flasher reboiler after each draining.

Check Points

a. Make sure noncondensables are not accumulating and building up pressure in the system.

b. Reboiler level.

c. Check feed pump and feed flow rate periodically.

13. Pre-Start-up Procedures--Purging, Drying, Filling: For the initial start-up, the vessels will contain air and moisture. The plant must be dried and purged before operations can commence. The following precedure is given for these pre-start-up operations, and will be applicable for initial plant operations after a long-term shutdown.

a. After the contractors have finished and turned the various pieces of equipment over to Operations, the Maintenance Department will go into each piece of equipment. At this time the equipment will be inspected and cleaned if necessary to remove such things as residual sandblasting sand, tools, or other kinds of trash.

b. When Maintenance completes their work and "buttons-up" the plant, a 30 psig air or nitrogen pressure test will be made to be sure flanges and other connections are not leaking.

c. After pressure testing, the pressure will be released at the low points in the system. The flow of pressurizing gas will be maintained until all possible low points have been purged. This will remove trapped water from the lines and equipment.

d. The pressure will be bled from the system and a gas sweep will be started through the plant. Dry air (instrument air) or nitrogen should be used for the purge gas. Start at the front end of the plant and work toward the back end. The purge gas will be exhausted at various places in the plant to allow each piece of equipment to be thoroughly purged with dry gas. It is preferable if the purge gas is heated. The reboilers can be turned on to facilitate the drying operation.

e. The chlorine and ethylene gas flow controllers can be checked out at this time.

f. The dry gas sweep will continue until it is ascertained that the plant has been completely dried. To check the degree of dryness of the plant, dew points will be measured on the exit gas.

g. The plant will then be completely purged with nitrogen to remove all air.

h. Begin filling the process equipment with EDC obtained from storage. Turn on pumps (follow pump start-up procedure) so that each line and process vessel is solvent-flushed. Wherever possible, the various pieces of equipment will be filled to the top to insure complete washing with EDC. For the EDC still, however, it is planned to cascade liquid from the top to the bottom.

i. Reboilers and condensers may be put into operation at this stage with the stills on total reflux or recycle. This is also an appropriate time to check-out and use the process instruments, so that operating personnel can familiarize themselves with the instruments and their actions.

j. After complete circulation, check the EDC for quality. If it is clean and only contains water, it will be circulated through the process drier in the stripper reflux line until the water content has been lowered to a satisfactory level (below 20 ppm). If the EDC solvent is not clean, it will be stored in the dump tank for eventual clean-up.

k. If the stripper and still are operated on total reflux during these flushing operations, the reflux receivers will have to be drained completely to insure that phase water is not trapped in them.

l. Start-up levels will now be established in all vessels with either the initial EDC charge or a new charge from storage.

m. The plant is now ready for start-up.

It is important that care is taken in the plant drying steps. Water is a very bothersome contaminant. Water will not only create off-specification product, but will cause equipment to corrode very rapidly.

After a long term shut-down, some of the procedure will have to be repeated. What steps are necessary will depend upon the nature of the shut-down. Any time it is necessary for a piece of equipment to be worked on, it will have to be drained and purged with nitrogen. After the repairs have been made, the piece of equipment that was opened will have to be dried and purged before it is put back on stream. Never put a piece of equipment on stream unless it has been purged with nitrogen and is absolutely dry.

VI. SHUT-DOWN PROCEDURES

A. Overall Shut-Down Procedure

The following procedure will apply whether the plant is to be down for a short or long time. Certain procedures will not be applicable for the short term shut-down, and will be eliminated by the operations supervisor.

Shut-Down Procedure

1. Notify the liquefaction department of the intended shut-down.

2. Notify PCI via the Shift Engineer of the intended shut-down.

3. Lower the chlorine and ethylene flows stepwise, reducing chlorine flow rate first.

4. Stop chlorine flow to the reactor. Close the manual block valves around the flow control valves and at the spargers. (Stop methane addition to the vent system.)

5. Leave a slight flow of ethylene going to the reactor until the reaction has stopped and the reactor temperature begins to drop. Stop the ethylene flow and close the block valves around the flow control valve and at the spargers.

6. When the low level alarm sounds on the reactor gas separator, turn off the stripper feed stream. Shut down the reactor reflux and stripper feed pump. Close the block valves around the reactor reflux and stripper feed control valves.

7. Shut off the steam to the stripper reboiler. Close block valves around the steam flow control valve.

8. Shut off the stripper reflux pump when the stripper gas separator level is low enough to close the automatic level control valve in the reflux line. Close the block valves around the level control valve.

9. Begin a small purge of nitrogen through the system. This prevents the system from going under a vacuum.

10. Shut off the feed to the still and stop the still feed pump. Close the block valves around the still feed flow control valve.

11. Shut off steam to the still reboiler. Close the block valves around the steam flow control valve.

12. Stop EDC product flow to storage. Close the block valves around the product flow control valve.

13. Shut off the still reflux and reflux pump. Close the block valves around the reflux flow control valve.

14. Make sure the still nitrogen pad system is in service.

15. If a short-term shut-down is anticipated, leave all utilities such as cooling water and steam on.

16. If a long-term shut-down is anticipated, the still reboiler contents should be flushed to the bottoms storage tank with fresh EDC.

17. For a long-term shut-down, shut down all utilities in the following order:

a. Refrigeration machine

b. Steam supply

c. Cooling tower system

d. Instrument air

e. Electrical system

B. Detailed Equipment Shut-Down Procedure

1. Ethylene System:

a. Lower stepwise the chlorine flow followed by the ethylene flow until the chlorine flow is zero. After a short ethylene purge stop the ethylene flow.

b. If ethylene continues to flow immediately close the block valves around the ethylene flow control valve.

c. Close all manual block valves in the ethylene feed line. This includes the control valve block valves and the valves in the sparger manifold.

d. Notify PCI that the ethylene system shut-down is complete.

e. During the shut-down maintain an ethylene pressure of at least 35 psig in the system up to the flow control valve.

2. Chlorine System:

a. Notify liquefaction of the shut-down and request that the lead operator stand by the chlorine pump to maintain the pump discharge pressure at approximately 125 psig.

b. Open valves to allow the MC liquid chlorine feed to by-pass the EDC vaporizers.

c. If a long shut-down is anticipated close the block valves in the liquid chlorine lines to the vaporizers. Shut off the steam to the vaporizer and let the chlorine pressure bleed into the reactor. When the pressure approaches the reactor bottom pressure close the reactor spargers. The final chlorine pressure will have to be bled to the sewer.

d. If a short shut-down is anticipated, the liquid chlorine lines and the steam supply will not have to be shut off. The FCV can be shut from the panel board. Just before the flow is completely off, close the spargers and a block valve on the PCV simultanepusly.

3. Reactor:

a. Shut down the feed gas systems as described above.

b. Be sure all sparger valves are closed to keep EDC from backing into the feed lines.

c. When the reactor gas separator level starts to drop and the stripper feed decreases, shut off the stripper feed flow. Secure the manual valves around the stripper feed control valve.

d. When the reactor cools down and the reflux flow stops, close the manual valves in the reflux line and stop the reactor pump.

e. Turn on a small nitrogen purge to the reactor system. This will prevent a vacuum from occurring in the reactor and keep it air free.

f. Shut off the cooling water to the reactor and separator condensers after the reactor cools, if a long term shut-down is contemplated.

4. HCl Stripper:

a. The stripper feed is turned off during the shut-down of the reactor. Proceed with the shut-down of the stripper at the time the feed is turned off. The block valves around the stripper feed control valve should be closed.

b. Shut off the steam to the stripper reboiler. Close the block valves around the steam flow control valve.

c. When the reflux flow control valve is essentially closed, shut off the stripper reflux pump and close the block valves around the control valve.

d. Shut off the cooling water to the stripper condenser after column cools down if a long term shut-down is contemplated.

5. Distillation Column:

a. When the level in the crude tank has reached the desired level shut off the still feed flow. Stop the still feed pump and close the block valves in the still feed line.

b. When the level control valve in the product line closes, turn off the steam to the still reboiler. Close the block valves around the steam flow control valve.

c. Gradually reduce the still reflux flow to keep a constant level in the still reflux drum.

d. When the reflux flow is essentially off, close the flow control valve to keep a constant level in the reflux drum. (Stop TCP Addition.)

e. The nitrogen padding system should be in service to prevent a vacuum from forming.

f. Close all block valves in the product lines.

g. Shut off the cooling water to the still condenser and product cooler after the column cools (only if a long term shut-down is contemplated).

6. Vent Gas Refrigeration System:

a. When the reactor and stripper systems have cooled the refrigeration system may be shut down.

b. Stop the compressor.

c. Close the suction and discharge valves around the compressor.

7. Utilities:

a. Electrical System

The following procedures should be used when shutting down electrical equipment.

(1) Motors: Select the service to be shut down and operate the stop pushbutton, then open the 440 v. starter breaker at the motor control center.

(2) Motor Control Center: Remove all connected equipment from service as directed in a-1, then open the 440 v. A.C.B. on the metalclad switchgear unit.

(3) 440 v. Metalclad Switchgear: Shut down all motors and the motor control center as directed in a-1 and a-2, then open the transformer secondary breaker.

(4) 13.8V/440 Transformer: Shut down equipment as directed in a-1, a-2, and a-3, then open fused disconnect No. 0-304.

b. Steam Supply System: Ordinarily the steam headers to the equipment will be left on during a shut down with a small steam bleed through the system. However, if it becomes necessary to shut down the steam supply system simply close all manual valves in the supply system and then close the pressure reducer valves.

8. Cooling Tower System: When a long term shut-down of all cooling tower water users is anticipated (includes both EDC plants and the MC plant) the following shut-down procedure should be followed:

a. When the plant has cooled down and all heat sources have been turned off, shut off the cooling water pumps.

b. Shut off cooling tower blow-down.

c. Turn off treatment chemical addition.

d. Stop well water make-up flow.

e. Turn off pH meter.

f. Shut down the cooling tower fans.

9. Instrument Air System: If it becomes necessary to shut the instrument air system down the following procedure should be followed:

a. Unload the compressor by turning the transfer switch to the "OFF" position.

b. Stop the compressor.

c. Turn off the cooling water to the compressor.

d. Turn off the cooling water to the aftercoolers.

e. Close the manual valves around the driers and shut off the reactivation timer.

f. Close the block valve above the air receiver.

10. Stack Seal: The stack seal shut-down simply involves shutting off the seal water.

11. Flasher: The following procedure should be used when shutting down the flasher.

a. Shut off the feed.

b. Turn off the steam to the reboiler.

c. Break vacuum with N_2.

d. Shut off the condenser cooling water.

C. Emergency Shut-Down of Equipment

In the event that trouble arises in the EDC plant that cannot be handled by normal operating procedures, it may be necessary to make an emergency shut-down of one or more pieces of equipment. Emergency situations may involve fire, explosion, vessel or line rupture, instrument air failure, electrical failure, water failure, steam failure, etc. Each emergency will dictate its own procedure to be followed and it should be handled accordingly. Following is an explanation of some situations that may arise and a general outline of what should be attended to:

1. Reactor: If an emergency arises in the reactor, the feed gases should be shut down. This can be readily accomplished with the feed stop switch on the panel board. This will shut off the feed gases and the reaction will stop as soon as the dissolved gases in the reactor are depleted. If possible, a manual valve should also be closed in each of the feed gas streams to insure complete shutoff of the gases.

If possible, the reflux stream should be left circulating until the reactor has cooled sufficiently to stop boiling. If it is necessary to shut off the reflux stream, the level in the reactor will drop until boiling stops. If possible, the stripper feed should be continued for a while in this case to prevent over-filling the reactor gas separator. The routine shut-down procedure can then be followed for the rest of the plant.

2. Stripper: If difficulty arises in the stripper, the feed and the steam should be immediately shut off. The reactor feeds should be stopped as soon as possible after the stripper feed and steam are shut off.

If the emergency is such that it is possible, the stripper reflux should be left on until the stripper cools below the boiling point. This will increase the rate of cooling of the column. If necessary, though, this flow can be shut off at the same time as the feed. This will reduce the volume of liquor retained in the column which may be advisable under some conditions.

If it is important for the heat to be removed from the column as quickly as possible, the volume of steam retained in the reboiler can be relieved by opening the by-pass around the steam trap and bleeding the reboiler to the atmosphere. This will greatly speed up the removal of excess heat from the column.

After the stripper is secure, the routine procedure should be followed for shutting down the remainder of the plant.

3. Distillation Column: If an emergency shut-down of the still is necessary, the steam and feed should be shut off immediately. Again, the reflux can be used to cool the column and the reboiler can be vented if it is desirable. Immediate shut-down of the reactor is not as important here since a surge is available in the crude tank. After the still is secure, the rest of the plant can be shut down routinely as necessary or desirable.

4. Loss of Refrigeration: No particular emergency is presented if a refrigeration failure should occur. The refrigeration compressor should be secured if the source of trouble is not readily corrected. When the surge volume of liquid Freon is exhausted, the vent condenser temperature will rise and the plant will suffer a loss of EDC.

At this point the situation will have to be evaluated to determine if operation at a loss is warranted, or if the plant should be shut down.

5. Loss of Cooling Water: If for any reason the cooling water flow should be interrupted, it is necessary that all heat supplied to the plant be shut off immediately. This includes shutting off the feed gases to the reactor. As rapidly as possible, shut off the reactor feed gases, and shut off the steam to the stripper reboiler, the distillation column reboiler, and to the reactor dopp kettle.

The remainder of the plant should then be shut down until the flow of cooling water is resumed.

If the cooling tower fans should fail, it is still possible to operate the plant. The cooling water temperature will rise and a corresponding rise in the condenser and cooler temperatures will result. If operation were continued under these conditions, the plant would suffer a large loss of ethylene dichloride in the vent stream. If the source of trouble can be corrected in a short time, it may not warrant shutting down the plant, but this operation should not continue for an extended length of time.

6. Loss of Steam Supply: If the steam supply should fail, all process streams should be shut down. This presents no particular emergency except for a level build-up in the stripper since the stripper feed cannot be vaporized and set forward. For this reason the reactor should be shut down and the stripper feed shut off as soon as possible.

7. Loss of Instrument Air: The loss of instrument air would immediately shut off most process flows in the plant. Manually shut down and isolate the plant equipment, closing block valves on all automatic valves to insure against leakage. Shut off all pumps as soon as possible to prevent continued running against a dead shut off. Make certain the reboiler steam supply is off. In general, all instruments "fail safe."

8. Loss of Electricity: The loss of electrical power will necessitate a rapid shut-down and isolation of all equipment. Some instrument air may be available for a brief period, but manual valves should be shut as soon as possible. Make sure steam to reboilers is shut off and the steam bled from the jackets.

9. Fire, Explosion, Line Rupture: Under any of these cases, the first consideration is to save the personnel in the plant area; the second consideration is to save the plant. REMEMBER THIS AND ACT ACCORDINGLY. The sprinkler system should be tripped (if it has not automatically) in case of fire or explosion.

In case of a chlorine line rupture, the area should be evacuted; the plant shut down. If an ethylene line ruptures, there may be danger of fire or explosion. Therefore, if safety permits the ethylene supply line should be shut off at the plant battery limits. In case of an EDC line rupture, the equipment will have to be isolated and the plant shut down if necessary.

VII. ANALYTICAL

A. Sample Points

The 44 sample points in the No. 1 EDC plant have been numbered and are described in Table 7.1. Some of the sample points will not be used routinely but are included for possible special samples.

In describing a sample, always use the sample point number as well as its description. This information should always be recorded on the sample tag to preclude the possibility of a sample mix-up.

B. Control Samples

1. Sampling Schedule: The schedule of control analysis is shown in Table 7.2. This schedule will be revised as needed to take into account the experience gained in operating the plant.

When there is any indication of trouble in the plant, extra analyses will be required. Do not hesitate to run the required analyses, because the answers so obtained may save a lot of useless effort later on or will help in making the right process correction sooner.

2. Liquid Samples: All liquid samples for control analyses will be taken in 6-ounce polyethylene bottles previously cleaned and dried by the laboratory. The schedule given in Table 7.2 will be followed.

The sampling procedure will be as follows:

a. Obtain the proper clean, dry bottle and take it to the sampling station. Leave the lid tightly closed on the bottle.

b. Purge the sample line to remove stagnant liquid from the tap and thus provide a good, fresh sample of the process stream.

c. Remove the lid from the sample bottle and take the proper amount of sample. Put the lid back on quickly and tightly.

> Special care should be used in keeping the sample and sample bottle from being contaminated with the outside moist air since most of the control samples will be for water in EDC.

3. Analytical Methods: The analytical methods used in the control tests are included in this manual beginning on page 7-8 . Table 7.2 gives the sample points and the analyses that will be run on the samples.

C. Laboratory Samples

1. Sampling Schedule: The determinations that the laboratory will make are indicated in Table 7.3. It will be necessary, usually, for the mid-night operator to take these samples.

2. Sampling Procedures:

a. General

It is quite important that samples for water determination be handled carefully to avoid contamination. The process streams contain so little water that the slightest trace of water from an external source will cause an incorrect determination.

b. Technique

All lab samples (except for cooling tower water and gas samples) will be taken in a dry brown glass bottle. These bottles have been cleaned and dried by the laboratory. If for any reason a bottle is suspected to be contaminated it should not be used.

To take a sample purge the sample tap of material to completely replace stagnant liquid in the fittings and dead line portion. Remove the bottle cap and fill the bottle completely full. Recap the bottle immediately.

Be sure to attach a numbered sample tag firmly to the sample. On the sample tag place the description of the sample, date, time and sample point number.

c. Who Takes the Lab Samples?

It is important that a systematic approach be followed in taking the plant analytical samples. Assignments for lab sample taking are given in Table 7.3.

d. Composite Sample

A daily composite sample of the EDC product will be taken. The scheme devised to accomplish proper compositing is as follows:

(1) Each operator will be responsible on his shift for "dripping" product EDC continuously in the 2" glass-pipe collection chamber. (Collect approximately one inch of liquid per hour.)

(2) The sample from the chamber will be transferred once per shift, at 6:30 a.m., 2:30 p.m., and 10:30 p.m., into the proper sample bottle.

(3) A numbered sample tag will be affixed to the sample bottle. The tag should show the sample point number and a description including the shift, date and time.

(4) These samples will be taken to the laboratory by the midnight lead operator by 7:30 a.m.

(5) Make sure that the sample chamber is empty before the next shift sample is started.

e. Gas Samples

Laboratory gas samples will be taken either in the pressurized ethylene container (for ethylene only) or in brown burettes in the case of vent gases.

D. Process Chromatograph

A semi-continuous on-stream process chromatograph has been provided to sample the off-gas from the separator condensers. A complete description of the chromatograph may be found in the operating instructions for the unit. The components to be resolved will be ethane, ethylene, oxygen, and methane.

This relatively expensive instrument gives the operator an essentially continuous analysis of the vent gases. The amount of unreacted ethylene and oxygen in the vent stream are the most important of the chromatograph results. The unreacted ethylene will be proportional to the excess ethylene used in the reactor. The oxygen analysis is used to adjust the percentage of combustables in the vent stream so that the gas does not become explosive.

The chromatograph will be used as a guide in setting the ethylene excess at which the reactor will be operated.

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TABLE 1
SAMPLE POINTS FOR THE No. 1 EDC PLANT

Sample Point No.	Container Type*	Sample Description	Sample Tap Location
1.	A	Ethylene Gas Feed	Sample Cabinet
2.	B	Chlorine Gas Feed	Feed Flow Control Valve
3.	B	Chlorine Vaporizer Liquid	Vaporizer Inlet
4.	D	Catalyst Solution	Dump Pump Suction
5.	E	No. 2 Reactor Off-Gas	Reactor Condenser Inlet
6.	F**, D***	Stripper Feed	Reactor Pump Suction
7.	E	No. 2 Reactor Condenser Off-Gas	Separator Condenser Inlet
8.	F**, D***	Stripper Condenser Off-Gas	Condenser Vapor Outlet
9.	E	No. 2 Separator Condenser Off-Gas	Condenser Vapor Outlet
10.	D	Separator Condenser Liquid	Condenser Liquid Outlet
11.	D	Vent Condenser Liquid	Condenser Liquid Line
12.	E	Vent Condenser Off-Gas	Condenser Gas Outlet
13.	E	Stripper Off-Gas	Stripper Condenser Inlet
14.	F**, D***	Stripper Reflux Before Drier	Reflux Pump Suction
15.	F**, D***	Stripper Reflux After Drier	Reflux Filter Disch.
16.	F**, D***	Still Feed Liquid	Feed Pump Suction
17.	E	Still Off-Gas	Still Condenser Inlet
18.	E	Still Condenser Off-Gas	Condenser Vapor Outlet
19.	F**, D***	Still Reflux	Reflux Pump Suction
20.	F**, D***	Product	Composite Point
21.	D	Still Bottoms	Still Bottoms Pump Suct.
22.	-	--	--
23.	D	Bottoms Storage Tank	Dip Sample
24.	F**, D***	No. 1 Transfer Tank	Dip Sample
25.	F**, D***	No. 2 Transfer Tank	Dip Sample
26.	F**, D***	No. 3 Transfer Tank	Dip Sample
27.	F**, D***	Product to Dock Storage	--
27.1	F**, D***	Before Transfer Pump	Pump Suction
27.2	D	From Line at Dock Storage	--
28.	D	Dock Storage Tank	Dip Sample
29.	D	Barge Loading Product Composite	Loading Line
30.	D	No. 2 Reactor Liquor	Reactor Bottom
31.	F	Cooling Tower Water	C.T. Basin
32.	E	Stack Seal Effluent	--
33.	F**, D***	Stripper Bottoms	Reboiler Bottom
34.	D	No. 2 Reactor Liquor at Sight Glass	Sight Glass Bottom
35.	F	Well Water Make-Up to C.T.	Make-Up Stream
36.	D	No. 1 Reactor Liquor	Reactor Bottom
37.	D	No. 1 Reactor Liquor at Sight Glass	Sight Glass Bottom
38.	E	No. 1 Reactor Off-Gas	Reactor Cond. Inlet
39.	E	No. 1 Reactor Condenser Off-Gas	Separator Cond. Inlet
40.	E	No. 1 Separator Condenser Off-Gas	Condenser Vapor Outlet

Sample Point No.	Container Type*	Sample Description	Sample Tap Location
41.	F**, D***	No. 4 Transfer Tank	Dip Sample
42.	F**, D***	Crude Storage Tank	Dip Sample
43.	B	Cell Gas	Before PCV
44.	B	Vaporized Chlorine	FCV on Vaporized Chlorine System

*Container Type A - Pressure Cylinder
B - Special-Lab Only
C - Clear Bottle
D - Brown Bottle
E - Brown Burette
F - Plastic Bottle

**Control Lab Sample

***Main Lab Sample

TABLE 7.2

SCHEDULE OF No. 1 EDC PLANT CONTROL TEST

Sample Pt. No.	Description	Frequency	Determination & Method
12	Vent Gas	1/Shift	Composition (M-3C)
6	Stripper Feed	2/Shift	H_2O (M-1), Iron (M-2)
14	Stripper Reflux	2/Shift	H_2O (M-1)
33	Stripper Bottoms	2/Shift	H_2O (M-1), HCl (M-4), Iron (M-2)
20	Product	2/Shift	H_2O (M-1), HCl (M-4), Iron (M-2)
31	Cooling Tower Water	2/Shift	Chlorides (M-313, pH (M-318) Chromate (M-315), Betz 107 (M-315A)
35	Cooling Tower Make-up	1/Shift	Chloride (M-313)

		Shift 1	Shift 2	Shift 3
Pt. 12	Time to Run Sample	8:30 AM	4:30 PM	12:30 AM
Pts.	Time to Run Sample	9:30 AM-1:30 PM	5:30 PM-9:30 PM	1:30 AM-5:30 AM
Pt.	Time to Run Sample	10:30 AM-2:30 PM	6:30 PM-10:30 PM	2:30 AM-6:30 AM
Pt.	Time to Run Sample	10:30 AM	6:30 PM	2:30 AM

Note: For start-up and on special control situations, determinations such as water or HCl will be done on a more frequent basis.

TABLE 7.3

SCHEDULE OF No. 1 EDC PLANT LABORATORY ANALYSES

Sample Pt. No.	Description	Sample Day	Analyses
30, 36	Reactor Liquor	Tuesday	GC, Fe, H_2O, HCl, Tars, Sp.Gr.
6	Stripper Feed	Thursday	GC, Fe, H_2O, HCl
16	Still Feed	Thursday	GC
21	Still Bottoms	Tuesday	GC, Fe, H_2O, HCl
20	Product	Daily	GC, Fe, H_2O, HCl
31	Cooling Tower Water	Wednesday	CrO_4, NaCl, SiO_4, PO_4, pH, Betz 107
35	Cooling Tower Make-up	Wednesday	NaCl, SiO_4

Note: For start-up and on special control situations, additional and more frequent samples will be necessary.

E. Analytical Control Test

The test procedures found on the next pages have been developed by the main laboratory. Occasionally changes will be made in these procedures. The new procedures should be substituted for the old ones to keep this manual up-to-date.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-1

1. Test: Water in Ethylene Dichloride containing no $FeCl_3$.

2. Sample Point: No. 13 - Still Feed, No. 15 - Still Reflux, No. 9 - Stripper Feed, No. 11, 12 - Stripper Reflux and No. 16 - Production, No. 20, 21 - VC Feed.

3. Safety Precautions: Ethylene Dichloride is an inflammable liquid, and its vapors will form explosive mixtures with air. Keep it away from flames and sparks, and use only under hood. Wear safety glasses, apron and gloves when handling EDC.

4. Interference: Free chlorine and iron.

5. Precision: $\pm$10 ppm.

6. Apparatus: Titration apparatus.

7. Reagents:

   a. Karl Fischer Reagent.
   b. Methanol - Phenol mixture.

8. Procedure:

   a. Turn instrument switch to the "on" position. Drain the titration vessel level with the drain spout. Make up the volume to the red mark on the vessel with alcohol. This is probably one of the more critical steps in the procedure. The degree of conductance through the solution is a function of the ratio of EDC to alcohol. Thus, the end point is established experimentally and is necessarily confined to a specific ratio. Failure to follow instructions in this step, therefore, may completely distort the true end point.
   b. Add concentrated K.F. reagent until, by visual observation, the neutral point is approached. The K.F. visual end point should be a very light iodine color.
   c. Stopper the vessel and finally adjust the solution to the exact neutral point by aid of the K.F. contained in the burette and the instrument end point. When the solution is neutral, the ammeter will register 12.
   d. Pipette a 20 ml. sample. Make sure the pipette is dry.
   e. Before adding the sample, check the micro ammeter. The needle should be either on or to the right of 13 drifting towards 14. If the reading is left of 12, or has exceeded 14, the neutral point will have to be readjusted. Otherwise, add the sample and replace the stopper. The importance of the neutral point cannot be over emphasized. The tendency, of course, is to over neutralize. The procedure incorporates a small over titration at the neutral point to allow the operator time to prepare the sample, etc., and normally, this excess is consumed by

atmospheric contamination before the sample is added. However, the instrument response at this point is weak, and it is easy to over titrate considerably, if a fine line is not emphasized as to the difference between being on 12 and somewhere to the left of 12. The same precaution should be taken regarding a drift beyond 14. In this case, the effect on the analysis is the reverse of that referred to above, but is equally critical.

f. Add the sample, replace stopper, reset the burette and start the titration. The burette assembly is designed with a double stopcock between the K.F. reservoir and the titration vessel. The stopcock nearest the vessel should deliver approximately a drop per second. This is necessary to allow for the lag in the instrument response, so that the needle deflection will be smooth and not erratic toward the end point. The second stopcock is merely used in the "on" and "off" position for refilling the burette and delivery of reagent to the adjusted stopcock. Serious error will result if this arrangement is employed in any other manner.

g. Turn the stopcock nearest the K.F. reservoir to the "on" position in the direction of the adjusted stopcock and allow the titration to continue, uninterrupted, until the needle of the ammeter coincides with 14.

h. Close the stopcock nearest the reservoir. If the needle does not drift beyond a reading of 16 in 20 seconds, record the titration. If it does, readjust the end point in the manner just described, and time the neutral point a second time. If it is necessary to readjust the end point over three times, the apparatus is not working properly, and the situation should be referred to the main laboratory.

i. ppm H_2O = Final K.F. Titer x N x 40,000.

j. Standardization of K.F. reagent.

The main laboratory will be responsible for maintaining a standard solution of CH_3OH and water in the plant laboratory. The water in the standard will be determined by the K.F. employed in the main laboratory, which, in turn, has been previously standardized against a weighted amount of water. The standard will bear a label stating the grams of water per 20 mls. of solution. The operator in the field will titrate a 20 ml. sample of the standard as described in the sample procedure above.

$$\text{Normality} = \frac{\text{grams } H_2O \text{ per 20 ml.}}{\text{K.F. Titer}}$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-2

1. Test: Iron.

2. Sample Point: Number 15 - Still Reflux, No. 13 Still Feed and No. 16 Product EDC.

3. Safety Procautions: Ethylene dichloride is an inflammable liquid, and its vapors will form explosive mixtures with air. Keep it away from open flames and sparks. Use only under hood. Wear safety glasses, apron and gloves when handling EDC.

4. Interference: Not determined.

5. Precision: $\pm$0.05 ppm Fe.

6. Apparatus and Equipment:

   a. 250 ml. separatory funnels.
   b. Separatory funnel stand.
   c. 100 ml. graduated cylinders.
   d. 50 ml. graduated cylinders.
   e. Glass funnels.
   f. 100 ml. volumetric flasks.
   g. Automatic 5 cc pipette.
   h. 50 ml. buret.
   i. Fisher electrophotometer equipped with 525-B filter.

7. Reagents:

   a. Approximately 1:1 HCl.
   b. 2% Thioglycolic Acid.
   c. Approximately 6 N NH_4OH.
   d. Distilled water.

8. Procedure:

   a. Add distilled water to the 90 ml. calibration mark on a 250 ml. separatory funnel. (The separatory funnel is calibrated and marked to indicate volumes of 90 and 200 mls.) Add 10 mls. of 1:1 HCl.
   b. Add EDC sample to the 200 ml. mark.
   c. Shake vigorously for two minutes, venting as needed.
   d. Place separatory funnel in rack and allow phases to separate.
   e. Drain EDC into waste container, leaving HCl phase in funnel.
   f. Transfer the HCl phase to a 100 ml. volumetric flask.
   g. Add 5 ml. Thioglycolic acid and mix.
   h. Add 10 mls. NH_4OH, dilute to volume with distilled water and mix.
   i. Place sample in electrophotometer cell and obtain reading on Scale A.

Method No. M-2 - Continued

j. Read ppm Fe from calibration chart.
k. For blank, add 10 mls. of 1:1 HCl to a 100 ml. graduated cylinder with a pipetter. Fill to the 100 ml. mark with distilled water. Pour 50 mls. of this solution into a 100 ml. volumetric flask and begin with Step "h" of procedure.

9. Calculations:

$$\frac{\text{Grams Fe from Calibration Curve} \times 10^6}{\text{Sample Size} \times 1.25^*} = \text{ppm Fe}$$

* Approximate specific gravity of EDC.

IRON CHART FOR ELECTROPHOTOMETER NO. 5011
EDC CONTROL TEST

Reading	ppm, Fe	Reading	ppm, Fe
0.5	0.02	13.0	0.60
1.0	0.05	13.5	0.63
1.5	0.07	14.0	0.65
2.0	0.09	14.5	0.67
2.5	0.12	15.0	0.70
3.0	0.14	15.5	0.72
3.5	0.16	16.0	0.74
4.0	0.19	16.5	0.77
4.5	0.21	17.0	0.79
5.0	0.23	17.5	0.81
5.5	0.27	18.0	0.84
6.0	0.28	18.5	0.86
6.5	0.30	19.0	0.88
7.0	0.32	19.5	0.90
7.5	0.35	20.0	0.93
8.0	0.37	20.5	0.95
8.5	0.39	21.0	0.97
9.0	0.42	21.5	1.00
9.5	0.44	22.0	1.02
10.0	0.46	22.5	1.04
10.5	0.49	23.0	1.07
11.0	0.51	23.5	1.09
11.5	0.53	24.0	1.11
12.0	0.56	24.5	1.14
12.5	0.58	25.0	1.16

Calculations: Grams Fe = Reading x 0.0000029 (Calibration Factor)

$$\text{ppm Fe (by Wt.)} = \frac{\text{Grams Fe} \times 10^6}{\text{Size Sample (50 Ml.)} \times 1.25 \text{ (Sp. Gravity EDC)}}$$

EDC ANALYTICAL CONTROL TEST
METHOD No. M-3

1. Test: Orsat Analysis of Vent Gas for HCl, C_2H_4, Cl_2 and Inerts.

2. Sample Point: No. 6, 8

3. Apparatus:

   a. 100 ml. brown gas bulb connected to a leveling bottle containing 150 ml. H_2O + 1 ml. Ortho Tolidine.
   b. Fisher orsat burette connected to leveling bottle containing ethylene absorbent.

4. Reagents:

   a. Absorbent for ethylene.
   b. Ortho Tolidine.

5. Sampling and Procedure:

   a. Purge 100 ml. gas bulb for two minutes. If possible, place the gas bulb in a vertical position. When removing the gas bulb from the purge line, disconnect the sample in this order:
      1. Turn the stopcock on the exit end of the bulb to the off position.
      2. Close the stopcock nearest the sample point.
      3. Disconnect the sample bulb from the line. This procedure provides the sample, which is under a slight pressure. Allow the gas sample to come to ambient temperature. Establish the prevailing atmospheric pressure by rapidly rotating the stopcock through the open position. The release of pressure will be evidenced by a hissing sound. Repeat the procedure, until this sound can no longer be heard.
   b. Attach leveling bottle containing 150 mls. water and 1 ml. Ortho Tolidine to the 100 ml. gas bulb. Be sure all air is out of rubber tubing, and connect to small end of gas bulb. Open the stopcock connecting the gas bulb and leveling bottle and allow the acid gases to dissolve. Shake gas bulb gently, until constant reading is obtained. Record this as % HCl.
   c. Fill Fisher orsat burette with ethylene absorbent to a point above connecting rubber tubing. Open cocks in gas bulb and orsat burette.. With aid of water leveling bottle, transfer remaining gas to orsat burette. Close both cocks and disconnect orsat burette at ball joint.
   d. Shake sample in orsat burette, until a constant reading is obtained. Record the mls. of gas left as inerts (i.e., 100 - reading).
   e. Allow water in gas bulb to flow back into the leveling bottle. If no color is detected, record % Cl_2 (0.1).

Method No. M-3 - Continued

6. Calculations:

100 - (% Inerts + % HCl) = % Ethylene

Note: Change ethylene absorbent once/shift.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-3A

1. Test: Orsat analysis of vent gas for HCl, C_2H_4, Cl_2 and unsaturated Organic gases, when operating with excess Cl_2 or when Cl_2 is found in vent gas.

2. Sample Point: No. 6, 8.

3. Apparatus:

   a. 100 ml. brown gas bulb connected to a levelling bottle containing about 150 ml. of 1.0 N caustic.
   b. Fisher orsat burette connected to levelling bottle containing ethylene absorbent.

4. Reagents:

   a. Absorbent for ethylene.
   b. 1.0 N NaOH or 1.0 N KOH.
   c. Acetic acid
   d. 0.1 N $Na_2S_2O_3$.
   e. Starch.
   f. KI crystals or 5% KI.

5. Frequency: Twice per shift, or as needed.

6. Sampling:

   a. Purge 100 ml. gas bulb for two minutes. If possible, place the gas bulb in a vertical position. When removing the gas bulb from the purge line, disconnect the sample in this order:
      1. Turn the stopcock on the exit end of the bulb to the "off" position.
      2. Close the stopcock nearest the sample point.
      3. Disconnect the sample bulb from the line. This procedure provides the sample, which is under a slight pressure. Allow the gas sample to come to ambient temperature. Establish the prevailing atmospheric pressure by rapidly rotating the stopcock through the open position. The release of pressure will be evidenced by a hissing sound. Repeat the procedure, until this sound can no longer be heard.
   b. Attach levelling bottle containing about 150 mls. 1 N NaOH to the 100 ml. gas bulb. Be sure all air is out of rubber tubing, and connect to small end of gas bulb. Open the stopcock connecting the gas bulb and levelling bottle and allow the acid gases to dissolve. Shake gas bulb gently, until constant reading is obtained. The reading obtained should be subtracted from 100 to give total acid gases which will be called B_1.

      Note: If this value cannot be read on the sampling burette, transfer the gas to the measuring burette and read, before the following step.

c. Fill Fisher orsat burette with ethylene absorbent to a point above connecting rubber tubing. Open cocks in gas bulb and orsat burette. With aid of the levelling bottle, transfer remaining gas to orsat burette. Close both cocks and disconnect orsat burette at ball joint.
d. Shake sample in orsat burette, until a constant reading is obtained. Record the mls. of gas left as inerts (i.e., 100 - reading). Record ethylene as 100-(inerts)-(acid gases).
e. Acid gases:
   1. Drain the NaOH contained in the levelling bottle and gas bulb into a 400 ml. beaker and add about 20 ml. of acetic acid.
   2. Add a small scoop of KI crystals or 10 ml. of 5% KI solution.
   3. Titrate the sample with 0.1 N $Na_2S_2O_3$ to a pale straw color. Add one dropper of starch solution and continue titration, until the disappearance of the blue color.

7. Calculations:

a. From the chart obtain the mls. of Cl_2 equivalent to the titer.
b. Mls. Cl_2 (from chart) = % Cl_2.
c. Total acid gases (B_1) - % Cl_2 = % HCl

$$\frac{\text{Titer} \times .100 \times .0355}{.00295} = \text{Titer} \times 1.20 = \text{Mls. } Cl_2$$

% Cl_2 in Vent Gas

Titer (ml)	Cl_2 (ml)	Titer (ml)	Cl_2 (ml)
.4- 1.2	1	13.0-13.7	16
1.3- 2.1	2	13.8-14.5	17
2.2- 2.9	3	14.6-15.4	18
3.0- 3.7	4	15.5-16.2	19
3.8- 4.6	5	16.3-17.0	20
4.7- 5.4	6	17.1-17.9	21
5.5- 6.2	7	18.0-18.7	22
6.3- 7.1	8	18.8-19.5	23
7.1- 7.9	9	19.6-20.4	24
8.0- 8.7	10	20.5-21.2	25
8.8- 9.6	11	30	36
9.7-10.4	12	40	48
10.5-11.2	13	50	60
11.3-12.1	14	60	72
12.2-12.9	15	70	84

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-3B

1. Test: Determination of Free Chlorine in Vent Gas

2. Sample: Vent Gas from Condenser (before addition of CH_4)

3. Interferences: Other Oxidizing Agents

4. Apparatus and Reagents:

   a. Manometer - (previously calibrated)[1]
   b. 1000 ml. Bubbling Jar
   c. 500 ml. Porcelain Casserole
   d. Stirring Rod
   e. 5% KI
   f. 0.01N $Na_2S_2O_3$
   g. Starch Solution Indicator
   h. Burette, 10 ml., Automatic "0"

5. Procedure:

   a. Purge vent gas for several minutes and then reduce flow. Cut off flow with small needle valve and connect to right side of the manometer.
   b. Connect the bubbling jar containing approximately 200 mls. 5% KI to the left side of the manometer.
   c. Adjust gas flow to a 2" differential on the manometer. Bubble gas for 5 minutes.
   d. Record temperature of gas and bubbling time and close needle valve.
   e. The reddish-brown KI solution is titrated with 0.01N $Na_2S_2O_3$ in the Casserole until a straw-yellow color is reached.
   f. Two droppers of starch solution are added and the titration is continued until a clear end point is reached.

6. Equations:

$$Cl_2 + 2KI \longrightarrow 2KCl + I_2$$

$$I_2 + Na_2S_2O_3 \longrightarrow NaI$$
(Colored) (Clear)

1. Manometer orifice set at time of calibration - DO NOT TAMPER.

Method No. M-3B - (Continued)

7. Calculations:

a. g Cl = Titer x Normality x 0.03545

b. Meq Cl_2 = g Cl/0.0709

c. ml Cl_2 = meq Cl_2 x 22.4 = V_1

d. ml vent gas = $\frac{114{,}500^2 \text{ x } 273 \text{ x t (Minutes)}}{60 \text{ x } 273 + \text{Temp. (Degrees F)}}$ = V_2

e. $\frac{V_1}{V_2}$ x 100 = % Cl_2 by volume

8. Typical Results:

Trace to 0.1% Cl_2

2. 114,500 ml. gas vent/hour at 0°F using HCl as standard

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-3C

1. Test: Orsat Analysis for HCl, CO_2, O_2 and C_2H_4. In Vent Gas

2. Sample Point: Nos. 6 and 8

3. Apparatus:

   a. 100 ml. brown gas burette.
   b. Leveling bottle.
   c. Orsat apparatus with absorption chambers for CO_2, O_2 and C_2H_4.

4. Reagents:

   a. Confining Solution
   b. CO_2 absorbant (29% KOH)
   c. O_2 absorbant (Pyrogallic Acid)
   d. C_2H_4 absorbant ($HgSO_4$ in H_2SO_4)

5. Procedure:

   a. Connect 100 ml. gas burette to sample tap and purge for two minutes. If possible, place the gas bulb in a vertical position. When removing the burette from the purge line, disconnect the sample in this order:

      (1) Turn the stopcock on the exit end of the burette to the off position.
      (2) Close the stopcock nearest the sample point.
      (3) Disconnect the sample burette from the line. This procedure provides the sample, which is under a slight pressure. When ready to run the test establish atmospheric pressure in the burette by rapidly rotating the stopcock through the open position. The release of pressure will be evidenced by a hissing sound. Repeat the procedure, until this sound can no longer heard.

   b. Attach the leveling bottle containing 150 mls. of confining solution to the burette. Be sure all air is out of the rubber tubing and connect to the small end of the burette. Open the stopcock connecting the burette and leveling bottle and allow the acid gases to dissolve. Shake the burette gently, until a constant gas volume reading is obtained. Record this reading as "A".
   c. Transfer the remaining gas to the measuring burette of the orsat apparatus. (Be careful not to allow air to enter the system.)
   d. Pass the residual gases through the CO_2 absorbant chamber slowly and back to the measuring burette until a constant volume reading is obtained. (This will take at least 10 passes.) Record the new volume reading as "B".
   e. Pass the remaining gas through the O_2 absorbant chamber and back to the measuring burette until a constant reading is obtained. Record this reading as "C".

METHOD NO. M-3C (Continued)

f. Pass the remaining gas through the C_2H_4 absorbant chamber and back to the measuring burette until a constant reading is obtained. Record this reading as "D".

6. Calculations:

a. % HCl = 100 - "A"
b. % CO_2 = "A" - "B"
c. % O_2 = "B" - "C"
d. % C_2H_4 = "C" - "D"

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-4

1. Test: Acidity as HCl, ppm.

2. Sample Point: No. 13, 16.

3. Safety Precautions: Ethylene dichloride is an inflammable liquid, and its vapors will form explosive mixtures with air. Keep it away from flames and sparks. Use only under hood. Wear safety glasses, apron and gloves when handling EDC.

4. Interference: None.

5. Precision: $\pm$0.10 ppm HCl.

6. Apparatus:

   a. 250 ml. separatory funnel.
   b. 250 ml. beaker.
   c. 10 ml. microburette.
   d. 100 ml. graduated cylinder.

7. Reagents: 0.01 Sodium hydroxide.

8. Procedure:

   a. Add 100 ml. of sample to a 250 ml. separatory funnel.
   b. Add 100 ml. of distilled H_2O to funnel and agitate thoroughly one minute. CAUTION: Release pressure in funnel by venting as required.
   c. Allow to separate into two layers. Drain bottom layer (EDC) into second 250 ml. funnel. Drain top layer (H_2O + HCl into 250 ml. beaker.
   d. Add phenolphthalein indicator and titrate with .01 N caustic to a permanent light pink end point. Record the titration to nearest .01 ml.
   e. If the titration requires more than 1.00 ml. of NaOH, the extraction procedure should be repeated using the sample retained from (Step 8c) and 100 ml. distilled H_2O. This second titer should be added to the one obtained in (d).

9. Calculations:

   a. $$\frac{\text{Total mls. NaOH} \times N \times .0365 \times 10^6}{100 \times \text{SP. Gr. EDC (1.25)}} = \text{ppm HCl}$$

      mls. NaOH x 2.92 = ppm HCl

   b. Locate total mls. NaOH used in titration on chart and read ppm HCl.

   c. If the $FeCl_2$ exceeds 1.0 ppm, the following correction should be made. Subtract 1.8 ppm HCl for every ppm Fe.

CHART FOR DETERMINATION OF HCl, ppm

Mls NaOH	ppm HCl	Mls NaOH	ppm HCl
0.33-0.35	1.0	1.91-1.93	5.6
0.36-0.39	1.1	1.94-1.96	5.7
0.40-0.42	1.2	1.97-2.00	5.8
0.43-0.46	1.3	2.01-2.03	5.9
0.47-0.49	1.4	2.04-2.07	6.0
0.50-0.53	1.5	2.08-2.10	6.1
0.54-0.56	1.6	2.11-2.14	6.2
0.57-0.59	1.7	2.15-2.17	6.3
0.60-0.63	1.8	2.18-2.20	6.4
0.64-0.66	1.9	2.21-2.24	6.5
0.67-0.70	2.0	2.25-2.27	6.6
0.71-0.73	2.1	2.28-2.31	6.7
0.74-0.77	2.2	2.32-2.34	6.8
0.78-0.80	2.3	2.35-2.38	6.9
0.81-0.83	2.4	2.39-2.41	7.0
0.84-0.87	2.5	2.42-2.44	7.1
0.88-0.90	2.6	2.45-2.48	7.2
0.91-0.94	2.7	2.49-2.51	7.3
0.95-0.97	2.8	2.52-2.55	7.4
0.98-1.01	2.9	2.56-2.58	7.5
1.02-1.04	3.0	2.59-2.61	7.6
1.05-1.07	3.1	2.62-2.65	7.7
1.08-1.11	3.2	2.66-2.68	7.8
1.12-1.14	3.3	2.69-2.72	7.9
1.15-1.17	3.4	2.73-2.75	8.0
1.19-1.21	3.5	2.76-2.79	8.1
1.22-1.24	3.6	2.80-2.82	8.2
1.25-1.28	3.7	2.83-2.85	8.3
1.29-1.31	3.8	2.86-2.89	8.4
1.32-1.35	3.9	2.90-2.92	8.5
1.36-1.38	4.0	2.93-2.96	8.6
1.39-1.42	4.1	2.97-2.99	8.7
1.43-1.45	4.2	3.00-3.03	8.8
1.46-1.48	4.3	3.04-3.06	8.9
1.49-1.52	4.4	3.07-3.09	9.0
1.53-1.55	4.5	3.10-3.13	9.1
1.56-1.59	4.6	3.14-3.16	9.2
1.60-1.62	4.7	3.17-3.20	9.3
1.63-1.66	4.8	3.21-3.23	9.4
1.67-1.69	4.9	3.24-3.27	9.5
1.70-1.72	5.0	3.28-3.30	9.6
1.73-1.76	5.1	3.31-3.33	9.7
1.77-1.79	5.2	3.34-3.37	9.8
1.80-1.83	5.3	3.38-3.40	9.9
1.84-1.86	5.4	3.41-3.44	10.0
1.87-1.90	5.5		

Method No. M-4

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-5

1. Test: Determination of Catalyst ($FeCl_3$) Concentration in Reactor Liquor.

2. Sample Point: No. 22.

3. Abstract: This method was devised for the rapid determination of catalyst concentration in the EDC reactor. The method involves the extraction of ferric chloride from the sample with hydrochloric acid, followed by an iron analysis by the colorimetric thioglycolic acid method.

4. Application: EDC reactor liquor.

5. Interfering Substance: Not determined.

6. Procedure: Place 25 ml. approximately 1 N HCl in a one liter volumetric flask. Add 10 ml. approximately 0.1 N $NaAgO_2$ to flask, then carefully pipette in 1 cc of sample. Mix the liquids thoroughly, shaking for at least 1 minute. Then sweep gaseous chlorine away by directing an air stream into the flask. Add 10 ml. 2% thioglycolic acid and mix. Make alkaline with approximately 6 N NH_4OH, adding a few drops in excess. Dilute to volume and mix. Pipette 50 ml. aliquot and dilute to 100 ml. Determine grams of iron from a previously standardized Electrophotometer.

   A distilled water blank will be adequate, unless the HCl extract is colored by organic materials. If so, a blank may be run on HCl extract, omitting the thioglycolic acid.

7. Calculations:

   gm. Fe x 2.9049 = gm. $FeCl_3$

$$\frac{\text{gm. } FeCl_3 \times 20 \times 100}{*1.25 \text{ g}} = \text{per cent } FeCl_3$$

*Approximate specific gravity of EDC.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-6

1. Test: Check for excess moisture in ethylene feed.

2. Sample Point: No. 1

3. Apparatus: Alnor Dew Point Analyzer.

4. Procedure:

   a. Turn off inlet valve, and depress button on top of ratio gauge. If ratio does not read exactly 1, adjust it by means of screw knob at bottom of ratio gauge.
   b. Open inlet valve 1/4 turn, allowing purging to start again.
   c. Read temperature on thermometer on instrument, and determine desired ratio by means of chart in cabinet.
   d. Close outlet valve, and pump pressure up to desired ratio.
   e. While observing expansion chamber through window on instrument, depress plunger.
   f. If no fog appears, record moisture as less than 100 ppm wt.
   g. If fog appears, notify foreman or shift engineer.

ETHYLENE DEW POINT CHART

Temperature (^{o}F)	Ratio
25- 29	.51
30- 34	.48
35- 39	.46
40- 44	.44
45- 49	.42
50- 54	.39
55- 59	.37
60- 64	.36
65- 69	.34
70- 74	.33
75- 79	.31
80- 84	.30
85- 89	.29
90- 94	.28
95- 99	.26
100-104	.25
105-109	.24
110-114	.23

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-6A

1. Test: Determination of Ethylene Purity.

2. Sample Point: No. 1

3. Apparatus:

   a. One 100 ml. clear absorption burette.
   b. One 250 ml. leveling bottle.

4. Procedure:

   a. Fill burette with ethylene absorbent or Burrell Sorbant "A" and allow to drain back into leveling bottle, keeping level in the burette above the bottom stopcock. This is done to remove all air from the rubber tubing.
   b. Connect burette to sample point and allow ethylene to purge through burette for approximately 1-2 minutes.
   c. Shut off bottom and top stopcocks, then needle valve in that order.
   d. Allow burette to reach ambient temperature and vent excess pressure.
   e. Open bottom stopcock and shake burette until no apparent absorption is taking place.
   f. Read % ethylene from graduation on the burette.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-311

1. Test: Alkalinity.

2. Sample Point: No. 40, 41.

3. Apparatus:

   a. 250 ml. beaker.
   b. 100 ml. graduated cylinder.
   c. Automatic 10 ml. burette assembly.
   d. Stirring rod.

4. Reagents:

   a. 0.02 N sulfuric acid.
   b. Methyl orange.

5. Procedure:

   a. Measure 100 ml. of cooling water into a 250 ml. beaker.
   b. Add .02 N sulfuric acid, stirring continuously, until the color changes from green to yellow.
   c. (If chloride test is desired, save sample and begin with Step 2 under chloride test.)

6. Calculations:

$$\frac{\text{ml. of Sulfuric Acid} \times .02 \times .05 \times 10^6}{\text{Sample Size}} = \text{ppm as } CaCO_3$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-312

1. Test: Calcium and Magnesium as CaO.

2. Sample Point: Number 40, 41.

3. Apparatus:

   a. 500 ml. porcelain casserole.
   b. 100 ml. graduated cylinder.
   c. Automatic 10 ml. burette assembly.
   d. Rubber policeman.

4. Reagents:

   a. Versenate solution.
   b. Monover indicator.
   c. 1 N hydrochloric acid.
   d. Methyl red.
   e. Ammonium hydroxide solution (6N).
   f. Distilled water.

5. Procedure:

   a. Measure a 100 ml. sample of cooling water into casserole and add 200 ml. distilled water.
   b. Add 10 drops methyl red.
   c. Titrate with 1 N hydrochloric acid to the red end-point and add exactly 2 ml. in excess.
   d. Stir gently for 30 seconds.
   e. Add 2 ml. of ammonium hydroxide solution and stir to mix.
   f. Add Monover indicator until the appropriate color develops.
   g. Add standard versenate solution, until the solution changes from wine-red to pure-blue.

6. Calculations:

$$\frac{\text{ml. Versenate Solution} \times .00056 \times 10^6}{\text{Sample Size}} = \text{ppm Ca and Mg as CaO}$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-313

1. Test: Chloride.

2. Sample Point: No. 40, 41.

3. Apparatus:

   a. 250 ml. beaker.
   b. 50 ml. graduated cylinder.
   c. Automatic 10 ml. burette assembly.
   d. Stirring rod.

4. Reagents:

   a. 0.02 N H_2SO_4
   b. 0.0282 N mercuric nitrate.
   c. Methyl orange.
   d. Diphenylcarbazone-bromophenol blue indicator.

5. Procedure:

   a. Measure 50 ml. of cooling water and approximately 100 ml. distilled water into 250 ml. beaker. Titrate with .02 N sulfuric acid using 5-8 drops methyl orange indicator. End point color is brass.
   b. Add 1/4 dropper of diphenylcarbazone-bromophenol blue indicator.
   c. Titrate with .0282 N mercuric nitrate, until solution turns from orange to wine.

6. Calculations:

$$\frac{\text{ml. mercuric nitrate} \times .0282 \times .05845 \times 10^6}{\text{Sample Size}} = \text{ppm NaCl}$$

$$\text{ml.} \times 32.96 = \text{ppm NaCl}$$

7-31

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-314A

1. Test: Chlorine.

2. Sample Point: No. 40.

3. Apparatus:

   a. Wallace and Tiernan Comparator.
   b. Chlorine color disc - chlorine residual range 0.1 ppm to 2.0 ppm.
   c. Sample cells with a 26 mm viewing depth.

4. Reagents:

   Ortho-Tolidine Reagent; 1.35 gms. of Ortho-Tolidine dihydrochloride dissolved in 500 ml. of distilled H_2O. Mix with 500 mls. of dilute HCl made by mixing 350 ml. of distilled H_2O with 150 ml. of concentrated HCl.

5. Procedure:

   a. Select two clean comparator cells.
   b. Fill one cell to mark with water under test.
   c. Insert this cell in the right cell space of the comparator.
   c'. Note: This cell, when filled to the mark with water under test, compensates for color and turbidity.
   d. Add 0.75 ml. Ortho-Tolidine reagent to the other cell.
   e. Fill this cell to the mark with the water under test.
   f. Insert the cell containing the water and the Ortho-Tolidine reagent in the left cell space of the comparator.
   g. Match, as closely as possible, the color developed in the left cell with the glass color standards by revolving the disc.
   h. When match has been made, take reading from upper left hand opening of comparator case.

6. Calculations:

   ppm residual Cl_2 read directly from comparator.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-315

1. Test: Chromate.

2. Sample Point: No. 40[illegible].

3. Apparatus:

   a. 250 ml. beaker.
   b. 50 ml. graduate.
   c. Automatic 10 ml. burette assembly
   d. Porcelain spatula

4. Reagents:

   a. 0.01N Sodium Thiosulfate.
   b. KI crystals.
   c. Concentrated HCl.
   d. Starch solution.

5. Procedure:

   a. Measure 50 mls. of cooling tower water into a 250 ml. beaker.
   b. Add 1 scoop of KI crystals.
   c. Add 2 mls. of HCl and mix.
   d. Let stand without agitation for 2 minutes.
   e. Add 0.01N sodium thiosulfate until sample turns straw yellow and then add 1 ml. of starch solution.
   f. Continue titration until blue starch compound disappears.

6. Calculations:

$$\frac{\text{ml. of sodium thiosulfate} \times 0.01 \times 0.0387 \times 10^6}{\text{Sample Size}} = \text{ppm } CrO_4^=$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. 315-A

1. Test: Betz 107 Concentration.

2. Sample Point: No. 40.

3. Apparatus:

   a. 250 ml. volumetric flask.
   b. 100 ml. graduated cylinder.
   c. Automatic 10 ml. burette.
   d. Spectrophotometer.

4. Reagents:

   a. 20% sodium carbonate.
   b. Distilled water
   c. Betz 107 indicator

5. Procedure:

   a. Add 50 mls. sample to 250 ml. volumetric flask.
   b. Add 2 mls. Betz 107 indicator, shake and let stand for 1 minute.
   c. Add 10 mls. 20% sodium carbonate (Na_2CO_3) and let stand for 3 minutes.
   d. Dilute to volume with distilled water.
   e. In a second 250 ml. volumetric flask prepare a blank of 10 mls. 20% Na_2CO_3 and 50 ml. sample and dilute to volume with distilled water.
   f. Zero spectrophotometer with blank using 650 mu "A" (red) filter or at 650 mu.
   g. Read sample on spectrophotometer.

6. Calculations: Read ppm Betz 107 from chart.

CHART FOR DETERMINATION OF BETZ 107 FOR C. T. H_2O - EDC

Reading	ppm, Betz 107	Reading	ppm, Betz 107
0	0	40	81
2	4	42	85
4	8	44	89
6	12	46	93
8	16	48	97
10	20	50	101
12	24	52	105
14	28	54	109
16	32	56	113
18	36	58	117
20	40	60	121
22	44	62	125
24	48	64	129
26	53	66	133
28	57	68	137
30	61	70	141
32	65	72	145
34	69	74	149
36	73	76	154
38	77		

Electrophotometer No. 5011

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-316

1. Test: Phosphate.

2. Sample Point: No. 40 - Cooling Tower.

3. Apparatus and Equipment:

   a. 100 ml. volumetric flask.
   b. 1 ml. pipette.
   c. 5 ml. pipette.
   d. 15 ml. pipette.
   e. Glass funnel.
   f. 41-H filter paper.
   g. Fisher Electrophotometer equipped with 650-A filter.
   h. Hot plate.

4. Reagents:

   a. 1:5 nitric acid.
   b. 10% potassium nitrate.
   c. Ammonium molybdate solution.
   d. Para-methylamino-phenol sulfate solution.
   e. Sodium acetate solution.
   f. Distilled water.

5. Procedure:

   a. Place 5 ml. of the filtered sample of cooling water in a 100 ml. volumetric flask.
   b. Add 3 ml. of 1:5 nitric acid to the sample and dilute to approximately 50 ml.
   c. Digest on hot plate for 30 minutes.
   d. To the cooled sample add:
      (1) 10 ml. of 10% potassium nitrate.
      (2) 2 ml. ammonium molybdate solution.
      (3) 1 ml. of para-methylamino-phenol sulfate.
   e. Allow to stand five minutes.
   f. Add 5 ml. of sodium acetate, and dilute to 100 ml.
   g. Allow to stand three minutes.
   h. Place sample and blank in Electrophotometer cells, and obtain reading using 650-A filter.
   i. For blank solution, place approximately 50 ml. of distilled water and 3 ml. of 1:5 nitric acid. Begin with Step 3.

6. Calculation:

$$\frac{\text{grams } PO_4 \text{ from calibration curve} \times 10^6}{\text{Sample Size}} = \text{ppm } PO_4$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-317

1. Test: Silicon.

2. Sample Point: No. 40 - Cooling Tower.

3. Apparatus:

   a. 100 ml. volumetric flask.
   b. 10 ml. pipette.
   c. Fisher Electrophotometer equipped with 650-A filter.
   d. 5 ml. pipette.

4. Reagents:

   a. HCl solution.
   b. Ammonium molybdate solution.
   c. Sodium sulfite solution.
   d. Distilled water.

5. Procedure:

   a. Pipette 10 ml. of cooling water into a 100 ml. volumetric flask, and dilute to volume with distilled water. Mix well.
   b. Pipette a 10 ml. aliquot of the dilution into a 100 ml. volumetric flask.
   c. Add:
      (1) 5 ml. of HCl solution.
      (2) 5 ml. of ammonium molybdate solution.
   d. Allow to stand for one (1) minute.
   e. Add 10 ml. of sodium sulfite solution, and dilute to 100 ml. with distilled water. Mix.
   f. Let stand one (1) minute, but not over three (3) minutes.
   g. Place sample and blank in electrophotometer cells, and obtain reading using 650-A filter.
   h. A blank is prepared using 10 ml. of the diluted sample, and all reagents except the ammonium molybdate reagent.

6. Calculations:

$$\frac{\text{grams of } SiO_2 \text{ from calibration curve} \times 10^6}{\text{Sample Size}} = \text{ppm } SiO_2$$

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-318

Operating instructions for Beckman Model H-2 pH meter.

1. Apparatus: One Model H-2 Beckman pH meter.

2. Procedure:

   a. Range switch should be in the neutral position.
   b. Temperature compensator knob should always be set at 27°C.
   c. Meter needle should always coincide with check pointer; if not, adjust with standardization knob, making sure range switch is in the neutral position.

3. pH Measurements:

   a. With switch in the neutral position, rinse electrodes and beaker with sample to be run.
   b. Make sure needle and check pointer coincide; adjust with standardization knob if necessary.
   c. Fill beaker half full with sample and switch to proper range.
   d. Allow needle to come to rest and take a reading. Then turn switch back to neutral position.
   e. Rinse electrodes with distilled water and wipe with Kimwipes.
   f. Immerse electrodes in distilled water.

4. Precautions:

   a. Keep range switch on neutral position whenever electrodes are removed, or lowered into solutions, then check to see if check pointer and meter needle coincide.
   b. Calomel electrode should never be less than half full of saturated KCl solution, and should be replaced if inner part is cracked or broken.

5. Standardization: Use procedure outlined on front of meter.

6. Replacement parts and solutions:

   a. Glass electrode - Laboratory Stock No. 1074-G.
   b. Calomel electrode - Laboratory Stock No. 1074-C.
   c. pH7 Buffer solution - Laboratory Stock No. 6096.
   d. Saturated KCl solution - Laboratory Stock No. 6350.

EDC ANALYTICAL CONTROL TEST
METHOD NO. M-318-A

Operating instructions for the Taylor pH Slide Comparator.

1. Apparatus:

   a. Taylor pH Slide Comparator.
   b. pH Slide - Range 5.2 to 6.8 (Chlorophenol red).
   c. pH Slide - Range 6.0 to 7.6 (Bromthymol blue).

2. Reagents:

   a. Chlorophenol red indicator solution.
   b. Bromthymol blue indicator solution.

3. Procedure:

   a. Remove the top from the base. Fill the three test tubes to the 5 ml. mark with the sample to be tested. Replace them in the holes back of the three slots in the base.
   b. To the middle tube, add 0.5 ml. of indicator solution (select solution for range expected) with calibrated dropper. (CAUTION: Avoid contact of the dropper with the sample.) Mix thoroughly.
   c. Place the color standard slide (select proper range to match indicator solution) on the base. Hold the instrument toward a light. Move slide in front of the test samples until a match is obtained. Read pH directly from values on slide.
   d. If match is not obtained, repeat Steps a, b and c using the other indicator and slide.

VIII. PRODUCT STORAGE FACILITIES

A. Storage Tanks

Six tanks are located north of the process area. Four of these have a capacity of 25,000 gallons each. The other two are 60,000 gallon capacity tanks. Two of the small tanks (No. 1 and the crude storage tank) and the two large tanks are used for product storage. The other two are used to store still bottoms from the EDC (still bottoms tank) and MC (No. 2) plant until they can be purified in the flasher system and sent to Per-Tri as feed.

Additional storage tanks are located at the dock storage area. These two tanks holding approximately 500,000 gallons each are used for final storage before barge boading.

The tanks are padded with nitrogen to give an inert gas for tank breathing which occurs during level and temperature changes in the tanks. This pad prevents a flammable mixture in the vapor space of the tank and prevents product contamination by damp air. This pad is maintained at 1.5 ounces pressure. A pressure vacuum relief valve provides for safety relief in event of pad system failure.

All storage tanks are equipped with a Varec float type level indicator which shows the tank level to 1/16" on the outside of the tank. This reading will be used for product inventory purposes. The area transfer tanks also have their levels transmitted to the control building and indicated on the panel board. These are indicated in percent of tank capacity and are to be used only as an indication of the level each tank contains. The tanks have high and low level alarms.

All storage tanks are surrounded by a dike. The volume enclosed by the dike is more than enough to hold the contents of the tanks within it. The purpose of the dike is to confine the contents of the tanks, in the event of a rupture, and limit the area which is exposed to possible fires.

All lines entering the EDC storage tanks enter at the bottom. It is possible to generate a static electricity charge when a liquid is allowed to fall through a vapor space. All tanks and pipe lines are equipped with electrical grounds to eliminate the chance for a potential to build up.

B. Product Lines

The EDC product goes from the process area to a header arrangement around the transfer pumps. The piping is arranged so that the product can be sent to either of the four transfer tanks. The header also serves as the suction manifold for the transfer pumps such that product can be transferred from either transfer tank to dock storage or the area shipping department. Care should be taken when pumping out of these tanks or switching production from one tank to another as off-spec product and inventory difficulties may result if a wrong valve is opened.

Several special arrangements at the transfer pump header allow a variety of transfer possibilities. The product from the No. 1 transfer tank can be pumped directly to the MC plant via the No. 1 still feed pump. The crude tank and No. 4 tank contents can be transferred directly to the docks or to Per-Tri as feed. In addition EDC can be transferred in either direction between the No. 1 and No. 2 plant transfer tanks.

The dock transfer line goes from the transfer pumps along the pipe rack to the Area B shipping building and across the Columbia Southern road. After crossing the road the line continues underground to the dock storage area where it enters both the dock storage tanks. A line comes from each of the dock storage tanks to the suction of the barge loading pumps. The inlet and outlet of the dock storage tanks are tied together through the barge loading pumps such that the contents of the tanks can be circulated.

EDC from the dock transfer line can be returned to the plant at the reactor, gas separator, or neutralizers for reworking purposes.

IX. CATALYST SOLUTION

A. General

The need for ferric chloride ($FeCl_3$) catalyst addition to the reactor will be determined by examinating laboratory analytical results of the $FeCl_3$ content of the reactor liquid. The reactor will normally be operated with a $FeCl_3$ concentration of 0.3 to 0.4 wt. %. When this value drops below 0.3% $FeCl_3$, a catalyst addition will be necessary. This should occur only occasionally.

To add catalyst, a master mix is prepared (see Fig. 14.5) that will then be added to the reactor to raise the iron concentration to the desired 0.4 wt. % $FeCl_3$ (see Fig. 14.4).

B. Preparation

When a catalyst addition is necessary, the quantity of $FeCl_3$ needed to raise the concentration to 0.4% is first determined by Figure 14.4. Figure 14.5 is then used to determine the amount of EDC needed for the master mix. Add the amount of EDC that is determined from Figure 14.5 to the rework kettle. This EDC is taken from the bottom of the reactor or the reactor recycle. After the EDC is in the kettle, line up the dump tank pump to pump from the bottom of the rework kettle back into the top. Start circulating the EDC and add the required catalyst through the kettle manhole.

C. Addition

After the catalyst solution is thoroughly mixed, it can be pumped into the reactor via the dump tank pump.

X. REACTOR DUMPING

A. Full Dump

In the event the contents of one of the reactors become contaminated, an emergency dump of a reactor is necessary; or, if a reactor requires dumping for maintenance purposes, the reactor liquor can be readily transferred to the dump tanks. There is a dump tank for each of the reactors. The dump tank capacities are approximately 10,000 and 6,000 gallons for number one and two tanks respectively. Since the normal reactor charges are 8772 and 3078 gallons (24 and 18 ft.) for number one and two reactors, the tanks will hold the full reactor charges.

To drain the reactor to the dump tank:

1. Open the valves between the reactor bottom and the dump tank except the bottom reactor valve.

2. Provide a supply of N_2 to the reactor to prevent pulling a vacuum on the reactor as it is drained.

3. Vent the dump tank to the reactor.

4. Open the bottom reactor valve and drain the reactor as much as possible by gravity flow.

5. Drain the reactor slowly to allow the dump tank to vent. Complete the dump by using the reactor dump tank pump.

B. Periodic Dump

It is anticipated that after an extended period of operation, the reactor liquid will accumulate a high concentration of tars and inactive catalyst. When this occurs, a purge stream will be withdrawn from the reactor to either the dump tank or the rework kettle. This material will be evaporated from the rework kettle into the No. 2 reactor vapor space to recover the EDC. The quantity of purge required or the frequency necessary will be dictated by operating experience. Past experience has shown a tar production of approximately 0.5 lbs./ton of EDC produced.

C. EDC Recovery

To recover the EDC, the reactor purge stream or any EDC requiring clean-up will be taken to the rework kettle from either the reactor or the dump tank. If the material was originally collected in the dump tank, the dump tank pump is used to make the transfer.

The following procedure should be followed when operating the rework kettle:

1. Fill the kettle to approximately the top edge of the jacket.

2. Turn on the steam supply. A pressure control valve is provided for the rework kettle steam control.

3. Open the kettle vent line and boil the EDC out of the kettle into the No. 2 reactor vapor space. Here the vapor will be condensed and fed to the stripper with the normal reactor vapor stream.

4. As the temperature of the vapor line from the rework kettle climbs above 185-195°F, turn off the steam to the kettle and close the vent line. The tars in the bottom of the kettle can then be dumped into drums for disposal. Be sure to dispose of these tars while they are hot and non-viscons. Do not let tars cool and harden in the kettle.

XI. SAFETY FOR THE ETHYLENE DICHLORIDE AREA

A. General

Due to the nature of the operation and the chemicals involved, the Ethylene Dichloride Area is restricted to authorized personnel only.

Company vehicles will be permitted to travel the main approach road during normal operations. Special permits will be required for vehicles to enter any of the other areas in this department.

The control building is pressurized for safety precautions, therefore it is to be utilized by authorized personnel only.

B. Definitions

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

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

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

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

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

C. Chemicals in the EDC Area

The chemicals encountered in manufacturing ethylene dichloride are listed below:

1. Chlorine
2. Ethylene
3. Ferric Chloride
4. Caustic Soda
5. Calcium Chloride

6. Sulfuric Acid

7. Natural Gas (Methane)

8. Inert Gases (Nitrogen)

9. Freon ~~22~~ 12

10. Ethylene Dichloride (EDC)

11. Hydrogen Chloride

12. 1,1,2 - Trichloroethane (TCE)

13. Sym. and Unsym. Tetrachloroethane

Some of the properties and safety hazards of these chemicals are given on the following pages.

NAME: Chlorine

FORMULA: Cl_2

MOLECULAR WEIGHT: 70.91

BOILING POINT: -30.1°F

MELTING POINT: -149.8°F

VAPOR PRESSURE: @ 23.9°C, 4,760 mm. Hg.

LIQUID DENSITY: @ 25°C, 1,391 g/cc

RELATIVE VAPOR DENSITY: 2.45 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 0.35 to 2 ppm

DETECTABLE ODOR CONC.: 3.5 ppm

HAZARDOUS PROPERTIES: Chlorine is an extremely powerful respiratory irritant. Because of its pungent odor and irritating effect, traces of chlorine in air are readily detected. Chlorine is not poison, but kills or burns tissues. It has no cumulative effect nor does repeated exposure produce immunity. In an atmosphere containing chlorine, short, shallow breathing should be used.

Liquid chlorine and high concentrations of gas in contact with the skin will cause irritation and blistering of the exposed area. Clothing contaminated with liquid chlorine should be removed immediately and the exposed areas flushed with water.

TREATMENT: Remove the person from the contaminated area and call for help or have the person report to First Aid, depending upon the severity of the circumstances. Administer artifical respiration if necessary.

Clothing contaminated with chlorine should be removed immediately and the exposed areas washed thoroughly with water.

All persons working with chlorine should be acquainted with the proper methods of handling this commodity, the use of gas masks, and emergency procedures.

Chlorine leaks can be located by wetting the suspected area with ammonia water. Dense, white fumes will indicate the point of leakage. Leaks must be given immediate attention because they become progressively worse. If the leak is extensive it should be reported so that persons in the path of the fumes can be warned. Chlorine is about 2½ times as heavy as air; therefore, it collects in low spots and stays near the ground.

NAME: Ethylene

FORMULA: CH_2CH_2

MOLECULAR WEIGHT: 28.052

BOILING POINT: -155°F

MELTING POINT: -272.9°F

VAPOR PRESSURE: @ 8.9°C, 38,000 mm. Hg.

LIQUID DENSITY: Normally a Gas

RELATIVE VAPOR DENSITY. 0.98 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: 3% to 34%

MAX. ALLOWABLE CONC.: 100 to 500 ppm

HAZARDOUS PROPERTIES: Ethylene is a flammable gas. It is a dangerous explosive hazard upon exposure to heat or flame. Carbon dioxide or dry chemical fire extinguishers should be used to put out an ethylene fire.

Ethylene is a colorless gas, but can be detected by its characteristic sweetish odor. It is not toxic but will result in asphyxiation by depleting the oxygen content of the air.

TREATMENT: A person that has been exposed to ethylene should be removed from the contaminated area. Administer artifical respiration if necessary.

NAME: Ferric Chloride

FORMULA: $FeCl_3$

MOLECULAR WEIGHT: 162.22

BOILING POINT: 606.2°F

MELTING POINT: 539.6°F

VAPOR PRESSURE: @ 194°C, 1 mm. Hg.

DENSITY: @ 11°C, 2.804 g/cc

FLASH POINT: None

EXPLOSIVE LIMIT: None

SUBLIMATION POINT: 572°F

HAZARDOUS PROPERTIES: Handling ferric chloride presents no particular problem. It will seriously stain clothing and skin so it is advisable to wear protective clothing when handling it.

In 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 be consulted in such cases.

NAME: Caustic Soda (Sodium Hydroxide)

FORMULA: NaOH

MOLECULAR WEIGHT: 40.0

BOILING POINT: 2534°F

MELTING POINT: 605°F

VAPOR PRESSURE: @ 739°C, 1 mm. Hg.

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 2 mg./m.^{3} of Air

HAZARDOUS PROPERTIES: Caustic attacks any body tissue upon contact. The degree of injury depends upon the extent and duration of contact and the temperature and concentration of the material. Caustic solution is detectable by a slippery feeling such as that of soapy water. Its corrosive action on tissue causes burns and frequently deep ulceration, with ultimate scarring. Contact with the eyes rapidly causes severe damage.

TREATMENT: Immediately remove all contaminated clothing and equipment, then flush the exposed area thoroughly with an abundance of water. If caustic contacts the eyes, they should be irrigated immediately with an abundant amount of water for at least 15 minutes. Hold the eyelids apart during irrigation. The eye should then be washed with a mild saline solution; if such solution is available. Wash the eye with water for an additional 15 minutes. A physician should be seen immediately after the eye is thoroughly washed.

NAME: Calcium Chloride

FORMULA: $CaCl_2$

MOLECULAR WEIGHT: 110.99

BOILING POINT: 2912°F

MELTING POINT: 1422°F

DENSITY: 2.512 g/cc @ 25°C

FLASH POINT: None

EXPLOSIVE LIMITS: None

HAZARDOUS PROPERTIES: The hydration of calcium chloride is exothermic. Thus, when driers are being flushed out with water there is the possibility of heat and some splashing. If calcium chloride enters the eye(s), a sustained water flush will be effective in removing the material.

NAME: Sulfuric Acid

FORMULA: H_2SO_4

MOLECULAR WEIGHT: 98.08

BOILING POINT: 626°F

MELTING POINT: 50.9°F

VAPOR PRESSURE: @ 146°C, 1 mm. Hg.

LIQUID DENSITY: @ 25°C, 1.834 g/cc

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 1 mg./m.^{3} of Air

DETECTABLE ODOR CONC.: 0.12 ppm

HAZARDOUS PROPERTIES: The greatest hazard of sulfuric acid is physical contact. Because of its affinity for water, it dries tissue; creating heat, and resulting in a painful slow healing burn. Repeated skin contact with diluted sulfuric acid solution can cause dermatitis.

Sulfuric acid reacts with water to produce heat. The acid should always be added to water upon dilution, never vice versa.

TREATMENT: Immediately remove all contaminated clothing and equipment. Flush the exposed area with an abundance of water. After thorough irrigation with water, applications of mild alkaline solutions may be used. Do not apply oils or ointments to burned area without instructions from a physician. If the eyes are involved they should be immediately irrigated with water for at least 15 minutes. Report to First Aid for further treatment if necessary.

NAME: Methane

FORMULA: CH_4

MOLECULAR WEIGHT: 16.04

BOILING POINT: -258.7°F

FREEZING POINT: -297.8°F

VAPOR PRESSURE: @ -82.1°C, 34,900 mm. Hg.

RELATIVE VAPOR DENSITY: 0.555 (Air 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: 5.3% to 14.0%

DETECTABLE ODOR CONC.: Odorless

HAZARDOUS PROPERTIES: Methane is a simple asphyxiant and a dangerous fire and explosion hazard. Methane fires should be fought with carbon dioxide or dry chemical extinguishers.

NAME: Freon-22 (Chlorodifloromethane)

FORMULA: $CHClF_2$

MOLECULAR WEIGHT: 86.46

BOILING POINT: -41.4°F

MELTING POINT: -230.8°F

VAPOR PRESSURE: @24°C 7,600 mm. Hg

RELATIVE VAPOR DENSITY: 3.87 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. 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 open flames to come in contact with Freon-22 or any vessels containing it.

NAME: Ethylene Dichloride (EDC) (1,2-Dichloroethane)

FORMULA. CH_2ClCH_2Cl

MOLECULAR WEIGHT: 98.966

BOILING POINT: 182.3°F

MELTING POINT: -31.9°F

VAPOR PRESSURE: @ 29 4°C, 100 mm. Hg.

LIQUID DENSITY: @ 20°C, 1.257 g/cc

RELATIVE VAPOR DENSITY. 3.55 (Air = 1.0)

FLASH POINT: 18.3°C open cut

EXPLOSIVE LIMITS: 6.2 to 15.9%

MAX. ALLOWABLE CONC.: 50 ppm, 200 mg./m.^{3} of Air

SL 008680

DETECTABLE ODOR CONC.: Much less than "MAC"

HAZARDOUS PROPERTIES: Ethylene dichloride has a distinctive odor which gives warning of its presence in relatively safe concentrations. It irritates the eyes and upper respiratory passages.

Short exposures to high concentrations of ethylene dichloride cause irritation of the eyes, nose and throat, followed by dizziness, nausea, mental confusion, depression, rapid pulse, and loss of consciousness in severe cases.

Chronic poisoning, where exposure occurs over an extended period, may cause loss of appetite, nausea, low blood sugar levels, and possibly dermatitis if there has been skin contact.

Employees exposed regularly to ethylene dichloride should be examined periodically by a physician acquainted with the occupational hazards involved. Physical examinations should be required when any symptoms of poisoning occur.

Employees who may be subjected to severe exposures to ethylene dichloride vapors should be provided with eye and respiratory protection.

TREATMENT. Immediately remove all contaminated clothing and equipment. All affected areas should be washed thoroughly with water and soap. After washing, an ointment containing lanolin should be applied to replace the natural skin oils. For serious or persistent cases of skin trouble and signs of generalized poisoning a physician should be consulted.

If EDC enters the eyes wash them promptly with copious quantities of water for at least 15 minutes. Medical attention should be obtained in all cases of eye contact.

A person showing symptoms of EDC vapor poisoning should be promptly removed from the contaminated area. In case breathing has stopped artificial respiration should be started immediately. Oxygen should be administered if oxygen inhalation equipment and a qualified operator are available If the patient is conscious, hot tea or coffee may be given as a stimulant. A physician should be called at once.

If EDC is swallowed vomiting should be induced at least three times followed by the administration of a tablespoonful of epsom salts.

Water, foam, carbon dioxide dry chemical or carbon tetrachloride should be used to fight ethylene dichloride fires.

NAME: Hydrogen Chloride

FORMULA: HCl

MOLECULAR WEIGHT: @ 36.47

BOILING POINT: -120.6°F

MELTING POINT: -173.7°F

VAPOR PRESSURE: @ 17.8°C, 3040 mm. Hg.

LIQUID DENSITY: Normally a Gas

RELATIVE VAPOR DENSITY: 1.268 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 10 ppm (for 10 Hr. Day)

DETECTABLE ODOR CONC.: Unknown

HAZARDOUS PROPERTIES: Anhydrous hydrogen chloride is a gas which has a corrosive action upon the skin and mucous membranes. It will cause rapid and severe burns and is particularly dangerous to the eyes. The vapors are irritating and will drive you away from the contaminated area before they are dangerous to you.

Hydrogen chloride 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: Immediately remove all contaminated clothing and equipment and flush exposed areas with large quantities of water. No attempt should be made to neutralize the acid with alkaline solutions. Report to First Aid if necessary.

NAME: 1,1,2-Trichloroethane (TCE)

FORMULA: $CHCl_2CH_2Cl$

MOLECULAR WEIGHT: 133.415

BOILING POINT: 236.8°F

FREEZING POINT: -31°C

VAPOR PRESSURE: @ 23.9°C, 22 mm. Hg.

LIQUID DENSITY: @ 25°C, 1.4319 g/cc

RELATIVE VAPOR DENSITY: 4.6 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 25 to 100 ppm

DETECTABLE ODOR CONC.: Unknown

HAZARDOUS PROPERTIES: Trichoroethane can cause burns of the eyes and has a harmful effect upon the liver. All contact with the eyes and skin should be avoided. This material should be handled with caution, because its toxicological properties have not as yet been adequately evaluated.

TREATMENT: Remove patient from the toxic area. Remove all contaminated clothing and wash all exposed skin thoroughly with soap and water. Flush eyes with copious quantities of water. Report to First Aid if necessary.

NAME: Symmetrical and Unsymmetrical Tetrachloroethane (Sym. & Unsym. TeCE)

FORMULA: $CHCl_2CHCl_2$ and CCl_3CH_2Cl

MOLECULAR WEIGHT: 167.864

BOILING POINT: 292.2°F and 266.9°F

MELTING POINT: -46.8°F and -94.4°F

VAPOR PRESSURE: @ 23.9°C, 4 mm. Hg. and 13 mm. Hg.

LIQUID DENSITY: @ 25°C, 1.588 g/cc and 1.533 g/cc

RELATIVE VAPOR DENSITY: 5.78 (Air = 1.0)

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAX. ALLOWABLE CONC.: 5 ppm

SL 008684

DETECTABLE ODOR CONC.: Approximately 5 ppm

HAZARDOUS PROPERTIES: The tetrachloroethanes are not flammable or explosive, but are the most toxic of the chlorinated ethanes that will be handled in the plant. They are toxic by inhalation, prolonged and repeated contact, or by oral intake. Prolonged or repeated exposures to TeCE in any form are hazardous.

Subacute poisoning is the form usually encountered. This develops gradually as a result of prolonged or repeated work in an atmosphere containing more than 5 ppm tetrachloroethane. Poisoning can also occur by TeCE being absorbed through the skin.

For some time, workers with subacute poisoning may show only such symptoms as fatigue, loss of appetite and abdominal pain. At any time more severe symptoms such as vomiting, dizziness, pain over the liver, and jaundice may occur. The symptoms of TeCE poisoning are due to systemic poisoning characterized by damage to the liver, kidneys, heart, blood cells, and nervous system.

TeCE may cause dermatitis after repeated or prolonged contact with the skin. The skin becomes rough, red and dry due to the removal of skin oils. Eye contact can cause serious damage if immediate care is neglected.

The first symptoms after toxic a unts of TeCE are taken by mouth are irritation of stomach, vomiting diarrhea with bloody stools. It is absorbed very rapidly and even mall amount produce unconsciousness and a deep flushing of the in

TeCE will be encountered in concentrated amounts in the still bottoms recovery streams.

TREATMENT: Most important in the case of any poisoning is quick removal from exposure. This means first removing the patient from the contaminated atmosphere and removing the TeCE from the skin, or gastro-intestinal tract, if those areas are involved. The patient should be kept quiet and comfortably warm.

A physician should be called immediately and told briefly and clearly what has happened.

If breathing has stopped as a result of TeCE vapor poisoning, artificial respiration should be started immediately. Oxygen should be administered if an inhalation apparatus and experienced operator are available. If the patient is conscious, hot tea or coffee may be given as a stimulant.

If liquid TeCE comes in contact with the skin the exposed area should be washed thoroughly with water and soap. After this an ointment containing lanolin should be applied to replace the natural skin oils. If TeCE enters the eyes, they should be washed promptly with copious quantities of water for at least 15 minutes. Medical attention should be obtained in all cases involving the eyes.

If TeCE is swallowed vomiting should be induced at least three times by drinking salty or soapy water. If necessary, the patient should be encouraged to stick his finger down his throat to induce vomiting. Following this a tablespoonful of epsom salt dissolved in a glass of water should be given.

D Electrical Equipment

The characteristics of EDC are such that it is classified in Group D, Class I by the U. S. National Electrical Code. Ethylene is classified in Group C, Class I. The method and materials of installation are those recommended by Factory Insurance Association. In general, the installation is Class I, Group D, Division 2. Motors are TEFC, (Totally Enclosed, Fan Cooled) lighting is vapor-tight, and all arcing devices are explosion-proof with seal offs. Since it is pressurized and isolated from the process area, general purpose equipment is used in the Control Building. The laboratory requires explosion-proof installations. Seal-offs are provided in all conduits leaving the Control Building and entering a hazardous area from a non-hazardous area.

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

2. Grounding: Grounding in the EDC plant has been given special attention due to problems peculiar to the handling of hydrocarbons and location of the process area.

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 followed to see that the ground has been connected.

Due to the tendency for hydrocarbons to build up a static electricity charge as a result of movement, agitation, or free fall through a gas space, a system of jumpers for pipe flanges has been installed to provide metallic continuity of the piping system. All vessels are grounded at two points. The results is a system of lines 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.

The nearness of the radio transmitting station north of the process area presents a problem. 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 still approaches this condition. The grounding system on the still should eliminate personal hazard and to some degree, act as a shield to equipment south of the still.

A particular hazard is created by the use of cranes with long booms. Under certain conditions, depending on the 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 hooks touch a grounded object.

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

E. Tools

Special spark-proof or alloy tools are not required in the EDC area. However, precautions should be utilized when handling tools and equipment. Do not strike a metal object that could cause a spark in the presence of EDC and air.

F. Pumps and Equipment

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

G. Safety Rules for the EDC Department

The following list of safety rules are applicable to the EDC area. They are to be used in conjunction with the Employee's Safety Manual

The EDC area has been designated as Zone 1o in the present zone system. The Directional Center is located inside the east door of the Control Building. The Emergency Code for this zone is 4-1.

Rules

1. Personnel entering the EDC area will deposit lighters, matches, regular flashlights, etc., at the gate to this area.

2. Smoking will be permitted in the control room only.

3. The usual safety equipment will be required in the EDC area, that is:

a. Safety hats
b. Safety glasses
c. Safety shoes
d Respirators
e. Neoprene-coated gloves

4. The established plant tagging procedure will apply in the EDC area.

5. Company vehicles only will be allowed to travel the road to the Control Building without a permit.

6. A vehicle permit signed by the operating supervisor will be required for any vehicle to go into the process area roadways.

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

a. Welding machines
b. Cutting torches
c. Electrically driven drills
d. Any electrical equipment except explosion-proof flashlights
e. Gasoline driven equipment
f. Lighters, torches or any flame producing equipment
g. Grinders or chipping equipment

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

9. Regular plant utility hoses are not to be used for EDC.

10. All light circuits outside the Control Building must be off and tagged before relamping.

11. Do not dump EDC into trapped sewers or openings where harmful vapors could be evolved.

12. Do not leave open containers of EDC sitting around to give off vapors.

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

14. Never mix chlorine and ethylene in the gaseous phase due to the possibility of a violent reaction.

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

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

17. In case of an emergency warning, all vehicles or equipment that is in the area on permits will be shut off immediately.

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

19. Do not permit ethylene and chlorine to be heated above 300°F. Decomposition and reaction with metals will occur.

20. No smoking or open flames will be allowed in the control laboratory.

21. The laboratory hood fan is to operate continuously.

22. Always put liquid EDC into tanks through standlegs or through bottom nozzles. Falling liquid can generate static electricity.

H. Clearing of Tanks and Process Vessels

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

Clearing Procedure:

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

2. The vapor contents of the tank will be purged with an inert gas.

3. Blinds will be inserted in all connecting lines.

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

5. Purge the equipment with plenty of air.

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

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

43A STD 4-66

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

_____ Unit.

To: _____ Date: _____

Location: _____ Equipment: _____

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

Remarks: _____

Approved: _____

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

_____
Employee's Signature

WELDING OR BURNING PERMIT

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

_____
(Location)

It is good for: Date _____ Time _____ AM PM

To: Date _____ Time _____ AM PM

Special Comments: _____

_____

Signed: _____
(Area Production Supervisor)

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XII. GENERAL DESCRIPTION OF FIRE PROTECTION FACILITIES IN THE EDC AREA

A. General

This section of the manual presents a general description of the fire protection facilities for the EDC area. This information is to be used for general indoctrination of operating personnel and supervisors, but for specific or detailed information concerning any part of the facilities the manufacturer's instruction booklets and drawings should be consulted. All of these booklets and drawings are available in the equipment files in the area supervisors's office, and all of the control valves have instruction booklets attached to the valves themselves.

The Area "B" Fire Protection Manual contains a complete description of the area fire protection facilities.

It should be remembered that all fire protection facilities in the EDC area are provided with water through an 8" cast iron fire main which consists of a single loop surrounding the process area. This loop is connected to the main plant's fire main system by a single 8" cast iron line. There are no fire pumps which provide a supply of fire water specifically for the EDC area alone--the area is totally dependent on the main plant's fire water supply.

The fire protection facilities for the EDC area will be considered in four separate sections: the cooling tower system; the process area system; the transfer tank system including its monitor nozzle station; and the dock storage tank system including its monitor nozzle station.

B. Cooling Tower System

The cooling tower fire protection system is an Automatic Sprinkler Corporation water deluge system which employs their Suprotex Deluge Valve. The system is <u>automatic</u>! The system may be considered to consist of a Controlling Gate Valve, a Suprotex Deluge Valve, a sprinkler piping network, and a Thermo-Pneumatic fire detection system.

The Controlling Gate Valve for the sprinkler system is the main water supply shut-off-valve, and is an indicator post gate valve located in the underground water supply to the cooling tower's sprinkler system. This valve is located due east of the small red fire house by the cooling towers. The Controlling Gate Valve to this sprinkler system does not have an electric Monitor Switch as described in the Automatic Sprinkler Corporation instruction booklets, and hence, no trouble alarm will be sounded if this valve is closed.

The Suprotex Deluge Valve is a mechanically operated valve which withholds fire water from the sprinkler piping until it is unlatched by the dropping of a weight which is held in place by a diaphragm-operated release mechanism. The Suprotex Deluge Valve has an instruction booklet attached which describes its operation and gives sketches of its parts.

The sprinkler piping network is attached to the outlet of the Suprotex Deluge Valve. It conveys the fire extinguishment water into the cooling towers and through open sprinkler heads. (NOTE that these are not fusible heads.) These heads are designed to cover 72 square feet with a density of 0.5 GPM of water per square foot.

The Thermo-Pneumatic system is designed to detect an abnormal rate-of-temperature rise, and by mechanical linkage cause a weight to drop and unlatch the clapper of the Suprotex Valve. This system consists of Heat-Activated Devices (H.A.D.'s) which are hollow shells located in the area to be protected and are connected by 1/8" copper tubing to the release diaphragm of the Suprotex Deluge Valve. The Thermo-Pneumatic system is suprovised with air under approximately 24 ounces per square inch pressure. This air is provided from the EDC area's instrument air system.

In actual operation an abnormal increase in temperature within the H.A.D.'s will cause an increase in pressure in the Thermo-Pneumatic system which will operate the Suprotex Deluge Valve and, thus, admit water to the sprinkler piping at the tower. There is a pull switch located on the Suprotex Deluge Valve which provides for manual operation of the system if desired.

A water motor gong is installed upstream of the Suprotex Deluge Valve. If the system is placed in operation this gong will automatically sound an alarm. A plug cock is installed in the piping to this water motor gong to provide for testing without placing the system in operation.

A trouble alarm is provided for the supervisory air pressure in Thermo-Pneumatic system. This alarm will sound if there is a loss of supervisory air.

CAUTION - If the cover of the release enclosing box on the Surpotex Valve is tampered with, so as to cause air to escape, the Suprotex Deluge Valve will trip and deluge the protected area with water.

Procedures for resetting and operating the Suprotex Deluge Valve are given on page 11 of the instruction book attached to the valve.

Two 15-lbs. CO_2 bottles provide auxiliary protection in the cooling tower area: These are located just to the east and south of the tower foundation. Also, a fire hydrant is located just southeast of the cooling towers with necessary hoses and wrenches located inside the red fire house by the cooling tower.

C.. Process Area System

Process area fire protection is provided by an <u>automatic</u> temperature rate-of-rise system that is very similar in operation to the cooling tower system except that it is a foam system rather than a water deluge system. In this type of system a Suprotex Deluge Valve governs admission of water into the sprinkler piping and another Suprotex Deluge Valve governs admission of

liquid foam into the piping. Pipe line orifices control the ratio of the two liquids. Once in the piping these two liquids flow to the sprinkler heads where they are aerated as they are sprayed into the area to be protected.

The small concrete brick house just north of the process area contains the water Suprotex Deluge Valve, two 250 gal. liquid foam supply tanks - one supply and one alternate supply - a separate nitrogen padding system for each liquid foam tank, the liquid foam Suprotex Deluge Valve, the supervisory air system for the Thermo-Pneumatic system associated with the two Suprotex Deluge Valves, the Controlling Gate Valves for the Suprotex Deluge Valves (in this case they are OS and Y gate valves attached directly to the Suprotex Deluge Valve inlet), and the piping associated with the above systems. The foam and water mixture from the house flows under the roadway and into the sprinkler piping in the process area and into the discharge heads that provide a coverage of 0.16 GPM of solution per square foot (approximately 0.65 GPM of foam). Each head covers approximately 80 square feet.

There are two remote control stations which may be used to manually operate the process foam system. They are located in the northeast and southwest corners of the process area. They have a breakable glass cover with a small hammer. The system may also be operated manually by the pull handles on the water and liquid foam Suprotex Deluge Valves. If the system is energized by using these two pull handles, the water Suprotex Deluge Valve should be operated first.

Several 15-lbs. CO_2 and Ansul dry chemical extinguishers provide additional protection in the process area. One of these is located near the concrete brick house and the others are located throughout the process area. Two portable foam buggies are also located in the process area, and four fire water hydrants are located around the outskirts of the process area to provide protection if necessary.

The process area protection system has a water motor gong and trouble alarm which operate in a similar manner to the alarms on the cooling tower protection system.

The monitor nozzle located north of the process area can be used to provide additional protection for the process area. This will be explained in the following section.

D. Transfer Tank System

The transfer tank area is protected by a manual foam system. Each individual transfer tank has a foam admission chamber located at the top of the tank. Liquid foam and water are supplied to this chamber through a single inlet pipe, and after entering the foam chamber, they are aerated and admitted into the transfer tank.

The foam system for the transfer tanks uses the same sources of supply for water and liquid foam as the process area protection system. As stated in the previous section, these supplies are located in the small concrete brick house just north of the process area, and consequently, the manual control

valves for the transfer tank protection system are located in the same building. Each transfer tank has a valve for water admission and a valve for liquid foam admission. The four water valves and the four foam valves for the tanks are all located on the west wall of the building. They are color coded similar to the individual tank colors, and in addition, a colored piping diagram is mounted on the piping.

In the event of a fire in one of the transfer tanks the water valve and liquid foam valve for the tank in question should both be turned completely open. NOTE: Always open the water valve first. Line orifices will control the quantity of water and liquid foam admitted to the foam chamber on the tank. The system is designed to provide coverage of 0.63 GPM foam solution per square foot (approximately 5 GPM foam).

To secure the system the individual valves in the concrete brick house must be closed. After operation the foam liquid and the water lines should be drained.

A foam monitor nozzle is located just to the west of the concrete brick foam house previously described. This is a manually operated station. Its operation is identical to that of the transfer tanks except that someone must be stationed at the nozzle to direct the stream of foam. NEVER open valves to the monitor nozzle until someone is holding the handles. The control valves for the station are located in the southwest corner of the concrete brick foam house. This station may be used to provide protection in the transfer tank area and in the process area.

It should be noted that the EDC and MC process areas, the transfer tank system, and the monitor nozzle station all use a common source for liquid foam supply - the two 250-gallon, nitrogen padded tanks in the foam house. One of the tanks is the actual supply tank and the other is an alternate tank. Both tanks have sight glasses to enable operators to determine the amount of liquid foam in the tank. As any of the above systems are operated the main supply tank of foam will empty and the operators will have to manually shift to the alternate supply tank. All operators and supervisors should be familiar with this piping arrangement.

E. Dock Storage System

Fire protection facilities in the dock storage area consist of three separate foam systems. Each dock storage tank has an automatic foam system that is identical in operation to the process area foam system. This means that each dock storage tank has its own liquid foam Suprotex Deluge Valve and water Suprotex Deluge Valve. They both have a common liquid foam supply which is a nitrogen padded 300-gallon tank. There is no alternate supply tank. Supervisory air for the two Thermo-Pneumatic systems is provided from the plant instrument air system. All four Suprotex Deluge Valve, Controlling Gate Valves, supervisory air systems, the liquid foam supply, and associated piping are located in the small concrete brick foam house just west of the diked area.

The foam admission chamber on the top of each dock storage tank is slightly different from the chambers on the tops of the transfer tanks. The chambers on the dock storage tanks contain a long rolled tube which rolls down to the EDC liquid level in the tank whenever pressure is applied to the inlet to the foam chamber. After use this tube must be re-rolled manually. The tube serves to convey the foam down to the EDC level without splashing the EDC itself.

The third system at the dock is a monitor nozzle station that is identical in operation to the monitor nozzle station north of the process area. The station at the dock storage area is located due west of the diked area, and its manual control valves are located in the concrete brick foam house. The nozzle uses the same foam supply tank as the storage tank automatic systems.

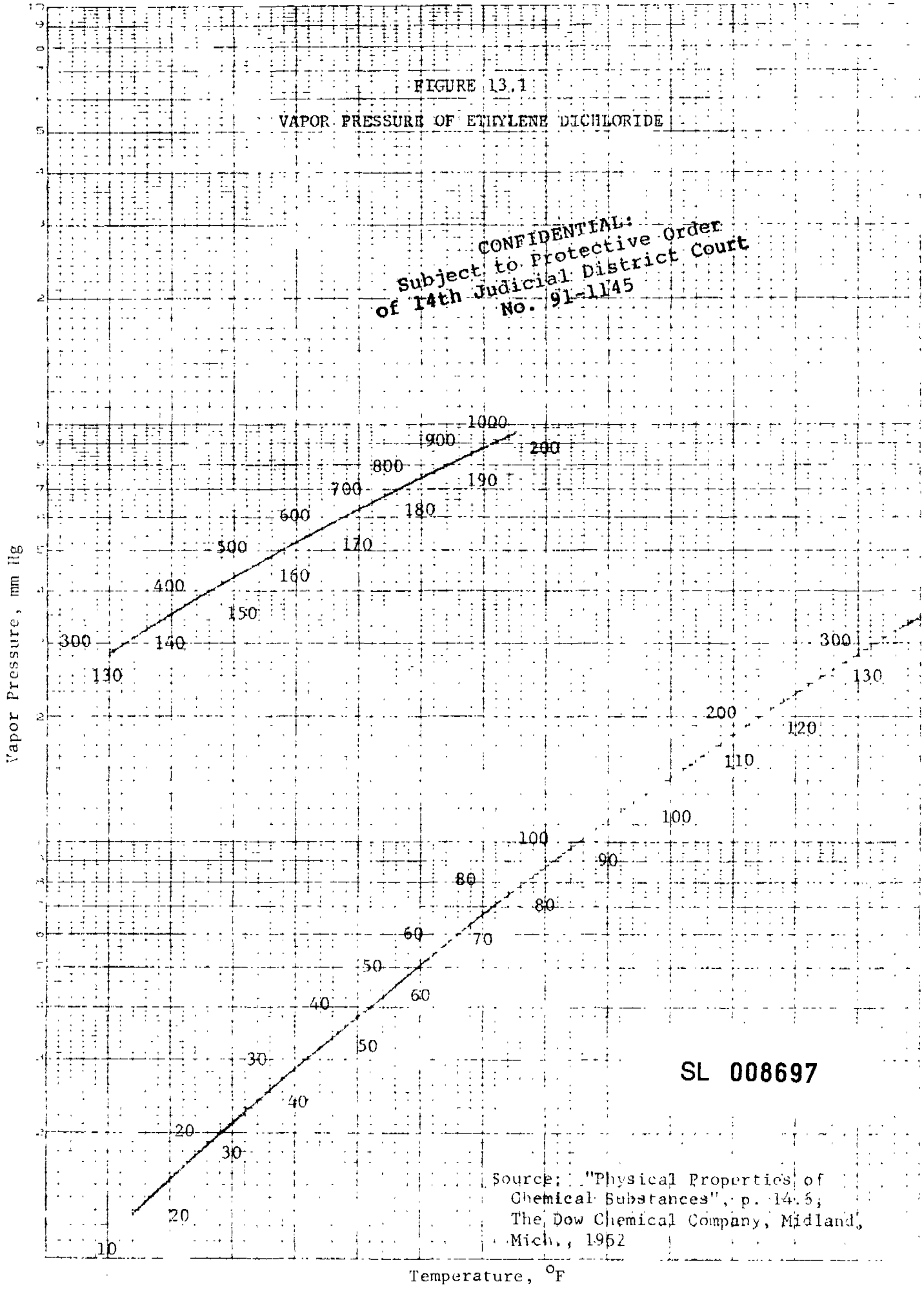
The manual Gate Valves in front of the water Suprotex Deluge Valves are maintained in the closed position. This is done to prevent the accidential tripping of the foam system and thereby ruining valuable product. In the event of a fire, the H.A.D. in the tank will respond and trip the water Suprotex Deluge Valve, which in turn sets off an alarm at the plant guard station The guard in turn notifies the EDC operator and the EDC operator immediately checks into the situation at the Dock Storage. If the fire is only at the vent valve outside the tank, the operator extinguish it with the monitor nozzle. If the fire is inside the tank and on the liquid surface, then the operator should manually open the Gate Valve in front of the Suprotex Deluge Valve which in turn will foam the entire liquid surface within the tank.

XIII. Properties of Compounds

Some physical and thermodynamic properties that may be useful in EDC plant operations are included in this section. The properties included are listed below:

FIGURE 13.1

VAPOR PRESSURE OF ETHYLENE DICHLORIDE



Source: "Physical Properties of Chemical Substances", p. 14.5, The Dow Chemical Company, Midland, Mich., 1952

FIGURE 13.2

Ethylene Dichloride Density

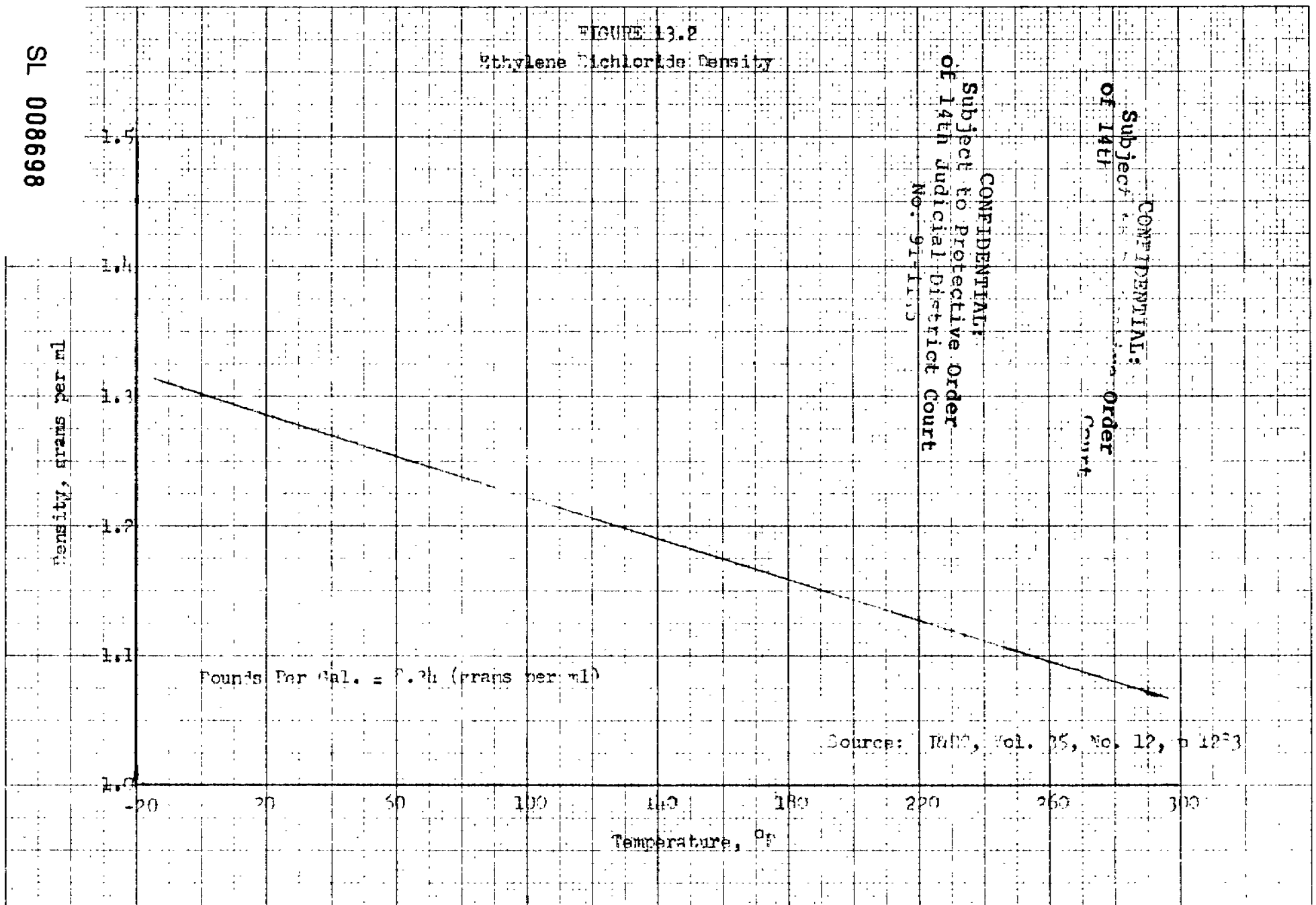


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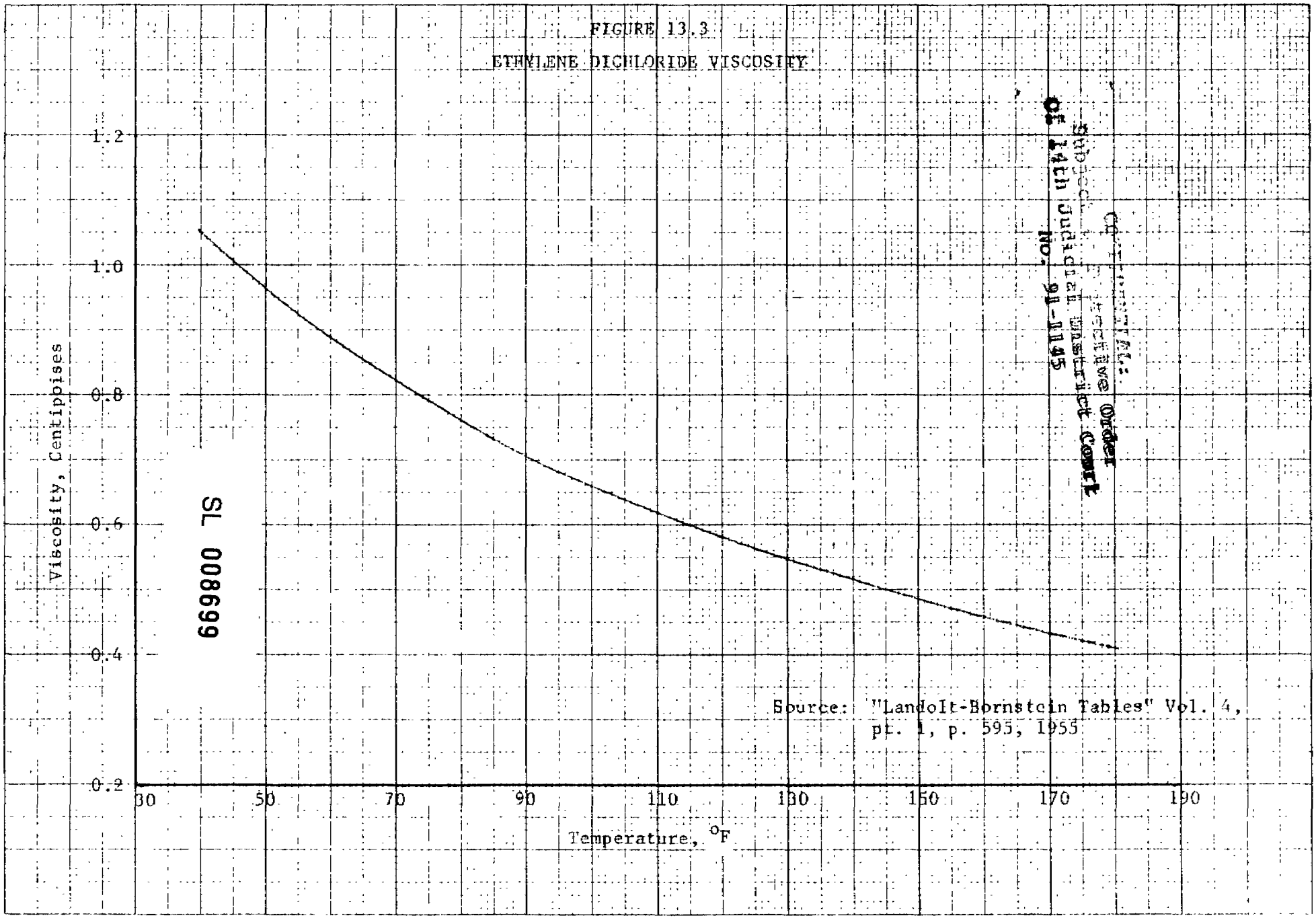
FIGURE 13.3

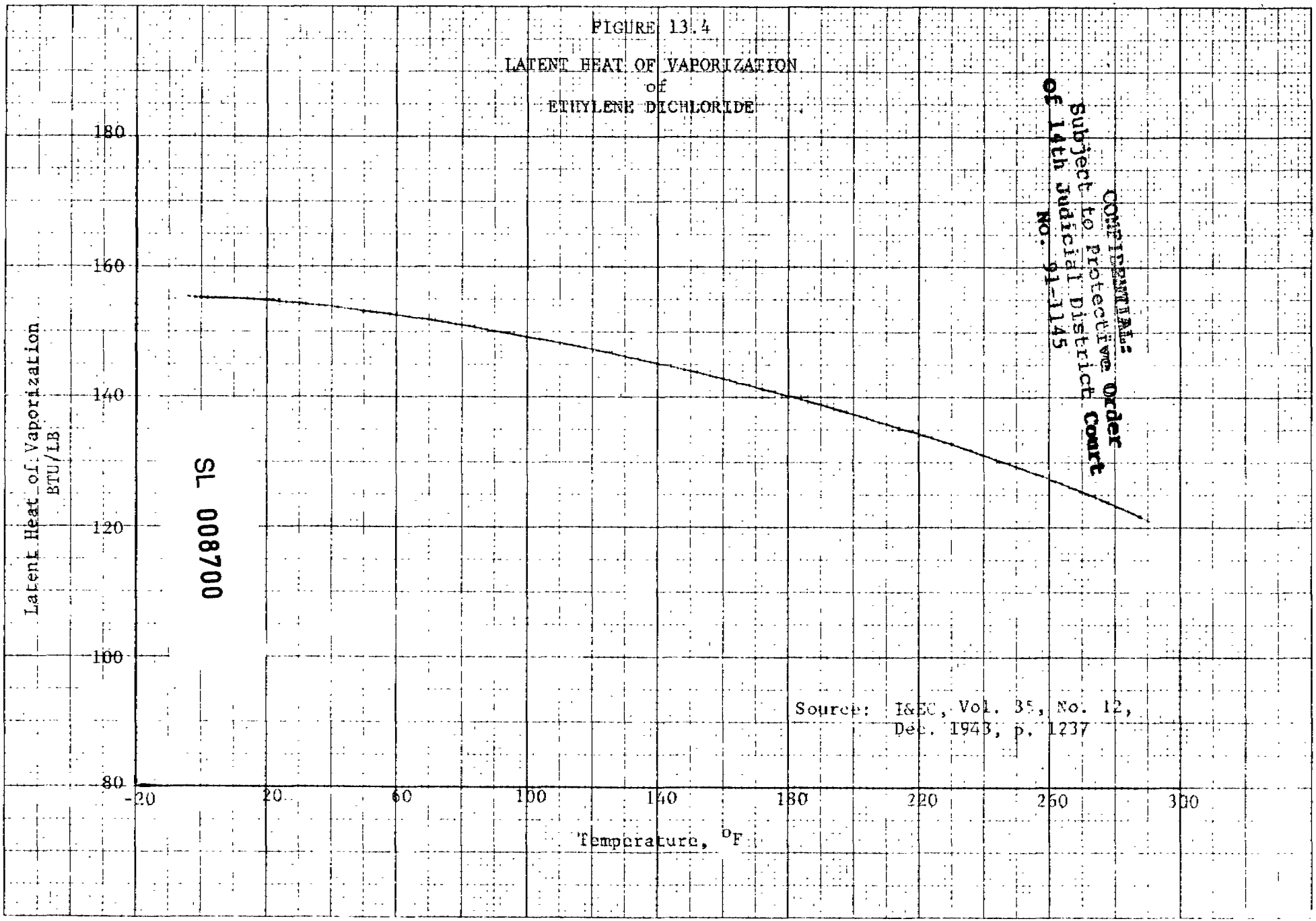
ETHYLENE DICHLORIDE VISCOSITY



Source: "Landolt-Bornstein Tables" Vol. 4, pt. 1, p. 595, 1955

FIGURE 13.4

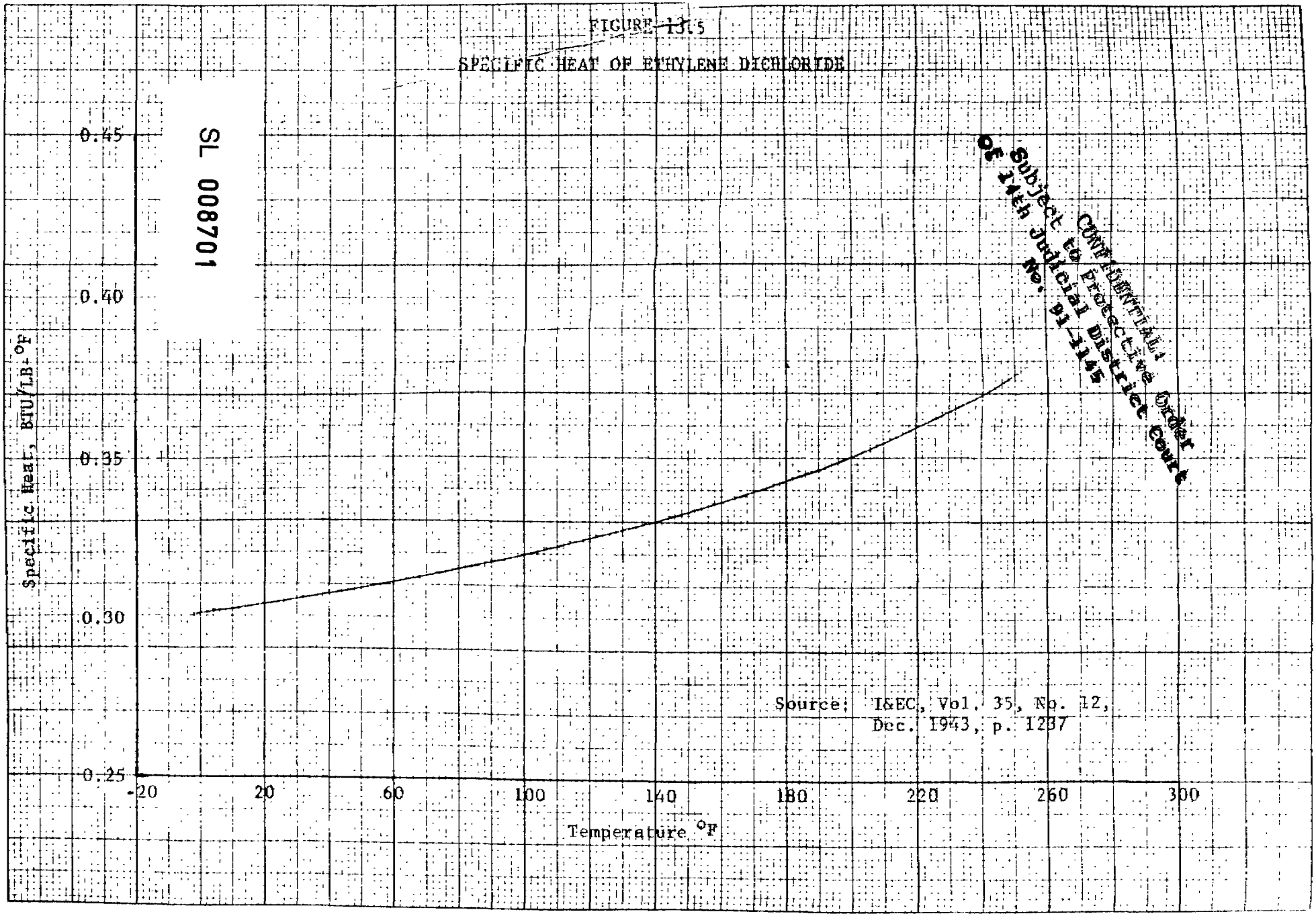
LATENT HEAT OF VAPORIZATION
of
ETHYLENE DICHLORIDE



Source: I&EC, Vol. 35, No. 12, Dec. 1943, p. 1237

FIGURE 13.5

SPECIFIC HEAT OF ETHYLENE DICHLORIDE



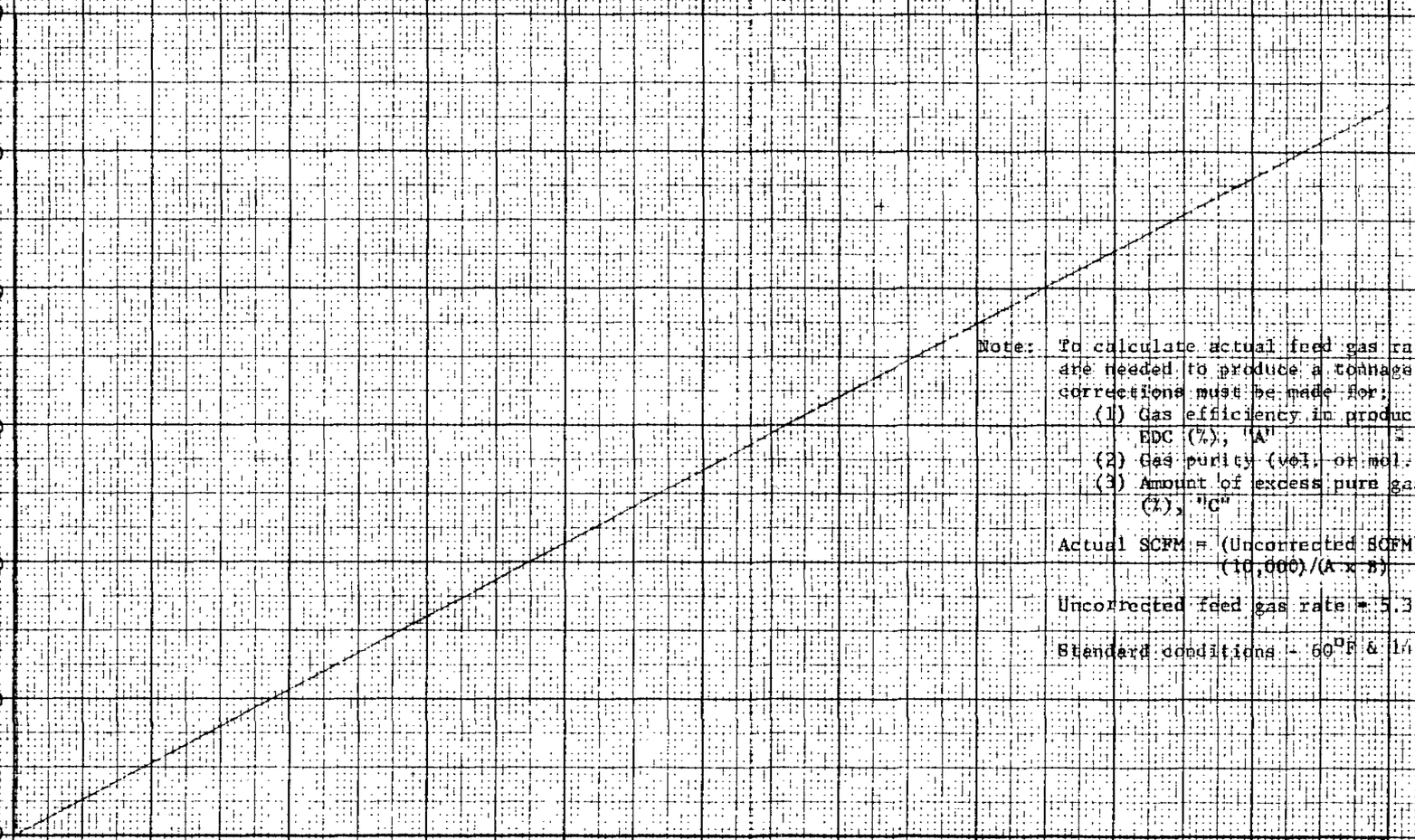
Source: I&EC, Vol. 35, No. 12, Dec. 1943, p. 1287

XIV. OPERATIONAL GRAPHS

This section contains graphs that will be useful in plant operations. The graphs included are listed below:

FIGURE 14.1

FEED GAS RATES versus EDC PRODUCTION



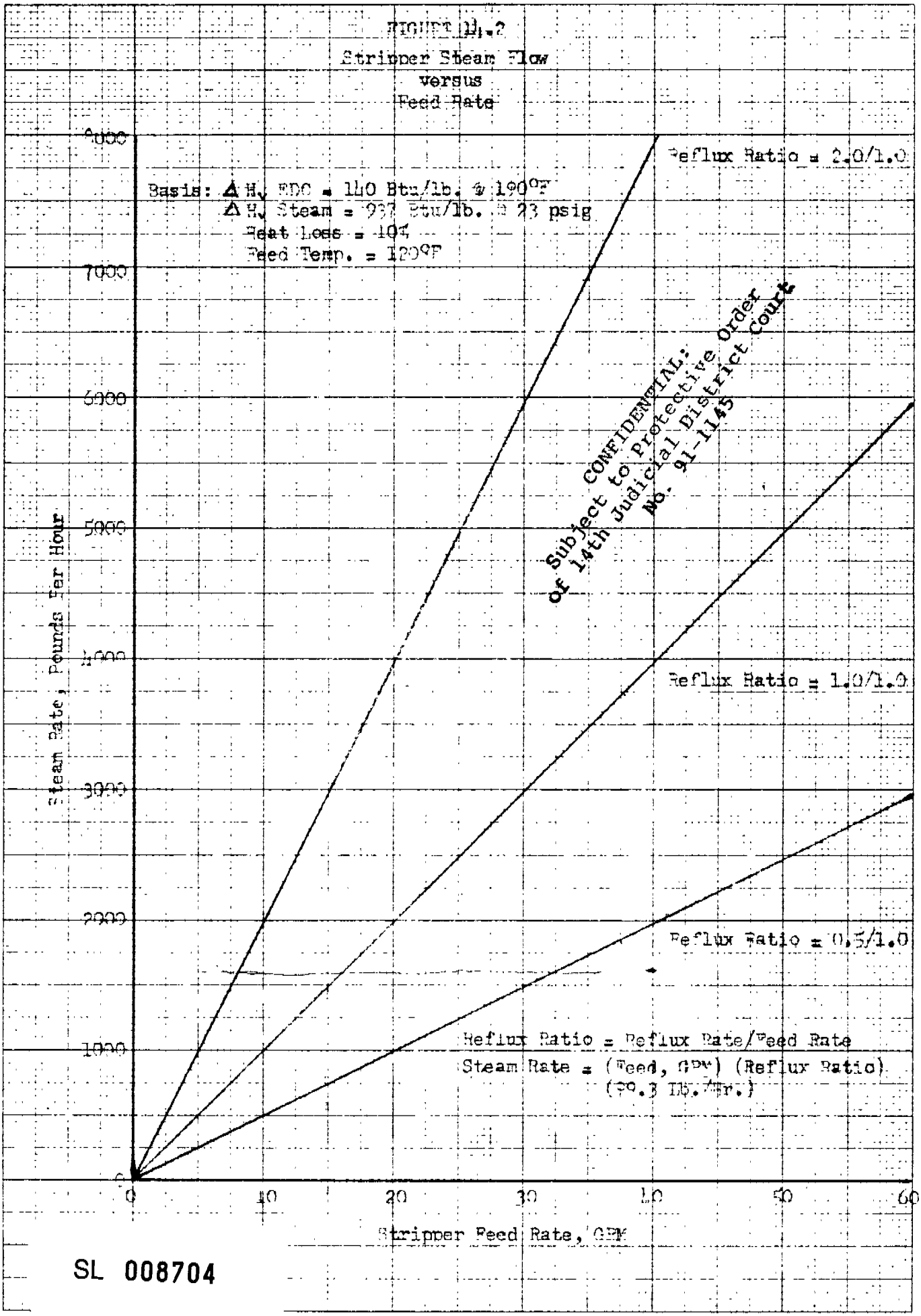
Note: To calculate actual feed gas rates that are needed to produce a tonnage of EDC, corrections must be made for:

(1) Gas efficiency in producing EDC (%), "A"
(2) Gas purity (vol. or mol. %), "B"
(3) Amount of excess pure gas desired (%), "C"

Actual SCFM = (Uncorrected SCFM)(1 + C/B)
(10,000)/(A x B)

Uncorrected feed gas rate = 5.32 (TPD EDC)

Standard conditions - 60°F & 14.7 psia



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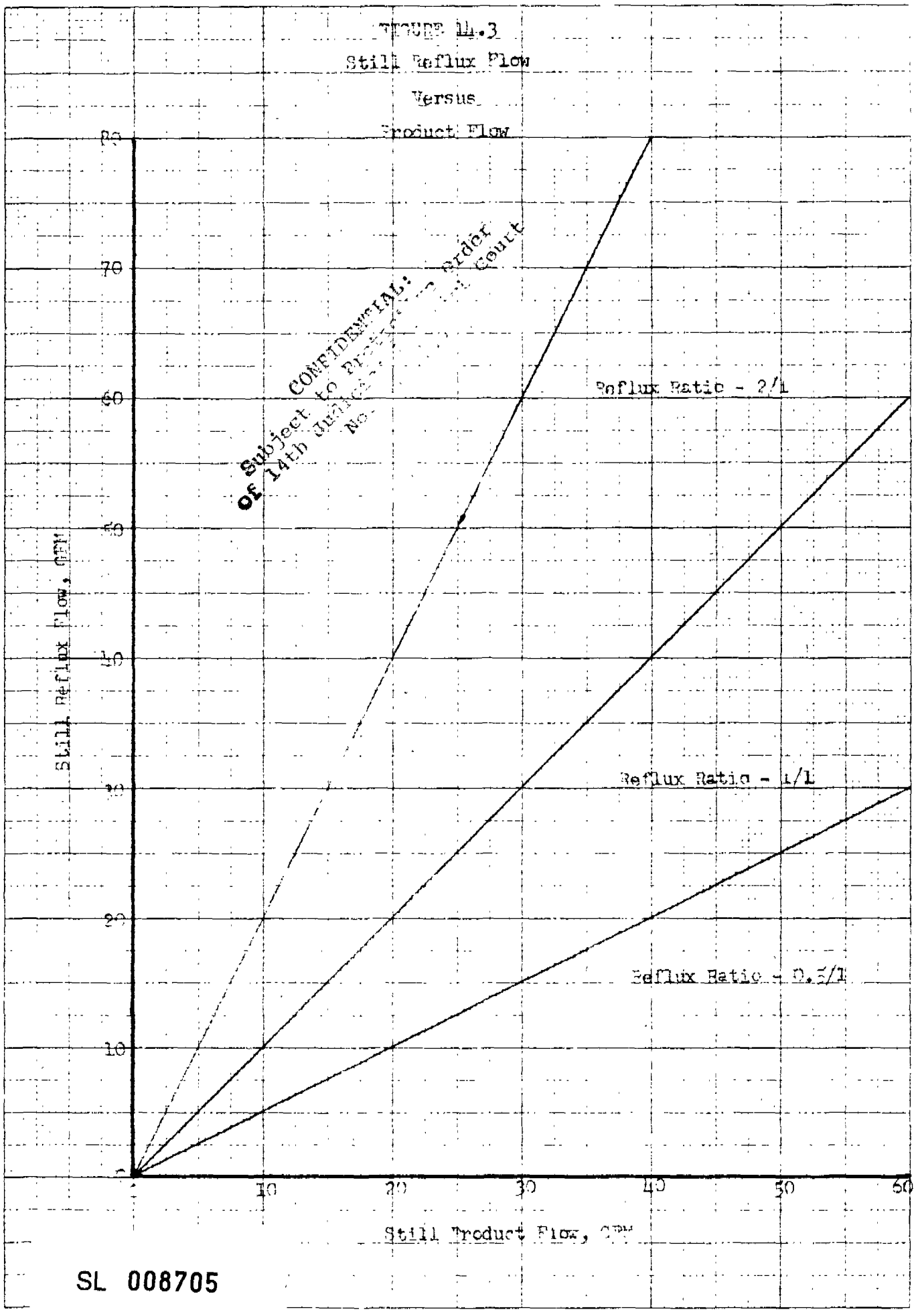


FIGURE 14.4

FERRIC CHLORIDE REQUIRED FOR
CATALYST ADDITION

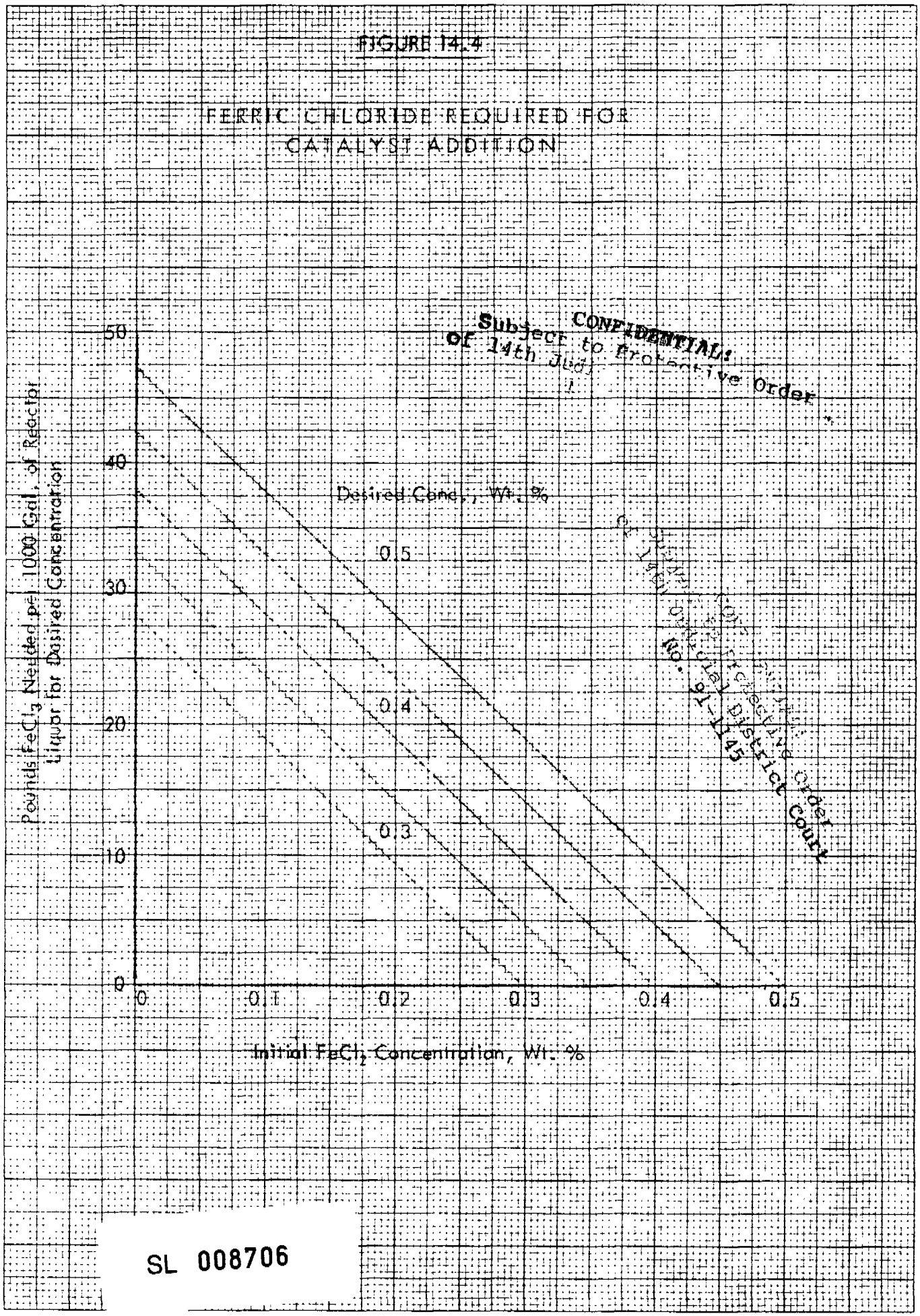
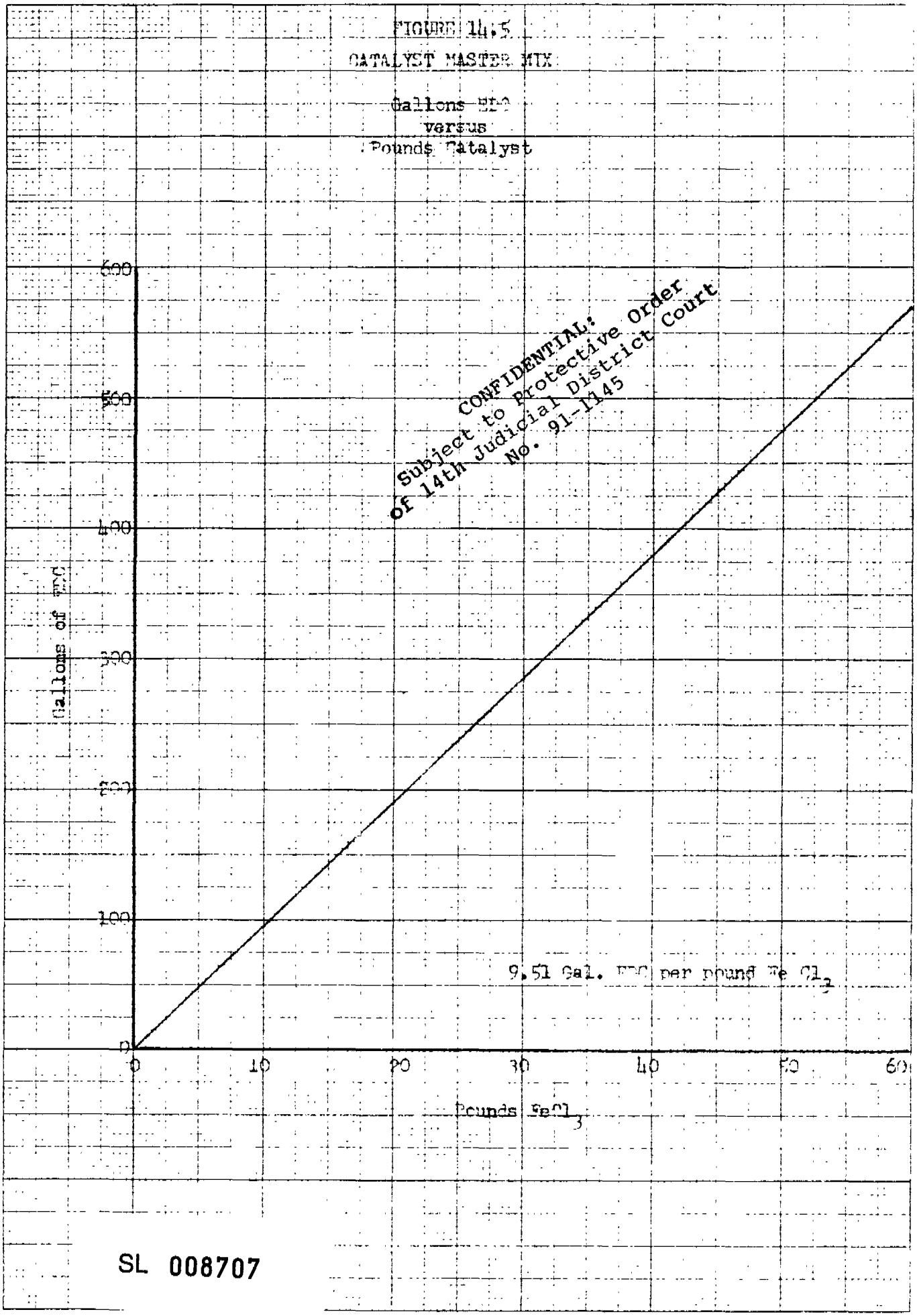


FIGURE 14.5

CATALYST MASTER MIX

Gallons [illegible]
versus
Pounds Catalyst



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