PPG INDUSTRIES, INC.

CHEMICAL DIVISION

LAKE CHARLES, LOUISIANA

CHLOROPIVALOYL CHLORIDE

OPERATING MANUAL

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CPC OPERATING MANUAL

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Record all revisions to this Operations Manual on the next page. Record also the date and reasons for any changes, along with the originator of changes.

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RECORD OF CHANGES

Date	Change No.	Description	Originator
9/19/86	1	Pages 5-65 thru 5-73 (vacuum system)	RM Hall

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NOTES ON PAGE NUMBERING

This manual is designed to permit revision or 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. Pages are numbered using a combination of two numbers, the first being the section number while the second represents a page, illustration, or table in that section. Thus, page 4-6 represents page 6 in Section IV; Figure 5-7 represents Figure 7 in Section V.

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CHLOROPIVALOYL CHLORIDE PROCESS

I. INTRODUCTION

This Operating Manual has been assembled to serve the following two purposes:

A. A reference manual which conveys operating concepts for the CPC Plant.

B. An aid in learning the plant through description of the CPC Plant operation.

This manual consists of concepts, generalities, and data that are not subject to much change. Specific details such as operating data are purposely omitted. The Operating Manual will reflect "Why" rather than specifically "How", with the intention of establishing better understanding of plant operations. It is the responsibility of CPC plant supervision to revise this manual when changes merit it.

Detailed standard operating procedures (SOP's) for startup, shutdown, troubleshooting, and normal operation of the CPC Plant are not included in the Operating Manual due to potential changes in methods of operations and frequent minor modifications of piping and equipment. These detailed procedures are made available to operating personnel in the Standard Operating Procedure Manual which can be easily altered as changes occur. These changes must be approved by CPC Plant supervision. The information contained in this

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Operating Manual is considered confidential. This manual is the prop rty of PPG Industries and may be recalled at any time.

Supplemental information for this Operating Manual is available in other documents. The information in these documents is too lengthy to include in the Operating Manual; nonetheless, it is essential and just as important.

These documents are kept available in the WTU control room to be used by all personnel on an as-needed basis. These are:

A. Documents Maintained By Others

1. CPC Plant Laboratory Procedures Manual
2. Employees Manual of Accident Prevention
3. Plant Emergency Control Plan
4. Job Training Manuals and Films
5. Hazard Communication Program Manual

B. Documents Maintained By CPC Personnel

1. Standard Operating Procedures Manual
2. Equipment Data Tabulations
3. General Plant Operation Training Manuals & Reference Books
4. Environmental SOP
5. Equipment Operating Manuals

This Operating Manual has been written with the thought in mind of establishing sound concepts for operating and maintaining chemical production units. The

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best way to operate a plant is to do so in as safe, efficient and nonpolluting a manner as is possible. Anything less is not operating.

Sound operating methods are those methods where anticipation of and reaction to process changes are performed in a safe manner that attacks and solves problems before problems become crisis situations. This is operating. The only way for operators and supervisors to accomplish this is to know how and to do their jobs.

The hope is that this Operating Manual, The Standard Operating Procedures Manuals, and all other reference materials mentioned will contribute greatly toward learning and thinking.

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(1) CPC 1

II. GENERAL DESCRIPTION OF THE CPC PROCESS

A. USES OF PRODUCT

Chlorinated pivaloyl chloride (CPC) is a raw material for making COMMAND(R), a pre-plant, pre-emergent herbicide for soybeans developed by FMC Corporation. PPG Industries provides FMC with 70-80% of their annual CPC needs. CPC is made by chlorinating pivaloyl chloride (PC), which is produced in LaPorte, Texas by PPG.

B. OVERVIEW OF PROCESS

The production and purification of CPC are conducted in a batch operation which is divided into the following steps:

1. PC Unloading and Storage
2. PC Chlorination
3. Distillation
4. Emergency Scrubber System
5. CPC Product Handling and Shipment

Much of the equipment is common to more than one of the above steps. The plant has been designed to produce 8MM pounds of CPC per year. One batch is produced each day. A 90% on-stream factor is assumed. A brief overview of the plant is presented below for a better understanding of the process and major sections.

The PC is shipped from PPG's LaPorte Plant in tank trucks and unloaded into a storage tank in the Lake Charles CPC Plant area. The PC make-up and a recycle stream, rich in PC from the previous batch, are pumped into the

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[redacted]vaporize the PC. The reactor is comprised of a distillation column including two condensers, a PC recycle tank, storage pot and bayonet reboiler. The vaporized PC is condensed and returned to the column as reflux to which the catalyst is added. Liquid chlorine is vaporized and introduced to the vapor space of the reactor and column. As the CPC is produced, HCl gas is generated. The non-condensibles, HCl and unreacted chlorine, along with a small amount of the PC and other light organics, are vented from the process to either No. 1 or No. 3 incinerator systems. In the incinerator, the chlorinated organics and chlorine gas are converted to inert gases and HCl, which is consumed in other processing units or neutralized. Only a portion of the PC is converted to CPC during each batch to keep the production of undesirable by-products to a minimum. Chlorine feed to the reactor is shut off at the end of the reaction step.

The separation of the desired CPC product from the crude reaction mixture is accomplished through three distillation steps using the same system which was used for the reaction. In the first distillation step the CPC is separated from most of the unreacted PC and other low boiling point impurities. In the second distillation step, which is termed the "mid-cut", residual PC in the system still pot and column are removed from the CPC and heavies. The "mid-cut" is returned to the distillation column during PC distillation in the subsequent batch. In the third distillation step the CPC product is separated from higher boiling point organics (heavies), the major constituents being isomers of dichloropivaloyl chloride, an unwanted by-product. These heavies are collected and burned in an incinerator, which converts them to inert gases and HCl. HCl is recovered for use in the Lake Charles Complex or neutralized to an environmentally acceptable pH.

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The CPC product, following clearance by analysis, is transferred from the CPC shift tank to the CPC product tank for shipment in tank trucks to the [redacted]

All process vessels are vented to an incinerator through a common CPC Plant header system which is nitrogen padded. The CPC shift and product tanks are also N_2 padded to prevent contamination of the product. The header runs directly to Nos. 1 and 3 incinerators without tie-ins from other Plant B processes. In the event that an incinerator is tripped off, the FCV to the incinerator automatically closes and the batch process is shut down; chlorine feed, catalyst and steam to the reactor/column is shut off via an automatic shut down system. If excessive pressure builds up, gases are processed through a PCV to an emergency scrubber, which uses cell liquor (NaOH) to neutralize all HCl, Cl_2 and organic acids. The emergency scrubber is only operated infrequently as needed when the vent is diverted from the incinerator and excessive pressure builds up in the vent header. It is anticipated that the only pressure build-up occurring after an incinerator trip is be due to residual chlorine remaining in the vapor phase or dissolved in the liquid in the reaction system. Hence, no vent releases are expected when an incinerator trip is experienced. The reactor and column contents should begin cooling immediately when chlorine and steam flow to the reactor are stopped.

If the incinerator trip occurs during one of the distillation steps, no process vent release to the emergency scrubber is anticipated. As the system cools, the header pressure drops.

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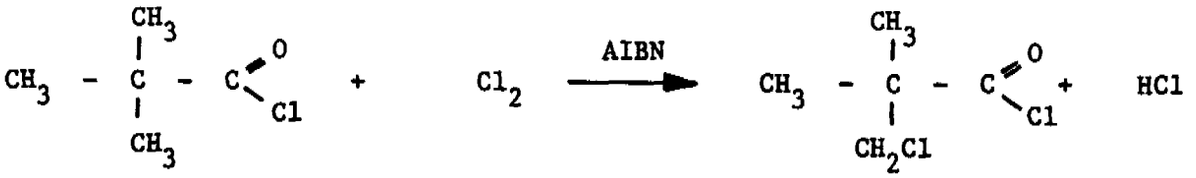
If the scrubber is used, the scrubber effluent is held in a r tention tank and analyzed to ensure destruction of organics has occurred befor discharging the aqueous stream to the sewer.

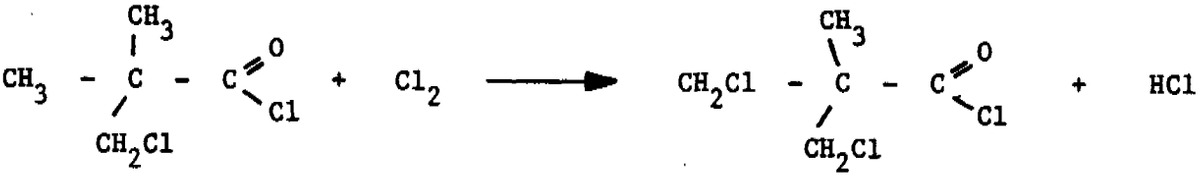
C. GENERAL CHEMISTRY

CPC is produced by reacting gaseous Cl_2 with PC. Reaction can take place in either the liquid or vapor phase.



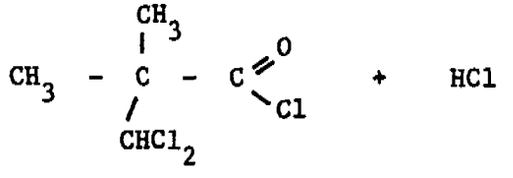
Pivaloyl Chloride (PC) Chlorine Chlorinated Pivaloyl Chloride (CPC)

This reaction is very slightly exothermic, which means it gives off heat. Chlorination in the liquid phase is enhanced by the addition of AIBN, a free radical initiator, to the liquid reflux to the column. As conversion of the PC increases, more CPC is available for subsequent chlorination to dichloropivaloyl chloride, an unwanted by-product.



CPC Dichloropivaloly Chloride

or



Dichloropivaloyl Chloride

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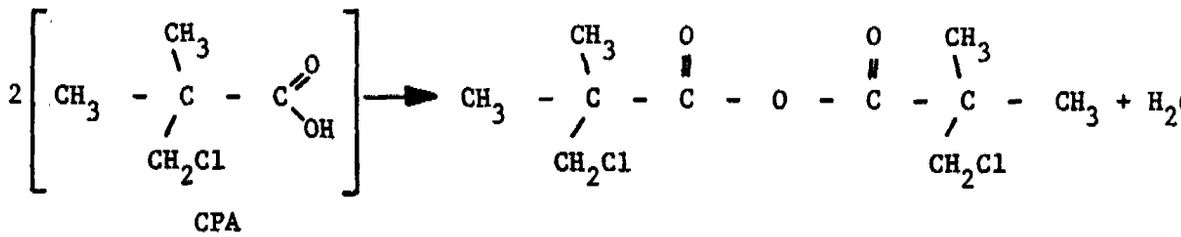
For this reason a distillation column is included with the reactor system. The CPC formed will be separated from the PC and the boil-up is kept high to maximize PC at the Cl_2 feed point.

If any of the acid chlorides come in contact with water they will be converted to the organic acid and HCl. With CPC, for example,

Chloropivalic Acid
(CPA)

PC, CPC and the products of this reaction are very corrosive and irritating to the eyes and mucous membranes. When heated, such as in the reaction or distillation steps, the acid is converted to an anhydride.



CPA

The anhydrides are high boiling compounds, which are disposed along with the DCPC remaining after the last distillation step. High levels of anhydrides indicate water is entering the system - a reboiler or condenser leak, for example.

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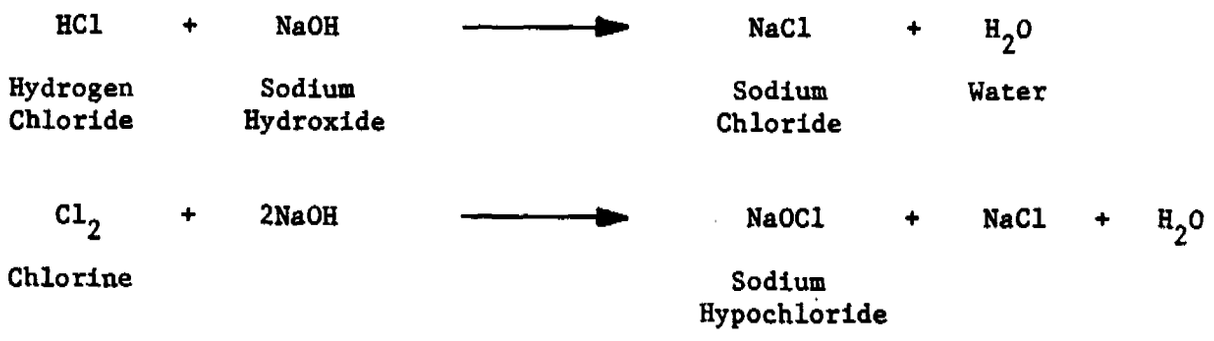
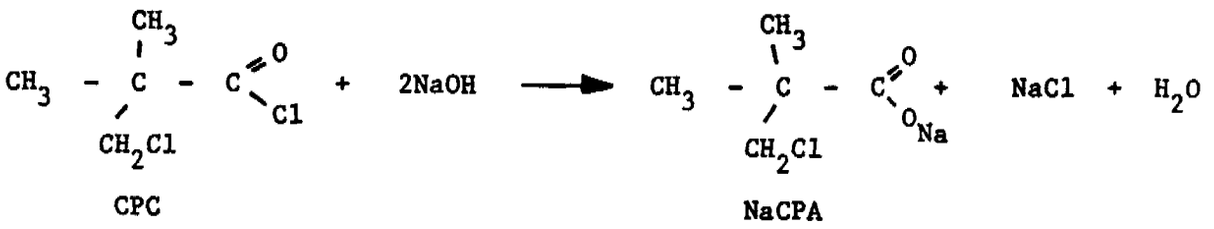
In the scrubber system the hydrogen chloride by-product stream, which normally is incinerated, is treated to remove harmful chemicals whenever necessary. The scrubber recirculation liquor contains sodium hydroxide (NaOH). The HCl and Cl_2 in the vent gases to the scrubber will be removed by the NaOH.



These reactions are both strongly exothermic and cause a significant temperature rise in the recirculation liquor.

Due to environmental regulations the hazardous chemicals, chloropivaloyl chloride (CPC) and chloropivalic acid (CPA) must be kept out of the aqueous effluent from the scrubber. CPC is present in the by-product HCl stream and rapidly hydrolyzes to the sodium salt of chloropivalic acid (NaCPA) when contacted with the aqueous NaOH stream.



In a less rapid reaction the NaCPA is converted to the sodium salt of hydroxypivalic acid (NaHPA).

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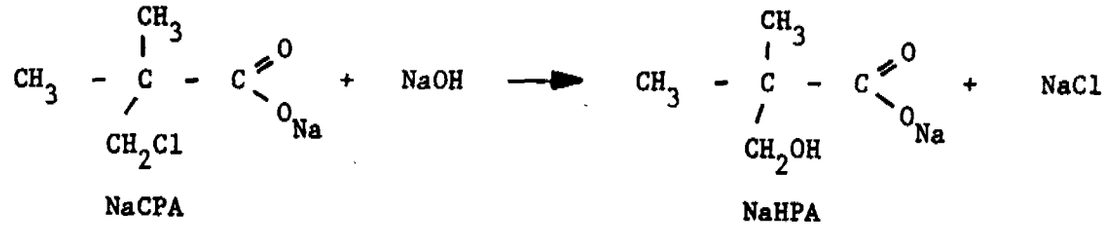
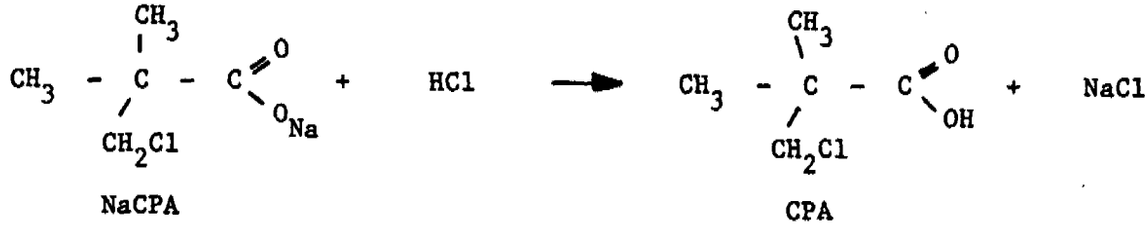


NaCPA NaHPA

If the CPC is not totally converted to the NaHPA, there is a chance that CPA will form when the scrubber effluent is neutralized with HCl.



NaCPA CPA

The NaHPA will <u>not</u> be converted back to CPA when the scrubber effluent is neutralized.

When cleared by analysis for acceptably low levels of CPA and CPC, the scrubber effluent is pumped to a tie-in point with the effluent from the sodium formate destruction unit (another alkaline stream) and subsequently neutralized to an acceptable pH level.

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III. SAFETY IN THE CHLOROPIVALOYL CHLORIDE PLANT

A. INTRODUCTION

Safety reflects attitudes. Development and maintenance of a safe work place depend on the safety attitude of everyone involved with anything in or around the work place. Accident prevention depends upon each individual's interest in observing safety rules, putting forth extra effort toward common sense things which prevent injury to himself and other people, and maintaining a positive attitude toward safety.

Safety rules can only represent minimum requirements or standards because it is impossible to list all safety rules or to write procedures for every situation that may arise. The greatest cause of accidents is failure to THINK. For safe working performance to occur, thought must be involved as to what actions must be taken in a given situation. The only person who is qualified to think about what actions to take to rectify a problem is the person who has put forth the extra effort needed to learn his or her job.

This section of the Operating Manual deals with safety in the CPC Plant. It must be understood now that each person involved with operating the CPC plant is required to know, understand, and follow all safety rules and procedures. There is no room for shortcuts. As previously stated, this is the minimum requirement ----- anything less is not acceptable!

There are many good sources of safety information; therefore, it is not the purpose of this section of the Operating Manual to attempt to give all safety rules and information. Instead, references and general concepts are presented.

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The best source of general plant safety information and rules is the Employees' Manual of Accident Prevention.

B. GENERAL SAFETY INFORMATION FOR THE CPC PLANT

Most of the chemicals in the CPC Plant are dangerous when mishandled. Some are suspected health hazards. General information on the rules and procedures concerning personnel protection from chemical exposure is presented in this Operating Manual. Detailed information for personnel protection from chemical exposure is outlined in the Standard Operating Procedures Manual for the CPC Plant.

Information on chemicals present in the CPC Plant is available in this manual. This information is also available from the Haz-Com System and from supervisors.

C. GENERAL SAFETY RULES FOR THE CPC PLANT

1. Due to the nature of the operations and chemicals involved, the CPC Plant is restricted to ONLY authorized personnel, activities, equipment, and vehicles.

2. Adhere to all job clearing and tagging/lock-out procedures.

3. All permits such as welding permits, vehicle entry permits, etc., are immediately voided if the CPC Plant evacuation alarm sounds or warning lights go on. All jobs have to be cleared again before permits can b reissued.

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4. The doors on the Control Room and all Electrical Switch Gear Rooms must be kept closed.

5. Adhere to all Personnel Exposure Prevention Procedures.

6. Follow all personnel protective equipment and clothing requirements.

7. Operating Personnel must be made aware of all permit jobs, such as welding or vessel entry, before starting any job.

8. Roadway barricades must be maintained closed.

9. Only explosion-proof or intrinsically safe equipment is allowed in the CPC Plant.

10. Smoking is permitted in the Control Room only. Personal lighters are not allowed.

11. Do not leave open samples of organics sitting around.

12. Do not dump any organics into the sewers.

13. All personnel, except operating personnel, are to leave the CPC Plant if the evacuation alarm sounds.

14. Adherence to Plant Hose Policy is required.

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15. Never permit air or oxygen to enter any of the process equipment containing organics.

16. Always purge air from a vessel with nitrogen before allowing flammables into the vessel.

17. All lighting circuits outside the buildings must be in the OFF position before relamping in the area.

18. Immediately remove all clothing, which has been wet with organics, and wash your body thoroughly with soap and water. Properly launder this clothing before wearing again.

19. Always put liquid into tanks through standlegs or bottom nozzles. Falling liquid can generate static electricity.

20. Proper equipment grounding must be maintained.

21. Use of proper breathing equipment is required when working around any organic vapors.

22. Do not permit chlorine to be heated above 300°F due to its reaction with metals.

23. Do not trap liquid chlorine in the Cl_2 vaporizer or surge drum, or in any isolated section of piping.

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24. Never feed liquid chlorine into reactors designed for chlorine vapor feed. This could lead to a greater-than-normal reaction rate and lead to equipment overpressurization.

25. Do not use broken or chipped glassware in the Plant Lab.

26. Do not allow steam lines to hammer.

27. Never bypass or change trip values on any safety shutdown system in the CPC Plant.

28. Do not allow anything to back up into the plant nitrogen system or plant air system.

29. The laboratory hood fan is to operate continuously. Do not attempt to run analyses unless the fan is in service.

30. Never operate any equipment without safety relief devices in place and functioning properly.

31. Never put your head in or enter any equipment before checking for adequate oxygen.

32. Never put your head above any equipment or piping points when purging with nitrogen.

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33. No open flames are allowed in the control room laboratory without a permit.

34. Never hammer, weld, or drop any heavy object on a glass-lined vessel.

The above rules are not intended to outline the total safety effort in the CPC Plant. Safety is a continuing and vitally necessary part of every job performed. Each of us must recognize our responsibilities toward safety. We must use our powers of observation and perception to seek out safety hazards and act to get problems solved. Each of us must TAKE PART. We must surely use good judgement in every case.

D. GENERAL CLEARING PROCEDURES

This is a general write-up concerning procedures for clearing vessels and tanks. Detailed procedures for individual cases are presented in the Standard Operating Procedure Manual for the CPC Plant.

Operations and Maintenance Supervision are responsible to see that all vessels and tanks are properly isolated, cleaned, and cleared of all hazardous materials before declaring them safe for work to begin. Under no conditions are the procedures set up for vessel entry or welding and burning to be bypassed in any form.

General Clearing Considerations

1. Empty the tank or vessel and close and tag all valves.

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

3. Insert blinds in all connecting lines. Blanket tagging/blinding is not sufficient if vessel entry is required.

4. Nitrogen purge the equipment to remove all flammable materials. Steam should not be used on glass-lined equipment since the condensate can attack and corrode the lining.

5. After all flammables are gone, purge the equipment with plenty of air.

6. Then check the equipment with an explosion meter before beginning work. Issue necessary welding and burning permits. See the Employees' Manual of Accident Prevention for complete rules.

7. If entry into the vessel is required, check the vessel with an oxygen meter and issue a vessel entry permit. Refer to the CPC Standard Operating Procedures Manual for complete rules to be followed for vessel entry.

8. Safety belts and safety lines are required.

9. Breathing air equipment must be available.

10. A manway watch is required while someone is in the vessel.

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11. Notify operating personnel of all permit work.

One of the responsibilities of operators is to clear equipment and piping for maintenance work. There are a few general rules that apply no matter what is being cleared. The main thing is that the equipment or piping must be cleared so that maintenance workers are not subjected to unsafe working conditions. It must be remembered that the maintenance workers are normally not as aware as operators are of how to treat various chemicals. They also do not have a good understanding of plant operating procedures and piping systems.

Another very important consideration is tagging of equipment and piping by the lead operator. The lead operator has the same responsibility toward tagging as a supervisor does when the supervisor issues a welding or vessel entry permit. He is saying in writing (hanging a legibly signed tag) that the equipment is then safe for maintenance mechanics to work on. One other thing that should be done when extensive piping systems are tagged is to trace out the piping carefully for all tagging locations. Do not depend on remembering all the block valve locations. It is not a bad idea to get someone (another lead operator or operator) to double-check the tagging of a complex piping system. A third factor that must be considered in all cases is to make sure that it is possible to purge completely through a cleared piece of equipment or piping. Also, purges at low points must be possible to make sure these drains are not plugged.

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E. EMERGENCY PROCEDURES

There is no substitute for an operator really knowing his unit. Many times, emergency situations can be avoided by quickly recognizing abnormal conditions (temperature, pressure, level, flow, etc.) and taking proper corrective action. It is very important for everyone to know and to do his job because it is not possible to specify all possible emergency situations that can develop in a chemical plant; nor is it possible to write detailed procedures for all possible emergency situations that can develop. Therefore, it becomes a must for everyone to know and to do their jobs.

Generally, the procedures are provided as guidelines for actions to be taken in the event an emergency condition does develop that allows PC, for example, to escape into the atmosphere in significant quantities.

When PC is escaping, the normal procedure is to think first of self-protection. The fire hazard involved is another consideration and ignition sources must be eliminated. Alarms for alerting persons outside of the CPC Control Room must be activated. Other control rooms should be alerted by use of the "RED" phone system. The sprinkler system may need to be activated, if automatic activation has not occurred. This will lessen the chances of ignition of any vapor cloud.

The second thought should be to use proper respiratory protection because of both short-term effects (eye/lung irritation) and potential long term effects. Scott air packs, air line masks, and emergency escape packs are available in the control room.

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The final action that should be taken (if the break is not catastrophic) is to isolate the leak source so that the escape of PC is minimized.

The section of this manual on emergency procedures presents guidelines and concepts that each person must know and understand to enable proper response to any situation that might arise. Detailed procedures for response to emergency situations in the CPC Plant are available in the Standard Operating Procedures Manual.

F. CPC/WTU EMERGENCY WARNING/EVACUATION SIREN

The emergency warning devices for the CPC Plant are:

OHC Emergency Evacuation Siren

Roadway Strobe Lights

The alarm system is an air horn as is used in the rest of the Lake Charles Plant. This alarm system is used to evacuate the entire CPC/OHC/WTU area. There is one horn in the CPC area located on the 4th deck north handrail. The mergency evacuation alarm system can be activated from the WTU Control Room. The roadway strobe lights are activated from the WTU Control Room also. The emergency evacuation alarm can be deactivated from the control room.

Detailed descriptions and procedures for these emergency warning devices are available in the Employee's Manual of Accident Prevention, and in the Standard Operating Procedures Manual for the CPC Plant.

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G. EMERGENCY EVACUATION SIGNALS IN THE COMPLEX

1. Plant A - Continuous Warbling Tone

2. Plant B - Intermittent Series of Straight Blasts

3. VCM II - Slow Whooping Sound

4. Plant C - Continuous Blast

5. Entire Plant - All Alarms Simultaneously

When an alarm sounds in a specific plant, only those employees in that area will evacuate.

In the event there is a spill or gas break involving flammable vapors or an unsafe condition is expected, the roadway strobe lights are activated and left on until the emergency condition is put under control. The strobe lights can be activated from the WTU Control Room or from the Main Guardhouse. Strobe lights are located southeast of the old control room, southeast of OHC catalyst building, next to Per-Tri acid pit, and northeast of the old Tetra Plant. All vehicles are to stop with ignitions off when the strobe lights are on. Vehicles responding to an emergency should stop at the plant perimeter and wait for clearance from supervision before entering the area.

H. GENERAL EMERGENCY EVACUATION PROCEDURE

1. When the evacuation alarm is sounded, employees should determine the wind direction and travel at right angles to the wind when moving out of the area. Windsocks are located throughout the area to aid in determining wind direction. You can also use the plume from the incinerator stacks, condensate tank stacks, or the cooling tower to determine wind direction.

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2. All personnel, except essential operating people, are to evacuate the CPC area immediately anytime the evacuation alarm is sounded. The alarm should be sounded in case of major unsafe happenings, or if there is reason to expect major unsafe conditions. When the alarm sounds, all equipment on permits, such as welding machines or portable air compressors, must be shut down.

3. The emergency evacuation alarm sounds for several minutes and then is silent. After the cause of the emergency has been put under control, two long blasts of the "all clear" signal sounds and everyone may re-enter the plant. The roadway strobe lights remain on until the "all clear" signal is sounded.

4. STOP all vehicles, including scooters and bicycles, on the side of the roads anytime the emergency evacuation alarm is sounded. Turn off the ignition and evacuate on foot.

5. All personnel, except essential operating people, are to evacuate on foot.

6. There is to be NO SMOKING during evacuation.

I. EMERGENCY FIRST AID OR FIRE-FIGHTING PROCEDURES

When emergency first aid or fire-fighting services are needed in the CPC Plant, the employee proceeds to the nearest plant telephone and dials 2700. He tells the guards whether first aid or fire-fighting services are

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needed. He also states the exact location of the emergency, if possible, and then proceeds or sends someone to direct the Emergency Squad to the scene.

During an emergency, all personnel not directly involved in the emergency must stay clear of the area.

Details on emergency first aid or fire-fighting procedures can be found in the Employee's Manual of Accident Prevention.

J. CRITICAL ISOLATION VALVE

The block valve on the Cl_2 header to the CPC Plant is the one block valve designated as "critical isolation valve" that is to be used to isolate the CPC Plant in case of major emergency situations, such as an explosion, that require total isolation ot the plant. Operation of this valve prevents chlorine flow into the plant. This critical block valve is covered in the CPC Emergency Equipment and Procedures Manual and in the CPC Standard Operating Procedures Manual.

K. COMMUNICATIONS EQUIPMENT

Rapid communication with other operating units when emergency situations arise is very important. The sooner everyone is made aware of emergency problems, the better the control over operating units. The CPC Plant can be upset by problems in other areas, or problems within the CPC Plant. The CPC unit can cause problems elsewhere. This is why emergency communications involving everyone is important.

The CPC Plant is equipped with different communications systems. These are:

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1. The emergency "RED" phone system. This is a telephone system which interconnects all of the operating units in Plants B-I and B-II with many of the units in Plants A and C, the Powerhouses, Shift Superintendents, and Shift Supervisors. The system can be used to communicate with all stations at one time, or just one station. Operating procedures for the Red Phone System are found in the S.O.P. Manual and by the phone (beneath the glass on the desk.)

2. The WTU Control Room Radio System is available for use involving emergency and non-emergency situations. The radio system is equipped with a back-up battery system to provide power to the control room console unit in the event of a power failure. When this back-up system is in service, a beeping tone will be heard over the control room speakers.

The above communications systems have a very important function. No playing around with any of the emergency communication systems is tolerated. Emergency situations are too important, because people's lives can be at stake. Never ignore any call coming in on any system; answer immediately; also, use the equipment to warn others of emergency problems in the CPC Plant as soon as possible.

L. FIRE PROTECTION IN THE CPC PLANT

Fire protection for the CPC Plant is provided in four forms---water, Halon, dry chemical, and CO_2. The dry chemical type is most effective for organic fires. Several dry chemical-wheeled units are located in the area. Portable, handheld, fire extinguisher units are located throughout the plant. The CO_2-type is good for small fires or small electrical fires. Water should

not be used to extinguish small fires in the CPC unit if possible, because CPC/PC react with water to liberate HCl gas. Water for fire protection is provided by piping connected to the existing fire water system in the Lake Charles Plant. The CPC fire water system consists of fire hydrants, monitor nozzle stations, and a sprinkler system connected by water mains and piping. The sprinkler system can be activated by vapor detectors located on ground level in the plant or manually from a switch on the panel board. Monitor nozzle stations are located east of the Bottoms Plant dike, east of the OHC tank farm dike, and just south of the OHC cooling towers. The sprinklers can also be activated by the pilot head system which detects a rapid rise in temperature. The main supply piping from the fire water main loop to the water deluge valves or the monitor nozzle stations is provided with post-indicator valves for isolation purposes.

The switchgear room is equipped with both Halon 1301 and CO_2 fire extinguishers located by the doorway.

M. ELECTRICAL SYSTEM SAFETY EQUIPMENT

The CPC switchgear room is located above the old OHC control room. A detailed description of the Electrical System is included in Section V.A.8. of this manual.

The switchgear room is pressurized and air-conditioned to maintain a purge. The purge pressure in the SGR is alarmed for low differential. There are alarms on the fresh air fan and motor. The SGR is also equipped with a vapor detector on the intake air to the blower. Detection of flammable vapors at the 40% LFL shuts down the pressurizing blower.

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The SGR is equipped with two portable fire extinguishers. One is a Halon 1301 extinguisher the other a CO_2 type.

The effects of Halon 1301 upon humans are minimal and short-term at the levels used in the fire protection system.

Caution should be exercised in discharging a CO_2 extinguisher in the SGR as a build-up of CO_2 in the confined space of the SGR could result in asphyxiation.

The SGR has a sealed beam, battery powered light unit for emergency lighting in the switchgear rooms.

The characteristics of the chemicals in CPC are such that the materials are classified in Group D, Class 1 by the U.S. National Electrical Code. The method and materials of installation are those recommended by Factory Insurance Association. In general, the installation is Class 1, Group D, Division 2. Motors are Totally Enclosed, Fan-Cooled (TEFC), lighting is vapor-tight, and all arcing devices are explosion-proof with seal-offs. Since the control building is pressurized and isolated from the process area, general purpose equipment is used. Explosion-proof equipment is required inside the laboratory hood. The laboratory itself is pressurized along with the rest of the control room.

N. RELAMPING AND GROUNDING

Vapor-tight lighting fixtures are used in the CPC unit. The relamping procedure will be to first determine the lamps which need replacing by

turning on all lights. Then, turn off all lights and relamp. Globes and guards must be replaced after relamping.

Equipment and piping grounding in the CPC Plant has been given special attention due to problems peculiar to the handling of hydrocarbons. Equipment and piping change-outs should be inspected to assure that proper grounding is maintained. All vessels are grounded at two points. The result is a system of piping, tanks, and vessels operating at ground potential. This does not prevent the generation of static electrical charges, but does provide adequate leakage to ground to prevent accumulation of dangerous static electrical charges. This is why it is so important for the grounding system to be maintained properly.

O. SAFETY CONTROL SYSTEMS

The CPC Plant contains many pieces of equipment equipped with safety control systems. This Operating Manual does not deal with details of the safety control systems. It names the systems. Detailed information concerning all of the safety control systems is available in the Standard Operating Procedures Manual. Detailed procedures for operation of the safety control systems are available there also.

Equipment with safety systems is as follows:

1. Chlorine Vaporizer
2. CPC Reactor
3. Emergency Scrubber
4. Liquid Waste To Incinerator
5. Vents To Incinerator
6. Water Deluge Systems

All of these safety systems are in the plant to protect personnel and/or equipment. The alarm and trip-off valves are not be changed without proper

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authorization. Do not bypass any safety system or any component of any safety system without proper authorization. All safety control systems must be in proper repair and in service at all times unless proper authorization has been granted.

While on the subject of equipment safety features, the importance of safety relief devices should be emphasized. Two safety relief devices used in the CPC Plant are rupture discs and safety (or pressure and/or vacuum) relief valves. Both devices perform a vital function, namely preventing overpressurization of equipment and piping. Another function is preventing too much vacuum to be pulled on tanks, for example.

Both over-pressure and too much vacuum are very dangerous to personnel and equipment. A vessel can literally burst outward at the seams when subjected to pressures higher than design. Vessels implode or burst inward when subjected to more vacuum than design. Disastrous results, such as a very large spill or large gas release can occur in both cases.

This general discussion is intended to emphasize the need for safety relief devices being in proper repair and in service. Equipment, piping, etc. equipped with safety relief devices of any nature must never be operated with the devices out-of-service in any fashion. A listing of the safety relief valves and rupture disc locations and settings is included in the S.O.P.

P. BREATHING AIR SYSTEMS

There are two breathing air systems in the CPC Plant. The control room is equipped with an emergency breathing air system consisting of

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compressed air cylinders, air line masks, and small emergency escape air packs. The systems must be maintained properly, i.e., the compressed air cylinders must be filled to maximum at all times. This emergency breathing air system is to be used in the event of a major release, such as chlorine, from another area, in which case the fresh air blower is shut down. Everyone must be familiar with the proper use of the control room emergency breathing air system.

There is a breathing air system located in the CPC process area. This system consists of compressed air cylinders, breathing air drops, and air line masks. This system is to be used when working on CPC process equipment, piping, etc. to prevent personnel exposure to fumes or other undesirable materials.

The CPC Standard Operating Procedures Manual contains detailed procedures for the CPC area breathing air system.

Q. CONTROL BUILDING AND PLANT LAB

The Waste Treatment Control Building is designed to be explosion-resistant. The building is pressurized to prevent accumulation of any vapors inside. Since it is a pressurized building, non-explosion proof electrical equipment is used and smoking is permitted inside.

The positive pressure is maintained by a blower which draws intake air across an activated carbon filter. If a major break occurs, the carbon filter cannot eliminate all contaminants. Therefore, the pressurizing blower must be shutdown.

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A blower shutdown switch is located in the control room. The blower is automatically shut down by a vapor detector located at the air intake if flammable vapors above 40% LFL are detected.

The Control Building must be kept pressurized at all times. Doors leading into the building must be maintained closed. The pressurizing blower is required to be in service unless shutdown for maintenance or because of outside vapors.

To prevent any vapors from entering the control room when the blower is shut down, instrument air bleeds should be opened. (Be careful to not bleed the instrument air system pressure too low by taking too much purge.) This air purge must maintain a positive pressure inside the control room until the blower can be put back in service.

The WTU Plant Laboratory has a nitrogen outlet under the hood used for purging sampling equipment. If this nitrogen is fully opened and allowed to purge into the laboratory, any personnel in the room can possibly be asphyxiated. Protection against this is provided by the lab vent hood fan.

One word of caution--- if you are working in the lab and become overly sleepy, go outside. This may be an indication of nitrogen in the lab. Notify your Supervisor.

The lab vent hood fan must be operating properly at all times when working in the lab. DO NOT work in the lab when the fan is out of service. The fan can be shut down by a switch located inside the lab.

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R. SAFETY SHOWERS AND BODY WASH STATIONS

Safety shower stations are located throughout the CPC Plant. Shower stations which are located near where caustic or cell liquor is used are equipped with containers of body wash. Safety showers are not to be used as sources for utility water. The area under and around shower stations must be kept orderly to allow for easy access to the showers. All shower pull ropes, eye wash stations, etc. must be maintained in good working order.

S. SAFETY SIGNS

Many safety signs are located throughout the plant. These signs are very important and must be maintained. The signs indicate such things as restricted areas, exits, special safety areas, safety shower locations, and fire extinguisher locations. These signs are a part of the safety program and proper attention must be paid to them.

T. TOOLS

Special spark-proof or alloy tools are not required in the area except where special precautions must be taken. However, precautions need to be utilized in the handling of tools and equipment even under normal conditions. Hand tools, for instance, if dropped can damage glass-lined equipment. Also, do not strike a metal object that can cause a spark in the presence of volatile chemicals. A permit is required for use of many tools, such as electric drills, in the CPC unit. Check with your supervisor or lead operator for direction if maintenance work requires use of any arcing devices.

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U. RESPIRATORY PROTECTION EQUIPMENT

Proper respiratory protection must be used in the CPC Plant to prevent personnel to exposure to organic vapors. Due to the nature of PC and CPC the mouth-bit escape respirator is not adequate for use for escape purposes in the CPC Plant. The Comfo-II half mask is used for escape. This respirator is for escape purposes only. It should not be used in the process of performing any jobs. No matter what the material exposure might be, proper respiratory protection must be obtained and used. Refer to the Employee's Manual of Accident Prevention and the Section IV of this Manual for a complete description of respiratory protection equipment needed for the CPC Plant.

V. COMPRESSED GAS CYLINDERS

A variety of compressed gases are used in the CPC Unit. These gases are stored in steel cylinders at pressures up to 2200 psig. Some of the materials stored in cylinders are: breathing air, hydrogen, helium, and nitrogen. Improper handling or use of gas cylinders can result in severe injury. Some basic guidelines for handling and using gas cylinders are given below. A detailed listing of safe handling procedures can be found in the Employee's Manual of Accident Prevention.

1. Store cylinders in groups according to type of gas.

2. Store empty and full cylinders separately.

3. Report cylinders found in poor condition to supervision.

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4. Cover cylinder valves with a cylinder cap when a cylinder is not in use or when being transported. If the valve of a high-pressure gas cylinder is broken off, the contents of the cylinder will jet from a hole about the diameter of a pencil. The escaping gas can have enough thrust to turn the cylinder into a rocket capable of doing significant damage to equipment and/or injuring people.

5. Store cylinders in an upright position and tie them off at all times.

6. Clearly mark cylinders to identify their contents. Be sure you are using the correct gas.

7. Transport cylinders using a hand truck designed for that purpose, or if carried by a winch truck, in an appropriate basket or rack.

W. HOUSEKEEPING

Housekeeping is a very important part of any good safety program. It has been previously stated that safety performance reflects attitudes. The level of housekeeping maintenance physically reflects the attitude levels toward safety, operating efficiency, plant maintenance efficiency, cost control, and pollution prevention.

Housekeeping standards are to be maintained at high standards in the CPC Plant. Operations and maintenance have identical responsibilities toward maintaining high housekeeping standards. Do not perform a job with the work area in an

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unsafe, cluttered condition. No job is complete until the work area is safe and clean. Whatever you mess up, expect to clean up.

There are many good results gained from good housekeeping. Some of these are:

1. Increased employee morale and efficiency.
2. Fewer accidents.
3. It costs less to operate a clean plant.
4. Production control is easier.
5. Employee productive time is higher.
6. Benefits the total safety effort.

All jobs must be planned with the thought in mind that housekeeping is an important part of all jobs. Anything less than high standards is not acceptable.

X. POLLUTION PREVENTION - A PART OF SAFETY

Safety deals with maintaining a work place that contributes to good health, and not just with accident prevention. This is why pollution prevention is so important in any good safety program.

Sound plant operating methods are those that attack and solve problems before these problems become major upsets or unmanageable. This is the way it is with pollution prevention. Prevention means to not allow something to occur. Pollution prevention is not possible once fumes are allowed to get int the atmosphere or liquid spills occur. Prevention is not possible after people are exposed to chemicals that are not conducive to good health. Pollution prevention has to be before the fact.

There have been many laws passed that deal with pollution of the air and waterways. This is all good BUT each person in this world has a much greater

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obligation concerning pollution and people. Each of us has a moral obligation to all people around us to prevent exposure of other people to harmful chemicals.

Pollution prevention is the intended method of operating the CPC Plant. Allowing pollution, or any other deviation from the safety program, to occur cannot exist in this intended way of operating. All jobs must be planned in such a manner that harmful exposure of personnel to chemicals is prevented. Supervisors and Operators have the same responsibility toward accomplishing this. This is the only acceptable standard to work under.

Never attempt to hide the fact that the CPC Plant has pollution problems, should any develop. Work diligently toward finding a solution to the problem and restoring the plant to normal operation.

Y. FREEZE PRECAUTIONS

During the winter months, there are days when the ambient temperature drops low enough to cause freezing of well water, Sabine water, steam condensate, fire water, steam traps, steam pressure and flow transmitter impulse lines, pumps, and others. On days when the temperature is low enough to cause freezing, special precautions must be taken to prevent freezing of lines, equipment, and instruments in the CPC Plant.

If freeze precautions are not taken, freezing in lines, equipment and instrumentation can result in emergency shutdown of the plant, process upsets, unusable safety equipment, piping ruptures, problems in other operating units resulting from CPC Plant problems, and many other such things. From a safety

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standpoint alone, it is very important that freeze precautions be a part of operating a plant.

Taking freeze precautions usually means opening small bleeds on piping, making sure steam tracing is in service, making sure things are insulated properly, and winterizing instrumentation. Water bleeds to the pad should be avoided to prevent dangerous slipping hazards. Bleeds should not be allowed to drip on stairs or ladders, or on heavily traveled paths.

The intent of this section of the Operating Manual is to state why freeze precautions are needed. Detailed check lists for taking freeze precautions are in the Standard Operating Procedures Manual.

Z. CHEMICALS IN THE CPC PLANT

Following is a list of compounds found in the CPC Plant, along with their formula and abbreviation. On the subsequent pages the more important chemicals are detailed as to their hazardous properties and physical characteristics.

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Abbreviation	Name	Formula
AIBN	Azobisisobutyronitrile	-
$CaCl_2$	Calcium Chloride	$CaCl_2$
NaOH	Cell Liquor (Caustic Soda)	NaOH
Cl_2	Chlorine	Cl_2
CPA	Chloropivalic Acid	$CH_2Cl\ (CH_3)_2\ CCOOH$
CPC	Chloropivaloyl Chloride	$CH_2Cl\ (CH_3)_2\ CCOCl$
DCPC	Dichloropivaloyl Chloride	$(CH_2Cl)_2\ CH_3\ CCOCl$
HCl	Hydrogen Chloride	HCl
N_2	Nitrogen	N_2
Per	Perchloroethylene	C_2Cl_4
$COCl_2$	Phosgene	$COCl_2$
PA	Pivalic Acid	$(CH_3)_3\ CCOOH$
PC	Pivaloyl Chloride	$(CH_3)_3\ CCOCl$

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OCCURRENCE:

Catalyst For CPC Reactor

NAME: Azobisisobutyronitrile (AIBN)

MSDS #: 49, 50

FORMULA: $NC(CH_3)_2 CN = NC(CH_3)_2 CN$

FLASH POINT: Not Applicable

MELTING POINT: 212°F (Decomposition can be violent)

EXPLOSIVE LIMITS: Not Applicable

MAXIMUM ALLOWABLE CONCENTRATION: OSHA limits not established. DuPont recommends less than 1 mg/m^3 for 8 hr. TWA.

HAZARDOUS PROPERTIES: AIBN is a solid, white, and odorless powder. It is a flammable solid. If exposed to heat above 122°F it may erupt and form an explosive dust mixture with air. Therefore, AIBN should be kept in a refrigerated area.

Overexposure to AIBN could cause nausea, headache, or weakness.

TREATMENT:

Inhalation: Remove immediately to fresh air. If breathing is difficult, give oxygen, call a physician.

Eye Contact: Flush eyes with water for at least 15 minutes. Call a physician.

Skin Contact: Wash skin with soap and water after excessive contact.

Ingestion: Induce vomiting immediately by giving two glasses of water and sticking finger down throat. Call a physician.

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OCCURRENCE:

Refrigerated Brine

NAME: Calcium Chloride

MSDS #: 96,104

FORMULA: $CaCl_2$

FLASH POINT: None

EXPLOSIVE LIMITS: None

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

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OCCURRENCE:

Emergency Scrubber Recirculation Liquor

NAME: Cell Liquor (Caustic Soda, Sodium Hydroxide)

MSDS #: 908,1052,1299

FORMULA: NaOH

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONCENTRATION: 1.2 ppm by volume

HAZARDOUS PROPERTIES: This material, both solid and solution, has markedly corrosive action upon all body tissue. Caustic solution is detectable by the slippery feeling of soapy water. Its corrosive action on tissue causes burns and frequently deep ulceration, with ultimate scarring. Prolonged contact with dilute solutions has a destructive effect upon tissue. Mists, vapors, and dusts of this compound cause small burns, and contact with the eyes, either in the solid or solution form, rapidly causes severe damage. Ingestion either in the solid or solution form causes very serious damage to the mucous membranes or other tissues with which contact is made. It can cause perforation and scarring. The effects of inhalation vary from mild irritation of the mucous membranes to a severe pneumonitis. It can cause an irritant dermatitis. Caustic soda will react with water or steam to produce heat and will attack organic tissue. Where large quantities are handled, the area should be well ventilated and face masks should be worn.

TREATMENT: Speed in removing caustic from contact with the skin is important. Remove all contaminated clothing at once, then flush the skin with water thoroughly. The use of small portions of water, such as by sponging, may cause more serious injury due to heat liberated on dilution. After flushing with water, use liberal quantities of a neutralizing solution such as a 0.5% solution of boric acid, citric acid, or ammonium chloride. If any caustic soda contacts the eyes, they should be irrigated immediately with an abundant amount of water for at least 15 minutes. The eyelids should be held apart during irrigation. The eye should then be washed with a mild saline solution if available. Wash the eye with water for an additional 15 minutes. A physician should be seen immediately after the eye is washed thoroughly.

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OCCURRENCE:

CPC Reactor Feedstock

NAME: Chlorine

MSDS #: 705,1868

FORMULA: Cl_2

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONCENTRATION: 1 ppm/8 Hr.

HAZARD PROPERTIES: Chlorine is a skin, mucous membrane and respiratory irritant. Because of its pungent odor and irritating effect, traces of chlorine in air are readily detected. As the duration of exposure or the concentration increases, general excitement follows as indicated by restlessness, irritation to the throat, sneezing and copious salivation. This is followed by retching and vomiting and by difficult or labored respiration. Respiratory distress increases until eventually death may occur from apparent asphyxiation.

In any atmosphere containing chlorine, short, shallow breathing should be used. Chlorine produces no known cumulative effects, and recovery from mild exposure usually occurs. Chlorine is particularly irritating to persons afflicted with asthma and certain types of chronic bronchitis.

TREATMENT: The following First Aid procedures are suggested, but it is recommended that emergency measures prescribed by your physician be followed.

1. Carry patient from gas area. Preferably, patient should be kept in a room at about 70°F. Supply blankets if necessary. Keep patient warm and quiet. Rest is essential.

2. Place patient on back with head and back elevated, or allow to remain seated.

3. Clothing contaminated with liquid chlorine should be removed promptly and the exposed areas flushed with water. Keep patient warm with blankets.

4. Cough syrup and an antacid such as Pepto Bismol may be given as a relief from throat irritation, provided the patient is conscious.

5. If the patient has inhaled chlorine but still has practical control of his own breathing, oxygen by mask or under intermittent positive pressure, may be administered. Be sure to follow the instructions which come with the oxygen-administering equipment which you are using.

6. CALL A PHYSICIAN IMMEDIATELY.

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OCCURRENCE:

Major Product Of CPC Plant

NAME: Chloropivaloyl Chloride (CPC)

MSDS #: 470

FORMULA: $CH_2Cl\ (CH_3)_2\ CCOCl$

FLASH POINT: 143°F

EXPLOSIVE LIMITS: Unknown

MAXIMUM ALLOWABLE CONCENTRATION: No OSHA limits established. Toxicity guidelines have ~~not~~ been established ~~as of this writing~~. at 1 ppm ceiling.

HAZARDOUS PROPERTIES: Chloropivaloyl Chloride (CPC) is a colorless to slightly yellowish liquid with a sharp odor. CPC is not a very flammable substance. Water should be avoided as an extinguishing agent since water hydrolyzes CPC thus liberating HCl which when in contact with metal can generate hydrogen gas.

Limited toxicity data are discussed in the Premanufacture Notice (PMN) for CPC. CPC is very corrosive. Any contact with skin should be avoided. Clean-up any spills immediately to avoid damage to equipment. Soda ash is excellent for cleaning up small spills of CPC. Washing with water will cause a cloud of HCl gas.

TREATMENT: Based on further toxicity data CPC is considered an inhalation hazard.

Inhalation: Remove to fresh air. If not breathing give artificial respiration. Give oxygen if necessary. Call a physician.

Eye Contact: Flush with plenty of water for at least 15 minutes. Call a physician.

Skin Contact: Wash with soap and water for at least 15 minutes while removing contaminated clothing and shoes. Thoroughly clean clothing before reuse or discard. Contaminated shoes should be discarded.

Ingestion: If conscious drink large quantities of water or milk. Do not induce vomiting. Take immediately to a physician. If unconscious take to hospital.

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OCCURRENCE:

By-Product Of CPC Reactor

NAME: Dichloropivaloyl Chloride (DCPC)

FORMULA: $(ClCH_2)_2$ (CH_3) CCOCl

FLASH POINT: Unknown

EXPOSURE LIMITS: Unknown

MAXIMUM ALLOWABLE CONCENTRATION: Toxicity guidelines have ~~not~~ been established ~~as of this writing.~~ at 1 ppm ceiling.

HAZARDOUS PROPERTIES: See CPC

TREATMENT: See CPC

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OCCURRENCE:

By-product of the CPC Reactor Operation

NAME: Hydrogen Chloride

MSDS #: 503,508

FORMULA: HCl

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONCENTRATION: 5 ppm for 8 hour working day.

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

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

Skin - Immediately wash with large amounts of water. Contaminated clothing and shoes should be removed while the patient is showering. Continue washing for a sufficient time to remove all muriatic acid.

Eyes - Flush with water immediately. The eyelids should be held apart during the irrigation to insure contact of water with all the tissues of the surface of the eyes and lids.

NOTE: No attempt should be made to neutralize the acid with alkaline solutions. Medical assistance should be summoned at the earliest possible moment.

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OCCURRENCE:

Inert purge gas for tanks and vessels.

NAME: Nitrogen

MSDS #: 2037, 1448, 1350, 845, 856, 155, 394

FORMULA: N_2

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

Therefore, every vessel that is entered not only must first be checked for flammability, etc., it must also be checked for sufficient oxygen. Not only that, nitrogen lines, as well as other toxic lines, must be isolated from the vessel before entry and a clean air sweep must be provided. The apparatus for testing for sufficient oxygen is an analyzing device. Become familiar with its use.

TREATMENT: Inhalation - Remove the person from the oxygen deficient area. Administer artificial respiration, if necessary.

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OCCURRENCE:

Circulating Fluid For Vacuum Compressor

NAME: Perchloroethylene (Per)

MSDS #: 1427

FORMULA: C_2Cl_4

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONCENTRATION: 50 ppm - TWA (8 Hr.)
200 ppm - STEL (15 Min. Short Term Exposure Limit)

HAZARDOUS PROPERTIES: Perchlor is a clear, colorless liquid with an ether like odor. Contact with the skin and eyes should be avoided. Overexposure to vapors can cause irritation of the respiratory tract, dizziness, nausea, headache, loss of coordination and equilibrium, unconsciousness and even death in unventilated areas. Perchlor is a central nervous system depressant and can cause central nervous system damage with overexposure.

Prolonged exposure above the OSHA limits may result in liver and kidney damage. There is no documented evidence that Perchlor causes increased cancer incidents in humans.

TREATMENT:

Inhalation: Remove to fresh air. If not breathing give artificial respiration. Oxygen may be needed. Contact a physician.

Eye Contact: Flush with plenty of water for at least 15 minutes. If irritation occurs, consult a physician.

Skin Contact: Flush with plenty of soap and water for at least 15 minutes while removing contaminated clothing. If irritation occurs, consult a physician.

Ingestion: If conscious drink large quantities of water. Do not induce vomiting. Take immediately to a hospital or physician.

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OCCURRENCE:

Possible Impurity In PC Feed

NAME: Phosgene

MSDS #: 704

FORMULA: ClCOCl

FLASH POINT: None

EXPLOSIVE LIMITS: None

MAXIMUM ALLOWABLE CONCENTRATION: 0.1 ppm (8 hr. - TWA)

HAZARDOUS PROPERTIES: Phosgene is a clear, colorless liquid or gas with a mild sweet odor like freshly mown hay. Phosgene is a highly poisonous gas which may cause pulmonary edema from 4-24 hours after overexposure. More immediate effects may be tightness in chest, shortness of breath, and nausea.

TREATMENT: Remove to fresh air immediately and remove all contaminated clothing and shoes. Do not allow person to walk. If breathing stops give artificial respiration. Otherwise give oxygen. Take immediately to a hospital.

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OCCURRENCE:

Feedstock For CPC Reactor

NAME: Pivaloyl Chloride (PC)

MSDS #: 911

FORMULA: $(CH_3)_3$ CCOCl

FLASH POINT: 80°F

EXPLOSIVE LIMITS: Not Determined

MAXIMUM ALLOWABLE CONCENTRATION: Toxicity guidelines ~~not~~ have been established ~~as of this writing~~. at 1 ppm ceiling.

HAZARDOUS PROPERTIES: Pivaloyl Chloride is a clear or slightly colored liquid with a strong pungent odor. Due to its flash point, PC is considered a flammable liquid. Water should be avoided as an extinguishing agent as water hydrolyzes PC thus liberating HCl, which in contact with metal can generate hydrogen gas.

Very irritating to the respiratory tract following inhalation of vapor or mists. Eye contact may cause extensive damage to corneal tissues, possibly resulting in loss of vision.

PC is very corrosive and any contact should be avoided. Spills of PC should be cleaned immediately with soda ash if possible. Cleaning a spill with water results in an HCl cloud.

TREATMENT:

Inhalation: Remove to fresh air. If not breathing give artificial respiration. Give oxygen if necessary. Call a physician.

Eye Contact: Flush with plenty of water for at least 15 minutes. Call a physician.

Skin Contact: Wash with soap and water for at least 15 minutes while removing contaminated clothing and shoes. Thoroughly clean clothing before reuse or discard. Contaminated shoes should be discarded.

Ingestion: If conscious drink large quantities of water or milk. Do not induce vomiting. Take immediately to a physician. If unconscious take to hospital.

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AA. CONCLUSION -- CPC GENERAL SAFETY

The general safety section of this Operating Manual has been written with the following ideas in mind:

1. To help each person associated with the CPC Plant make their work place accident-free and security-safe.
2. To provoke each person into THINKING safety.
3. To provide written materials and point out references that will aid each person in learning safety.
4. To display written guidance that safety is <u>equal</u> to production, costs, yields, and pollution.

The cause of most accidents is PEOPLE. Human failure is at the root of most all accidents. The following is a list of unsafe practices that are people-caused, which will allow for a better understanding of what "Human Failure" means.

1. Failure to properly secure equipment.
2. Failure to warn others of intended actions or unsafe conditions.
3. Failure to wear protective clothing.
4. Failure to use safety equipment.
5. Operating equipment or using tools without proper training.
6. Operating at unsafe speeds.
7. Using unsafe equipment or tools.
8. Failure to maintain housekeeping standards.
9. Working in an unsafe position.
10. Bypassing safety devices.
11. Indulging in horseplay.
12. Failure to learn.
13. Failure to think.
14. Failure to ask questions.
15. Failure to insist that others work safely.
16. Failure to just plain care.

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This list can, no doubt, be expanded, but it points out what is intended. "Human Failure" is really the failure of people to accept personal responsibility toward safety.

Adherence to safety rules and procedures is the expected minimum standard of safety. This is the least that you can do---this is not a reflection of sound operating standards. Sound operating standards are standards where written safety rules and procedures are encompassed with safe, knowledgeable thinking. The best way to stay out of emergency situations is not to get into these situations. Job knowledge, the ability and desire to think, and quick reaction provides the key for safe operation of any plant ------- ANTICIPATION.

The real purpose of any Operating Manual is to set acceptable standards under which a plant is operated. A good operator is one that sets high standards for himself ---and for everyone around him. The aim of this manual is to provoke everyone into working safely. There is no other way to work.

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IV. PERSONNEL EXPOSURE PREVENTION TO CHLORINATED ORGANICS

A. INTRODUCTION

PPG Industries has a very real interest concerning the health of its employees. With this in mind, this section of the CPC Plant Operating Manual deals with preventing exposure of personnel to chlorinated organics. In the past, much effort has been directed toward avoiding and preventing people from being exposed to chlorinated organics. Concentrated effort toward this remains the norm in the CPC Plant.

B. GENERAL DISCUSSION

The Occupational Safety and Health Administration (OSHA) has set forth base standards and regulations dealing with exposure limits of personnel to some chlorinated organics. PPG Industries has adopted these limits as the guidelines to be followed in doing business day-to-day.

In today's business world, prevention of exposing people to many chemicals, not just chlorinated organics, is a must, and this should be the way of doing business.

C. PREMANUFACTURE NOTICE

Through an arrangement with FMC Corporation and the U. S. Environmental Protection Agency, PPG was allowed to begin the manufacture of CPC in Barberton in mid-October, 1985. This arrangement is called a Premanufacture Notice (PMN) consent order. In summary, this allows PPG to manufacture CPC for sale to FMC through an established EPA procedure. This procedure specifies steps that both PPG and FMC must take for compliance with the consent

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order, which is mandatory. In general, the PMN includes labeling requirements, personal protective equipment requirements, toxicity study summaries and fire fighting procedures.

A copy of the PMN is included in the Standard Operating Procedures Manual. Details of the PMN must be presented to all personnel associated with the CPC Plant as part of their training.

D. RESPIRATORY PROTECTION PROGRAM

Procedures concerning the respiratory protection program are available in the Standard Operating Procedures Manual. Locations, or situations, where respiratory protection is required are shown in the manual along with the respiratory equipment needed.

Proper respiratory protection must be used at all times. There is a breathing air system in the CPC Plant that is to be used when working around PC or CPC. Information on this system is available in the General Safety section of this Operating Manual and in the CPC Standard Operating Procedures (SOP) Manual.

E. EMERGENCY PROCEDURES

Reaction to emergencies is very important. In order to do this, each person in the CPC Plant has to know his job and, during an emergency, perform his individual job in teamwork fashion along with the other operating people in the Waste Treatment Plant. Job knowledge and performance in team fashion minimize emergency situations, should any arise.

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It is very important that emergency situations be taken seriously in the CPC Plant because of the health and fire hazard of CPC and PC, for example.

Further emphasis is given in the Safety section of this Operating Manual concerning emergency situations. Detailed procedures for response to emergencies in the CPC Plant are stated in the Standard Operating Procedures Manual.

F. SAMPLE COLLECTION AND HANDLING

Sample points have been piped up in the CPC Plant in a fashion that eliminates or significantly reduces personnel exposure to chlorinated organics. It is expected that samples are caught using the piping and equipment as intended. Analyses should be performed in the lab hood. Detailed information is available in the Standard Operating Procedures Manual on sampling.

G. VESSEL ENTRY

Procedures for vessel entry have been prepared in accordance with the PPG Work Practice Guidelines and are available in the CPC Standard Operating Procedures Manual. These procedures are not intended to replace the standard PPG vessel entry procedures given in the Employee's Manual of Accident Prevention. But rather, they give additional requirements for vessels that formerly contained CPC or PC in any concentration.

H. OTHER EQUIPMENT

Detailed procedures for taking other equipment such as pumps out of service, clearing, and putting such equipment back in service are available in the Standard Operating Procedures Manual for the CPC Plant.

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I. CLEARING OF VESSELS AND LINES

In accordance with the PPG Work Practice Guidelines, procedures for the clearing of vessels and lines have been prepared and are available in the CPC Standard Operating Procedures Manual. Such things as hazards, treatment, protective clothing required, clearing procedures, and special precautions are discussed.

J. RESTRICTED AREAS

The CPC Plant has been established as restricted area on a continuous basis. All persons who enter the restricted area must be authorized to do so. A review of the PMN is required before a person can be allowed to enter the area.

K. MONITORING PROGRAM

The CPC Plant monitoring program consists primarily of two means of sampling the ambient air in the restricted area. These are:

1. Individual Personnel Monitoring
2. Portable Detector Monitoring

Individual Personnel Monitoring is accomplished by the use of small sampling devices which are worn by individuals. These sampling devices sample the ambient air in close proximity to what the individual is actually breathing. These samples must be analyzed in the Main Laboratory to determine if an individual has been exposed to CPC or PC and, if so, how much.

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Portable Detectors are used to make routine checks throughout the area. They are also used to search out exact locations of leaks should Individual Personnel Monitors indicate problems in a given area or for a given task, such as sampling.

L. MEDICAL SURVEILLANCE

Medical surveillance consists primarily of medical examinations, including blood work, of persons working in areas where they could be exposed CPC or PC. Because of the lack of toxicity data for PC and CPC we intend to use the same medical surveillance procedures which are currently in effect for persons working in vinyl chloride areas. Basically, this involves pre-employment physical examinations, preplacement physicals (before a person starts working in the CPC Plant), periodic examinations, examinations of employees formerly assigned to the area, examination of employees involved in an emergency situation where large quantities of PC or CPC were released, and examinations of ex-employees.

The examinations consist of tests that are required by law (for VCM) as well as those deemed necessary by the company physician. Approval for working in the restricted area is the company physician's decision.

M. TRAINING

Training for CPC has been established by the PMN. This training program is carried out in conjunction with the usual operator training program which is given prior to an employee being approved as an operator in the CPC Plant and/or in conjunction with the normal plant safety training program for all employees. This program consists of:

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For Plant Operators:

1. Operator training school, testing and trial period prior to being classified as a CPC Plant operator.

2. Annual review of the program with all operators approved for the CPC Plant.

3. Safety meetings.

For All Employees Associated or Possibly Associated with CPC:

1. Safety meetings.

N. PERSONNEL EXPOSURE STANDARDS

In Derivatives there are many chemicals to which people can be exposed. Each has a different exposure standard. Exposure standards for some chemicals, specifically PC and CPC, have not been developed, so toxicity guidelines are used for these compounds. The Safety Department has a listing of these standards. The standards are to be the control used for any question related to personnel exposure to chlorinated organics. A copy of these standards is maintained in the control room. PPG has set the toxicity guideline for PC, CPC at 1 ppm ceiling.

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V. DESCRIPTION OF SYSTEMS AND OPERATION

A. GENERAL

1. Cooling Tower Water System

The cooling tower water system is a vital part of CPC operations. Proper care and operation of this system are essential. If neglected corrosion and fouling of process equipment results and leads to shutdowns and costly repairs. The OHC cooling tower system is designed to supply 5,500 GPM of water at 90°F.

The cooling tower water system is a closed loop in which chemically treated water is circulated from the cooling tower basin through process heat exchangers, and then back to the tower. The cooling tower water system consists of the two OHC cooling towers, four cooling tower pumps, acid and treatment chemical storage and feed systems, and the water distribution system.

The major users of cooling tower water are the primary condenser in CPC, the #2 DH still condenser, and the Bottoms Plant.

After removing heat from the process heat exchangers, the hot water returns to the top of the cooling tower and is distributed through the towers. As the water flows downward over the fill material inside the towers, it contacts the upflowing air that is pulled from the bottom sides of the towers by the fans on top. A portion of the downflowing water evaporates, thereby cooling the remaining water. The cooled water drains into the basin where it is pumped through another recirculation cycle.

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2. Nitrogen System

The purpose of the Nitrogen System is primarily to provide nitrogen for padding process equipment such as CPC storage tanks and system vent for pressure control, and purging equipment to clear it for maintenance.

Nitrogen is very important in its role in pressure control throughout the plant. If pressure control fails due to lack of nitrogen, serious operating difficulties as well as unsafe conditions may result. Nitrogen is very costly. Keep its use to the minimum required.

The nitrogen system consists of a network of piping to the individual users and to the utility drops. Nitrogen is identified at each of the utility drops and has the connection per the plant specification. The plant specification has been developed to alert personnel that it is nitrogen. The purge connection on equipment for hooking up nitrogen meets the plant criteria. All connections on nitrogen or tie-ins to equipment must be replaced with the exact same type of fitting.

The nitrogen for the CPC Plant is taken from the nitrogen system that supplies the entire Lake Charles complex. The CPC supply comes off of the system through a six-inch line and enters the unit on the east piperack. The nitrogen flow for the area is transmitted to the WTU control room and alarms and indications are provided there.

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3. Steam and Condensate Collection System

The purpose of this system is to provide and maintain a steady reliable source of heat for the various heat exchangers in the CPC Plant. It also provides a system of collecting steam condensate for reuse. A discussion of steam trap operation, header warm-up, and troubleshooting is included in the SOP Manual.

There are two steam systems in the CPC Plant: 150 PSIG and 50 PSIG. The 150 PSIG steam comes off the 150 PSIG steam header feeding the area from Per-Tri. The tie-in is located in the pipe rack north of the old OHC control building.

The plant-wide steam system has a steam load shedding system incorporated in it to protect critical steam-driven equipment in case of upsets at the Powerhouses. In this load shedding system, steam users are automatically cut back or cut completely off to keep from drawing down the steam line pressures.

Most of the time the 150 psig steam will come from the Per-Tri Dowtherm system and be "saturated" (366°F). On occasion, the 150 psig steam entering the area is superheated steam around 600°F to 650°F. Before going to the reactor heater, the 150 PSIG steam temperature is reduced to about 380°F by a desuperheater. This is done by injecting condensate from Per-Tri into the steam header.

The 50 PSIG steam originates from the 175/50 PSIG steam reducing station located north of #3 incinerator. Makeup steam for the 50 psig system is

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supplied from the 175 psig system. The #3 incinerator flash tank, the CPC flash tank, and the Per-Tri flash tank all produce 50 psig steam into the header also.

Condensate formed in the process is collected in one of two condensate collection headers, high pressure or low pressure. The low pressure condensate collection header returns condensate to the low pressure condensate storage tank. This condensate is pumped to the plant-wide condensate collection system that returns condensate to the Powerhouse.

The high pressure condensate collection header returns condensate to the high pressure condensate flash tank. Steam produced as a result of the reduction of pressure is added to the 50 psig system. Condensate from this tank flows into the low pressure condensate storage tank.

Block diagrams of the steam feed systems for the CPC Plant are presented in Figures 5-1 and 5-2 on pages 5-6 and 5-7 of this manual.

4. Sabine Water System

The Sabine water system is a support system for CPC. Sabine water is not potable. It is used for process water and is not supplied to any utility stations or safety showers.

Sabine water is supplied from the plant distribution system. Sabine water is brought into the CPC area on the east-west piperack. Ultimate responsibility for the Sabine water system lies with Powerhouse "C" personnel. In the

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FIGURE 5-1

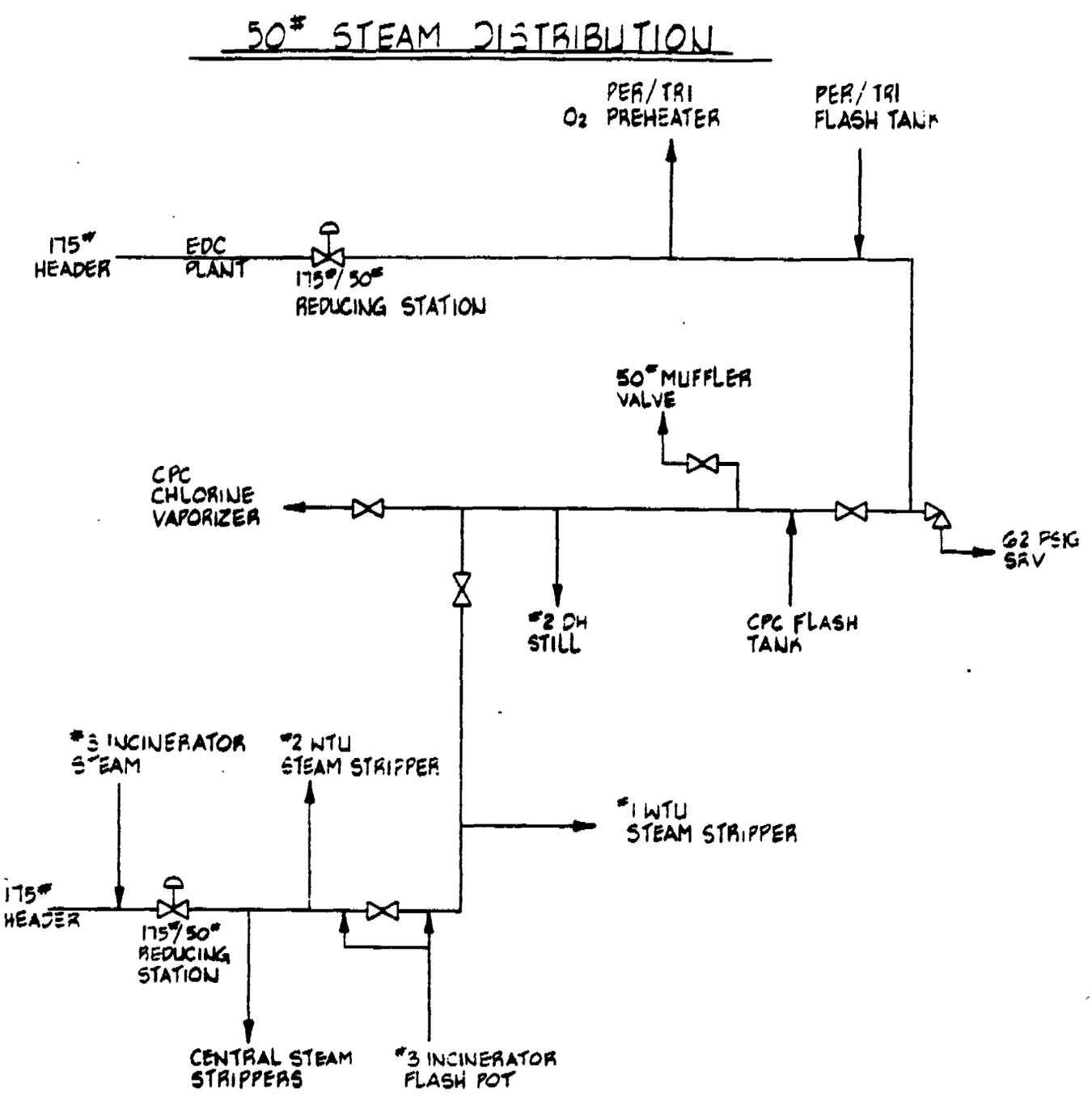

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FIGURE 5-2

150# STEAM DISTRIBUTION

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event of problems such as low pressure or inadequate flow, the WTU control room should notify "C" Power personnel. In the event of a power failure, Sabine water flow is lost, as the power to the distribution system is supplied from the WTU unit.

Sabine water is distributed to the reactor bayonet heater in the CPC Plant at the end of the last distillation step. This allows the contents of the reactor to be cooled before transferring to another tank.

5. Well Water System

Well water is supplied from the plant well water distribution system. Well water is piped to CPC along the east-west piperack through a four-inch pipeline. There is no process use of well water. Safety showers and utility drops on the upper decks of CPC are supplied with high pressure well water by the well water booster pumps. The high pressure well water header pressure is maintained at approximately 100 psig by recirculating a portion of the discharge back into the suction header. All other well water is piped to utility stations, safety showers, the control room and the maintenance shop. The well water system is not potable. Drinking water is pr vided in the control room.

6. Plant Air System

The main function of the plant air system is to provide air for operating the control instruments. In the CPC Plant, instrument air and plant air are the same. The air is also used to purge vessels of nitrogen when entry is required, but it is not to be used for breathing air, which is discussed in the next section.

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The plant air system is supplied to the plant by Liquid Air and the Elliott air compressors located west of VCM-I.

The air from Liquid Air is delivered at control conditions of 80 psi and -40°F dewpoint. PPG's check meter is located in the meter station area and is tied into the meter station area computer. Liquid Air has a meter and a flow control valve on the line at their plant. In order to protect their air separation process from upsets, the flow control valve will close on either high flow or low pressure. PPG's normal take is a flow rate of 2500 SCFM. The take can be up to 4000 SCFM. Per-Tri handles the communication with Liquid Air.

Liquid Air has a moisture analyzer on the line and will notify PPG when the dewpoint gets higher than -20°F. There is also a moisture analyzer located at VC-I that measures the dew point of the plant air.

The air supplied by Liquid Air enters the Plant Air System at the meter station area. The air from the Elliott air compressors enters at the west end of the header which runs through Plant B. The Elliots are operated by the personnel in the VCM-I Control Room. The compressors are set to maintain a pressure of about 80 PSIG. The air is dried to approximately -40°F d w-point and filtered. Air from Plant C can also be supplied into the Plant B system. Normally the Silica Products Unit is supplied from Plant C, but can be supplied from Plant B.

The supply line for CPC is three inches in diameter. It enters CPC on the east-west piperack.

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7. Breathing Air System

The PPG and OSHA regulations require respiratory protection as defined by the guidelines in Personnel Exposure Prevention to Chlorinated Organics. Also, the Premanufacture Notice for CPC discussed in Section IV of this manual requires respiratory protection. The Breathing Air System provides one method of protection.

The distribution system is a network of piping to fixed air drops. The fixed drops are located in the areas of high potential use. The most frequent usage is maintenance work on pumps.

A supply of masks and hoses are furnished at the Control Room. Hoses are also in the process area. The fixed air drops can be used at the spot if the need is for protection when a piece of equipment is being opened up or cleared. If the leak is small and in a contained area, a drop in an upwind area can be plugged into and then the leak approached. A contaminated area is not to be entered prior to having respiratory protection.

The system has a bank of breathing air cylinders → located in the old otic catalyst bldg. as the source of air. With all four cylinders at maximum working pressure, they will provide approximately 975 SCF of breathing air. If it is assumed that the system is operating at near full capacity (40 CFM) the breathing air system will be depleted in approximately 24 minutes.

The air is often used by shipping and maintenance employees.
~~The use of the bottles should be logged~~. The amount of air should be regularly checked.

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Proper maintenance and routine checks [redacted] ensure a source of air when it is needed. The detailed procedures on these items are in the SOP.

8. Electrical Power System

Electrical power to CPC is supplied from the OHC switchgear room located above the old OHC control room. The SGR houses the three 480V motor control center buses and transformers. It also houses the lighting and instrument power supply.

a. Ground Detection

All three 480V motor control centers have individual ground detection equipment on each motor control system. Any ground detected activates an alarm in the WTU Control Room. If a single ground occurs, operations can continue normally for some time. If a second ground occurs on the same bus on a different phase, a fuse blows and disconnects; therefore, anytime a ground is detected, the area electrical personnel should be contacted to locate, isolate, and repair the ground.

b. Emergency Switching

The CPC plant electrical system has been designed so that every motor driver for pumps, compressors or fans, and its spare are on different feeder systems. If any portion of one system fails, the other unit for those affected should be able to be started immediately. Starting the spares and notifying supervision should be the extent of the operator's electrical responsibilities in the event of a system failure. Trip of a feeder may cause a serious plant operating upset. Refer to the emergency procedures section of this manual for more information on this situation.

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By design, all power transformers will normally be loaded to a 45-55% rating. If a transformer is out of service, the other transformer on the system can be loaded to 90-110% by closing the tie between the buses. Due to unbalanced loading of the buses and feeders, these two systems may be operating at slightly different voltages or out of synchronization. The area electrical foreman must be contacted before making any ties.

c. Emergency Power

There is no emergency power, as such, to the CPC plant. Either one of the feeders to the plant is sized large enough to handle the total plant by itself after proper switching. All lighting and non-critical instruments are fed from lighting transformers. The outdoor sodium vapor lights do not relight instantaneously after a transfer, but there are strategically located incandescent lamps which do give instant light. All outdoor lighting is switched by lighting contactors which are photoelectrically controlled. The pressurizing blower for the SGR is fed by the transfer device at 480V.

d. Switchgear Room

The switchgear room is equipped with a flammable vapor detection system, and portable fire protection. These safety systems are described in section III-L of this manual. The intake air has a vapor detector which shuts down the pressurizing blower and alarm the control room when flammable vapors are detected.

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The SGR is equipped with an environmental control unit for filtering the incoming air, heating/cooling/dehumidification, and maintaining SGR pressurization.

Schematics of the electrical system can be found on the electrical one- line drawings. A set of these drawings is kept by the Electrical Group.

The electrical system must be properly maintained and monitored if safe, trouble-free operation is to be expected. Regular operator rounds are required to check voltage and amperage levels on buses and transformers, transformer conditions (level, temperature, pressure), ground detection system. The rounds are performed by the Tetra operators.

9. Chlorine System

Chlorine is used in chlorinating pivaloyl chloride in the CPC reactor.

The CPC plant is supplied liquid chlorine from the Liquefaction Unit. The chlorine is pumped from a liquid chlorine storage tank through a two-inch line which enters the CPC plant on the east side from Per-Tri. No other plant is on the line to CPC. A dedicated scale tank is isolated from the rest of the systems at Liquefaction.

When maintenance is required on the dedicated tank or pump, the CPC plant is fed from another tank which is tied to the rest of the Liquefaction system. During this period, the chance of losing the chlorine supply is greater; i.e. the switching of tanks is a potential cause of loss of feed due to

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swings in flow, causing the excess flow check valves in the discharge of the pump to seat.

Liquefaction produces into the dedicated tank and pumps to CPC at the same time. The supply pressure is set by adjusting recirculation back into the tank. At the discretion of Liquefaction, the flow-in is balanced to match the flow-out, or the tank is filled and then allowed to go to a low level. The former is the normal procedure. The personnel at Liquefaction need to know immediately of changes in the usage rate at CPC. CPC personnel supplies this information to Liquefaction.

The isolation valve for the chlorine supply line is located just south of #1, 2 EDC Plants. Between this valve and Liquefaction are several expansion bottles. Also downstream of this valve, north of the Per-Tri Control Room, is another expansion bottle assembly. Chlorine has a very high coefficient of thermal expansion. If liquid chlorine is trapped between two valves, high pressure will develop upon increase in the temperature of the chlorine. This pressure may lead to hydrostatic rupture of the line. The bottles are designed to protect their respective sections of line if it is blocked in. The block valves between the bottles and the chlorine supply line are not to be closed except for repairs. When the battery limit isolation valve is closed, make a check of the expansion bottle block valves to ensure they are open. Except for emergencies or maintenance on the line, the isolation valve is not to be closed. The two bottle assemblies have a rupture disc rated at 400 PSIG between the bottles. Normally the secondary bottle should have essentially no pressure on it. Pressure on the secondary bottle is an indication that either the disc has failed prematurely, or the line has been

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blocked in. In either case, the system should be checked thoroughly and the disc replaced. Per-Tri monitors their bottle assembly.

By following proper procedures, the trapping of liquid chlorine in lines is avoided. When chlorine is not needed, it should be blocked downstream of the vaporizer. Liquefaction is to be notified. If it is necessary to isolate the vaporizer, Liquefaction evacuates the line. Liquefaction does not isolate the supply line at the scale tank without evacuating the line. Notify liquefaction if liquid is trapped in a section of line or the vaporizer. This is especially important when the trapped liquid is opened back into a section which has been or is being evacuated. Liquefaction is able to take care of liquid chlorine, if they are aware of what is needed and when. An unexpected dumping of liquid chlorine into the sniff system can lead to a chlorine release to the atmosphere at Liquefaction.

More information on the chlorine vaporizer is found in the PC Chlorination Reaction section of this manual and the SOP manual.

10. Cell Liquor System

Cell liquor (10-12% NaOH solution) is pumped to the Plant B cell liquor distribution system from Plant C Caustic. CPC is fed from the Plant B system. The line to CPC enters the plant from the west on the piperack from the OHC tank farm.

Cell liquor is distributed to the emergency scrubber recirculation tank. It is needed to prepare an initial charge of recirculating liquid for the

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scrubber and may be necessary during actual operation of the scrubber to maintain pH.

11. Refrigerated Brine System

The purpose of the refrigerated brine system is to provide a cooling medium for use in process condensers that require a lower temperature than is provided by cooling tower water. By using refrigerated brine, the amount of useful product lost to the incinerator can be minimized.

The refrigerated brine is supplied to the CPC Plant from Per-Tri. A refrigeration unit is used to cool $CaCl_2$ brine to about 20°F. The unit is designed to maintain a constant brine temperature. The Per-Tri operators run samples on the brine and make any needed adjustments to the refrigeration unit.

Refrigerated brine is used on the CPC secondary condenser and the brine chiller on the CPC vacuum compressor. Since these exchangers are at a higher elevation than the brine condensers in the Per-Tri unit, the CPC Plant is equipped with a brine head tank. This atmospheric tank floats on the outlet of all the brine condensers.

The level of brine in the head tank should be monitored. Too low a level in the system can allow air into the system thus greatly reducing their cooling ability. If a low level is detected it is necessary to make-up brine to the system. This is done be mixing $CaCl_2$ and water and adding to the system to raise the level in the head tank. Do not overfill the tank because when the

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refrigeration unit is shut down the volume of brine will expand as the temperature rises and overflow the head tank.

The Per-Tri operators are responsible for mixing the brine. Coordination between the CPC operator and Per-Tri operator is necessary when filling the head tank.

12. CPC Plant Materials Of Construction

Due to the extremely corrosive nature of the chemicals used in the CPC Plant there are special considerations as to materials of construction. Suitable materials are glass, glass-lined steel, Teflon-lined steel, or tantalum. A new packing (for the distillation column) made of liquid crystal polymer (LCP) is being used. Under certain conditions nickel, Inconel and Hastelloy B2 or C as well as Derakane vinyl ester are used. Some of these materials require special care in their operation and maintenance. The CPC Plant SOP has details on these materials.

(Also, Kynar or stainless steel be used in low temperature service

13. Condensate Collection System For CPC Condensers

Throughout each batch operation, three different tanks are used to collect condensate from the two distillation column condensers. Furthermore, since the batch is at times operated under vacuum, the vent from each of the condensate tanks can be routed to either the secondary condenser or the CPC Plant incinerator header (downstream of the vacuum pump). Proper vent routing is essential to equalize pressure on the tanks with the condensers. Three-way valves are used on the vents.

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As can be seen, numerous options are available for directing condensate flow and aligning tank vents. This section is devoted to providing a clearer understanding of the proper orientation of condensate drain and tank vent lines during each of the following four steps in the CPC batch:

Reaction

PC Distillation

CPC Pre-Cut

CPC Distillation

Figures 5-3 and 5-4 on pages 5-20 and 5-21 are provided to clarify the following narrative. Supplementary information is presented in Sections V.C. and V.D.

Reaction - The system is operated under enough pressure (2-6 psig) to transfer the process vent to the incinerator. The distillation column is operated under total reflux. Condensate is lined up to the reflux pump only. The block valves on the condensate to the PC recycle, still recycle and CPC product shift tanks are closed. Vents from the still recycle and CPC product shift tanks are routed to the incinerator header. Note that when the CPC product shift tank is vented to the incinerator, a PCV on the vent from the tank keeps the tank free from back-flow of contaminated vents so CPC product purity will not be affected. The PC recycle tank is vented to the secondary condenser so it is equalized with the reactor and any PC vapors are condensed during transfer of PC from the tank to the reactor at the beginning of the reaction step.

PC Distillation - This step may be conducted under vacuum. ~~is also operated under pressure~~. The only change in valving of the condensate or vents from the reaction step is to

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open the condensate drain to the PC recycle tank. The reflux splitter is set to the desired reflux ratio and flow alternates to the PC recycle tank and the reflux pump. During PC distillation, the contents of the still recycle tank are drained to the reactor pot.

CPC Pre-Cut - This step is conducted under vacuum. Condensate is drained to the still recycle tank so the block valve to that tank is opened and the block valve to the PC recycle tank is closed. The PC recycle and CPC shift tanks are vented to the incinerator header, but the still recycle tank is vented to the secondary condenser to equalize pressure for proper draining of the condensate.

CPC Distillation - Vacuum operation continues. The still recycle tank is placed back under pressure by slowly opening the vent valve to the incinerator. The CPC product tank is slowly switched to vacuum operation and the block valve on the condensate to this tank is opened. The block valve on the still recycle tank is closed. At the end of the CPC distillation step the entire system is placed under pressure by shutting down the vacuum pump and adding N_2 to the vent header. After the system is under pressure, the vent and condensate valve arrangement is reset for the reaction step. The waste bottoms left in the reactor can then be drained to the incinerator feed tank (see Section V.E.).

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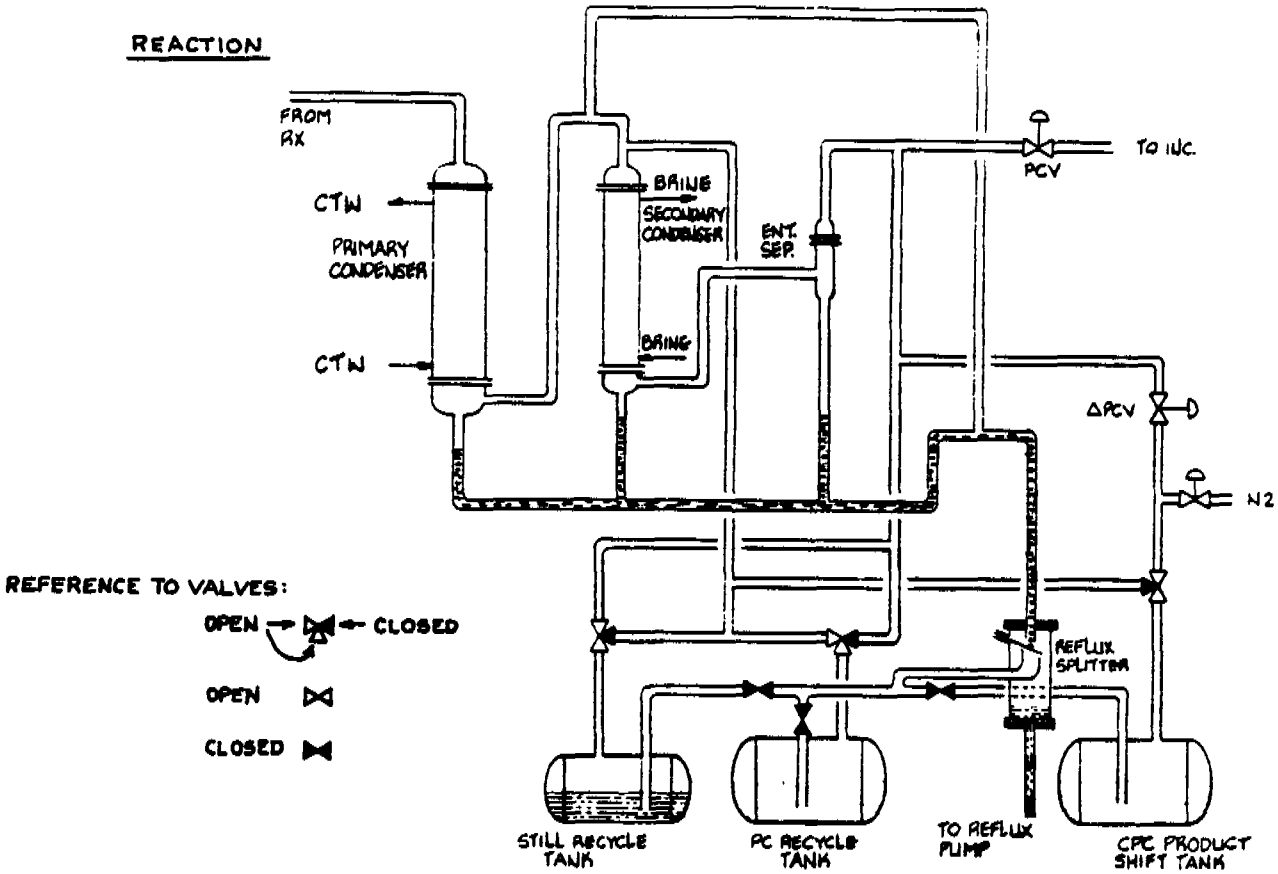
FIGURE 5-3
FLOW ARRANGEMENT FOR REACTION AND PC DISTILLATION STEPS



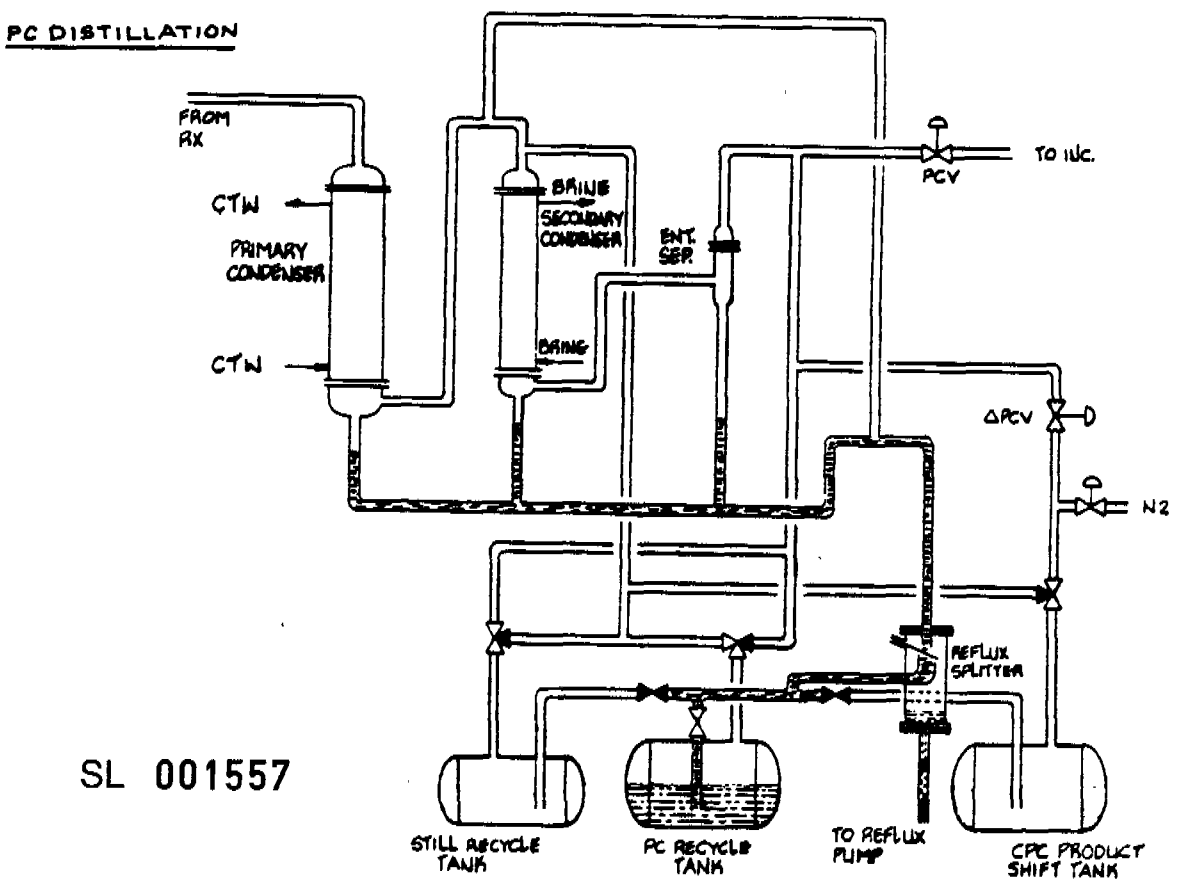


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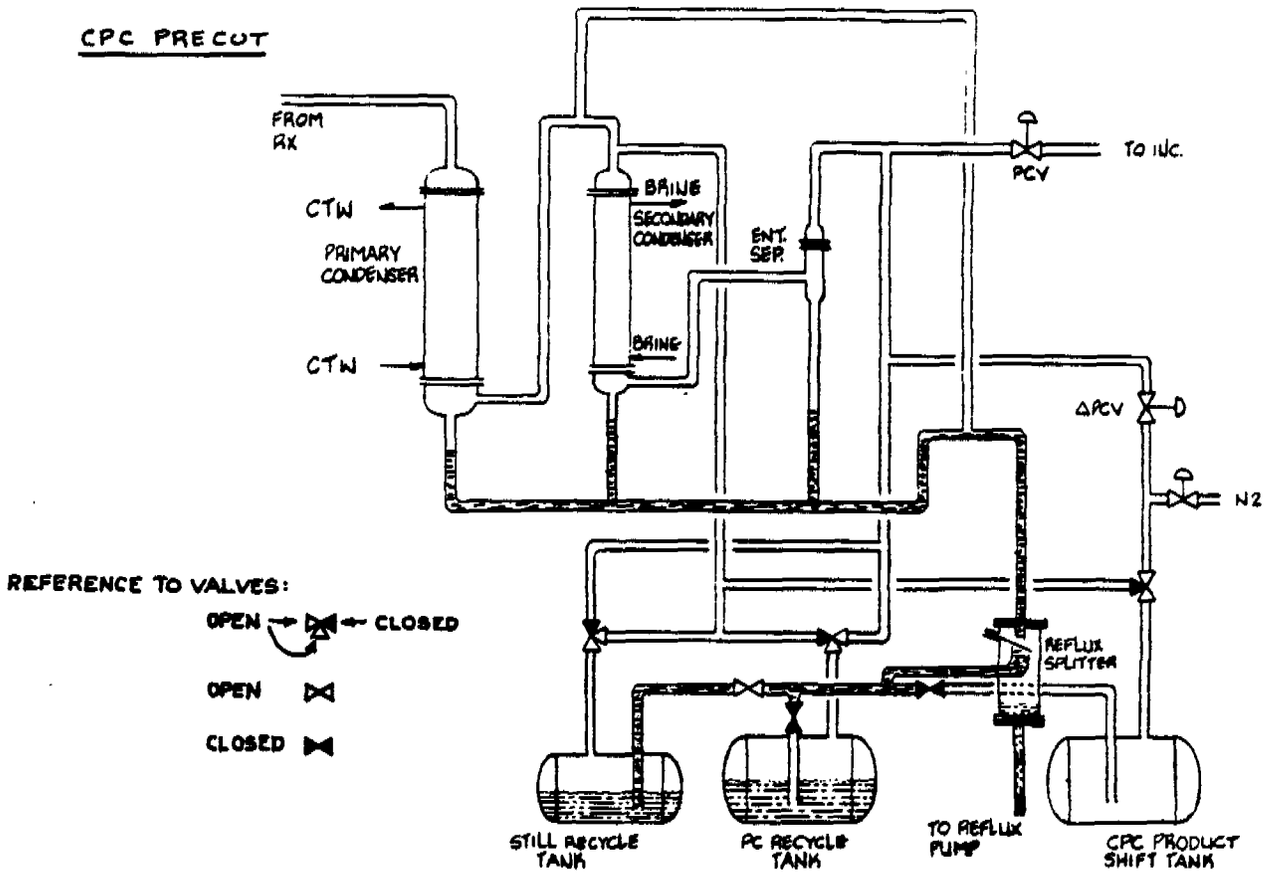
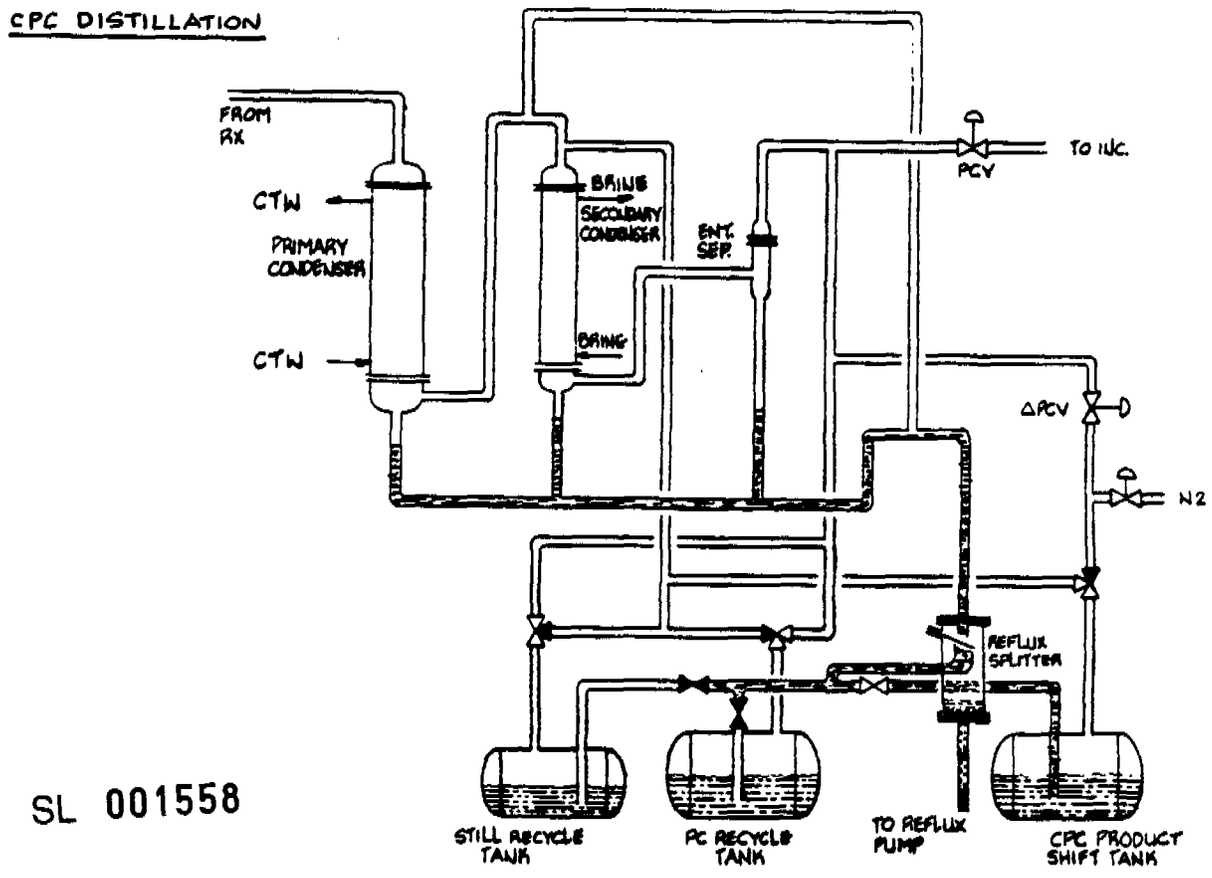
FIGURE 5-4
FLOW ARRANGEMENT FOR CPC PRE-CUT AND CPC DISTILLATION STEPS





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B. PC UNLOADING AND STORAGE

The main purpose of the PC unloading system is to provide an adequate method to transfer PC from the incoming 40,000 pound (net) truck to the CPC reactor feed tank. PC is shipped from the PC Plant in LaPorte, Texas in Derakane 470 lined trucks which are dedicated to this service. The trucks are cleared by analysis prior to leaving LaPorte. Prior to unloading PC at Lake Charles, the analysis certification sheets are verified.

The main purpose of the CPC reactor feed tank is to provide sufficient process surge time for uninterrupted operation of the CPC Plant. Another purpose of the tank is to provide storage capacity for out-of-spec material produced during any of the steps in the batch operation. This secondary use is employed only if all other tanks, which can used for storing out-of-spec material, are full. For easier process control and accountability of raw material, it is better if this tank is kept free of recycle streams.

The CPC reactor feed tank has a capacity of 10,000 gallons and holds up to 80,000 pounds of PC (full). At design rates of 8MM lb./yr. CPC, this provides enough feed for about 3½ days. Trucks arrive with PC at least every three days. A drawing of the PC unloading process is presented in Figure 5-5 on page 5-24.

The unloading area is [redacted] protected from rain. [redacted] precautions have to be taken when unloading a truck in inclement weather since PC and water react to form pivalic acid and HCl gas. Contamination of the CPC reactor feed tank occurs if the PC gets wet. If contamination is not too great the reaction is probably not affected but any pivalic acid formed results in a yield loss.

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Flexible hoses are used to hook-up the truck to the CPC feed tank. A breathing air station is located in the truck loading/unloading area for use with a full face mask when needed.

PC is unloaded by using nitrogen to pad the truck thus pressurizing the PC to the CPC reactor feed tank. The nitrogen in the area is regulated at 15 psig. This is important, as the SRV on the feed tank is set to relieve at 30 psig. Once PC is unloaded the line is cleared of PC by purging to the truck and to the tank independently with nitrogen.

Included in the PC unloading and storage area are the CPC reactor feed tank, an unloading line with nitrogen hook-ups for purging, a nitrogen pad and vent system, a pump and associated piping for recirculating the tank, a sampling system, an SRV on the tank and associated instrumentation for monitoring tank level and pressure. The pump is provided to feed PC to the reactor or to recycle material from the reactor and other process vessels back to the tank.

All piping in this area is Teflon (TFE) lined steel except for the vent, which is ~~FRP (vinyl ester resin)~~ derakane lined FRP. The reactor feed tank is glass lined steel. The reactor feed pump is a magnetic drive sealess pump with all wetted parts being either Teflon TFE or Tefzel(R). The tank is rated for 30 psig full vacuum. It is protected from overpressure by an SRV/rupture disc set at 30 psig.

Tank level in T-1 is monitored by a sight glass. No level indication or high level alarm on T-1 is available in the control room. It is essential

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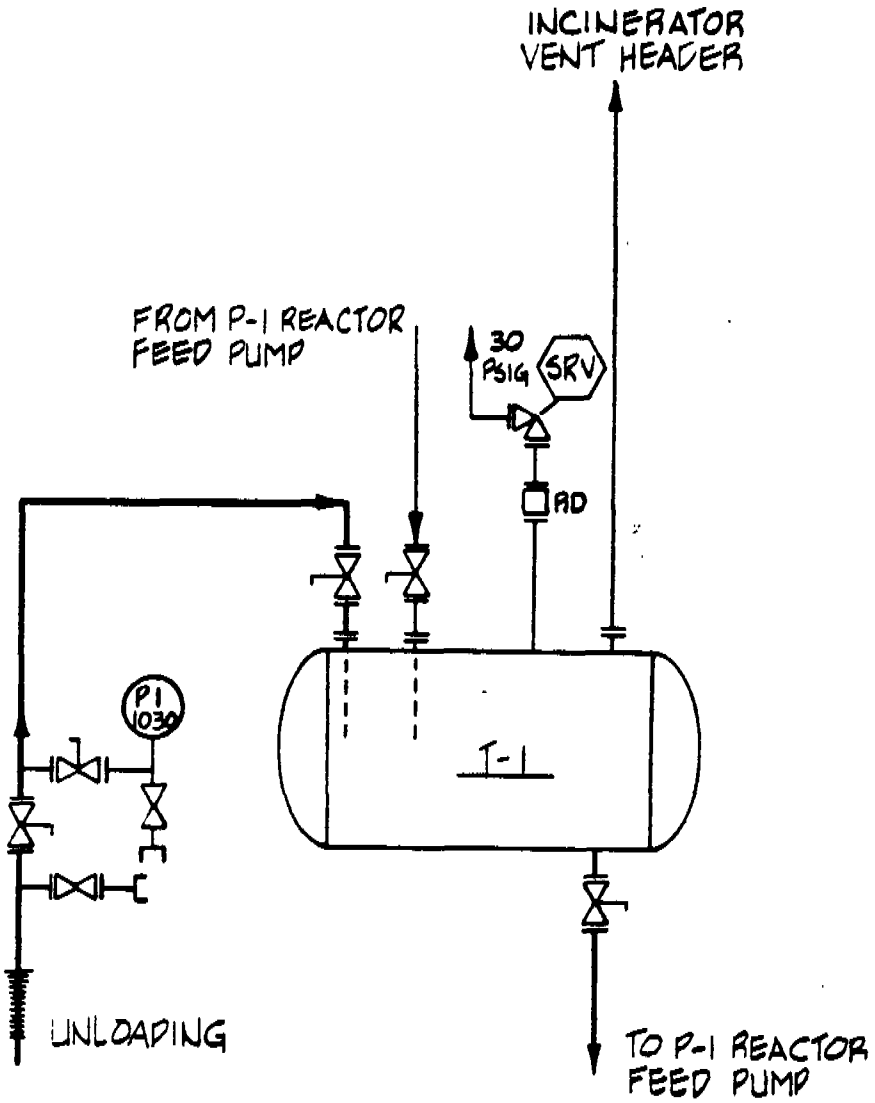
FIGURE 5-5

PC UNLOADING AND STORAGE



T-1
CPC Rx FEED TANK

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that operators are present in the area when the tank is being loaded. Prior to unloading the PC, the tank has to be checked for adequate available space by using level indications from the sight glass.

The reactor feed tank, like all other vessels in the CPC Plant except those in the emergency scrubber system, is rated for full vacuum. It is vented to the CPC Plant vent header, which goes to the incinerator. It can be isolated from the vent header for clearing with nitrogen if necessary. The reactor, still, condensers and all process storage vessels are kept at a pressure sufficient enough to get the vent to the incinerator. This is expected to be in the range of 3-6 psig. The vent header is padded with nitrogen to keep it above a minimum pressure. The vent header system will be discussed in more detail in the Section V. F. in this manual on the "Emergency Scrubber System".

The feed pump can be used to recirculate the reactor during heat-up but only if the reactor liquor temperature is below 200°F, the upper limit of the feed pump. Temperatures above 200°F may damage the Tefzel lining in the pump.

While the make-up and recycle PC are being added to the reactor, a sample of each stream is collected and analyzed to calculate material balances and yields.

The CPC reactor feed tank is surrounded by a concrete dike designed to contain organics spilled from the storage tank. Prior to draining any

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organics from the diked area to the process sewer, the spill is neutralized with soda ash and analyzed to ensure destruction of pollutants.

C. PC CHLORINATION

1. BATCH PREPARATION

In batch operations such as with the CPC Plant, equipment is often used for more than one type of operation or may not be used at all during some stages of the operation. When preparing for any batch it is important to realize that, at the end of the previous batch, equipment and utilities are not oriented properly for the start of the subsequent batch. Thus, it is essential that all equipment, lines, valves and utilities are set up for the reaction step at the beginning of each batch.

In this section of this manual a general overview of batch preparation is provided to acquaint the operator with the numerous steps which are taken prior to initiating the chlorination step. The SOP Manual provides a detailed check list for batch preparation and chlorination as well as other steps in the process.

At the end of the previous batch Sabine River water (SRW) was used to cool the heavy organic waste (heel) left in the reboiler and reactor. Before taking 150# steam the SRW is drained from the steam and condensate lines. SRW to the reboiler is then isolated (from the steam line) with block valves. The 150# steam system is then readied for reactor operation. ~~This involves notifying the Per-Tri operator that you are ready to take steam.~~ If high pressure condensate to the desuperheater is available, unblock the 3" steam header upstream of the SRW tie-in, making sure the pressure

differential control valve is closed. The block valve to the steam traps and flash tank are also opened at this time making certain the flash tank is lined up properly to the 50 psig steam header and atmospheric flash tank. Check the steam temperature and pressure before opening the ΔPCV to the reboiler. The header is then warmed up slowly before taking the full steam requirement.

During the final distillation step, when CPC product is being recovered to the CPC product shift tank, part the CPC system is under vacuum and at the lowest pressure of any of the processing steps. The reaction takes place under a slight pressure - enough to get the vent to the incinerator - so the system is slowly brought to atmospheric pressure (0 psig) with nitrogen at the end of the CPC distillation step (Section V.D.6.). A detailed procedure for doing this is in the SOP Manual. Prior to the reaction step, the operator checks the pressure throughout the system to ensure that the vacuum pump is bypassed and all vessels and piping are operating under atmospheric or slight pressure.

The reaction takes place while the still is on total reflux so valves to all three of the tanks used for collecting the recycle PC, mid cut, and CPC product are isolated from the reflux splitter. The vessels are vented to the incinerator header. Each of these three tanks is equipped with a three way valve on the vent to allow independent operation under either vacuum or pressure depending on the process step. When necessary the tank vents are routed to the secondary condenser to equalize pressure in the tank and condensing system.

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When the plant is operating under pressure, the vent can be routed to the secondary condenser or downstream of the vacuum pump. Specific routings will be covered in applicable sections.

The Cl_2 vaporizer should be in the "float" mode. If it is not, refer to the S.O.P. Manual for specific instructions on starting up a cold vaporizer. The Cl_2 vaporizer will be discussed further in the next section.

In the previous batch, the solvent, PC or a mixture of PC and CPC, was transferred to the catalyst mix tank during one of the distillation steps. The catalyst mix tank can be filled using the reflux pump. The solvent can be transferred to the catalyst mix tank during the reaction or one of the distillation steps. ~~To prevent unstable still operation~~, The tank ~~can be filled while loading the reactor with recycle PC or while adding the mid-cut (CPC + PC) to the reactor pot at the end of the distillation step~~ is normally immediately before charging the AIBN addition tank while the column is on total reflux during heat-up. However, it is ~~acceptable~~ acceptable to load the mix tank during the PC distillation step (see Section F.D.3.).

The optimum catalyst addition rate will be established during operation. As previously discussed, the catalyst addition per ton CPC produced may be greater in the commercial plant due to higher temperature in the revised reaction system design.

If the required catalyst addition rate is high, a mixture of CPC and PC can be used to dissolve the AIBN. AIBN is much more soluble in CPC than in PC. However, if the AIBN addition is low, PC is preferred as the solvent. This lowers the amount of CPC at the Cl_2 injection point and, thus, lessens the

by-product DCPC formation. Additional batches of catalyst can be prepared during the reaction step if necessary. The Cl_2 feed is shut off while an additional catalyst mix is prepared.

AIBN is removed from the cold box, measured (by volume) and added to the catalyst addition pot which is setting on the level above the mix tank. The catalyst addition pot has to be cleared, isolated from the mix tank, purged with N_2 to the incinerator, isolated from the incinerator header, and depressurized before opening and adding catalyst. Both the catalyst addition pot and mix tank are vented to the incinerator header. The addition pot is then closed, left isolated from the vent header and pressurized with nitrogen. A N_2 regulator is used to keep N_2 pressure at a level safely below the 75 psig SRV setting on the addition pot. The tank is then isolated from the nitrogen system.

The catalyst solution circulation pump is rated for 40 gpm at 50' TPH. In order to artificially load the pump, the recirculation line has a restricting orifice ($\Delta P = 30'$) and a ball valve. The ball valve should be left nearly closed prior to starting the recirculation pump. If these restrictions are not present, the motor on the pump could burn out due to high flow when started.

With the solvent circulating through the recirculation pump and back to the tank, the AIBN is padded into the mix tank by opening the block valve at the bottom of the addition pot. When the pressure on the addition pot has dropped to less than 5 psig (or equal to incinerator head pressure) it can be assumed all the AIBN has been transferred to the mix tank. When the

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pressure drops on the addition pot, it should drop slowly at first and then suddenly to the vent header pressure. A continual slow drop or no drop in pressure indicates plugging of the nozzle into the mix tank with AIBN.

If there are any other indications that all the AIBN has not been dumped to the mix tank, such as lower Cl_2 utilization during reaction, the catalyst addition pot can be inspected after it has been isolated from the mix tank, purged with N_2 for several minutes, isolated from the vent header and depressurized. Breathing air is available, if needed, in the area of the catalyst addition pot.

The AIBN solution should be circulated for some period of time before it is assumed ready for addition to the reactor. The circulation pump is then used to pump the mixture into the reflux line, thus supplying AIBN to the system. ~~shut down. Other batch preparations should be made during this period. The catalyst solution is now ready for addition to the reactor. A Teflon lined metering pump is used to add the catalyst solution to the reflux stream.~~

During the reaction the reactor vent from the entrainment separator downstream of the secondary condenser is lined up to the incinerator, bypassing the vacuum pump. The vent ~~reactor~~ pressure control valve is used to control a constant reactor pressure which is sufficient to transfer the vent to the incinerator. No other plant vents tie into the CPC Plant vent, which ties into the incinerator vent header just upstream of the main burner nozzle. This vent can go to either No. 1 or No. 3 incinerator. The CPC Plant vent can be isolated from the incinerators by a FCV located on the CPC header just upstream of the tie-in to the main burner nozzle at either incinerator. The vent line to each incinerator is equipped with a flame arrestor.

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With the reactor PCV closed, the CPC Operator asks the Incinerator Operator open the FCV on the CPC vent to the incinerator. Preferably, the vent is sent to the No. 3 incinerator. The reactor pressure control valve can then be set to control at a specified pressure (2 - 6 psig).

The oxygen analyzer is not included in the reactor system. Any oxygen which enters the system is diluted with the HCl by-product to a level well below the lower explosive limit. The oxygen analyzer is operational only when the system is operated under vacuum (during part of the distillation steps) when any leak may allow air to enter the system, thus creating the potential for an explosive mixture. The O_2 analyzer is located downstream of the vacuum pump.

The emergency scrubber system is prepared and periodically checked for a proper state of readiness in case a vent release occurs. Critical checks are the alkali solution level in the recirculation tank, pH and temperature and proper lining up of recirculation piping. Vents are released to the emergency scrubber through a PCV only if the system pressure reaches 20 psig. An alarm forewarns the operators to start the circulation pumps, which are normally left off. Once the alkali liquor is circulating through the scrubber, chemicals in any vents relieving to the scrubbers are neutralized. The emergency scrubber system will be discussed in more detail in Section V. F.

Prior to the reaction, levels in the feed, PC recycle and still recycle tanks are checked and recorded. ~~Also, the liquor in each of these tanks is sampled and analyzed.~~ The PC recycle is also added to the reactor prior to

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the reaction step. Sufficient make-up PC has to be available in the feed tank for a reactor charge. Contents of the still recycle tank are added to the reactor pot during the PC distillation step.

2. CHLORINE VAPORIZER

The purpose of the chlorine feed system is to provide chlorine feed for the CPC reactor. The chlorine feed system is designed to convert liquid chlorine, supplied by Liquefaction, to a gas and feed it to the reactor at a rate that maintains a safe and efficient production of each batch of CPC. The Cl_2 feed system is illustrated in Figure 5-7 on page 5-[redacted].

The chlorine feed system begins with the liquid chlorine feed header as it enters the CPC Plant on the north-south piperack from Per-Tri, and ends with the chlorine entering the CPC reactor. The system includes the expansion bottles, vaporizer, surge drum, associated piping, and instrumentation.

The chlorine vaporizer (see Figure 5-6 on page 5-33) converts the liquid chlorine to gaseous chlorine by transferring heat from 50 psig steam to the chlorine. The vaporizer is a bayonet type heat exchanger.

Liquid chlorine enters the bottom of the shell side of the vaporizer through a distributor ring. It is vaporized and superheated by the steam in the vertical tubes. The chlorine gas exits at the side of the vaporizer near the top.

The chlorine vaporizer operates under a variable heat transfer area principle. The vaporizer runs with a level of liquid chlorine in the shell

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FIGURE 5-6

CHLORINE VAPORIZER

(SHOWING ONE TUBE)

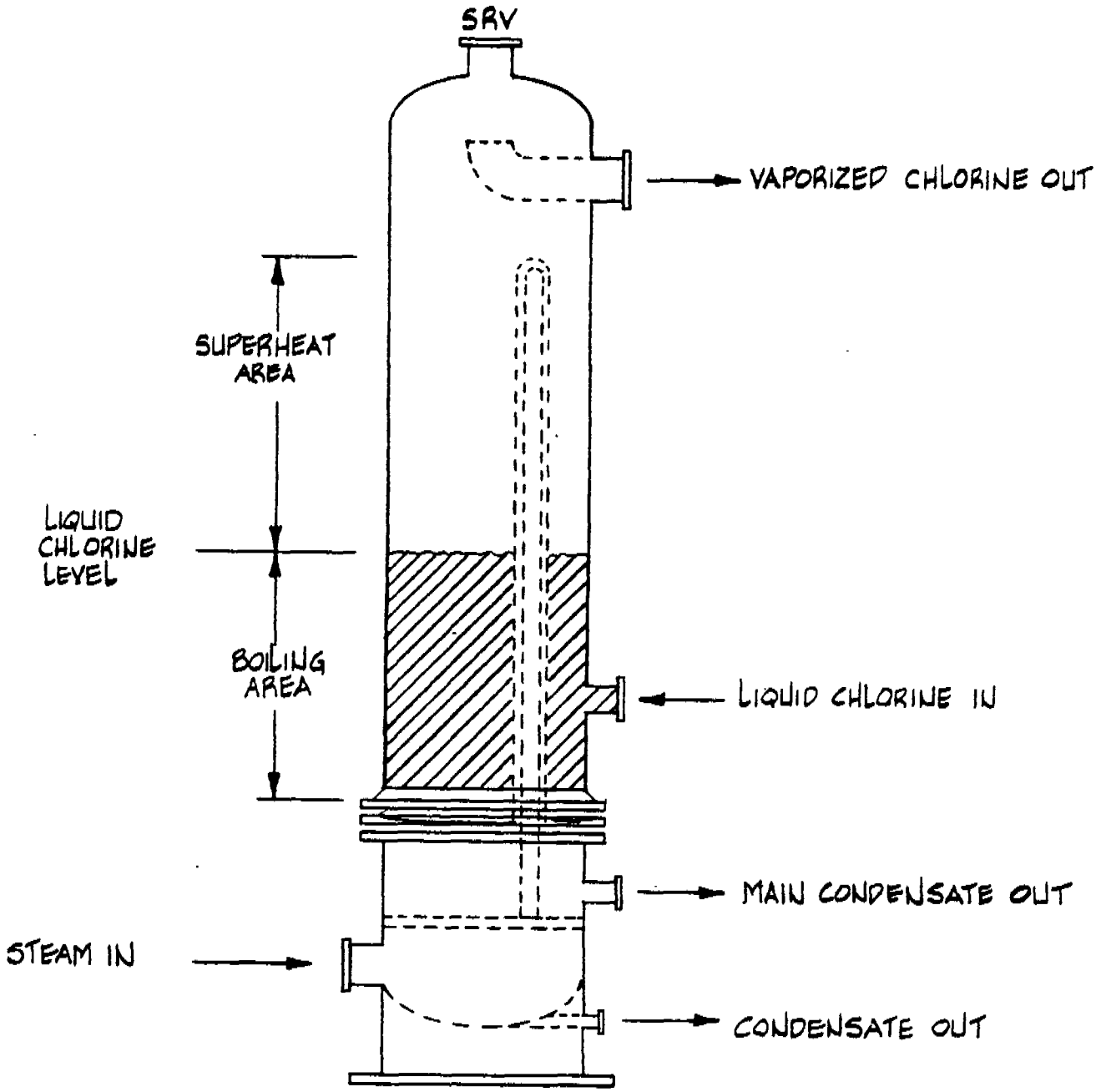

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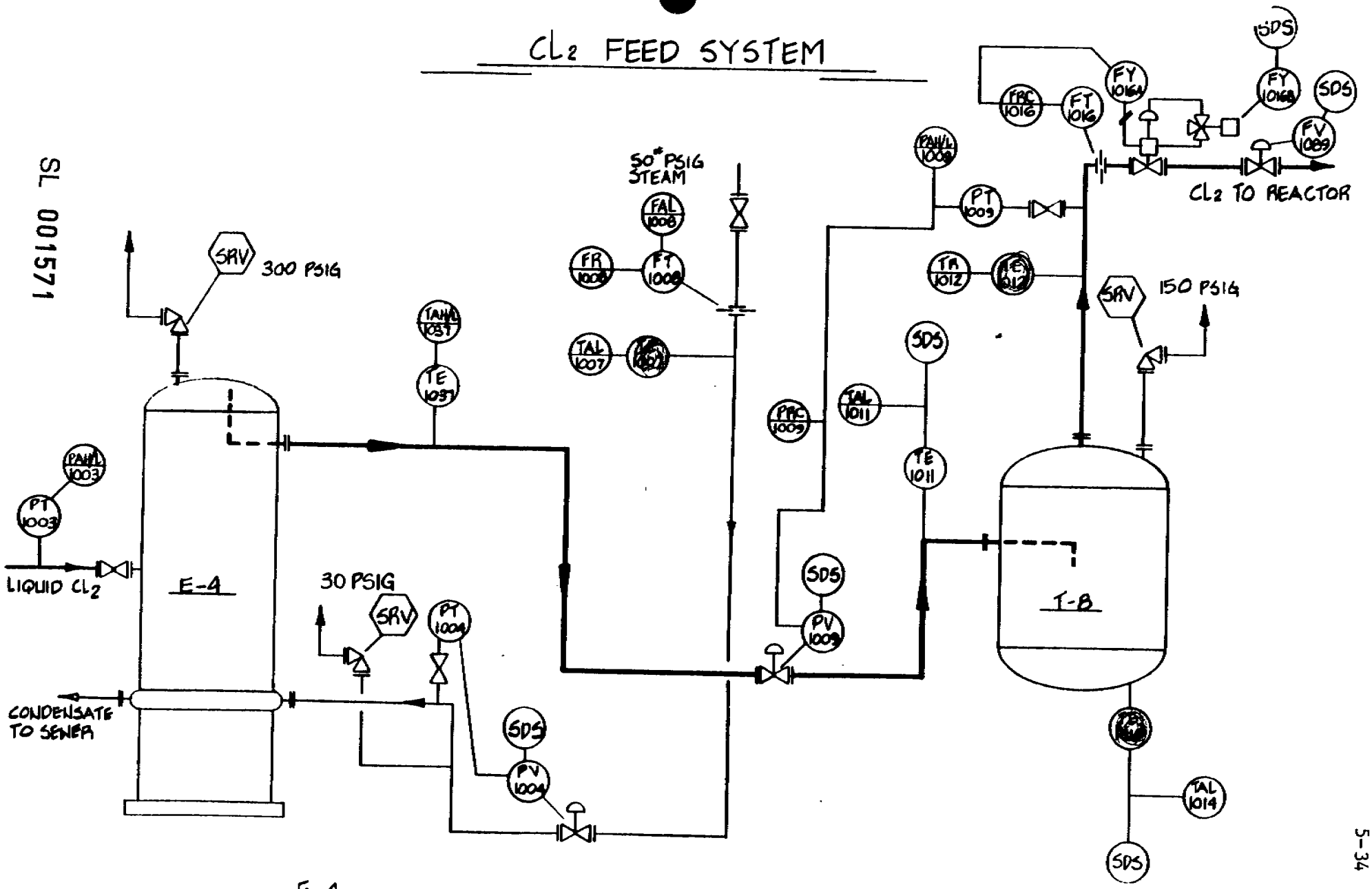
FIGURE 5-7

Cl_2 FEED SYSTEM



E-4
Cl_2 VAPORIZER

T-8
Cl_2 SURGE DRUM

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side. The chlorine chest pressure is near the liquid line pressure since the pressure control valve throttles the outlet vaporized chlorine. Steam chest pressure is maintained by a pressure control valve on the inlet steam. As chlorine throughput increases, more area is needed to transfer the additional heat; the liquid level builds up until enough additional tube area is covered to boil off the additional chlorine. Conversely, if the chlorine throughput drops, the chlorine level drops.

The amount of superheat added to the vaporized chlorine is critical to operation of the system. Liquid chlorine must never be fed to the reactor. (A discussion on flooding is included in Part c of this section.) The vaporizer shutdown system is designed to protect against this event. The result of getting liquid chlorine into the reactor is the possibility of an uncontrolled reaction. The shutdown system closes the chlorine PCV, the chlorine FCV, the anti-backup valve and the steam to the vaporizer if low temperature is detected either in the surge drum or the chlorine vapor line into the surge drum. The shutdown system is also activated by a low pressure differential across the FCV or manually from the control room. Furthermore, an incinerator trip during the reaction step trips the shutdown system for the chlorine vaporizer, and trips the steam to the reactor heater.

The chlorine flow is regulated by a flow control valve on the chlorine gas feed to the reactor. The pressure on the vaporizer is a function of the supply pressure of the liquid chlorine from Liquefaction. Between the vaporizer and surge drum is a pressure control valve which takes its impulse downstream of the surge drum and controls the pressure on the surge drum.

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When an increase in flow is called for, the FCV opens, lowering the pressure on the surge drum. The PCV opens to re-establish the pressure, which in turn allows more chlorine to flow into the vaporizer. A large change in flow or pressure could cause the excess flow check valves in the pump at Liquefaction to seat off, halting chlorine flow and shutting down the reactor.

The chlorine vaporizer uses 50 psig steam to heat the chlorine. The steam comes from the 50 psig header which runs through the unit. This supply comes from the main desuperheated 175 psig system through a pressure reducing control valve located north of #3 incinerator. A back-up supply of steam is available. EDC can make-up 175 psig steam through a pressure reducing control valve. The header can be isolated from WTU leaving CPC and the #2 DH still on steam from EDC. In either case, do not use steam on the vaporizer without the desuperheater in service. The result could be a chlorine/steel fire inside the vaporizer or high rates of corrosion. The steam flow to the vaporizer is metered. The flow of steam is regulated by setting the pressure desired in the chest. A pressure control valve maintains the pressure by letting steam in as the steam in the chest condenses and leaves by the two steam traps in parallel. Each of the traps is sized to handle the condensate flow at full rates.

The steam enters the tube side at the bottom of the vaporizer. Any condensate entrained in the steam drops out and is removed via a small trap on the bottom. The steam flows up through each bayonet tube into the main tube. The steam condenses as the heat is transferred to the chlorine. The condensate falls to the condensate chamber and is removed via the steam traps.

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The condensate is at a pressure too low to be economically recovered in the plant condensate system so is discharged to the sewer. Sabine water is injected into the condensate prior to its discharge to reduce the generation of flash steam.

The use of two traps minimizes the potential for flooding the chest with condensate. The two most common trap failures are flooding and blowthrough. Traps may flood or quit allowing condensate to flow out of the system. This blocks the steam flow, fills the chest with condensate and reduces heat transfer area. When flooding occurs, the chlorine temperature falls and, if allowed to continue, vaporization stops. (If one trap floods, the other handles the flow). Traps may become stuck in the open position causing blowthrough, or allow steam to flow through without being condensed. When a trap blows through, sufficient back-pressure is not being held and an adequate ΔT for heat transfer is not available. Again the temperature will fall until vaporization stops. The operation of the traps should be checked on a routine basis.

a. Start-up

The chlorine vaporizer has to be started up and lined out prior to starting up the CPC reactor. The vaporizer is started up and left "floating on the line", which means there is steam on the vaporizer with the inlet wide open and chlorine outlet blocked. While the vaporizer is floating on the line, liquid chlorine is moving into the vaporizer, flashing and moving back into the liquid line.

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As soon as the reactor is up to temperature and total reflux flow is established, chlorine may be fed to the reactor. ~~Prior to feeding the reactor, a nitrogen purge has to be established from downstream of the flow control valve into the reactor. The nitrogen purge clears any organic vapors or chlorine from the feed system.~~ The chlorine is started to the surge drum slowly, watching the vaporizer outlet temperatures, pressure and steam draw. ~~At this point the N_2 purge is discontinued.~~ Steam adjustments have to be made carefully to avoid pressure surges on the vaporizer which can result in lifting the SRV.

Detailed start-up procedures are available in the CPC Plant SOP.

b. Shutdown

During the distillation steps or when the plant is shut down for a short period of time, the chlorine vaporizer is left floating on the line; that is, the chlorine feed to the reactor is shut off by blocking chlorine upstream of the PCV, leaving the inlet to the vaporizer open and steam on the chest. Chlorine feed piping to the reactor is purged with N_2 to remove organics and the chlorine feed piping is blocked at the reactor to prevent organics from entering the steel chlorine piping. A special "float" mode is provided on the Cl_2 SDS to allow the vaporizer to remain in this condition.

If the plant outage is for several days or more, the vaporizer has to be shut down. The chlorine vaporizer shutdown is accomplished by slowly shutting off chlorine flow to the vaporizer while there is still some chlorine going into the reactor. This method leaves the vaporizer at the

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same pressure as the surge drum. When the steam is cut off, the pressure drops even further and no liquid forms in the vaporizer. Important: If the vaporizer is not going to be left floating, shut off the steam as soon as the inlet to the vaporizer is blocked.

c. Flooding

Flooding of a vaporizer, i.e., overfilling of the process side to the point where liquid chlorine flows to the surge drum, is a very serious problem. The vaporizer/surge drum system is equipped with a shutdown system to stop chlorine flow to the reactor if this occurs, but good operating practices can prevent flooding from occurring in the first place. A discussion of the vaporizer shutdown system is included in Part d of this section. Some of the causes of flooding are:

1. Condensate In The Steam System - 50 psig steam is recovered in the CPC unit from the 150 psig steam condensate via the flash tank. If the flash tank overfills, condensate will enter the 50 psig system and flood the chest (steam side) of the vaporizer. There is not sufficient heat in the condensate to boil the chlorine at desired rates nor can the steam traps handle the higher condensate load. There is a low temperature alarm on the 50 psig steam supply to warn of this problem.

2. Inadequate Heat Supply - If the chest pressure is too low or if steam flow is too low, flooding can occur.

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3. Chlorine Feed Rates Too High - If the chlorine feed rates exceed the design rate of the vaporizer then all superheat is lost and liquid carry-over occurs.

4. Excessive Inlet Chlorine Pressure - High inlet pumping pressure can overcome the pressure generated by the vaporized chlorine.

5. Severe Pressure Or Flow Swings - These can cause a 'burp' or temporary flooding. Make all changes slowly.

6. Liquid Filling While Down - Leak-by of the inlet chlorine valve while a vaporizer is sitting idle with no steam in the chest results in a flooded vaporizer.

A flooded vaporizer has to be started up very carefully as the introduction of only a small amount of heat could result in lifting the SRV. If time permits, Liquefaction can sniff down the vaporizer and liquid header. A vaporizer can be depressurized into a drum of water/caustic but this is a lengthy process if much liquid is to be removed. Another alternative is to use N_2 cylinders to pad the chlorine back into the header. Do not use plant nitrogen as its pressure is too low.

A procedure for starting a flooded vaporizer can be found in the CPC Standard Operating Procedures Manual.

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The chlorine vaporizer is protected from overpressure by a safety relief valve/rupture disc arrangement set at 300 psig. The chlorine surge drum is protected by a similar SRV/RD arrangement set at 150 psig. The vaporizer is also protected by an SRV on the steam feed. This SRV relieves at 30 psig.

Drawings of the chlorine feed system can be found on process flow sheet 66C-10010 and P & ID's 66C-10002 and 66C-10003.

3. REACTION

a. Overview

The purpose of the reaction step is to convert a portion of the raw material, pivaloyl chloride (PC), into the product, chloropivaloyl chloride (CPC). The reaction is carried out by adding (vaporized) Cl_2 gas to PC in the presence of a catalyst, 2,2-azobis (isolutyronitrile), which is commonly referred to as AIBN. The reaction takes place at incinerator header pressure (sufficient to get the vent to the incinerator) or 2-5 psig and 220°F - 240°F.

The reactor step is the first step in a four step batch operation. Three distillation steps follow. The plant is designed to produce about 12 tons of CPC in one batch per day. Much of the plant design is based on data from pilot plant operation. However, the reaction step has been revised to improve yields by reducing by-product formation.

If the reaction were carried out by feeding gaseous chlorine directly into the reactor liquor, the CPC, as it forms, is available to react further with Cl_2 to produce isomers of dichloropivaloyl chloride (DCPC). As CPC forms,

the reactor liquor becomes richer in CPC and leaner in PC. This leads to an increased formation of by-products. DCPC is a waste product. Its formation is costly to the plant as one mole of chlorine (71 pounds) and one mole of CPC (120.5 pounds) are depleted for every mole of DCPC (189.5 pounds) formed. Furthermore, DCPC has to be incinerated and the by-product HCl neutralized.

Hence, the commercial plant reactor is designed to maximize the PC concentration at the chlorine injection point. This is accomplished by utilizing the distillation column to separate PC from CPC while the reaction takes place. Initially, Cl_2 is fed into the ~~vapor space of the reactor pot~~ column at the middle addition point. As CPC forms and begins moving up the column, the Cl_2 feed point ~~is~~ may be raised to ~~one of two other locations up~~ the top of the column. The goal is to minimize the DCPC formation.

The reaction gives off (exothermic) a small amount of heat but the heat release is not enough to significantly reduce steam consumption.

One disadvantage of utilizing the column to separate CPC from the reaction ~~mixture may~~ occurs. At the temperatures needed to separate PC from CPC in the still the "half life" of the catalyst is much less than it is if the Cl_2 is fed to the reactor liquor at a temperature below the boiling point of the liquor. Half life is the time required for 50% of the AIBN to decompose. Therefore, catalyst consumption may increase.

As reflected in the material balance, the plant is designed on proven technology. The reaction step lasts about 9-12 hours, which gives about to 70%

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conversion of the PC to CPC and a crude which containing from 5-8% DCPC. By feeding chlorine into the column, it is expected that the conversion can be increased without experiencing high levels of DCPC formation. This probably lengthens the reaction step but at the same time may shorten the distillation steps due to the lower PC recycle required. The conversion, reaction time, Cl_2 addition rate, PC boil-up, etc. have to be optimized as experience is gained through operating the plant.

b. Equipment

The following equipment are used during the reaction step. Please refer to the reactor system in Figure 5-8 on pages 5-47 and 5-48.

P-1 CPC Reactor Feed Pump - This pump is used to add make-up PC from the CPC reactor feed tank to the reactor. It is a sealess magnetic drive pump with all wetted internal parts constructed of TFE Teflon or Tefzel(R). It is rated for 47' TPH at 150 gpm.

T-1 CPC Reactor Feed Tank - This is a 10,000 gallon glass-lined steel tank. It is 10½' Ø x 14' T/T. It is used to store the PC being shipped from the LaPorte, Texas plant in tank trucks. The trucks contain 40,000 pounds or about 5,000 gallons of PC.

R-1 CPC Reactor - This is a 10,000 gallon glass-lined steel tank. It is 10' I.D. x 185" T/T. It is a vertical vessel with 36" Ø and 30" Ø flanges on the top and bottom, respectively for the distillation column and bayonet h ater. It is rated at 90 psig and full vacuum and is protected from

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overpressure by an SRV on the vapor line set at 40 psig. The SRV pressure is lower than design since the SRV also protects other equipment with lower design pressure.

E-3 Reactor Bayonet Heater - This exchanger is constructed of 107 - 1½" Ø x 6' vertical bayonet tantalum tubes. The heat transfer area is 252 ft^2. The tube sheets are clad with .020" tantalum on the process and steam sides. The exchanger is located inside a 30" Ø x 4½' glass-lined steel spool piece attached to the bottom of the reactor. Two 4" Ø downcomers from the bottom of the reactor to the bottom of the exchangers are present to provide increased circulation (see 66C-10003). The exchanger utilizes 150 psig steam to heat the reactor liquor. The tantalum tubes are thin walled (.015") and cannot be subjected to an external pressure greater than 15 psi above the internal pressure. ΔP is measured across the tube walls and N_2 is automatically added to the chest at high ΔP to prevent tube collapse.

C-1 CPC Reactor Column - The column is constructed of six 8' x 36" Ø glass-lined steel spool pieces. Two of the spool pieces are used for each 13' packed section. The top section has a ceramic (Alurdur) distribution tray, packing support plate and redistributor. The mid-section has a packing support plate and redistributor and the bottom section has a packing support plate. Each section is packed with plastic, liquid crystal polymer (LCP), No. 2 super intalox saddles.

The column is separated from the reactor pot by a 36" Ø expansion joint constructed of Hastelloy B2.

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E-1 Primary Condenser - This is a vertical shell and tube condenser. The 110 tubes are thin walled (.015") tantalum, 3/4" Ø x 144" long. The shell is 16" Ø carbon steel. The heat transfer area is 259 ft^2. The tube sheets are clad with .015" tantalum on the process and water sides. Cooling tower water (CTW) is used as the cooling medium.

E-2 Secondary Condenser - This is a vertical shell and tube condenser. It has 31 thin walled (.015") tantalum tubes, 3/4" Ø x 168" long. The tube sheets are clad with .015" tantalum on the process and brine sides. The shell is 8-5/8" Ø carbon steel. The heat transfer area is 85 ft^2. Chilled brine is used as the cooling medium.

SP-1 Reflux Splitter - This is a flow divider to distribute reflux to the column and distillate to the collection tank. ~~It is Pyrex glass and TFE Teflon construction,~~ complete with an explosion proof solenoid and rectifier and a solid state explosion proof timer. The inlet and outlet flamges are 3" I.D. and the overall length is 27½". It has a capacity of 42 gpm at 21" head. ~~For safety a ½" thick plexiglass protector surrounds the reflux splitter.~~

T-7 Catalyst Mix Tank - This is a 50 gallon glass-lined steel tank. It is 2' Ø x 28" T/T. It is vented to the incinerator vent header. The AIBN from the catalyst addition pot and the solvent, PC or a mixture of PC and CPC charged during one of the previous distillation steps are mixed in this tank by circulating the contents with the catalyst mix pump.

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T-13 Catalyst Addition Pot - AIBN is transferred from cold storage to this pot prior to the reaction step. The AIBN is padded into the mix tank, with N_2. The pot is also vented to the incinerator except when AIBN is being added to the mix tank. The pot is an FRP (Derakane) vessel, 12" Ø x 2'6" T/T. It is rated for 75 psig and protected by a RD and SRV set at 75 psig.

P-6 Catalyst Mix Pump - This is a ~~sealless magnetic drive pump~~ centrifugal pump with all wetted internal parts constructed of ~~TFE Teflon or Kynar~~ Hastelloy (R). The recirculation line has an orifice to artificially load the pump and a TFE ball valve to control flow. The pump is equipped with tandem mechanical seals and barrier fluid. The pump is rated for 30 gpm at 52' TDH.

~~P-11 Catalyst Addition Pump - This is a positive displacement Pulsafeeder metering pump with Teflon internals. It is rated for 0-10 gph. It is made by Pulsafeeder and is a ½" x ½" Model L68085.~~

P-2 Reflux Pumps - ~~This is an~~ These are air operated diaphragm pumps. It is a Model ~~#DL 4011 PPP PP~~ M-8 Champ by ~~Dopa~~ Wilden. ~~All internal wetted parts are Teflon.~~ They have teflon diaphragms and kynar bodies. ~~It has a~~ They have a capacity of ~~20~~ 40 gpm. The discharge head and flow are dependent on the available air pressure. The main advantage of a diaphragm pump is that it is not damaged when liquid feed to the pump is interrupted. This occur in the CPC Plant since liquid feed is taken directly from the condensers instead of a reflux drum.

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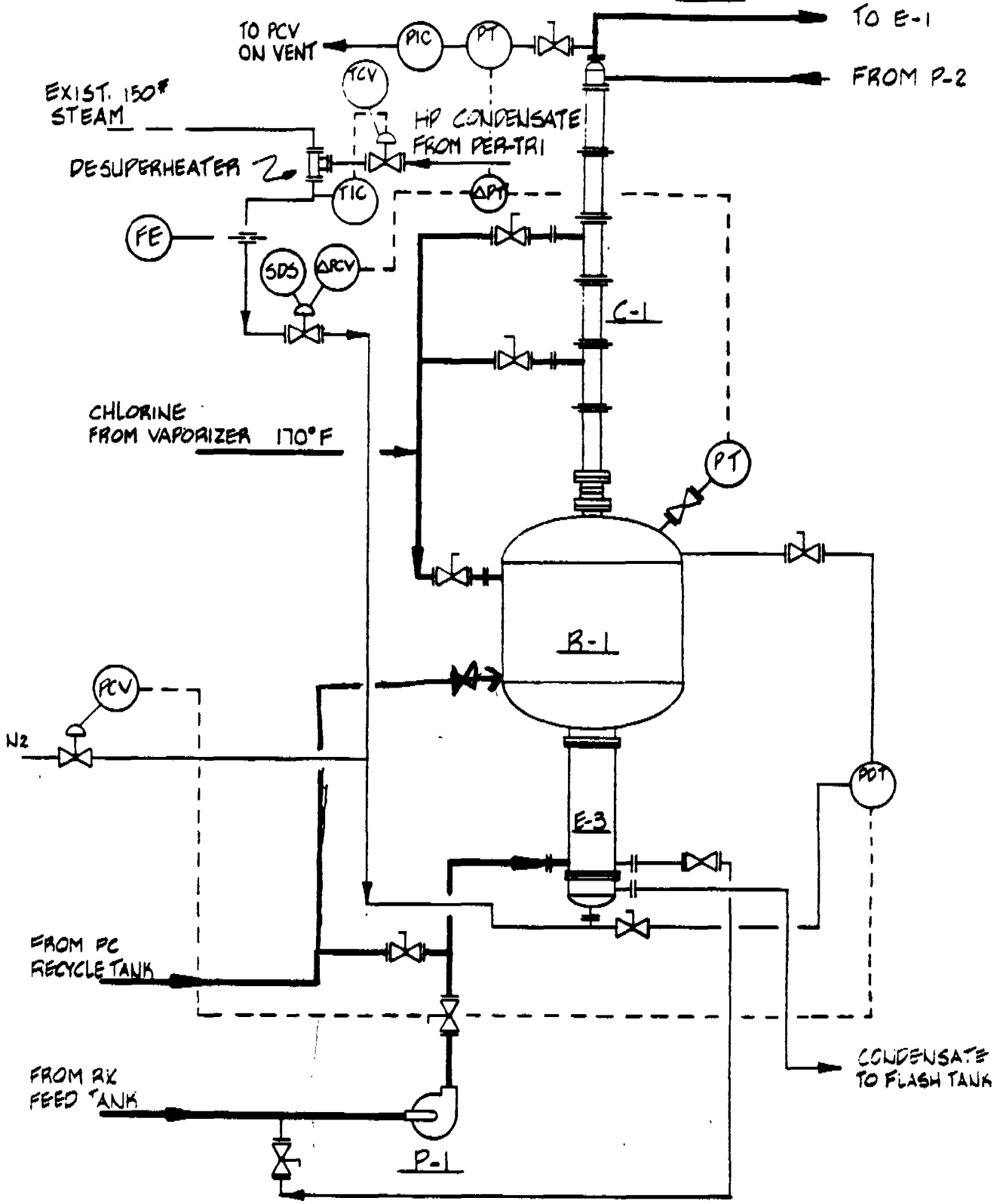
FIGURE 5-8

REACTOR SYSTEM



C-1	B-1	P-1	E-3
CPC REACTOR COLUMN	CPC REACTOR	CPC RX FEED PUMP	REACTOR BAYONETTE HEATER

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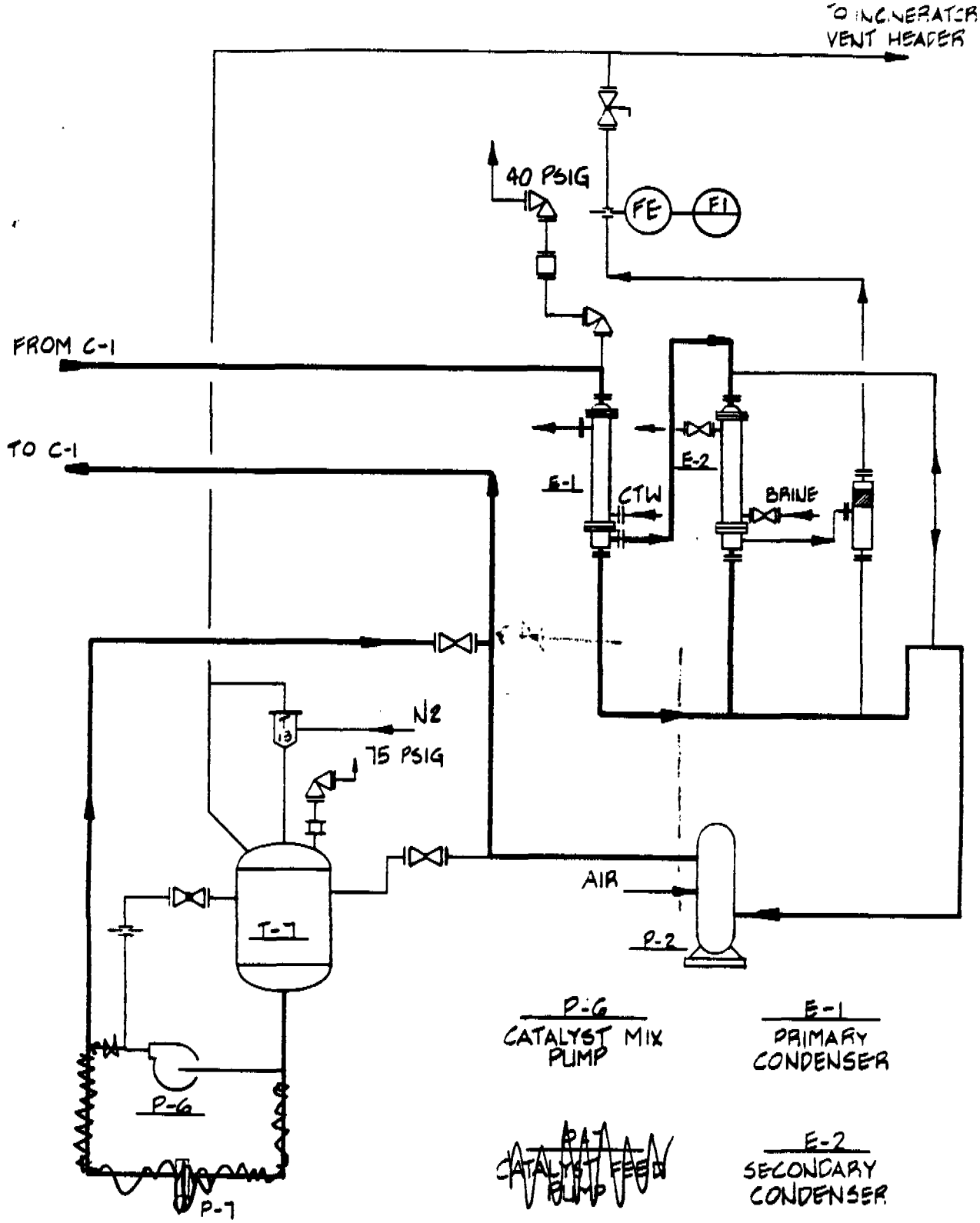
FIGURE 5-8



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c. Start-Up, Operation and Shutdown

After all batch preparations have been made, the reactor is ready for charging. The recycle PC from the PC recycle tank is transferred to the reactor first. Recycle PC is collected during the first distillation step and contains most of the unreacted PC from the previous batch. Recycle PC can be added by gravity flow through the ~~bayonet reboiler~~ sparger in the side of the reactor. Recycle PC can also be added by using the reflux pump to feed it to the reactor through the column. ~~Although gravity flow will take longer it may decrease the overall charging and heat up time as this method will pass cool PC through the reboiler. The feed pump and tank must be isolated from the feed pipe to the reboiler while the contents of the recycle tank are being transferred to the reactor. The downcomer loops from the reactor to the reboiler should also be blocked off to force PC feed through the reboiler.~~

Once the liquid transfer from the recycle tank to the reactor begins, the 150 psig steam feed can be put into service. With the PCV closed the 3" block valves at the inlet to the reboiler and downstream of the PCV should be opened. Steam flow can then be manually controlled to the reboiler while heat-up is proceeding. Steam should be introduced slowly at first to warm up the header.

Liquid PC must be present on the process side of the reboiler before introducing steam or thermal shock of the glass lining in the spool piece surrounding the reboiler may occur. If the proper steps were taken in batch preparation, the condensate system should be lined up through the steam traps to the condensate flash tank.

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~~If the PC recycle tank has not yet been analyzed, a sample should be taken while transferring its contents to the reactor~~. Based on the initial level reading on the PC recycle tank ~~and an analysis of the PC recycle~~, the required amount of make-up PC from the CPC reactor feed tank can be determined. Once this is done, the final level (following transfer to the reactor) ~~of make-up PC~~ in the CPC reactor feed tank can be calculated. When the PC recycle tank is empty, close off the block valve on the drain line to the reactor.

Valving on the feed line and around the reactor is arranged so that the PC being fed passes through the reboiler and up into the reactor to enhance heat-up of the PC.

The reactor feed pump should be started up on recirculation to the PC feed tank. After the recirculation rate has stabilized, the block valve to the reboiler can be opened. The block valve on the recirculation line to the feed tank can then be closed.

The reactor feed pump can transfer PC at a rate of 150 gpm, so the loading time is short. The operator remains in the area, keeping an eye on the level in the Reactor feed tank, and shuts off the feed pump when the reactor is charged. If the reactor is overfilled, the feed pump can be used to pump excess PC back to the PC feed tank or, preferabley, the PC recycle tank.

Once all the PC has been loaded into the reactor, the block valve on the feed line at the reboiler is closed. Increased recirculation from the reactor through the reboiler can then be achieved by opening the block valve on the downcomer(s). Steam flow is increased.

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As heat up continues PC begins to boil and vapors will pass up the column and enter the water cooled (shell side) primary condenser. The column is operated under maximum boil-up during chlorination to minimize DCPC formation. At atmospheric pressure (0 psig), PC condenses at 221°F, so all vapors will condense in the primary, water cooled condenser at this time. When reaction begins, the by-product HCl gas lowers the condensation temperature of the mixture and, hence, transfers some of the condensing load to the secondary, chilled brine condenser.

The condensed PC passes down through a seal loop to the reflux splitter which is set for total reflux at this time and throughout the reaction step. When condensation begins, the reflux pump is started up. The double diaphragm, air operated, reflux pump can be run dry indefinitely, so it is not critical that liquid be in the pump suction during start-up.

With the PC being refluxed to the column, the AIBN addition is started. AIBN is metered into the reflux. ~~The metering pump is set for the desired addition rate (0 - 10 gph).~~ Initially, the AIBN addition rate is set slightly higher than anticipated. As Cl_2 addition progresses, the catalyst addition may be cut back very slowly, watching for signs of loss of Cl_2 utilization, such as yellow-green liquid/vapor appearing in the reflux splitter.

When the column lines out under total reflux at the desired boil-up with catalyst being added to the column, Cl_2 addition is begun. Initially, Cl_2 is fed to the ~~vapor space of the reactor pot~~ column at the middle addition point. The two other feed points (to

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the column) are blocked off. The Cl_2 feed is gradually increased to the desired CPC production rate.

As discussed on pages 5-41 and 5-42, by-product formation has a tremendous effect on chlorine utilization, batch size and recycle flows. In Table 5-1 below, this effect is illustrated by comparing Cl_2 feed to reactor pot and Cl_2 feed to the distillation column for one batch. The commercial plant equipment size and material balance are based on feeding Cl_2 to the pot liquor since Cl_2 feed to the column was not proven technology at that time. Results when feeding Cl_2 to the distillation column are theoretical and are intended only as guidelines to operating the plant.

TABLE 5-1 EFFECT OF Cl_2 FEED LOCATION ON YIELDS AND CAPACITY

	Cl_2 To Reactor Pot	Cl_2 To Distillation Column
Production Rate, Tons CPC	12.18	12.18
Reaction Time, Hours	6	4.6
Cl_2 Feed, Pounds	16,457	12,566
Molar CPC/DCPC Ratio	6.5	32
DCPC Formed, Lbs	5,025	937
PC Boil-Up, Lbs/Hr	0	19,280
PC Recycle, Lbs	30,154	13,878
Final Wt. % PC in Rx Crude	47	35

The key variable in minimizing DCPC formation is the concentration of CPC at and above the Cl_2 feed point. In Figure 5-9 below this concept is illustrated. The data in this Figure are predictions from pilot plant operation for a specific set of reaction conditions; the most important of

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these being: (1) a CPC production rate of 24,353 pounds, and (2) a 6 hour reaction time. Two cases can be used to clarify the graph.

In the first case a CPC/DCPC molar ratio of 6.5 results at a boil-up rate of 64 moles/hour and a Cl_2 feed rate of 38 moles/hour. At these conditions, a PC concentration of 53 mole percent is at the Cl_2 injection point.

In the second case, a CPC/DCPC ratio of 32 results at a boil-up of 160 moles/hour and a Cl_2 feed rate of 29.5 moles/hour. At these conditions a PC concentration of 83% is at the feed point.

As boil-up increases at a given CPC production rate, the concentration of PC at the feed point increases (% CPC decreases) thus reducing DCPC formation, which is the product of the reaction of Cl_2 with CPC. At the same time the Cl_2 requirements are reduced as less Cl_2 is being consumed in by-products.

The maximum boil-up on the column is limited by one or more of the following variables: (1) steam flow to reboiler, (2) heat transfer area of reboiler or condensers, (3) pressure drop across the column and (4) reflux pump and system capacities.

At a specified boil-up, the Cl_2 feed rate is limited by (1) capacity of the Cl_2 vaporizer; (2) concentration of AIBN at the Cl_2 feed point; (3) by-product DCPC formation, (4) unreacted Cl_2 appearing in the plant vent and (5) excessive pressure drop in the column. Chlorine feed can be adjusted to maximize production of CPC while minimizing production of DCPC.

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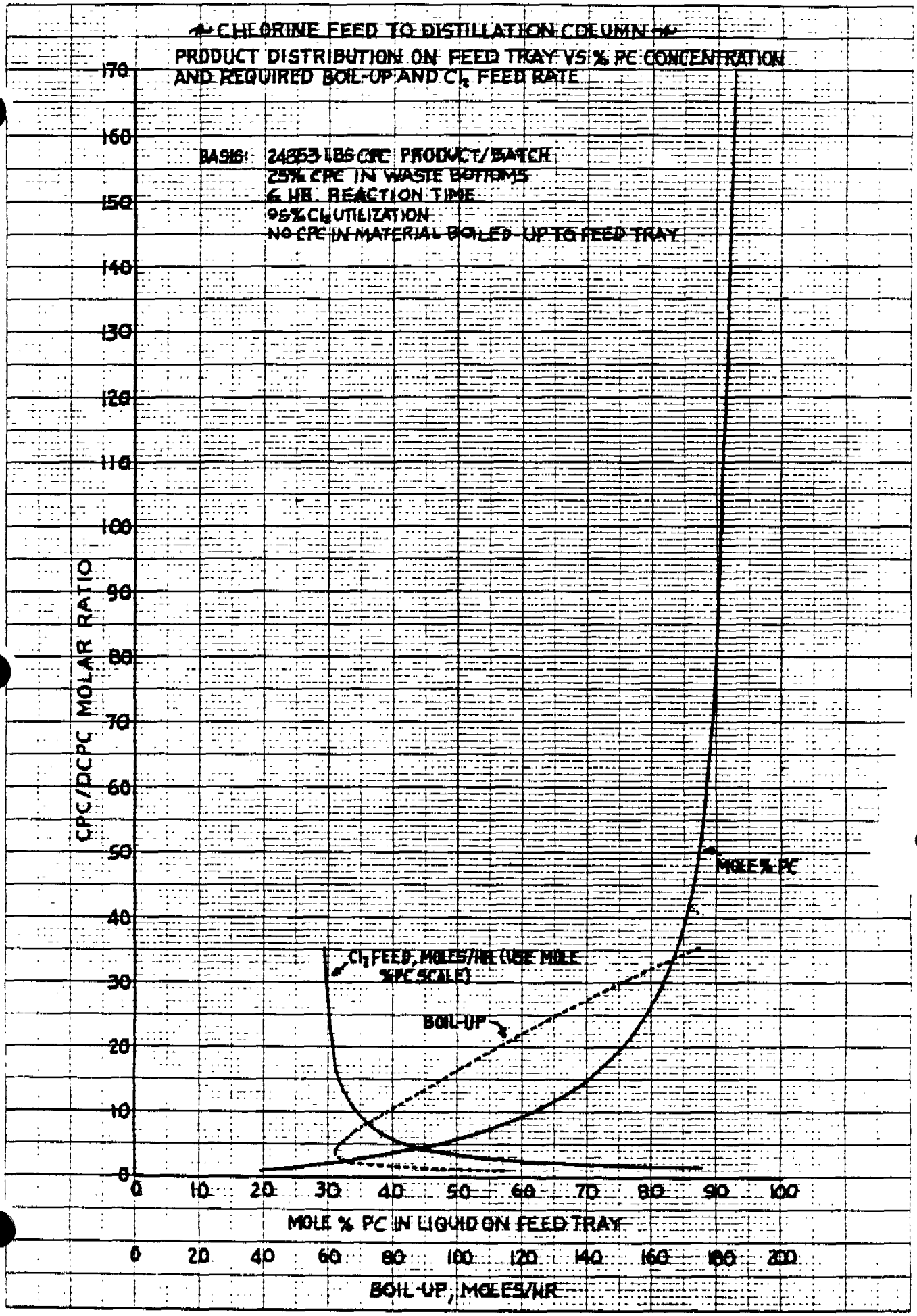
FIGURE 5-9



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As the reaction proceeds, the concentration of CPC and DCPC in the pot liquor increases. Gradually, the CPC becomes more concentrated in the lower section of packing. If the Cl_2 feed location ~~were to remain at the lower Cl_2 injection point, the rate of DCPC formation would begin to increase~~ may be raised to reduce the DCPC formation as the reaction proceeds.

~~A pot analysis or the temperature profile on the column can be used to determine at what point in time the Cl_2 feed location is to be moved to the mid-point on the column. This can be done without interrupting reaction by first opening the mid-point block valve and then closing the lower block valve.~~ If the Cl_2 feed point is raised, less time is available for the Cl_2 to react with PC. The Cl_2 feed rate may have to be lowered or catalyst addition increased if evidence of Cl_2 breakthrough, such as yellow-green liquid in the reflux splitter, appears.

~~This same procedure can be used to raise the Cl_2 feed point to the upper section of the column~~. Reaction continues until DCPC formation becomes economically prohibitive. Guidelines on how far to carry PC conversion are given in the SOP Manual. An analysis of reactor crude is ~~probably~~ required to make this judgement, although column temperatures may be used to make a fairly reliable estimate. The reactor pot sample is collected from the sample apparatus on the reboiler recirculation line. (Specific sampling procedures are given in the SOP Manual.)

Steam flow to the reboiler is controlled by the pressure drop across the packed sections of the column. The column is designed for 0.5" H_2O/ft. packing pressure drop or about 55% of flooding vapor velocities at the maximum design boil-up. The column has 39 feet of packing.

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Ten column/reactor temperatures are automatically recorded on the data logger in the control room. These include readings from six, equally spaced, thermocouples in the packed sections and one thermocouple in each of the following locations: reactor liquor, reactor vapor, column overhead and column reflux. The reactor pot (vapor) and column differential pressure are also indicated on the control board.

The reactor liquid level above the bayonet heater tubes is transmitted to an indicator on the control board or measured from a sight glass in the field. A bulls eye on each of the reboiler recirculation lines can be used to check for the presence of organics in the spool piece around the reboiler.

As the reaction progresses, the changing composition of the liquid in the reactor pot leads to an increase in pot temperature. This in effect lowers the available ΔT to transfer heat from the steam. PC boils at 234°F at 3 psig. A 50:50 mixture of PC:CPC boils at 267°F. With 150 psig steam (365°F) the T is reduced from 131°F to 98°F or 25%; but, the heat of vaporization of PC vs a 50:50 PC:CPC decreases from 155 to 136 BTU/lb. or 12%. Thus, at the same steam feed rate, the boil-up will decrease gradually as % PC conversion increases. To keep the same boil-up, the steam flow has to be gradually increased. This is important to understand when trying to maximize the CPC/DCPC formation ratio.

The reaction step is terminated by closing Cl_2 FCV. The Cl_2 vaporizer is left "floating" on line (see Section V.C.2.) unless that CPC Plant is to be down for an extended period.

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d. Abnormal Operation

The appearance of the yellow-green color in the reflux splitter is an indication of poor chlorine utilization. This can be caused by a number of variables:

1. Loss of or insufficient catalyst
2. Poor distribution in the packed section
3. Insufficient boil-up
4. Excessive chlorine feed

In any of the cases, the problem should be first corrected by lowering the chlorine feed rate. After this is done the cause should be investigated and corrected before raising the chlorine feed rate. Free chlorine in the vent does not cause an immediate danger in this process; however, it is avoided as it does result in a yield loss and, if excessively high, can lead to unsteady incinerator secondary scrubber operation.

Insufficient boil-up or loss of reflux causes poor distribution in the packed sections and can lead to poor chlorine yield. If boil-up is the problem, it can be increased to the limit of heat transfer of the reboiler by increasing steam flow. High steam chest pressure indicates the maximum steam flow has been reached.

If the reflux flow is cut off by pump shutdown or failure, a more serious problem can occur as condensate will quickly begin backing up into the condensers and vent piping. If this occurs, steam to the reboiler should be shut off immediately until the cause of the problem is corrected and the

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reflux flow is restarted. Loss of reflux can also be caused by a malfunctioning reflux splitter.

Reactor pressure build-up is indicative of a major process problem. The cause should be identified and remedied as quickly as possible. The reactor vent will be directed to the CPC Plant emergency scrubber (See Section V. F.) if the system pressure reaches 20 psig. An alarm will warn the Operators of increasing pressure so the pump on the scrubber can be started to begin circulating alkali solution through the scrubber. If the vent is relieved to the scrubber and alkali solution is not circulating, HCl, Cl_2 and PC is released to the atmosphere. This leads to a hazardous environment and violates the air permit.

In the case of an emergency incinerator shutdown, the CPC Plant is automatically shut down by the shutdown system (SDS). See Section V. H. for a description of the SDS. If, after shutdown, the vent header pressure rises to 20 psig, the vent is pressure controlled to the emergency scrubber.

The CPC reactor, column and overhead piping are glass-lined steel, which is susceptible to damage by thermal shock. Extra precautions should be taken when transferring hot liquor to a cold vessel or introducing steam into the reboiler, which is surrounded by a glass-lined spool. Cool hot liquor in the reactor (see Section V. E.) before transferring the liquor to the incinerator feed tank. Check to see that the spool surrounding the reboiler is full of liquid before steam is introduced.

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D. DISTILLATION

1. Overview

The purpose of the distillation section is to separate CPC from the unreacted PC and heavier chlorinated by-products such as DCPC. This separation is accomplished in three steps, the latter two taking place under vacuum.

Especially in a smaller production unit such as the CPC Plant, batch distillation is the most economical method for separating a pure component(s) from a multicomponent mixture. In continuous operation, a separate still is required for each pure liquid (less one). With batch distillation only receivers (tanks) need to be switched. In small production units, receivers are less expensive than additional distillation systems.

Batch operation also provides more flexibility than continuous operation. Varying reactor liquor compositions can be easily handled. Reflux rate and throughput can be changed without having to adjust several instruments. No balance of feed and draw-off is necessary.

Operation of batch and continuous distillation columns are quite different. The following general guidelines apply to batch distillation.

(a) For optimum separation efficiency, reflux hold-up is minimized. A reflux splitter has been used in place of a reflux drum. All condensate is put either directly back to the still or to a "product" receiver.

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(b) The column consists solely of a rectification section. When the low boiling component drops to a specific concentration in the pot, an acceptably high concentration of the low boiler in the overhead can no longer be achieved.

(c) Capacity to produce at a given rate and product specification is only minimally affected by changing the reflux ratio or length of a cut.

(d) Separation becomes progressively more difficult as the more volatile component is being removed from the pot.

(e) The composition of the pot continually changes throughout the distillation.

(f) The column operate best under loaded (maximum boil-up) conditions.

A batch still has two main modes of operation. The still can be operated at constant reflux ratio, which results in a varying overhead composition, or at constant overhead composition, which requires a varying reflux ratio. In the CPC process the constant reflux ratio is the primary mode of operation. However, the reflux ratio does vary from one distillation step to another. The first step is operated at the lowest reflux ratio and the last (third) step is operated at the highest reflux ratio. Towards the end of each step, the reflux ratio probably has to be increased to maximize the low boiling component in the overhead.

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The column design is based on data generat d from the pilot plant and tests on liquid-vapor equilibrium from the laboratory. All physical properties for PC, CPC, DCPC and related compounds are not well defined. Operating instructions in the SOP Manual and general guidelines discussed in this manual are for the best predicted mode of operation. Available equipment was used in the pilot plant and, thus, it was not specifically designed for the CPC process. There should be room for process optimization once the commercial plant is on stream.

2. Equipment

Except for the catalyst addition system, all major equipment used during the reaction phase is also used during distillation. In addition, the following major equipment are a part of the distillation process.

T-6 CPC Product Shift Tank - This is a horizontal, 5000 gallon, glass-lined steel tank. It is 100-5/8" I.D. X 128" T/T. It is used to store the CPC Product from the third distillation step. The vessel is equipped with a 70 psig RD and SRV. The tank can be vented to the incinerator or secondary scrubber, but normally is vented to the incinerator except during the third distillation step (vacuum).

T-10 PC Recycle Tank - This tank is identical to the CPC Product Shift Tank. It is used to collect and store the unreacted PC recovered from the first distillation step. The tank is vented to the incinerator or secondary scrubber depending on the required mode of operation.

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T-12 PC Still Recycle Tank - This is a 2000 gallon glass-lined steel tank, 7' O.D. X 6' S.S. It is used to collect and store the mid-cut overhead product. It has a 70 psi RD and SRV and can be vented to either the incinerator or secondary condenser.

Vacuum System - This is a package unit which will be described in full in Section V.D.4.

A schematic of the distillation system is presented in Figure 5-10 on pages 5-64, 5-65, and 5-66.

3. PC Distillation

The purpose of the PC distillation step is to recover and recycle the unreacted PC from the reactor crude. The CPC product must be essentially free of PC contamination. The PC distillation and CPC pre-cut steps are used to accomplish this.

The transition from the reaction to the PC distillation step proceeds smoothly as the column is under total reflux at the end of the reaction step. In only a few minutes, the CPC formed in the still during reaction is worked down the column. The PC distillation takes place at a low reflux ratio (theoretically less than 1). The reflux ratio is defined as the moles of liquid recycled (refluxed) to the column divided by the moles of condensed liquid which are removed from the system. A mole is defined as the total weight of a component or mixture divided by the average molecular weight of the mixture. In most cases for this system either a weight ratio or gpm ratio can be used to approximate the reflux ratio.

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For ease of switching distillate from one tank to another, a manifold station with shut-off valves on lines to each of the three distillate tanks is located near the reflux splitter. During PC distillation, the valves to the still recycle tank and the CPC product shift tank closed. Using the three-way valve, the vent from the PC recycle tank is lined up to the secondary condenser. This allows pressure equalization of the vapors in the reflux system so there will be no back pressure on the PC recycle tank. The vent from the still recycle tank and the CPC shift tank are routed to the vent header downstream of the vacuum pump. Refer to Figures 5-3 and 5-4 on pages 5-20 and 5-21.

The reflux ratio is controlled by the reflux splitter. After analyzing the reflux for acceptably low levels of CPC, the block valve to the PC recycle tank is opened. Then, the reflux splitter is set to provide the desired reflux ratio. The solenoid valve on the reflux splitter is set to operate at a predetermined "time on" and "time off". When the valve is closed, the condensed liquid will drain to the suction of the reflux pump, which returns the liquid to the column as reflux. When the valve is open the condensed liquid drains part of the time to one of the three distillate tanks. During PC distillation the distillate is drained to the PC recycle tank.

At the beginning of the PC distillation step the distillate is nearly 100% PC. As the PC is removed from the still, the pot becomes leaner in PC and the pot temperature rises. The column temperatures increases as PC distillation continues, first at the bottom and then progressively towards the top. The eight temperature readings on the column, including one on the pot and one on the overhead are used to watch the temperature profile. There will be

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FIGURE 5-10

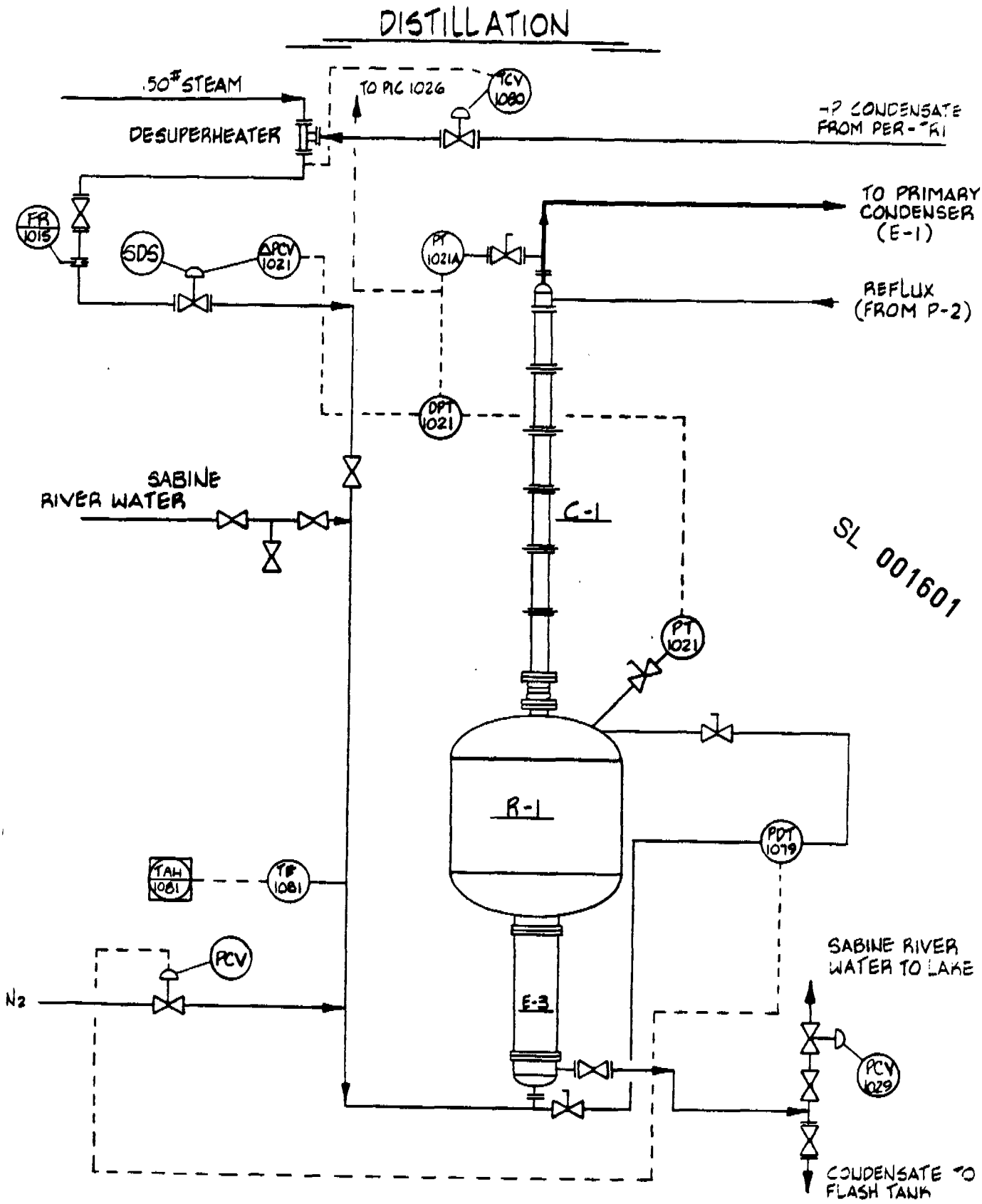

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FIGURE 5-10

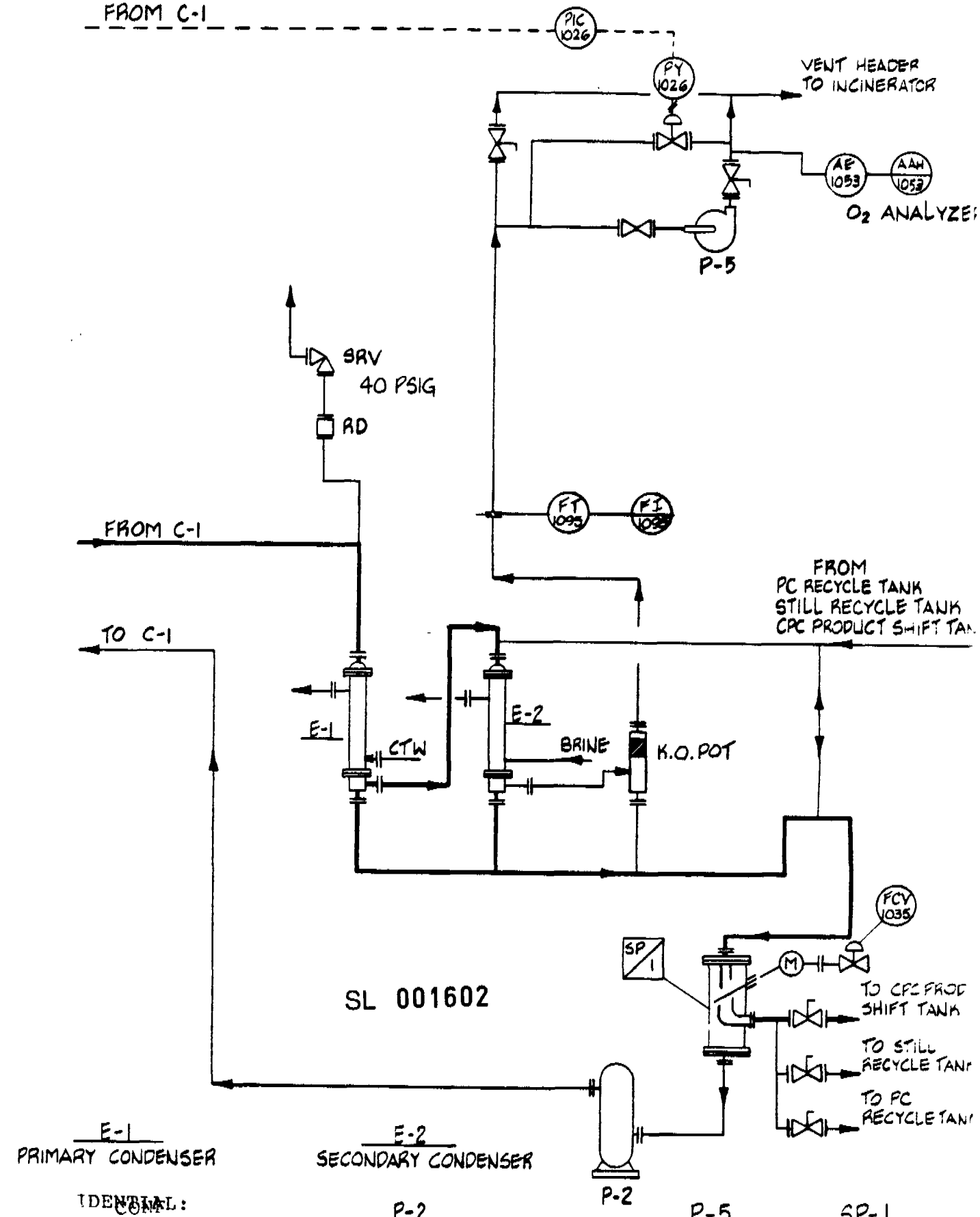

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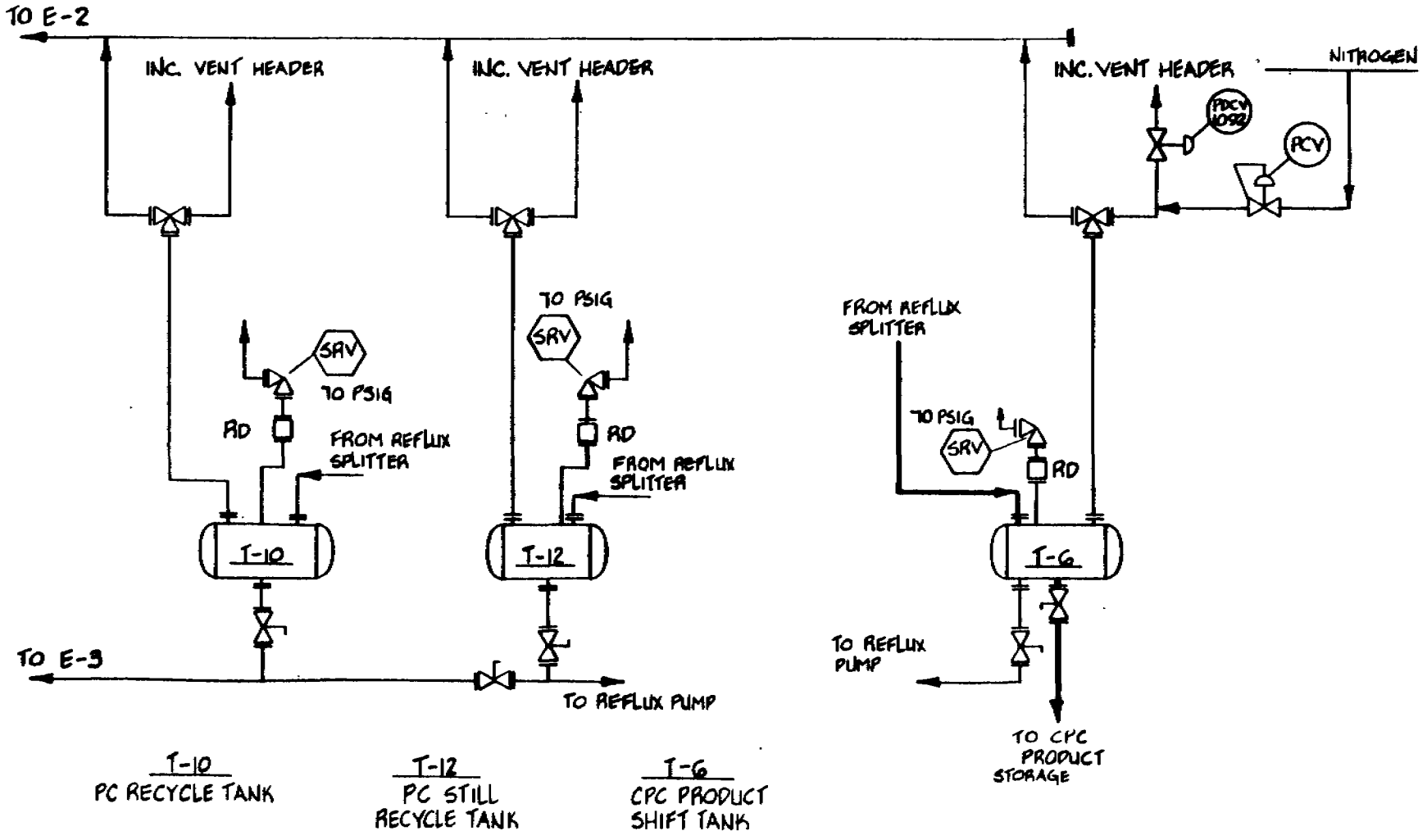
FIGURE 5-10

DISTILLATION



T-10
PC RECYCLE TANK

T-12
PC STILL RECYCLE TANK

T-6
CPC PRODUCT SHIFT TANK

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a fairly well defined break in temperatures as the CPC begins moving up the column. When the overhead temperature increases, begin analyzing the reflux for CPC content.

Until experience is gained, reflux analysis is the primary tool used to determine when to switch distillate tanks. It probably is not possible to switch directly from the PC distillation to the CPC product cut because it takes a while for the PC to be depleted from the CPC in the overhead. Liquid hold-up in the column is the cause of this delay. Hence, the PC left in the column, when CPC begins coming overhead in fairly high concentrations, must be removed before beginning the CPC distillation step.

The residual PC is removed in the "CPC pre-cut" step (Section V.D.5.). The CPC pre-cut is stored in the still recycle tank. The CPC pre-cut (from th previous batch) is returned to the still pot is during the PC distillation step. The optimum time occurs when the pot composition (in CPC and PC) equals the composition of the liquor in the still recycle tank. It is best to transfer the CPC pre-cut back to the still pot slowly (by gravity) through the reboiler recirculation line. During this time the column may have to b operated under total reflux, as unsteady operation may occur. It's possible, however, if the mid-cut is returned slowly enough, distillation can proceed normally. Once the CPC pre-cut has all been returned, PC distillation continues until the CPC in the overhead increases to an undesirable level.

Near the end of the PC distillation or the beginning of the CPC pre-cut, the reflux ratio may have to be gradually increased to keep the CPC content in the overhead low, but theoretically remains less than one.

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~~During PC distillation, the catalyst mix tank is slowly filled with reflux. The timing is arbitrary. If catalyst usage is high, transfer the reflux to the mix tank when the CPC level is high as the catalyst is more soluble in CPC than it is in PG.~~

The percent CPC in the overhead when the distillate is switched from the PC recycle tank to the still recycle tank is arbitrary since product will not be made following this switch. The main consideration is to minimize CPC in the PC recycle tank. The contents of the PC recycle tank are returned to the reactor prior to reaction, so the CPC present may lead to DCPC in the product.

4. Vacuum System

The purpose of the vacuum system is to lower the operating pressure of the CPC distillation column and pot. This is required to keep the boiling point of mixture being distilled less than about 320°F. Above 320°F the upper temperature limit of the various materials of construction is being approached and the driving force for heat transfer provided by the 150 psig steam at 365°F is reduced significantly. A diagram of the vacuum system is presented in Figure 5-14. The following major equipment are included:

Vacuum Pump - This is a single stage liquid ring pump manufactured by SIHI. It is constructed of Hastelloy B2 and delivers 163-168 CFM(1) at 4 to 20" Hg vacuum and 105 CFM at 26" Hg vacuum. The service liquid is perchloroethylene (Perchlor). A 15 HP, 1800 rpm cast iron motor is used to drive the pump.

(1) Dry Air @ 68°

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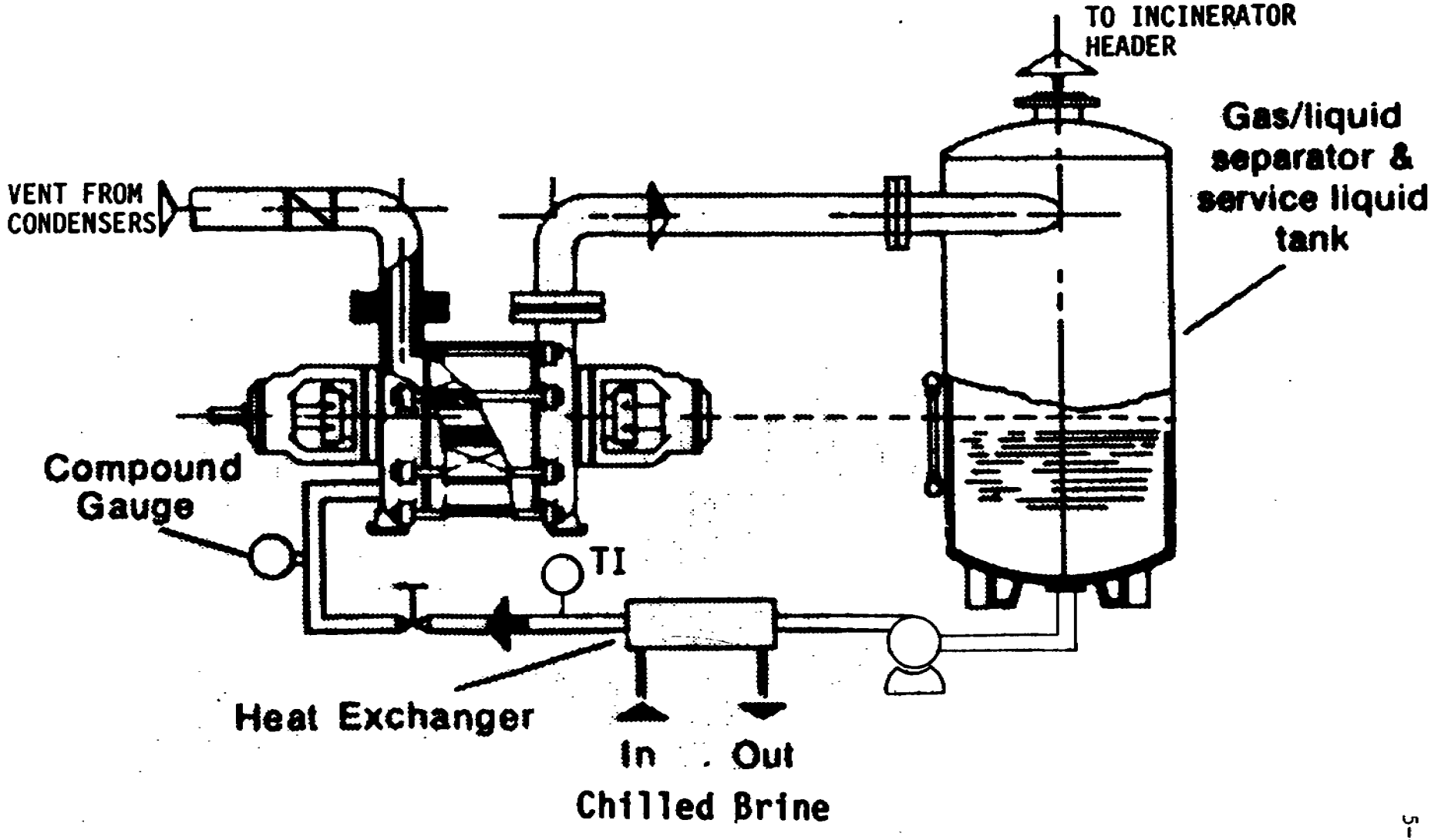
FIGURE 5-14. VACUUM SYSTEM



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Service Liquid Tank - This is a carbon steel vessel with a PTFE liner. The tank is 16" I.D. by 2'6" overall length. It has the dual purpose of providing storage for the perchlor and acting as an entrainment separator to remove the liquid perchlor in the gases discharged from the vacuum pump.

Circulation Pump - This is a magnetic drive centrifugal pump. It has a capacity of 20 gpm at 2' head. It is constructed of PTFE and FEP teflon. This pump is used to circulate the perchlor from the service liquid tank through the cooler to the vacuum pump and back to the tank.

Perchlor Cooler - This is a U-tube cooler utilizing chilled brine as the cooling medium. It is 4½ O.D. by 4' long and has a surface area of 6.5 ft^2. The tubes and shell are made of Hastelloy 2B. It is designed to cool the perchlor from 123°F to 90°F at a flow of 7.5 gpm. Perchlor is on the shell side.

The principle of the operation of the SIHI vacuum pump is illustrated in Figure 5-15. An excerpt from SIHI Bulletin 110, Section 1000 (issued 7/85 provides a description of the operation.

> In a round pump body (A) we position a rotating element, namely a shaft mounted impeller (B) at a point eccentric to the center line of the pump body. The amount of eccentricity is related to the depth of the liquid ring (C). The liquid ring is formed by introducing service liquid, perchlor, via the pump suction casing (L) and through the channel (D) positioned in the suction port plate (E). The centrifugal action of the rotating impeller forces the liquid towards the periphery of the pump

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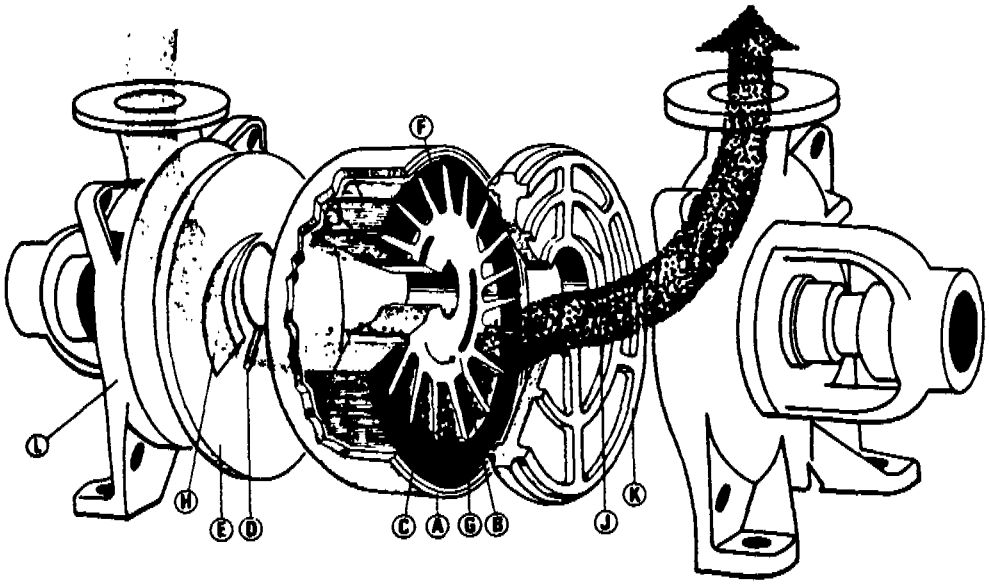
FIGURE 5-15. VACUUM PUMP OPERATION

SIHI
A SIHI International Company

the principle of SIHI single acting liquid ring pumps



= gas
= service liquid
= gas plus service liquid

● *The illustration is intended to depict the operating principle of the SIHI liquid ring pump only and should not be considered for engineering details of construction.*

body. By controlling the amount of service liquid within the pump body to where the impeller blades are completely immersed to their root at one extreme (F) and all but their tips exposed at the other extreme (G), optimum pumping performance will be attained.

When this pumping action is achieved, the vapor to be handled is induced through the suction port (H) when the depth of impeller blade immersion is being decreased. Then as the immersion increases, the vapor is compressed and discharged through the discharge port (J) in the intermediate port plate (K). As there is no metal to metal contact between the impeller and the pump body and intermediate plates the need for lubrication is eliminated and wear is reduced to a minimum.

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During the compression cycle heat is being imparted to the liquid ring. In order to maintain a temperature below the vapor point, cooling must be applied. This cooling is achieved by continuously adding a cool supply of perchlor to the liquid ring. The amount of coolant added is synonymous with that discharged through the discharge port (J) together with the compressed vapor. The mixture of vapor and liquid is then passed to the service liquid tank for separation.

Detailed start-up and operating steps are provided in the SOP Manual. Three main points in operating the unit are important:

1. Prior to starting the pump, the perchlor level in the storage tank should be at the center line of the motor shaft to the pump. A sight glass is provided.

2. During operation, the pressure of the perchlor at the inlet of the vacuum pump is to be kept at or just slightly below atmospheric pressure (14.7 psia, 0 psig).

3. To minimize perchlor loss and to maximize pump capacity, the perchlor is to be kept as cold as possible at the inlet of the vacuum pump.

5. CPC Pre-Cut

The purpose of the CPC pre-cut is to remove the residual PC from the distillation column and pot. At the end of the ~~PC distillation~~ Reaction step the system is operating slightly above incinerator header pressure (2-6 psig)

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and the pot contains mostly CPC. The effect of pressure on the boiling point of CPC is as follows:

PRESSURE	BOILING POINT, °F
300 mm Hg (=18" Hg Vacuum)	254
0 psig (760 mm Hg)	302
4 psig	318

The temperature of saturated 150 psig steam is 365°F.

The CPC distillation system is put under vacuum during the PC distillation step. Initially the system pressure is lowered to 300 mm Hg (18" Hg vacuum). This pressure will be reduced further as the percent DCPC in the still pot increases to keep the temperature at an acceptably low level. To allow for pressure drop in the system, the pressure of the process vent at the vacuum pump inlet has to be lower than the pressure on the column.

During the CPC pre-cut, the vent from the still recycle tank is routed to the secondary condenser. The vents from the PC recycle and the CPC shift tank are routed to the vent header downstream of the vacuum pump. This equalizes pressure on the condensing system and the still recycle tank. The block valve on the condensate to the still recycle tank is opened and the block valve on the PC recycle tank is closed.

Prior to starting the vacuum system, the oxygen analyzer is placed into operation (see detailed instructions in the SOP Manual). The oxygen analyzer

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is located downstream of the vacuum pump and upstream of the recycle loop for pressure control. If oxygen enters the system (most likely during vacuum operation), an explosive mixture may develop. An alarm warns the Operator of the presence of a high level of oxygen in the system. The O_2 analyzer is not tied into the shutdown system. So, if oxygen enters the system, the vacuum pump must be shut down manually. The leak can then be located and repaired. Locating a vacuum leak can be difficult. The entire plant may have to be shut down and purged with nitrogen, in extreme cases, before procedures can be implemented to locate the leak.

As the vacuum pump is put into service the block valve on the by-pass around the vacuum pump is closed. As the system pressure is slowly reduced, gas expands causing the vapor load on the column to increase significantly. The column can be flooded easily if certain precautions are not taken. Prior to reducing the pressure, column is placed on total reflux. The steam ΔP control valve lowers steam flow as pressure is reduced.

The pressure must be reduced very slowly in accordance with standard operating procedures. If the pressure is dropped too rapidly, a high ΔP will be experienced across the column. Even with no steam flow, the liquid in the pot may continue to boil as the pressure is reduced.

The system is put under vacuum for two main reasons:

1. The process temperature must be kept below about 320°F to extend the life of the materials of construction.

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2. The process temperature must be low enough to enable use of 150 psig steam for heating.

To save time and to minimize the potential of operational upsets, the pressure is reduced to the lowest anticipated level in one step; i.e., the system is not be operated at different pressures during the CPC pre-cut. Once the lower pressure level has been reached, steam flow to the reboiler can be increased and, after the column is stabilized under total reflux, the steam ΔPCV is placed back on automatic operation. The proper reflux ratio is then re-established.

The CPC pre-cut is continued until essentially all of the PC is removed from the system. The reflux ratio may have to be increased during this step. However, it may be necessary, towards the end of this step, to put no reflux back to the column for a short period of time to rid the system of PC; i.e., totally draw off the condensed overhead to the PC still recycle tank. Towards the end of the CPC pre-cut, while the column is under higher reflux, the distillate contains greater than 90% CPC. Samples of reflux are taken periodically to check PC and CPC concentrations. The CPC pre-cut is terminated when the PC level is low enough to keep the CPC product in spec on PC concentration.

6. CPC Distillation

The purpose of the CPC distillation step is to recover the product CPC from the remaining liquor in the batch still. The still pot now contains a very small amount of light impurities, CPC and heavier chlorinated by-products.

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When the distillate becomes low enough in PC to meet the CPC product specification, the distillate tanks are switched. The CPC product shift tank is slowly placed under vacuum. At the manifold station near the reflux splitter, the block valve to the CPC product tank, which is now part of the vacuum system, is opened and the block valve to the still recycle tank is closed.

Since this is a batch operation, the CPC product drawn off initially is higher than specification in PC. But, as distillation proceeds, the PC nearly or completely disappears. After switching the distillate tanks, the column is again put on reflux ratio control. Proper reflux ratio is optimized as operating experience is gained. Design calls for a higher reflux ratio than in either one of the other steps. Near the end of the batch, the reflux ratio may have to be increased significantly to keep the DCPC at a minimum in the overhead CPC product.

CPC distillation as designed takes longer than either one of the other distillation steps. The system is kept under vacuum and the column temperatures are observed, especially in the pot, to make certain sufficient ΔT between the liquor and the steam is high enough to achieve proper boil-up. The system is designed for 300 mm Hg pressure at this point, but the vacuum pump can be used to decrease the pressure to 100 mm Hg if necessary to recover maximum CPC. Be careful when lowering pressure further to watch for excessive ΔP across the column to keep it from flooding. Boil-up will increase at the same steam feed rate. Samples of distillate are collected as necessary to evaluate still operation.

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As CPC distillation continues the pot and column temperatures will increase since concentration of DCPC is increasing. Towards the end of the CPC distillation, the CPC Product can be kept in specification on DCPC if the reflux ratio is increased. As the reflux ratio is increased, the distillate will decrease so at some point it becomes ineffective to continue this operation. At this point the reflux ratio is lowered and opened back to the still recycle tank to remove some of the CPC left in the column and increase yields. This is done for 15-20 minutes. The still is then shut down by shutting off steam to the reboiler at the PCV and blocking condensate at the trap.

The vacuum pump is shut down and blocked off. The system under vacuum is then gradually repressurized to vent header pressure by adding nitrogen to the vent header downstream of the secondary condenser entrainment separator. When header pressure is reached, the by-pass around the vacuum pump is opened.

In preparation for the next batch, the vents from the still recycle and CPC product shift tanks are lined up to the vent header downstream of the entrainment separator. The distillate tanks are all blocked off from the reflux splitter at the manifold.

The CPC product shift tank is sampled and analyzed ~~put on recirculation using the CPC product transfer pump, and a sample is taken and analyzed~~. If cleared, the CPC product is transferred to the CPC product tank (see Section V.G.). If out of spec the CPC may have to be redistilled.

E. BOTTOMS PROCESSING

Before starting the next batch, the bottoms left from the CPC distillation step must remov d from the reactor and bayonet heater spool

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piece. These waste bottoms are transferred to the DCPC incinerator feed tank. From this tank the waste is pumped through a transfer line, dedicated to the CPC Plant, to the No. 1 or No. 3 incinerator. There is insufficient head between the reactor and the incinerator feed tank to allow gravity drainage. The incinerator feed pump, located in the CPC Plant, is used to make the transfer.

Figure 5-11 on page 5-81 presents a diagram of the waste bottoms processing system.

In addition to the reactor and bayonet heater, other equipment in the bottoms processing step are:

T-4 DCPC Incinerator Feed Tank - This is a 2093 gallon glass lined steel tank. It is 85-3/4" I.D. x 96" crown to crown. It is rated for 25 psig and full vacuum at 400°F and is equipped with a rupture disc and SRV set at 25 psig. It is continuously vented to the incinerator header downstream of the vacuum pump.

P-3 Incinerator Feed Pump - This pump is used to transfer waste from the CPC Plant to the incinerator and to transfer the bottoms remaining in the reactor at the end of the CPC distillation step to the DCPC incinerator feed tank. It is a ~~sealless magnetic drive~~ centrifugal pump with ~~all wetted parts made of Tefzel or TFE Teflon~~ double mechanical seals and Hastelloy wetted parts. It is rated for 40 gpm at 132' TPH.

The bottoms left in the reactor at this point in time are over 300°F and must be cooled before being pumped out of the reactor. This is done for two major

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reasons. ~~The upper temperature limit on the pump is 200°F and, under certain conditions,~~ liquid above 300°F can cause thermal shock and possible damage to the glass lined incinerator feed tank, although with the steam tracing on the Incinerator Feed Tank this is unlikely.

Sabine river water is circulated through the bayonet heater to cool th waste bottoms prior to transferring it to the incinerator feed tank. Refer to Figure 5-11, a diagram of the waste bottoms processing, on page 5-81. A common line is used for steam and cooling water. It is essential that all valves are properly positioned when using either of these utilities. When using cooling water the steam and steam trap systems must be blocked off.

The bayonet heater is made of "thin" wall (.020") tubes and, as such, demands special precautions. Even though the tubes can take an internal pressure of 200 psig, they cannot take an external pressure greater than 15 psi above the internal pressure. If the pressure differential exceeds 15 psi the tubes may collapse. It's conceivable that, if the steam were blocked off, the pressure in the steam header could drop below atmospheric pressure due to condensation of the steam. With the reactor under pressure (up to 20 psig before relieving to the scrubber) and some liquid head, the ΔP can rise above 15 psi. To prevent this from occurring, nitrogen is automatically be added to the steam/SRW header if the ΔP, measured between the reactor vapor space and steam chest, becomes excessive. This subject has been discussed here since it is at this time in each batch that the steam is cut off.

During SRW addition, pressure is kept on the condensate/SRW header with a back pressure control valve on the SRW return line to the Sabine water surge pond. When the bottom temperature has dropped below 200°F, preferrably about

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150°F, the bottoms can be transferred to the waste tank using the incinerator feed pump. After the bottoms have been cooled and transferred, SRW must be drained from the condensate header prior to steam addition to prevent SRW from entering the condensate system.

When the incinerator feed pump is being used to transfer bottoms to the incinerator, the incinerator feed tank liquor is also being recirculated through a three-way FCV on the pump discharge. At this time, the bottoms transfer from the reactor is made by opening the block valve(s) on the transfer line to the incinerator feed pump. This is followed by closing the block valve on the waste liquor from the waste tank. If the waste tank is not isolated from the suction of the pump, the level in the reboiler/reactor will equalize with the level in the waste tank, making it impossible to empty the reactor.

Since the pump and motor can be damaged if the pump is allowed to run dry, the pump is automatically cut off by a flow switch, which senses (by frequency) no flow in the transfer line. If CPC bottoms are being fed to the incinerator while liquor is being transferred from the reactor to the incinerator feed tank, the feed to the incinerator will be abruptly shut off when the pump automatically shuts down. Therefore, the incinerator feed from the CPC Plant is shut off prior to transferring waste from the reactor to the waste tank. The Incinerator Operator must be advised of this temporary shutdown of liquid feed from the CPC Plant.

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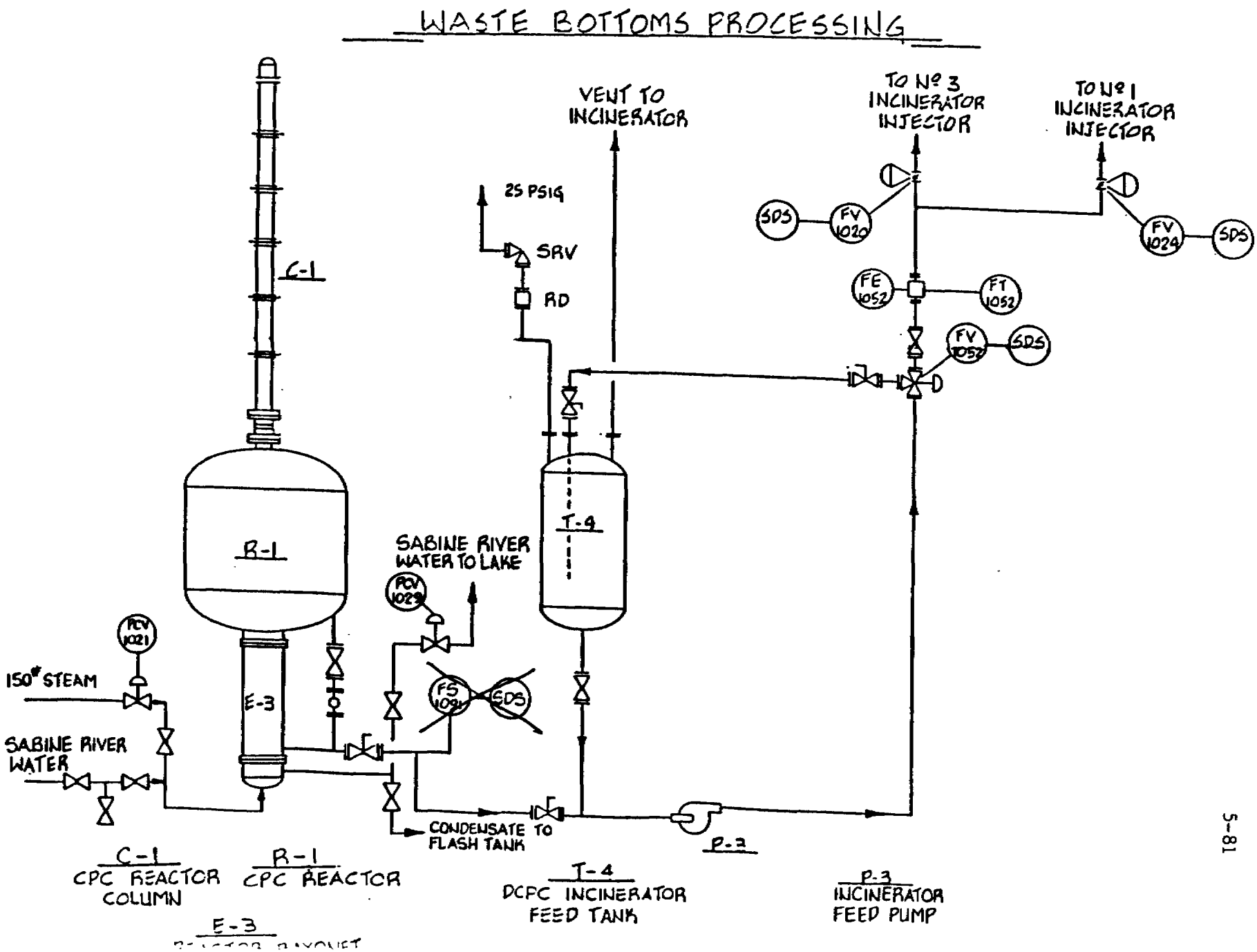
FIGURE 5-11



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If the CPC waste is not being transferred to the incinerator when the reactor needs to be emptied, the incinerator feed pump is started up with the incinerator feed tank on total recirculation while the transfer of waste from the reactor is made.

After the pump automatically shuts down, the valves on the transfer line from the reactor are blocked off and the valve on the line from the waste tank to the pump is reopened. After bypassing the sensor switch, the pump is restarted with the tank on total recycle. Once recycle flow has lined out, feed to the incinerator can be re-established.

Liquid waste is transferred to either No. 1 or No. 3 incinerator. If the incinerator being fed liquid from the CPC Plant shuts down, no action needs to be taken at the CPC Plant as the incinerator feed tank will simply operate on total recirculation. If both incinerators are to be down for an extended period, the incinerator feed pump can be shut down. CPC Plant operation can be continued until the incinerator feed inventory reaches maximum level.

F. EMERGENCY SCRUBBER SYSTEM

The purpose of the emergency scrubber system is to effectively and efficiently destroy all hazardous chemicals in the CPC Plant vent whenever the incinerator, which is burning the vent, shuts down. All hazardous chemicals in the vent can be eliminated through reaction with sodium hydroxide. FMC Corporation has obtained approval of the revised premanufacturing notice (PMN), which allows for destruction of the hazardous chloropivalic acid (CPA) by sodium hydroxide treatment. CPA is formed when CPC contacts water and is converted to the sodium salt of hydroxypivalic acid (a non-hazardous chemical) in the presence of sodium hydroxide. For this reason

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a weak cell liquor solution, which contains a minimum of 4% sodium hydroxide, is circulated through a packed absorption column (scrubber) whenever necessary to destroy CPC and, subsequently, CPA.

The weak caustic solution also destroys the HCl and Cl_2 present in the vent. The caustic recirculation rate is kept high to keep the scrubber temperature from becoming too high in a localized area. Absorption of HCl and Cl_2 are highly exothermic reactions (give off heat) and can cause boiling in the bottom of the scrubber if the recirculation flow is too low. The temperature out of the scrubber is controlled at less than 170°F. Above 170°F the sodium hypochlorite, which has been formed by reacting sodium hydroxide with Cl_2, begins to decompose and can lead to a Cl_2 release to the atmosphere from the scrubber vent. This abnormal operation must be avoided. The caustic recirculation rate to the scrubber must be sufficient to keep the scrubber effluent temperature less than 170°F under the worst anticipated release case.

The scrubber system is vitally important to the CPC Plant. If the system does not properly function, it can lead to a violation of the air and/or water permits as well as create an unsafe condition in the CPC Plant and outlying areas. The emergency scrubber system must be, at all times, in a state of readiness so that hazardous chemical releases do not occur.

The major equipment included in the emergency scrubber system are:

C-2 Emergency Scrubber - This vessel is 18" x 2' I.D. and is constructed of FRP-Derakane 411. The column is packed with 12' of #2 CPVC Tellerettes. The

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support plate is made of titanium. The recirculation liquor is introduced into the column through a 3" Hastelloy B2 sparger containing fifteen 3/4" holes. The bed limiter is made of fiberglass grating.

T-11 Emergency Scrubber Recirculation Tank - This is a flat bottom tank, 6'0" I.D. x 7'6" to the tangent line, and is constructed of FRP-Derakane 411. It is used to store the 1200 gallons (about 3/4 full) of weak alkali solution needed for treating the CPC vent if diverted to the scrubber.

P-8, 9 Emergency Scrubber Recirculation Pumps - These are epoxy resin (Durcon 6) pumps made by Durco. They are 2 x 2E-10 with a 9-1/8" impeller and are rated for 150 gpm at 84' TPH. This pump is used to circulate the weak alkali solution from the recirculation tank to the scrubber.

Figure 5-12 on page 5-88 presents a diagram of the emergency scrubber system.

Under normal CPC Plant operation the plant vent (from the reactor and process vessels) goes to either the No. 1 or No. 3 incinerator. The incinerator vent header is exclusively used by the CPC Plant. A PCV on the header in the CPC Plant area controls plant operating pressure. A FCV on the header at the incinerator is tied into the incinerator shutdown system.

In the event that an incinerator trips off, the FCV to the incinerator automatically closes and the batch process shuts down. Chlorine feed, catalyst and steam to the reactor/column are shut off automatically. The PCV to the emergency scrubber is set to control at 20 psig. This setting is probably high enough to prevent relief to the emergency scrubber even if the

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trip occurs when the reaction step is taking place. In the chlorination of PC one mole of HCl (36.5 pounds) is produced per mole of chlorine (71 pounds) consumed. The reaction is only slightly exothermic (minimal heat release). It is anticipated that the only pressure build-up occurring after an incinerator trip is due to residual chlorine remaining in the vapor phase or dissolved in the liquid in the reaction system. Hence, no vent releases are expected when an incinerator trip is experienced. The reactor and column contents begin cooling immediately when chlorine and steam flow to the reactor are stopped.

If the incinerator trip occurs during the distillation no process vent release to the emergency scrubber is anticipated since no net production of vapors occurs. As the system cools the header pressure drops.

The emergency scrubber is designed to handle the vent flow during two types of severe releases: (1) a sudden system depressurization from 20 psig or, (2) a vent flow from the reaction step at 1.2 times the design rate. The severe depressurization would occur only if system pressure built up to near 20 psig (operating pressure is 2-6 psig) and the PCV failed open (instrument air failure, for example). The depressurization lasts about 3 minutes. The release for 1.2 times the design vent flow is based on a five minute period, which provides a safety factor of 2½ times the predicted flow duration. A high pressure alarm sounds if the CPC vent pressure (upstream of the PCV) reaches a predetermined upper limit. The operator then initiates caustic flow to the scrubber by starting the recirculation pump from the control room. If no release to the scrubber occurs and the pressure of the CPC Plant is brought under control, the recirculation pumps can be shut down.

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CPA is difficult to destroy. At a pH of 14 (approximately 4% NaOH) and a temperature of 149°F, the half life of CPA (the time necessary to destroy ½ of the initial amount) is 3 minutes. The scrubber effluent collected from the release is held in the recirculation tank until the CPA is less than 10 gm/l, the maximum level set by the EPA. This tank is provided for another reason also. If the release to the scrubber is of short duration, only a small portion of the caustic reacts. In other words, if the weak alkali solution is used on a once through basis most of the caustic is wasted.

The recirculation tank must be, at all times the CPC Plant is operating, charged with cell liquor and water to provide a minimum of 1200 gallons of at least 6% NaOH. If the NaOH strength is too high "salting out" (precipitation of dissolved solids) may occur when the vent is scrubbed. If the volume is too low, the scrubber temperature will climb above the control point. The SOP Manual provides a table for amounts of cell liquor and water to be added. The water must always be added first.

Once the recirculation tank is charged, the contents are circulated through the scrubber for proper mixing and then sampled to verify adequate caustic strength. On a periodic basis the operation of the system is to be checked by recirculating the tank liquor through the scrubber.

The cell liquor make-up is flow recorded. Initial water addition is measured using the sight glass on the recirculation tank. When circulating the alkali solution the following information is available in the control room:

(1) pH measured by an on-stream analyzer.

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(2) Recirculation flow rate.

(3) Temperatures of liquor in the recirculation tank and out of the scrubber.

This information is essential for reliable operation of the system. If the recirculation tank has been properly charged with cell liquor and water and the liquor is circulating at the correct flow rate, the NaOH strength will remains above the minimum 4% after the worst anticipated release. The temperature out of the scrubber remains below 170°F (the tank temperature will be less). When the release has subsided, the tank can be sampled after some period of time to verify CPC and CPA have been destroyed. This sample is analyzed by the main lab. The CPC Plant cannot be restarted until the CPC and CPA are destroyed and the recirculation tank liquor is adjusted to the specified tank level and NaOH strength. See the SOP Manual for details of this operation.

The destruction of CPC and CPA is greatly affected by temperature. A minimum temperature of about 140-150°F is needed to hasten the destruction process. If the temperature in the recirculation tank is too low, steam can be added very slowly through a sparger in the bottom of the tank until the proper temperature is reached.

Once the CPA and CPC are destroyed, some of the liquor is pumped out of the recirculation tank to the 118 sewer and the minimum NaOH strength is re-established in the tank by adding water (first) and cell liquor.

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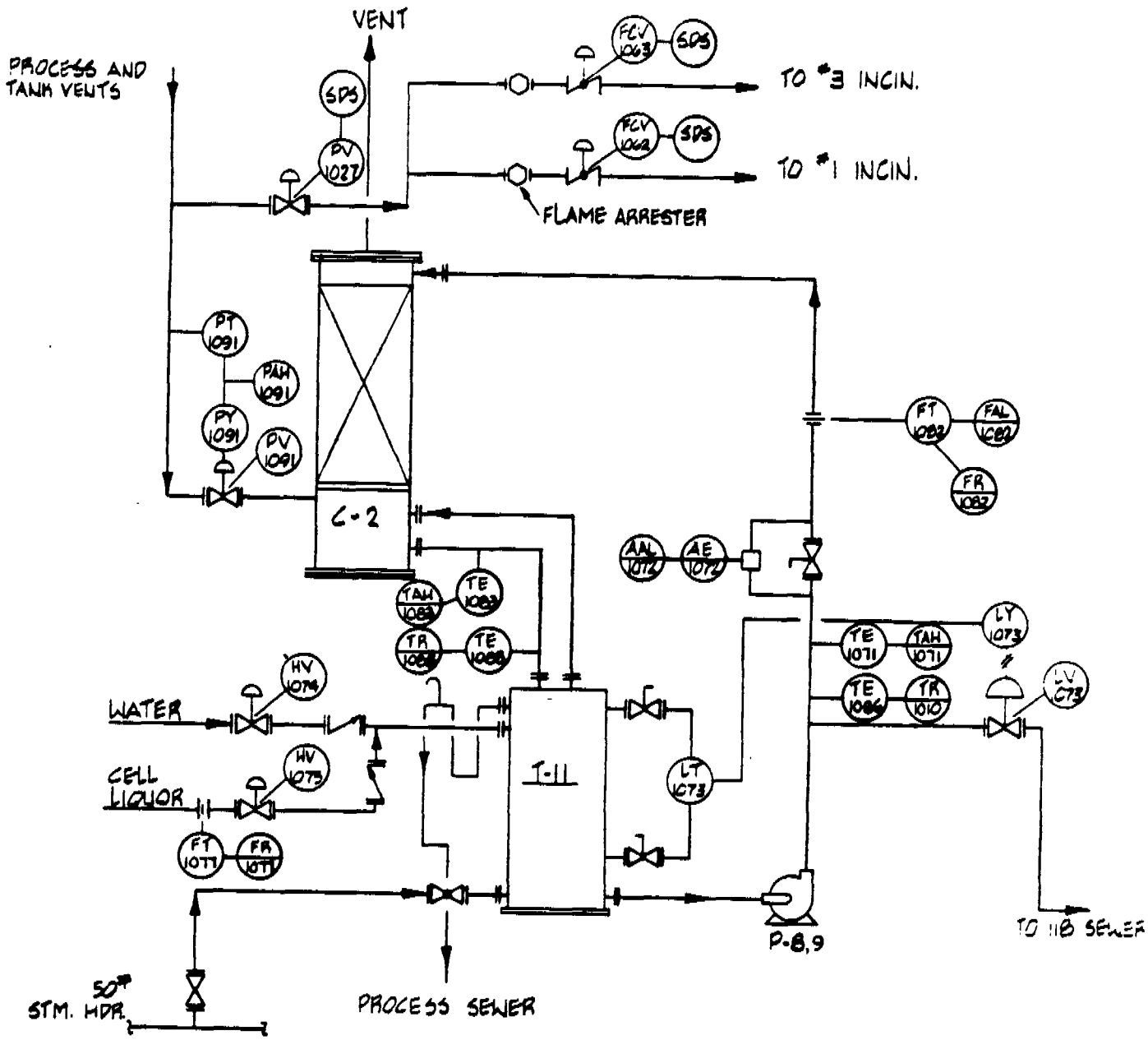
FIGURE 5-12

EMERGENCY SCRUBBER



EMERGENCY SCRUBBER C-2

EMERGENCY SCRUBBER REC.RC. TANK T-11

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If the vent release is greater than anticipated, the temperature of the recirculation liquor may rise above the maximum set for the process or the pH may drop below the acceptable minimum. If either of these occur, water and cell liquor are added (the ratio is given in the SOP Manual) to the recirculation tank. Tank level is controlled by an automatic level control valve. If alkali solution is removed from the tank by the LCV, a sample must be caught and analyzed by the main lab to check for acceptable levels of CPC or CPA.

Whenever the vent is released to the scrubber, all efforts must be directed to (1) minimizing or stopping the vent release, and (2) properly operating the scrubber system to destroy the pollutants and hazardous chemicals.

G. CPC PRODUCT HANDLING AND SHIPPING

In this operation the CPC product will be qualified by analysis and transferred to the CPC product storage tank. The CPC operators will assist the Shipping Department in loading the CPC in trucks to the customer. Refer to Figure 5-13 on page 5-92 for a diagram of the CPC loading system.

The following equipment is used in this operation:

T-2 CPC Product Storage Tank - This is a glass-lined tank, 9'8-3/4" I.D. x 16'4-1/8" T/T. It is rated for 30 psig and full vacuum at 400°F. It is equipped with a rupture disc and SRV set to relieve at 30 psig. This tank is used to store the CPC product transferred from the CPC shift tank after product qualification. The shift tank specifications are given in the "Distillation" section of this manual. The product storag tank is twice as

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large as the shift tank and can hold up to 100,000 pounds (10,000 gallons) of CPC.

P-4 CPC Product Transfer Pump - This pump is used to load CPC product into trucks and to transfer rework material from the shift and/or product tanks back into the CPC process. It is a sealess magnetic drive pump with all internal wetted parts constructed of Tefxel(R) or TFE Teflon. It is rated for 150 gpm at 67' TPH.

At the end of each batch after repressurization of the system with nitrogen, the CPC in the CPC product shift tank is sampled and analyzed. CPC product purity is controlled by independent N_2 pad systems on each of these two tanks. If the sample passes the qualification test, the CPC is transferred from the shift tank to the product tank. At this point in time the vents from the still recycle and CPC product shift tanks are routed to the vent header downstream of the vacuum pump. The PC recycle tank is vented to the secondary condenser. All block valves from the reflux splitter in the tanks are closed.

When the product is drained (by gravity) from the shift tank, a constant positive pressure is kept on the shift tank by introducing nitrogen into the tank through a pressure regulator. As the product tank is being filled a ΔPCV on the vent from the tank keeps the pressure slightly above the vent header pressure.

At peak CPC production rates, shipments to the customer are made at 2-3 day intervals. Shipping is responsible for loading the trucks but the CPC

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Operator will assist as necessary. The CPC product tank liquor is recirculated so CPC can be sampled and analyzed by the main lab for qualification prior to loading the truck.

The truck is vented to the CPC product tank during CPC loading. This keeps the truck and tank equalized so N_2 padding, although available, is probably not be necessary. The CPC truck must be cleared and purged with N_2 prior to CPC loading. A breathing air station is located at the loading station for use during loading if necessary.

During CPC transfer and loading, extra care must be taken to prevent contamination of the CPC product with water. Water will react with CPC forming HCl and chloropivalic acid (CPA), a mutagen and suspected carcinogen. If the CPC product becomes wet, it will have to be reanalyzed and possibly reworked through the still, resulting in a significant loss of production as there is no storage for off spec material.

If the product does not pass the quality analysis, it will have to be reworked, again at a significant production loss. Since only CPC product will be handled by the CPC transfer line (to Shipping) and vent line (from Shipping), the lines will not normally have to be cleared after each transfer to control product purity. However, the lines must be purged with N_2 to remove CPC so no personnel exposure is encountered when removing the flexible hoses from the truck. Nitrogen hook-ups are available on the loading and vent lines.

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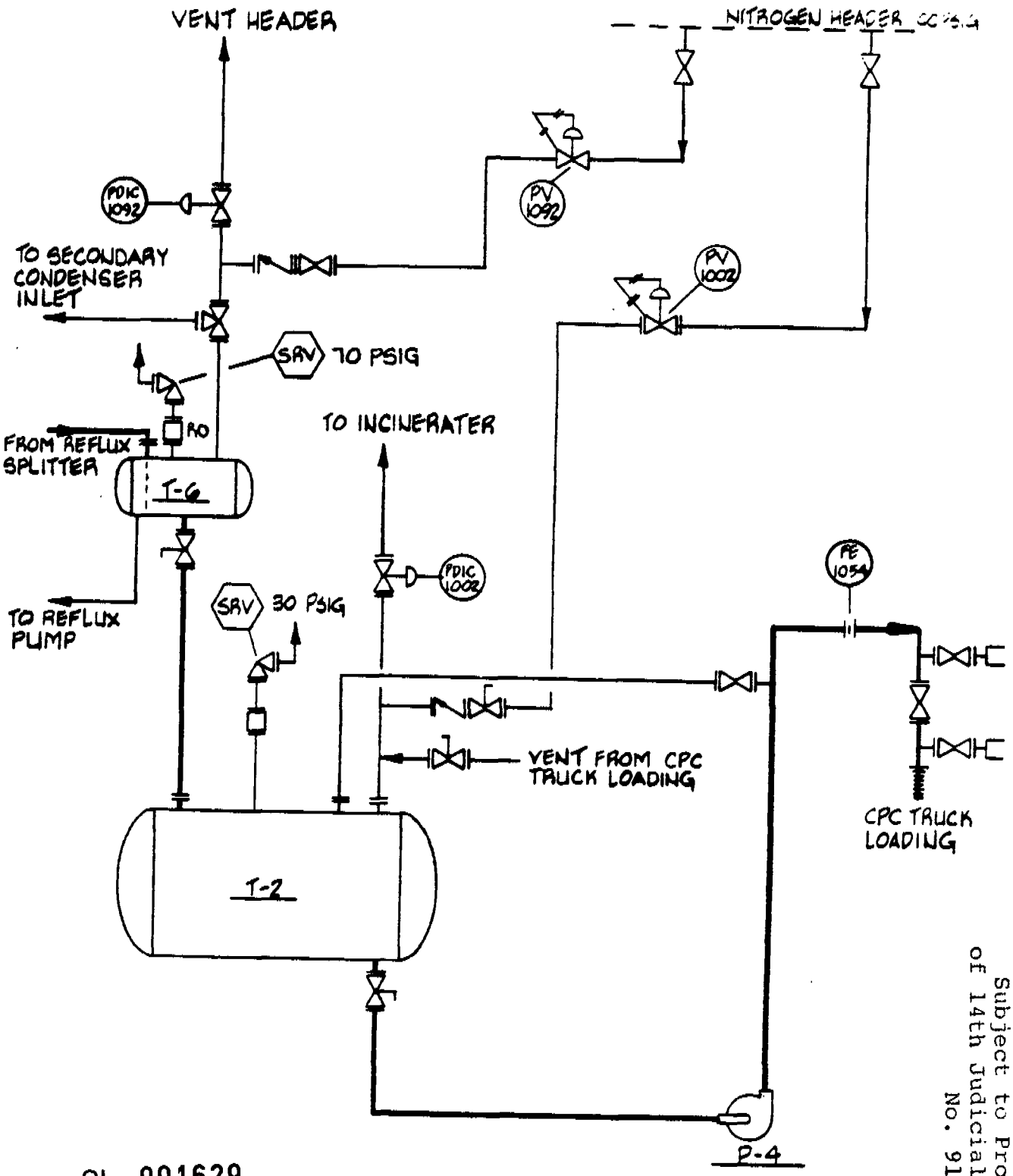
FIGURE 5-13

CPC LOADING AND SHIPPING



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T-6
CPC PRODUCT SHIFT TANK

T-2
CPC PRODUCT STORAGE TANK

P-4
PRODUCT TRANSFER PUMP

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H. CPC PLANT VENT MANAGEMENT SYSTEM

The CPC Plant vent management system is designed to prevent the release of CPC or PC into the atmosphere. It performs this function by shutting off sources of heat and reaction when the incinerator that is burning the plant vents shuts down.

When the incinerator shuts down, the vents from the CPC Plant are automatically isolated. In order to prevent the system from pressurizing to 20 psig and thus venting to the emergency scrubber, the plant shutdown system activates. It has been calculated, that after the SDS activates, the system pressure should not climb to 20 psig.

The SDS consists of a set of solenoids and a programmable logic controller. When the incinerator burning CPC Plant vents shuts down, the chlorine vaporizer SDS is activated (unless it is in the "float" mode) and the steam PCV to the reactor/still heater and the incinerator vent header PCV are closed. The transfer of liquid wastes is not affected if the liquid were being burned in a different incinerator than the vents.

Once the incinerator is restarted or the vents switched to another incinerator, the SDS can be reset, and normal operations resumed. In order for the CPC Plant to maintain stable, continuous operation, it is imperative that nuisance trips of the incinerators be minimized. Periodic prooftesting of this system is required to ensure proper system function.

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VI. EMERGENCY PROCEDURES

The potential exists in any chemical plant such as the CPC/WTU Plant for emergency situations or process upsets to develop with little or no warning. It is essential that everyone know the proper corrective action to take in these situations so that injury to personnel, damage to equipment, product contamination, exposure of personnel to hazardous chemicals, and/or pollution of the air or waterways are avoided or minimized. The following procedur s are presented as guidelines and concepts. These guidelines represent the minimum response to a given situation. Operators must think and act beyond the general guidelines to overcome the problems presented by each emergency. Detailed checklist procedures for response to emergency situations are included in the SOP Manual.

A. RESPONSE TO HAZARDOUS CONDITIONS

In the event of a major catastrophe, self-protection and protection of your co-workers must be your primary consideration. Ignition sources must be eliminated. The evacuation alarm, the red phone, and roadway strobe lights are be used as necessary to warn others of the situation. Activation of the deluge system can be considered. Proper respiratory equipment must be used. The plant is secured to minimize the hazard potential. The control building is designed to be explosion resistant and is equipped with an emergency breathing air system. Evacuate the control building only as a last resort.

Response to an emergency in the plant is limited to the operator for that area and the lead operator and/or the auxiliary operator, if necessary. If

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everyone left the control room to respond to the situation, or just out of curiosity, and was overcome by fumes, for example, there would be no one left to respond to alarms and secure the plant from the control room.

1. Major Break, Gas Leak, or Explosion

a. Activate the CPC Plant B Evacuation Alarm the roadway strobe lights.

b. Shut off the control room air intake.

c. Set off the sprinkler system if they have not activated automatically.

d. Notify other units via the red phone system.

e. Notify the guards. Request fire and/or first aid assistance as required.

f. Put on an emergency escape mask or Scott Air Pack.

g. Secure, stabilize, and isolate the affected areas of the plant if possible.

h. Trip the CPC shutdown system if necessary.

i. Block steam to the plant to reduce flammable vapor generation.

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j. Leave the area only as a last resort.

Make an evaluation with supervision about corrective action to be taken. Isolation valves to consider are: chlorine and liquid waste to incinerator. Other lines to be considered are air, nitrogen, and water so bleeding down these utilities does not affect other units. Shutting down electrical power to the plant can reduce the amount of flammables being pumped out through damaged or ruptured lines. Ensure that power is not needed for other reasons, such as pumping flammables out of the area, before shutting it down.

The Lead Operator is responsible to make contact with the Emergency Squad, direct them to the affected area and apprise them of the general situation. The Lead Operator is also responsible for making a "head count".

2. Minor Spill or Leak

a. Activate the CPC Plant evacuation alarm and roadway strobe lights.

b. Make sure that maintenance and construction have shut down all welding machines, air compressors, vehicles, and all other possible ignition sources.

c. Activate the sprinkler system if necessary. Remember PC and CPC generate HCl on contact with water.

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d. Notify other operating units of the situation via the red phone.

e. Notify the guards of the situation. Keep them informed in case further blocking of roadways is required.

f. Put on a Scott Air Pack.

g. Bypass and isolate the leak source, if possible.

h. If bypassing is not possible, take the leaking equipment out of service and isolate it. If taking the equipment out of service requires shutting down the plant, follow procedure in the SOP Manual for shutting down. Try to keep as much equipment as possible operating so everyone is not tied up in a major shutdown effort.

i. If possible, route spills to pad drains and away from surface drainage to prevent or minimize water pollution.

NOTE: A vapor release in the area may cause the switch-gear room to trip off if sufficient vapors enter the room. Respond to this situation as described under "Loss of Switchgear Room."

3. Fire

In the event of a fire, the size of the fire has a considerable effect on the action to be taken. Small fires, especially trash, oil, motor, electrical, or pump seal fires must be extinguished immediately. Use

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of fire extinguishers is preferred for PC or CPC fires as use of water liberates HCl. In general, cool down larger fires with water and allow them to burn out by consuming all available fuel. If the fire is put out prior to isolating the fuel source, the flammable vapors can spread and re-ignite, causing further damage and possibly injury to personnel. In any fire situation, notify the fire chief. The key to minimizing fire losses is to react quickly.

In all cases except for the smallest fires, take the following steps:

a. Activate the CPC evacuation alarm.

b. Set off the sprinkler system if it hasn't already tripped. Turn on monitor nozzles as necessary to cool structures and adjacent equipment.

c. Report the fire to the guards.

d. Alert other units via the red phone.

e. Put on Scott Air Packs or emergency escape masks.

f. Isolate the equipment or section of the plant.

g. Secure and control the plant.

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h. The lead operator is responsible to make contact with the Emergency Squad, direct them to the fire, and apprise them of the general situation.

For a very large fire, take the following steps:

a. Trip the CPC shutdown system.

b. Block the Cl_2 valve and steam into the plant.

c. The lead operator is responsible to make contact with the Emergency Squad, direct them to the fire, and apprise them of the general situation. He also makes a "head count".

Remember that when CPC and PC burn or contact water, HCl is liberated. You must, therefore, wear respiratory protection and stay upwind of the fire. (Note that burning VCM, EC and some other light organics float on water. Be aware that a fire can possibly be spread by this floating action.)

4. Emergencies in Other Units

In the event of an emergency (fire, explosion, vapor release) in another unit within the Lake Charles complex, attempt to keep CPC operating unless loss of chlorine, air, steam, power, incinerator etc. dictate otherwise. It takes less effort and manpower to keep the plant operating than it does to shut it down. Operators remain at their jobs unless requested by shift supervision or the Disaster Coordinator to aid in the emergency, or unless the emergency requires a total plant evacuation.

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Isolate any lines going to or from the affected unit as necessary. For a major gas release in another unit, evacuation of the CPC Plant may be necessary. Shut down the control room air intake and use the emergency breathing air system. If evacuation is required, follow those steps (as applicable) as given in the procedure for Major Break, Gas Leak, or Explosion.

B. OPERATIONAL UPSETS

These procedures are guidelines for a number of various operational upsets or abnormal operating conditions. In most cases, operational upsets do not involve the hazardous conditions discussed in the previous section. It must be remembered, though, that failure to act promptly and correctly during an operational upset to quickly bring the plant under control will almost surely leads to a much more serious emergency.

NOTE: In many cases of operational upsets, chlorine and/or HCl may be released as a result of the upset. Wearing of respiratory protection (Scott Air Pack) is advised.

1. Momentary Loss of Electrical Power

Results:

a. All motors (pumps, cooling tower fans, compressor, etc.) shut down.

b. Relay logic shutdown systems (chlorine vaporizer, steam to heater, etc.) trip.

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c. If the power loss is complex wide, Sabine water and plant air may be momentarily lost until these systems can be restored.

Actions to take:

a. Restore cooling - cooling tower pumps and fans, (WW booster pumps).

b. Reset control panel instrument power (resets behind panel board).

c. Restart process pumps.

d. Re-establish "float" on Cl_2 vaporizer

e. If reaction was in progress during power loss re-establish Cl_2 feed in accordance with the reaction start-up procedure in the SOP Manual.

f. If distillation was in progress, re-establish vacuum if necessary.

g. Put distillation column on total reflux.

2. Long-Term Power Loss

Results:

a. Same immediate results as when momentary power loss occurs, i.e., loss of all motors, relay logic control systems, etc.

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b. Plant air may be lost.

Actions to take:

a. Shut down the plant.

b. Put plant in non-operating safe condition, i.e., block all feeds, and heat inputs.

c. Isolate vacuum compressor.

d. Block discharge valves of pumps that were operating.

e. Block control valve stations as necessary. Block any control valve bypasses that were open.

f. Make frequent rounds through the plant to monitor pressures.

g. Block all deluge valves. Log in logbook that valves are blocked. Unblock valves as soon as power is restored.

3. Loss of One 13.8 KV Feeder to CPC

Results:

One-half of the CPC Plant electrical equipment is essentially lost until feeder is restored or tie-breaker is closed.

Actions to take:

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a. Contact maintenance electrical duty man (week days) or shift superintendent (after 4 p.m. and on weekends). Electrical supervision must be present to determine cause of trip and to restore feeder or close tie-breaker.

4. Loss of Air

Results:

Control valves move to "fail-safe" position as air pressure decreases.

Actions to take:

a. Shut down THE PLANT.

b. Block control valve stations as necessary. Do not rely upon control valve "fail-safe" positions to prevent flow through a line.

c. Close steam control valves from the control room and block steam to the plant.

d. Shut down all process pumps and block them off.

e. Shut down and isolate vacuum compressor.

f. Leave on cooling tower pumps and fans until plant is secured.

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5. Loss of 150 psig Steam

Process interruption due to loss of steam is generally much slower than one caused by an electrical or air failure. If loss of steam becomes likely, you should begin preparing to shut down, but keep the plant operating until there is no hope that steam will be available. If it does become necessary to shut down, even if steam is completely out, the shutdown can be orderly. Some equipment can be left in operation, which makes restart much easier.

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Results:

a. Loss of steam to bayonet heater.

b. Distillation column dumps.

Actions to take:

a. Stop Cl_2 feed and "float" the chlorine vaporizer if reaction was in progress. The reaction can not be initiated without the column on total reflux.

b. Put the column on total reflux if distillation was in progress.

c. Close steam control valve from the control room. Block steam to plant.

Block AIBN addition and column reflux.

e. Leave cooling tower pumps and fans in service.

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f. Shutdown vacuum compressor and isolate.

6. Loss of 50 psig Steam System

Total loss of the 50 psig steam system is unlikely unless the 175 psig steam system is lost while #3 incinerator is down or the 50 psig system suffers some mechanical damage.

If only the 50 psig system is lost:

Results:

a. Chlorine vaporizer floods and shuts down.

Actions to take:

a. Shut down CPC reaction if in progress. Block 150# steam to heater and AIBN and reflux.

b. Secure chlorine vaporizer per shutdown SOP.

c. If distillation was in progress, continue plant operation.

d. It may be necessary to route high pressure condensate to atmospheric condensate tank to isolate the 50 psig header.

7. Loss of Well Water

Loss of well water results in safety showers and utility drops. This causes no immediate major problems.

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8. Loss of Cooling Tower Water

The Tetra operator is responsible for proper operation of the OHC cooling towers and will attempt to restart fans/pumps, if these had shut down.

Loss of CTW results in a rise in process temperatures and the pressure on the system (loss of condenser) SRVs may lift. The secondary brine condenser may provide some cooling.

Actions to take:

a. It is necessary to reduce steam and chlorine rates to prevent overpressurizing the system.

b. If cooling is not restored soon, the plant must be shut down.

9. Loss of Chlorine

Results:

a. Loss of chlorine feed to CPC reactor.

Action to take:

a. Block AIBN flow if in reaction phase.

b. Secure the chlorine vaporizer and put in float mode when chlorine is restored.

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10. Loss of Cell Liquor

Loss of cell liquor will affect the ability to produce a new charge of emergency scrubber recirculation liquid.

Actions to take:

a. If the emergency scrubber had been in service and the scrubber recirculation liquid has been used, then the plant must remain down until the scrubber recirculating liquid is replenished.

11. Loss of Low Pressure Nitrogen

The most likely time for loss or lack of sufficient low pressure nitrogen is during a complex-wide upset such as a steam or power failure. When this occurs, there can be as many as two OHC reactors and five Per-Tri reactors trying to fluidize. Block of all nitrogen purges into process streams/vessels to ensure that process streams do not back up into the nitrogen system.

12. Loss of Condensate Supply

Loss of the high pressure condensate supply from Per-Tri can be caused by a number of factors.

Results:

Loss of desuperheater.

Action to take:

Monitor 150# steam temperature. This temperature may become excessive thus requiring a shutdown of the reaction or distillation.

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13. Loss of Refrigerated Brine From Per-Tri

Results:

Vent temperature increases and product losses to the vent increase. Vacuum compressor may overheat.

Action to take:

Watch temperatures and shut down if required.

14. Incinerator or Vent Header Trip

The CPC Plant cannot operate without an incinerator to burn the plant vents. See Section V.H. on the CPC Plant Vent Management System.

The plant will shut down. Start the scrubber recirculation pumps if the system pressure increases to the alarm point.

C. SPILL MANAGEMENT PROCEDURES

Federal and state regulations and plant policy require that certain materials not be allowed to enter our waterways or be discharged to the air. Common sense also tells us not to pollute. The following procedures are guidelines to follow in the event that there is a spill of some material in the plant. The best spill management procedure, of course, is to prevent the spill in the first place by everyone knowing and performing their jobs properly. When a spill does occur, direct efforts toward containing the spill and preventing it from reaching the waterways or overloading the plant treatment systems.

In all cases, take the following steps:

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a. Stop the leak/spill at its source or stop the action causing the problem.

b. Activate the roadway warning lights or evacuation alarm, if necessary.

c. Contain the spill to prevent it from reaching surface drainage which leads directly to the storm sewer.

d. Inform supervision. Supervision will inform the Environmental Affairs Department, who in turn will contact any necessary governmental agencies.

e. Operations supervision and the Environmental Affairs Department will determine corrective action and make arrangements for clean-up.

1. Spills of Solid Materials

Examples: AIBN and soda ash.

General Guidelines:

a. Shovel up and dispose in hazardous waste box.

2. Spills of Liquids

Examples: CPC, PC, $CaCl_2$, brine, oil, cell liquor, etc.

General Guidelines:

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a. CPC Plant sump valves are to be kept closed at all times. Draining to the lift station may then be permitted. Take action to neutralize the spill with soda ash.

b. Cover oil spills immediately with an absorbent such as Hi-Dri and swept or shovelled up.

c. Cover small spills of PC or CPC with soda ash and swept up or shoveled.

d. Inform the Waste Treatment operator if a large spill is routed to the lift station so Plant B steam stripper operation is not upset.

e. Dispose of used reagents and process samples in the lab sinks only.

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VII. PROCESS AND MECHANICAL FLOW SHEETS

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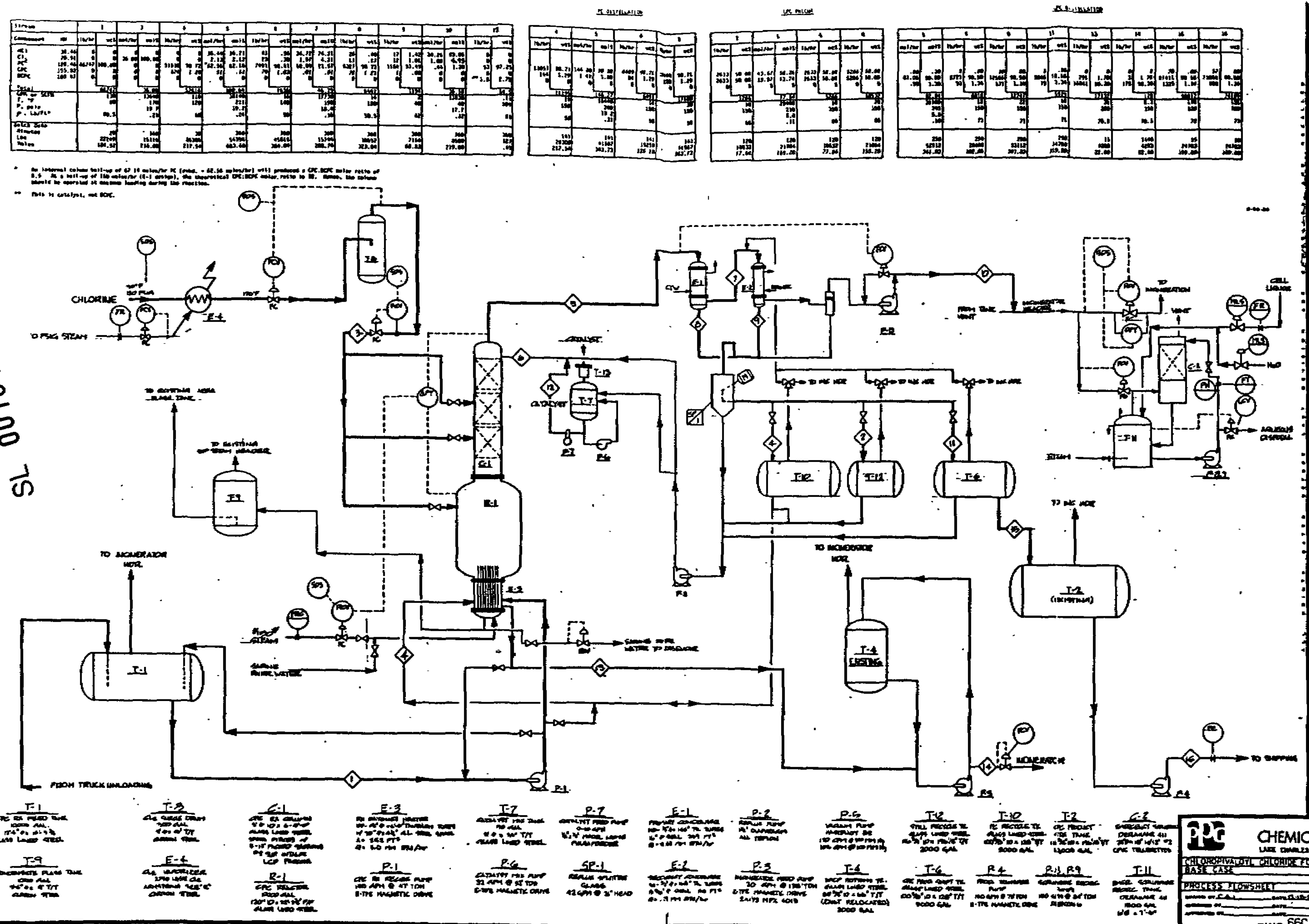

* An internal column boil-up of [illegible] moles/hr PC [illegible] will produce a CPC:BCPC molar ratio of [illegible]. At a boil-up of [illegible] moles/hr (E-1 design), the theoretical CPC:BCPC molar ratio is [illegible]. Hence, the column should be operated at maximum loading during the reaction.

** This is catalyst, not BCPC.

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PPG CHEMICALS
LAKE CHARLES, LOUISIANA
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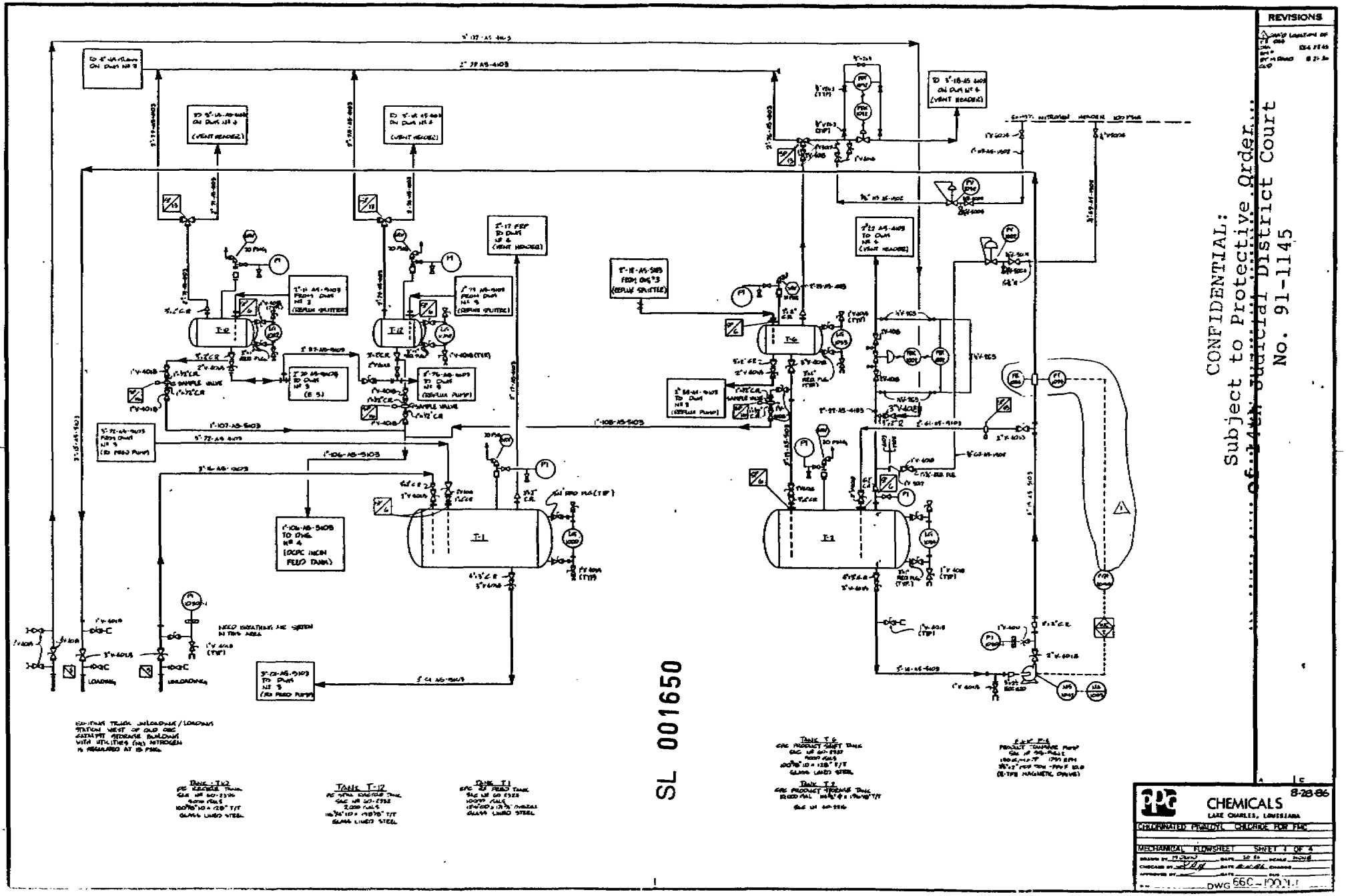
CHLOROPIVALOYL CHLORIDE FOR FMC BASE CASE

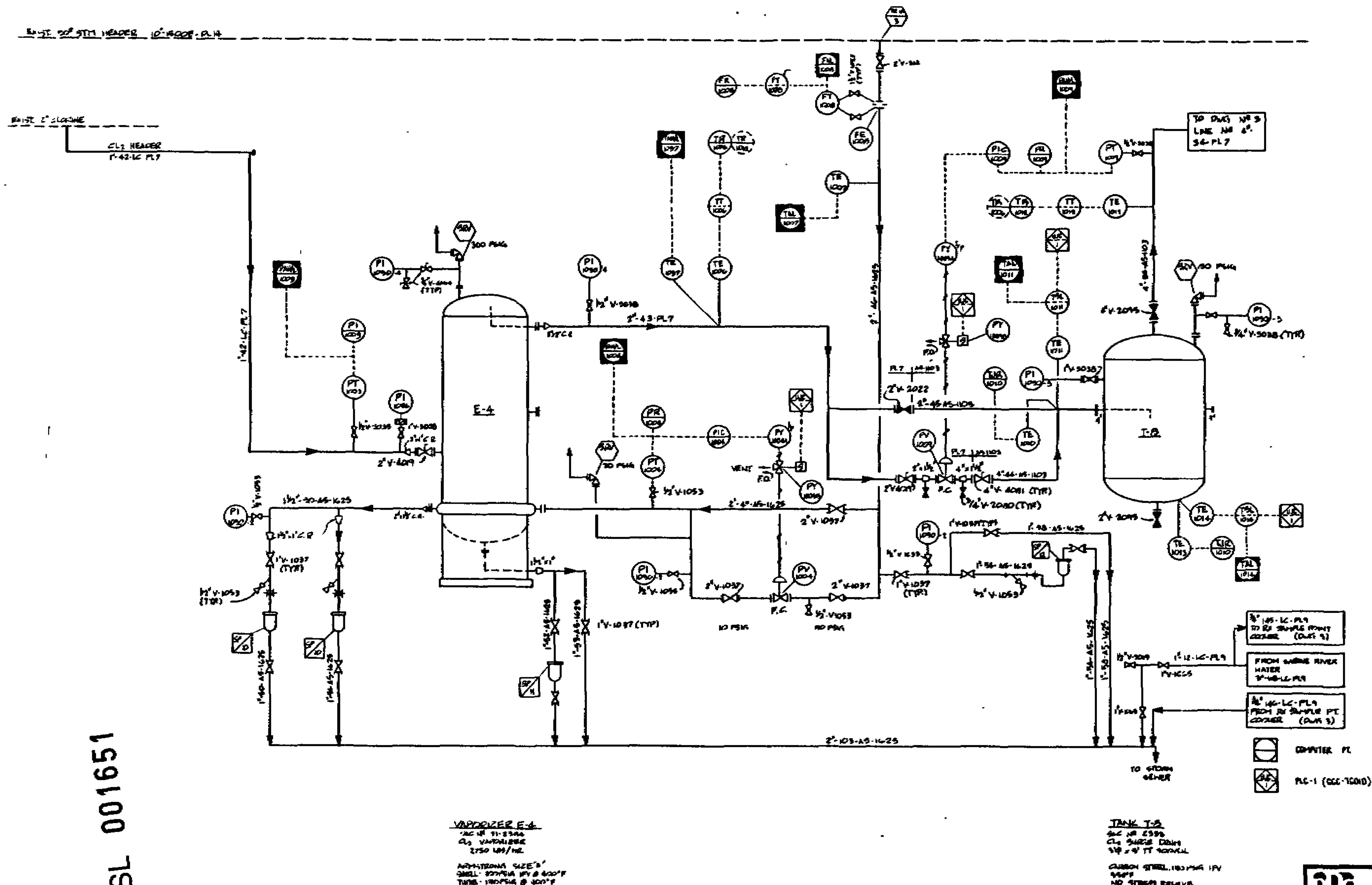
PROCESS FLOWSHEET

DWG 66C-10010


REVISIONS
CONFIDENTIAL:
Subject to Protective Order...
of the Judicial District Court
No. 91-1145
SL 001650
T-1
T-2
T-12
T-6
CHEMICALS
LAKE CHARLES, LOUISIANA
CHLORINATED PHENOL CHEMICALS FOR FMC
MECHANICAL FLOWSHEET
SHEET 1 OF 4
8-28-86
DWG 660-10011

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

REVISIONS



PPG CHEMICALS
LAKE CHARLES, LOUISIANA

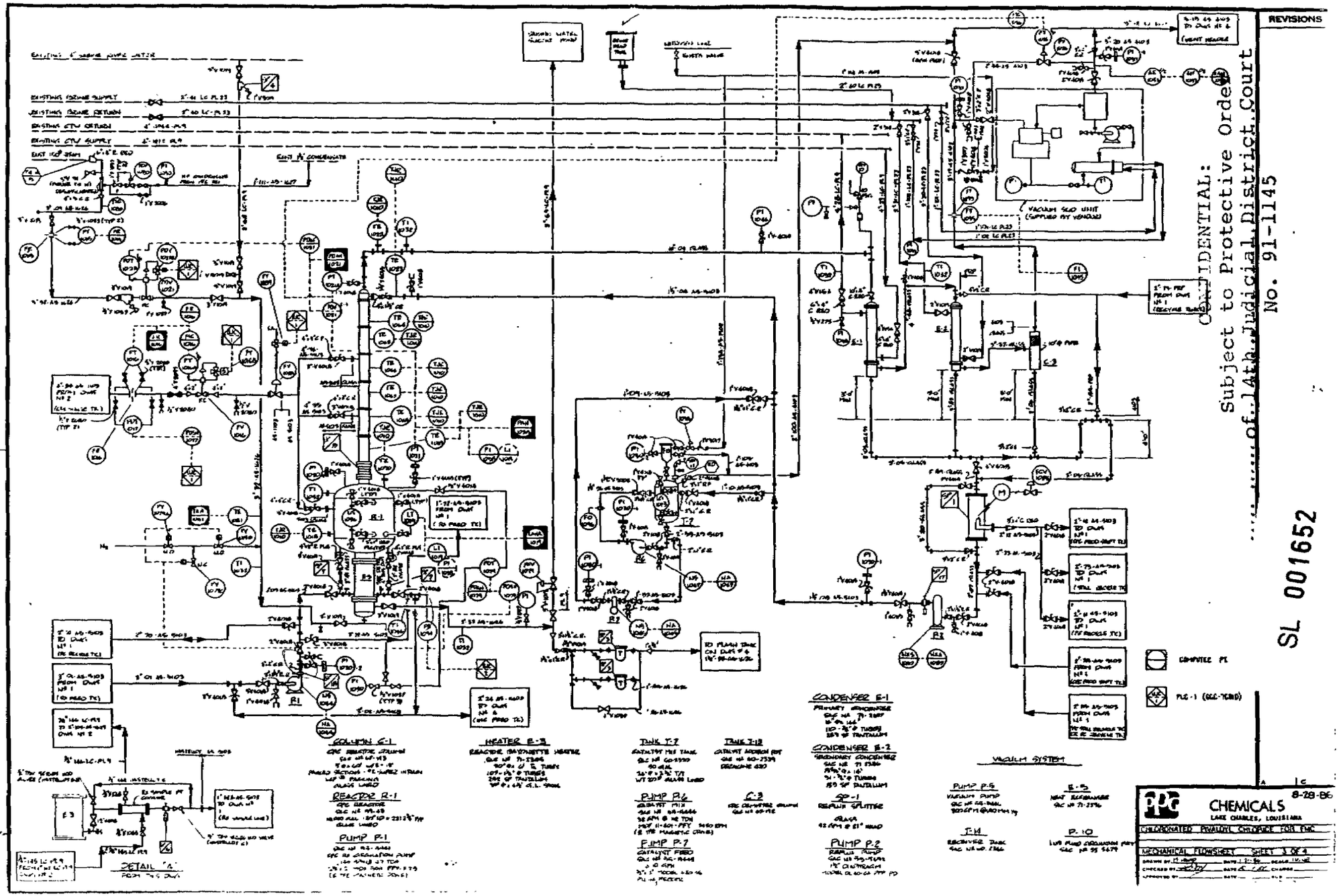
8-28-86

CHLORINATED PIVALOYL CHLORIDE FOR FMC

MECHANICAL FLOWSHEET SHEET 2 OF 4

DRAWN BY ____ DATE ____ SCALE ____
CHECKED BY ____ DATE ____ CHANGED ____
APPROVED BY ____ DATE ____

SL 001651


REVISIONS
CONFIDENTIAL:
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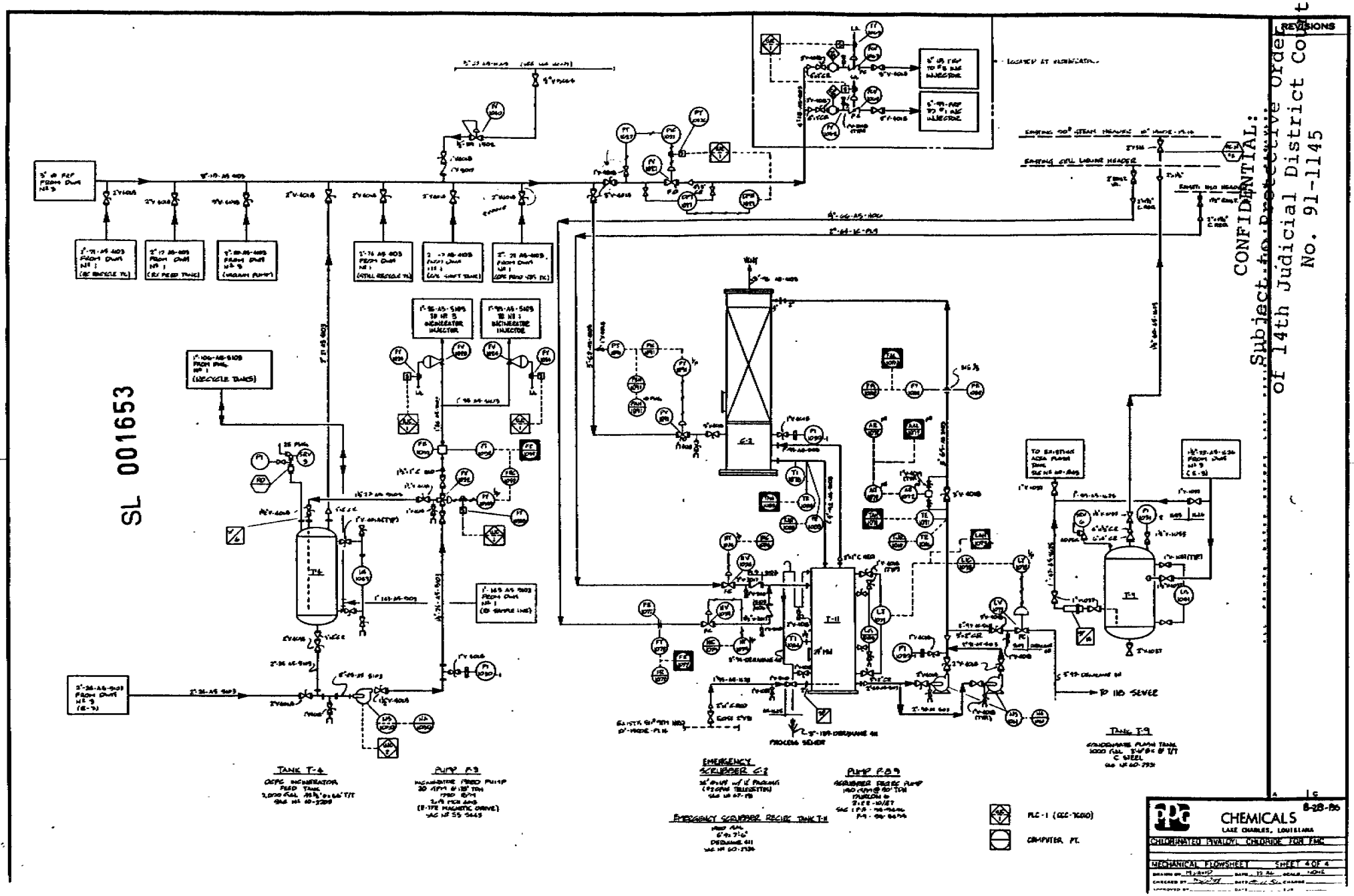
of 14th Judicial District Court
No. 91-1145
SL 001652
CHEMICALS
LAKE CHARLES, LOUISIANA
CHLORINATED PENTANE CHLORIDE FOR EMC
MECHANICAL FLOWSHEET SHEET 3 OF 4
8-28-86
REACTOR R-1
PUMP P-1
COLUMN C-1
HEATER E-3
TANK T-7
TANK T-12
PUMP P-4
PUMP P-7
C-3
CONDENSER E-1
CONDENSER E-2
SP-1
PUMP P-2
VACUUM SYSTEM
PUMP P-5
T-11
E-5
P-10
DETAIL "A"


REVISIONS
CONFIDENTIAL: Subject to Protective Order of 14th Judicial District Court No. 91-1145
SL 001653
TANK T-4
PUMP P-3
EMERGENCY SCRUBBER C-2
PUMP P-89
EMERGENCY SCRUBBER RECIRC TANK T-11
TANK T-9
TO HO SEWER
PROCESS SEWER
PLC-1 (CCC-7000)
COMPUTER, PT.
PPG CHEMICALS
LAKE CHARLES, LOUISIANA
CHLORINATED ETHALYL CHLORIDE FOR EMC
MECHANICAL FLOWSHEET
SHEET 4 OF 4
8-28-90