CEA 1668

BIPHENYL AND
AROCLOR

JAPAN

YOKKAICHI

PROJECT MANUAL

Central
Engineering
Department

Monsanto

MONS 095266

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MANUAL NO. 1

CONFIDENTIAL

PROJECT MANUAL

Monsanto

Central Engineering Department

MONS 095267

PROJECT MANUAL
CENTRAL ENGINEERING DEPARTMENT
MONSANTO COMPANY
ST. LOUIS, MISSOURI

PROCESS DESIGN PACKAGE FOR NEW 5.5$\bar{M}$ LBS/YEAR BIPHENYL AND 10$\bar{M}$ LBS/YEAR AROCLOR MANUFACTURING FACILITIES FOR MONSANTO MITSUBISHI KASEI (MMK) AT YOKKAICHI, JAPAN

CEA-1668

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1 PROJECT SCOPE REPORT

MONS 095269

3. Blow air into the blow tank for two hours before transferring to the still pot.

4. When blowing aroclors 1154 and 1160 adjust steam pressure to coil to hold temperature at 140°C.

   When blowing aroclors 1142 and 1148 steam should be off the coil to prevent air distilling these aroclors into the scrubber.

B. Control and Alarm System

1. Temperature of the batch is controlled by a steam pressure regulator to the aeration coils.

2. A float type level gauge is provided to show product level in the tank. This is covered in detail on instrument schematic drawing I-9.

C. Emergency Shutdown

1. Shut off air blower.

2. Shut off steam to coil.

D. Scheduled Shutdown

1. Examine sparger to determine erosion or corrosion of inlet holes.

Section C Distillation

I. Still Pot, Cyclone, Still Heater, Still Condenser, Still Receiver

A. Startup and Normal Operation

1. Design Considerations:

a) Time cycle considerations:

	For 1142	For 1160
Heat up	1.9 hr.	2.1 hr.
Heat up and distill	22.0 "	25.0 "
Add lime	0.5 "	0.5 "
Heat up and distill	22.0 "	25.0 "
Distill to Montar	3.5 "	4.0 "
Draw off Montar	1.5 "	1.5 "
Total time	51.4 "	58.1 "

	For 1142	For 1160
Actual Distilling Time	44.0 hrs.	44.0 hrs.
Distilling Rate	2300 lb/hr.	2300 lb/hr.
Capacity	15.3$\overline{M}$ lb/yr.	13.5$\overline{M}$ lb/yr.

2300#/hr.x8750 hrs.
x 44 x 90%

58.1 or 51.4

b) Heater design is based on an absorption rate of 400,000 Btu/hr. into the circulating mass to give a distillation rate of 2300 lb/hr. This rate allows for initial batch heatup, heat up during continuous operation and for vaporization.

c) The still vapor condenser is designed for a heat loading of 400,000 Btu/hr. Calculated loading is:

Desuperheat	37,000 Btu/hr.
Condense	230,000 "
Cool	122,000 "
Total	389,000 "

This is based on distilling aroclor 1142 with an inlet temperature of 210°C and condensate outlet temperature of 100°C.

d) Heat removal from the condenser is accomplished with water in a high volume circulating system. This will allow better control over condensate temperatures and should prevent fouling of the condenser.

e) Distillation rate is controlled by controlling the oil feed rate to the external heater.

2. Still Operation

a) Close bottom outlet valve on still. Leave ram jet valve open about one inch.

b) Align valves to charge from No. 1 or No. 2 Blow Tank. The automatic level control valve and flow integrator will pass 30 gallon/minute.

c) Start blow tank transfer pump and charge 578 gallons into still pot. This is equal to 33" on the gauge in the still pot leaving 48" of freeboard for disengaging height. When still is at proper level shut off valve to still and transfer pump.

d) Add 125 pounds of lime directly to the still. Close up still and still receiver.

e) Energize the steam ejector.

1. Close 4" valve to deadhead the jet.

2. Open steam valves to first and second stages.

3. Vacuum should pull down steadily to about 4mm Hg. absolute and hold steady.

4. Open water valve to the after condenser and adjust to point where no steam is escaping from the after condenser vent.

f) Align valves to still receiver from ejector and condenser.

g) Energize the still heater.

1. Leave the "auto-man" switch on FRC-311 in the "auto" position with set point at minimum flow for tempering the heater coils.

2. Check that heater damper is open.

3. Circulate oil until header is completely filled with fuel. Burners normally require a viscosity of 15-25 SSF at 200°F.

4. Light gas pilot.

5. Open steam valve and cross over valve to oil gun until both oil and steam lines to the burner are completely warm with steam.

6. Close cross over valve and adjust steam valve to proper pressure.

7. Open oil valve gradually and adjust to proper burner pressure.

8. After burner lights, open block valve in oil line wide, the FRC-311 will be the heat control. Adjust steam valve for clean, bright fire.

9. Adjust primary and secondary air for stability.

10. Steam pressure should run 15-25 lb. above oil pressure.

h) Energize the water circulating system to the condenser.

1. Align valves at the water circulating pump to put water in at the bottom of the condenser, out at the top and back to pump suction.

2. Adjust TIC-240 to allow water into pump suction.

3. Start pump. As soon as water is circulating properly set TIC-240 to temperature required. Expected temperature set points will be:

For 1242 - 50°C
1248 - 50°C
1254 - 55°C
1260 - 65°C

i) Align valves in still pump line to circulate through the furnace coil and back into the still.

j) Set vacuum-recorder to maintain 40 mm Hg. absolute pressure. Observe the schedule start of stills to insure that operating vacuum is reached before boiling temperature is reached. If this is not done properly, the overheated aroclor may flash and belch over crude material if the vacuum is lowered quickly.

k) Slowly increase the flow of fuel oil to the still heater by adjusting FRC-311. If the furnace has an efficiency of 70% then the increase should follow approximately the table established below:

GPH	Btu Input
1	150,000
2	300,000
3	450,000
4	550,000 - Design Point
5	700,000

l) When the still first starts to flow, start the blow tank transfer pump and open the block valve in the line to the still. Record the reading on the input flow integrator, FQ-223. The level control will now maintain a level in the still dependent on the amount of oil burned at the heater and the amount of condensate flowing into the still receiver. It may be possible to operate the transfer system with the transfer pump down but valves open to the blow tank.

m) When the still is under proper operating conditions the following data can be expected:

1. Temperature in Still Pot

Aroclor	Degree ^{o}C
1242	200
1248	215
1254	230
1260	250

2. Vacuum — 40 mm Hg. Absolute

3. Flow Rate — 2300 pounds/hour

4. Coil Temperature — Steady with a difference of about 8°C between inlet and outlet temperature.

5. Still Condenser — Foward flow temperature at 100°C for 1242, 140°C for 1260.

6. Still Pump	Output pressure about 50 PSIG.
7. Oil Feed	About 5 GPM maximum rate.

n) When the still receiver is full align valves to pump to surge tank.

o) Start receiver transfer pump. The level control system will allow continuous pump-out without dead-heading the pump.

p) When 5000 gallons of feed material has been distilled shut off feed valve to still and valve in condensate flow line to still receiver. This is measured on FQ-223. Shut off oil flow to heater.

q) Release vacuum and add 125 pounds of lime to the still pot.

r) Restart still system by opening valve to still receiver, opening valve in feed line to still and starting flow of oil to still heater.

s) Continue to distill into the still receiver until an additional 5000 gallons has been distilled. At this point shut off the still feed and begin still boil down.

t) Still boil down procedure:

1. Reduce circulation rate of still pump by closing outlet valve on pump. Regulate to a pump pressure 10-15 PSIG below the pump operating pressure.

2. Continue to boil down to one or all of the following endpoints:

a) Observe pump pressure. If it drops below 10 PSIG the distillation is finished.

b) Observe temperature spread between inlet and outlet of coil. When this spread reaches 35°C or more the distillation is finished.

c) Observe forward flow. When flow stops the distillation is finished.

3. Shut down oil flow to heater. With vacuum still on close valve under bottom of still and open vent valve at pump. Suck vacuum through pump and pump discharge pipe until line is clear. Shut down pump. Close block valve at pump. Close vent valve at pump.

4. The ram valve should be open about 1" during the distillation. Now close to break the crust and open to allow air to suck into the still pot. Close immediately.

5. Release vacuum at still receiver.

u) To drop residue:

1. Turn water and steam on fume ejector.

2. Drop bottoms into open top drums using a thermometer or measuring stick to tell when the drums are 2/3 full.

3. When the draw-off is complete, close bottoms dropping valve to within 1" of being completely closed.

4. Shut off water and steam to fume ejector.

B. Control and Alarm System

1. Still Pot

a) Covered in detail on instrument schematic drawing I-10 which covers the following loops:

LRCA-229	Level control on the still
PRC -231	Vacuum control on the still

b) Set level in still pot to alarm as follows:

High level - 60" from bottom bend line
Low level - 12" from bottom bend line

c) If circulating pump shuts down, alarm will sound.

2. Still Heater

a) Covered in detail on instrument schematic drawing I-13 which covers the following loop:

FRC-311	Fuel oil control for heater

b) Set alarms as follows:

High coil outlet temperature	300°C
Low inlet oil pressure	100 PSIG

3. Water Circulating System

a) Covered in detail in instrument schematic drawing I-10 which covers the following loop:

TIC-240	Water temperature control to condensers

4. Still Receiver

a) Covered in detail on instrument schematic drawing I-10 which covers the following loop:

LCA-236	Level control in still receiver

b) Set level in receiver to alarm as follows:

High level	50" from bottom bend line
Low level	6" from bottom bend line

C. Emergency Shutdown

1. Shut off feed to the still pot.
2. Shut off oil flow to the heater.
3. Release vacuum by readjusting the vacuum controller to 760 mm Hg. Absolute.

D. Scheduled Shutdown

1. Check for corrosion in the heater, still pot and pump system.

Section D Absorption

I. Absorbers, Surge Tanks, Polish Filter, Finished Product Storage

A. Startup and Normal Operation

1. Design Considerations:

a) Absorbers

1. Based on experience in the Anniston experimental column. This is covered in reports for Job 370-322.

2. Flow rate - 2300 lb/hr.
Viscosity - 40-45 SUS
Height to diameter ratio - $7\frac{2}{5}$/1 (3/1 minimum)
Design mass flow - 1 GPM/Ft.^{2} of bed

3. Vibrators should be installed on the absorbers to facilitate charging ~~and~~ to and to prevent channeling.

4. Porocel - Regular grade 20/60 is the charge material for the absorbers.

5. Absorbers must be charged with dry porocel before starting the distillation system. Charging should be made with vibrators operating. It is absolutely essential that the porocel be dry before treating of the aroclor begins. The bed may have to be heated with steam coil provided and blown with nitrogen to accomplish this.

6. Provide individual absorbers for each aroclor to prevent product contamination.

b) Surge Tank (Item 223)

1. A coil with agitator is provided for control of temperature to the absorber.

2. It is desirable to have the goods at a temperature of 100°C when charged to the surge tank.

3. The main function of the tank is to control product inlet temperature to the absorbers.

c) Filter (Item 227)

1. This unit collects any porocel that passes through the support plate in the absorber.

2. Filter elements are replaceable fiberpaks.

2. Absorber Operation

1. Set controller on crude surge tank to regulate temperature to the proper value for viscosity control and start agitator. These temperatures are:

Aroclor 1242	70°C
1248	85°C
1254	110°C
1260	135°C

NOTE: The temperature of the product stream from still condenser will determine whether water cooling is to be used or steam heating.

2. Align valves to proper absorber, from absorber through filter to finished product surge tank.

3. Set flow recorder to the desired flow rate. This should not be over 3½ GPM.

4. Start flow into Item 223 from still receiver and when temperature is adjusted to proper figure start flow through system.

5. For initial operation it may be desirable to circulate back to the crude surge tank until lab tests show product is within the standard range.

6. When product stream is within proper specification start collecting product in finished product surge tank.

7. When finished product surge tank is 80% full align valves to proper storage tank.

8. Pump surge tank to storage.

B. Control and Alarm System

1. Crude Surge Tank

a) Covered in detail in instrument schematic drawing I-11 which covers the following loops:

TIC-255	Temperature control system
LIA-253	Level control crude surge tank

b) Set level in crude surge tank to alarm as follows:

High level	75" from bottom bend line
Low level	12" from bottom bend line

2. Absorbers

a) Covered in detail in instrument schematic drawing I-11 which covers the following loops:

FRC-256	Feed flow control to absorber
TIC-258	Temperature control for the absorber

b) Set flow on FRC-256 at a figure to equal the forward flow from the still condenser. This can be judged by readings taken on FQ-223.

c) Set TIC-258 to maintain proper temperature into and out of the absorbers based on the product being treated.

3. Finished Product Surge Tank

a) Covered in detail in instrument schematic drawing I-12 which covers the following loop:

LIA-261.1	Level control finished product surge tank

b) Set level in finished product surge tank to alarm as follows:

High level	75" from bottom bend line

C. Emergency Shutdown

1. Shut off feed to absorbers.

2. Shut off feed to crude surge tank.

3. Shut off water or steam.

D. Scheduled Shutdown

1. Check for corrosion in the surge tanks, absorbers and polishing filter.

Section E Blending

I. Blend Tank, Filter, Raw Material Storage

A. Startup and Normal Operations

1. Design Considerations:

a) Blends required are as follows:

	Interteen KA	Interteen PPO
Aroclor 1260	-	60%
Aroclor 1254	70%	-
Trichlorbenzene	30%	40%
PPO	0.2%	0.2%

NOTE: % by weight

b) To produce the interteens pump the desired amount of trichlorbenzene into the mix tank. Treat with Attapulgus Earth. Pump in the desired amount of aroclor, mix thoroughly and have analysis made for specific gravity, refractive index, moisture and chlorides.

2. Blending Operation

a) Operation of the blending tank

1. Close valve under the blend tank.

2. Align valves from TCB storage to blend tank and pump to desired measurement.

3. Start agitator. Align valves to circulate, bypassing the filter.

4. Add 10 pounds of Attapulgus Earth. Circulate and mix for two hours.

5. Realign valves to go through filter press and remove Attapulgus Earth.

6. Repeat Items (4) and (5). Shut off circulating pump.

7. Pump in the proper amount of Aroclor 1260 or 1254 on top of the TCB. When the measurement is 6" from the cut-off point turn off the agitator so an accurate measurement can be made. Shut off the charge valve at the desired measurement.

8. Agitate for two hours. Then take a control sample. Results desired are usually, specific gravity, refractive index, moisture and chlorides.

9. Take a sample from the PPO drum (Glycidal Phenyl Ether) to be used in the mix. Results desired are usually, specific gravity and chlorides.

10. After receiving the laboratory report make any adjustments necessary with TCB or Aroclor 1254 or 1260. Blend again and resample. Repeat until the mix is within specification range.

    TCB will lower specific gravity and refractive index.

    1254 or 1260 will raise specific gravity and refractive index.

    If moisture is high blow with dry air or nitrogen.

11. Add the exact amount of PPO to the batch mix by sucking it in through the suction side of the circulating pump.

12. Agitate for two hours. Take final sample for complete analysis.

13. If electrical properties of the aroclors used are low it is not necessary to treat the entire batch, aroclor and TCB, with Attapulgus Earth. If electrical properties are high use the following procedure:

a) Add 25 pounds of Attapulgus Earth to the batch.

b) Circulate and agitate the mix for four hours. Bypass the filter press. Take sample for analysis.

c) Make necessary adjustments with TCB or 1254 or 1260 as required.

d) Add specified quantity of PPO. Mix for two hours. (Circulate and agitate).

e) Realign valves to go through filter press and remove Attapulgus Earth. Circulate for one hour before taking final samples. Continue to circulate for two hours.

f) Blow filter with air or nitrogen and clean.

g) While press is being cleaned, add 25 pounds of Attapulgus Earth, and agitate. Circulate through press for 30 minutes before drumming or loading tank cars. Blow filter press with air and clean.

B. Control and Alarm System

1. This is covered in detail on instrument schematic drawing I-12 which covers the following loops:

TIC-301	Temperature control, blend tank
LT-305	Level in blend tank
TIC-304	Temperature control, raw material storage tank

2. Controls and alarms are set up as follows:

a) Steam is controlled by means of a temperature controller on the plate coil of the raw material tank.Set at 90°C.

b) Steam is controlled by means of a temperature controller on the plate coil of the blend tank. Set at 90°C.

C. Emergency Shutdown

1. Shut down agitator.

2. Shut down circulating pump.

D. Scheduled Shutdown

1. Examine tanks and agitator for corrosion.

PROJECT SCOPE REPORT

CEA 1668

BIPHENYL & AROCLOR
MANUFACTURING FACILITIES
FOR
MONSANTO MITSUBISHI KASEI (MMK)
YOKKAICHI, JAPAN

March 1968

A. J. Heinze

MONS 095270

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A. Process Flow Sheets

B. Equipment List and Summary Sheets

1.1 Introduction

This scope report describes the facilities required to manufacture biphenyl and Aroclor at the Yokkaichi, Japan plant of MMK. The biphenyl facilities will be similar to those installed and operating at Monsanto's Anniston, Alabama plant. The Aroclor facilities will be similar to those installed and operating at Monsanto's WGK plant at Sauget, Illinois, and at the Anniston plant.

1.11 Project Objectives

The object of this project is to provide manufacturing facilities to produce 5.5M lbs/year of biphenyl and Aroclor 1242, 1248, 1254 and 1260 at an aggregate total rate of 10M lbs/year. This project will include an Aroclor blending system and warehouse, drumming and storage facilities for finished goods.

1.2 Process Technology

1.21 Process Description

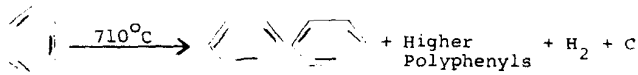
a) Biphenyl process:

Benzene is converted into biphenyl, higher polyphenyls, carbon and hydrogen by raising its temperature above 650°C in a direct fired tubular furnace and holding the reaction mass until a portion of the benzene has reacted. Acetone is added to the furnace feed and acts as a reaction promoter. The vapors from the furnace pass to a fractionating column where the unreacted benzene and by-product hydrogen are distilled overhead. The benzene is condensed and recycled to the furnace. The hydrogen is compressed and cooled to recover benzene for recycle. The compressed hydrogen is subsequently vented to the atmosphere.

The crude biphenyl from the bottom of the benzene stripping column is fed to the refining column where product biphenyl is distilled overhead.

The ratio of biphenyl to higher polyphenyls is basically controlled by varying the benzene conversion per pass or the amount of biphenyl recycled to the furnace with the benzene.

The overall reaction which takes place in the pyrolysis furnace is as follows:



Benzene Biphenyl Hydrogen Carbon

b) Aroclor process:

Aroclor is a trade name used to designate chlorination products of biphenyl, higher polyphenyls or a mixture of biphenyl and higher polyphenyls.

Biphenyl is chlorinated at 150°C in the presence of ferric chloride catalyst to a specified specific gravity which is a measure of chloride content. Hydrogen chloride gas is liberated during this reaction and can be recovered or absorbed in water and sewered.

The crude Aroclor is then air blown to remove the residual hydrogen chloride and transferred to a batch still. The crude Aroclor is then distilled under vacuum in a semi-continuous fashion in a direct take-over still. Heat input to the still is supplied by an external oil-fired heater.

The still residue contains lime, high boilers, tars, ferric chloride and Aroclor which is drummed and discarded or sold as Montar.

The distilled material is the finished Aroclor product and is further refined by treatment with porocel, filtered, then stored in a nitrogen atmosphere.

1.22 Yields and Raw Materials

	Lbs/100 Lbs Biphenyl
Benzene	115.4
Acetone	1.28

	Lbs/100 Lbs Aroclor			
	1242	1248	1254	1260
Biphenyl	61.02	55.02	49.01	43.00
Ferric Chloride	0.03	0.03	0.03	0.03
Chlorine	90.32	103.23	116.13	129.03
Air	22.56	28.67	16.25	30.97
Lime	0.50	0.50	0.50	0.50
Porocel	0.15	0.15	0.15	0.15

	Lbs/100 Lbs Blend
Aroclor 1254 or 1260	60.00
Trichlorobenzene	40.00
Glycidal Phenol Ether	0.21
Attapulgus Earth	0.05

1.23 Material Specifications

Raw material and product specifications as indicated below are appended.

Raw Material or Finished Product	Code No.
Benzene	14700
Acetone	82905
Ferric Chloride	33500
Chlorine	82638
Lime - $Ca(OH)_2$	38800
Attapulgus Earth	19400
Trichlorobenzene	60200
Glycidal Phenol Ether	36100
Biphenyl	82601
Aroclor 1242	82612
Aroclor 1248	81102
Aroclor 1254	82613
Aroclor 1260	82614
Inerteen 54201-KA	82681
Inerteen PPO	82649

1.24 Major Basis for Flow Sheets

The Anniston biphenyl plant and the WGK and Anniston Aroclor plants will form the basis for the flow sheets and engineering flow diagrams generated for this project. The only modifications to design will be those needed to solve specific problems such as waste disposal at the Yokkaichi plant.

1.25 Packaging

The major liquid products from the plant (Aroclor 1242, 1248, 1254, and 1260 and Inerteen) will be shipped in tank cars or tank trailers, or will be packaged in 55-gallon steel drums. No product flaking facilities are included in this project.

1.26 Waste Streams

During the production of biphenyl, small amounts (approx. 51 lbs/hr.) of hydrogen off-gas will be vented to the atmosphere. No liquid waste streams are

produced during normal operation. The residue (higher polyphenyls) from the biphenyl refining step is assumed to be drummed for disposal.

During Aroclor production, approximately 630 lbs/hr. (average rate) of a mixture of chlorine and hydrogen chloride gas is liberated from the chlorinator and aeration tanks. This gas mixture is scrubbed with water in a drowning jet and sewered. During the distillation operation, about 70 lbs/hr. (average rate) of Montar (higher polyphenyls) discharges from the still pot. It is assumed that this material is drummed for disposal. Solids disposal at the filter amounts to about 7 lbs/hr. of cake consisting of approximately 70% Aroclor product and 30% clay. During the Inerteen blending operation, about 25#/batch of attapulgus clay is discharged from the filter. It is assumed that these solid wastes will be drummed off prior to disposal.

1.27 Personnel, Property and Public Hazards

1.271 Process Hazards

Hydrogen and benzene are present above their auto-ignition temperatures in the benzene pyrolysis furnace and transfer piping and therefore a leak will result in a fire. The furnace tubes and transfer piping are constructed of high temperature alloy steel to minimize this danger.

The pyrolysis furnace will be of the multiple-burner type fired with fuel oil. Interlocks will be provided to shut off the oil flow to the burners in the event of high or low benzene feed rate, high furnace stack temperature and process outlet temperature, combustion air blower low pressure and low fuel oil pressure.

1.272 Fire Hazards

Storage and process vessels containing the following chemicals above their flash points indicated are potential fire hazards:

Material	Flash Point, ^{o}F
Acetone	4
Benzene	12

Hydrogen gas leakage is a potential hazard because of its explosive properties and non-luminous characteristic when ignited.

1.273 Personnel Hazards

No special handling precautions are required for Porocel, lime or attapulgus clay.

Acetone

Breathing acetone vapors can result in a strong narcotic effect on severe exposure. Recommended maximum atmospheric vapor concentration is 1000 PPM by volume.

Benzene

Benzene is a toxic organic chemical and contact with liquid benzene and breathing of the vapors should be avoided. The maximum allowable concentration of vapors in air is 100 PPM. Both acetone and benzene liquid will be stored and handled outside and under normal conditions vapor concentrations will be less than the maximum allowable.

Biphenyl

Biphenyl is a toxic organic chemical and breathing of biphenyl vapors should be avoided. The maximum allowable concentration of vapors in air is 1.5 PPM. During processing biphenyl liquid is generally hot (100°C or above) and can cause serious thermal burns on contact with the skin. Therefore, skin contact with biphenyl must be avoided by the use of gloves and protective garments.

Chlorine Gas

Chlorine gas is a hazardous substance which is very dangerous to the eyes and lungs. Maximum allowable concentration of Cl_2 in air is 2 PPM. Concentrations in the order of 15 PPM will cause throat irritation. Canister gas maskes, Scott Air Paks or similar apparatus should be available in the area in case of abnormal chlorine concentrations.

Hydrogen Chloride Gas

HCl gas is a corrosive, poisonous substance and very dangerous to the eyes. The gas also has an irritating effect on the nose and throat, although the nature of the irritation is usually transient except in cases of prolonged exposure. Maximum allowable concentration of HCl gas in the air is 10 PPM.

Ferric Chloride

This compound, which is a solid at room temperature, is not toxic but can be an eye irritant on contact. Normal precautions of wearing safety goggles when handling this material should be followed.

Trichlorobenzene

Trichlorobenzene is a colorless liquid which, on contact with vapors, is irritating to the eyes and respiratory system and can cause damage to the liver. Normal safety precautions should be followed when handling this material.

Aroclors

During processing, Aroclors are at elevated temperatures (100°C or above) and can cause serious thermal burns on contact. Breathing of vapors from this material should be avoided. At elevated temperatures, the vapors are quite toxic.

1.3 Manufacturing Facilities

1.31 Capacity

a) Biphenyl production design capacity 5.5$\overline{M}$ lbs/year.

b) Aroclor production design capacity 10$\overline{M}$ lbs/year broken down as follows:

Aroclor	$\overline{M}$ lbs/year	Approx. hrs/year
1242	5.70	3825
1248	1.06	830
1254	2.46	2110
1260	0.78	705

c) Operating on-stream time: Approximately 85%.

1.32 Description of Facilities

1.321 Biphenyl Unit

The following major items of equipment will be installed:

a) Pyrolysis furnace - The pyrolysis furnace is a direct fired, multiple-burner, box type tubular heater for the vaporization and pyrolysis of benzene. The furnace will operate in conjunction with external feed preheaters (heat exchangers) to take advantage of the high level heat in the reaction mass effluent from the radiant section of the furnace.

b) Benzene stripping column - The benzene column is a vertical, cylindrical, valve type tray distillation column which functions to separate unreacted benzene from crude biphenyl from the pyrolysis furnace. The benzene, which is taken overhead in the column, is condensed and recycled to the furnace. The benzene-free biphenyl and higher polyphenyls from the bottom of the column are pumped to two new 3,000 gallon storage tanks. These tanks also serve as feed tanks for the biphenyl refining facilities.

c) Hydrogen compression facilities - The benzene-saturated hydrogen, which is formed during the furnace reaction, is compressed in a two-stage reciprocating compressor to 300 PSIG. The compressed gas is cooled and the condensed benzene is recycled to the furnace.

d) Biphenyl refining facilities - The biphenyl refining column is a vertical, cylindrical tray-type column which functions to separate biphenyl from higher boiling polyphenyls. The biphenyl overhead product is sub-cooled and pumped to storage. This column operates in conjunction with an oil-fired reboiler furnace.

1.322 Aroclor Unit

The following major items of equipment will be installed.

a) Chlorinator system - The chlorinator is a 1400 gallon batch vessel used to convert biphenyl to crude Aroclor. The chlorinator operates in conjunction with an 800 gallon biphenyl mix tank and two 2000 gallon aeration tanks.

b) Aroclor distillation - The distillation facilities consist of a 1350 gallon still pot which is heated by an oil-fired still furnace.

c) Clay treatment - The crude Aroclor from the distillation unit is further refined by Porocel treating. The treating system consists of a 1250 gallon surge tank, four vertical adsorption vessels, a polishing filter and a 1250 gallon product receiver.

d) Aroclor blending - The Aroclor blending system consists of a 5000 gallon agitator blend tank and filter.

1.33 Major Storage Facilities

Storage facilities to be provided are as follows:

a) Benzene - one 15,000 gallon tank.

b) Acetone - one 800 gallon tank.

c) Crude Biphenyl - two 3,000 gallon tanks.

d) Refined Biphenyl - one 10,000 gallon tank.

e) Finished Aroclor Product - one 23,000 gallon, one 10,000 gallon and two 8,000 gallon tanks.

f) Trichlorobenzene - one 10,000 gallon tank.

g) Fuel oil - one 10,000 gallon tank.

1.34 Flexibility and Future Expansion

The operation of the biphenyl unit is based on producing minimum cost biphenyl when there is no outlet for higher polyphenyls. Additional biphenyl capacity is achievable if higher polyphenyls are desired. Higher rates can be obtained at the following conditions.

	Design	Alternate Operations			
Feed, lbs/hr.	9250	8440	8440	8440	8440
Outlet, °C	715	740	740	740	740
Biphenyl recycle,%	0.5	~~10.0~~ 0.5	~~7.0~~ 0.5	~~3.0~~ 0.5	0.5
Conversion, %	8.5	12	13	14	15
Acetone, %	0.1	0.15	0.15	0.15	0.15
M lbs/yr. Biphenyl	5.5	6.4	6.9	7.3	7.7
M lbs/yr. SANTOWAX C	0.63	0.99	1.15	1.31	1.49

No specific provisions for future expansion are included in either the biphenyl or Aroclor units.

1.4 Service Requirements

1.41 Utilities

The approximate utility requirements are as follows:

	Biphenyl	Aroclor	Total
Steam (170 PSIG), lbs/hr.	500	2300	2800
Cooling Tower Water, GPM	350	100	450
Fuel Oil, M Btu/hr.	9.5	0.75	10.25
Potable Water, GPM	10	205	215
Power, Installed HP	65	118	183
Air (75 PSIG) SCFM	50	25	75
CO_2 (200 PSIG) lbs/day	150	-	150

It is assumed that all utilities are available in the plant.

1.42 Services

This project includes a 50'x30' warehouse for drumming the Aroclor products and drum storage. It is assumed that no additional storeroom, shop or locker room facilities are needed for this project.

1.5 Plans and Schedules

The targeted completion date for the process design package is March 1, 1968.

1.6 Reference Reports

a) "Standard Manufacturing Process for Biphenyl" - Anniston Plant - December 1966 - B. O. Severson. (A complete list of reports pertaining to the production of biphenyl is contained in appendix N of this reference).

b) "Standard Operating Instructions for Biphenyl Tubular Units" - April 1964 - B. O. Severson.

c) "Standard Manufacturing Process for Aroclors" - Anniston Plant - September 1966 - M. J. Williams.

d) "Standard Operating Instructions for Aroclors" - Anniston Plant - April 1966 - M. J. Williams.

e) "Process Description for the Manufacture of Aroclors" - Department 246 - WGK Plant - October 1966 - D. E. Harris.

f) "Process Description for the Manufacture of Pyranols and Inerteens" - Department A246 - WGK Plant - October 1966 - D. E. Harris.

g) "Standard Operating Instructions for Aroclors and Pyranols. Department 246 and A246 - WGK Plant" August 1966 - V. Brawley.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
14700	Benzene - Nitration Grade
PLANT	USING DEPTS.
Anniston	20
DATE EFFECTIVE	SUPPLIER
7-20-66	Enjay Chemical Co.

APPROVALS

PRODUCTION	RESEARCH
R. G. Moody /s/R. G. Moody 6/9/66	N. F. Mueller /s/N. F. Mueller 7/13/66
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
H. L. Phillips /s/H. L. Phillips 6/10/66	O. E. Dolin /s/O. E. Dolin 6/9/66

GENERAL DESCRIPTION

Colorless, clean liquid

CHEMICAL FORMULA

C_6H_6

Mol. wt. 78.108

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
Sept. 11, 1957	2 x 16 oz. NN bottles	J. A. Liddle - 5/18/66

CHARACTERISTIC	LIMITS	Typical Values	METHOD
UNRESTRICTED INFORMATION			
Appearance	Clear liquid with no clear free water or suspended matter		G-1
Acidity	No free acid		D-847
Acid wash test, Barrett	2 Max.		D-848
Color, APHA	25 Max.	5	046
(R) Crystallizing Point	5.30°C. Min. wet basis 5.39°C. Min. dry basis	5.42	D-852
Distilling Range 100% over in range to include	1 °C. 80.1°C		D-850
Specific Gravity 60/60°F 25/25°C	 0.8820 to 0.8860 0.8740 to 0.8780		G-11
Copper Corrosion	Negative		D-849
Sulphur Compounds	Free of H_2S and SO_2		D-853
Total Sulfur, ppm	2 Max.	1	D-90
Total Chlorine, ppm	- - -	1	093
Aliphatics, ppm		3000	
Toluene, ppm		25	
Thiophene, ppm		<50	

The prefix "D" in method numbers indicates an ASTM method.

MONS 095281

(R) Routine Analysis. All others Analysed by request only.

IN 169

MONSANTO CHEMICAL COMPANY

AGRICULTURAL CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

RAW MATERIAL	
Acetone	
FOR USE IN	MATERIAL NO.
GRADE	
Anhydrous	

DATE EFFECTIVE	ISSUE NO.	SUPERSEDES
12/18/64	1	All other

SUPPLIERS

Monsanto, Chocolate Bayou, Tennessee Eastman, and Carbide & Carbon

APPROVALS

PRODUCTION (Plant Manager)	RESEARCH (Asst. Director)
	/s/ J. A. Stephens
PROCUREMENT (Procurement Manager)	QUALITY CONTROL (Chief Chemist)

AUTHORIZED (Quality Control Director)

/s/ CHR 12/18/64

GENERAL DESCRIPTION

A clear water white liquid

CHARACTERISTICS	LIMITS	METHOD NO.
Appearance	Clear, water white	CB-393
Water solubility	No turbidity when diluted with water	CB-393
Sp. Gr. @ 20/20° C.	0.791-0.794	CB-405
Dist. range @ 760 min.	1.0° C. to include 56.1° C.	CB-406
Non volatile matter	0.002 gm/100 ml max.	-407
Color	5 max. APHA	CB-395
Acidity as acetic acid	0.002% max.	CB-408
Alkalinity as NH_3	0.0005% max.	CB-409
Permanganate test	$KMNO_4$ color retention for 30 minutes minimum	CB-235
Moisture	0.5% max.	CB-410
Foreign odor	None	CB-394

Note: Chocolate Bayou and other suppliers test each shipment on the above. Anniston normally makes no tests.

NEW MATERIAL SPECIFICATION

MATERIAL: Ferric Chloride Anhydride

PLANT: WGK

USING DEPTS.: 233

DATE EFFECTIVE: JAN 7 1963

SUPPLIER: McKesson & Robbins

APPROVALS

PRODUCTION: T. W. Dalton	RESEARCH: M. A. Terpstra
PROCUREMENT: C. A. Pratte	QUALITY CONTROL (Chief Chemist): W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

$FeCl_3$

mol wt. 162.21

SUPERSEDES SPEC'S OF	SAMPLE FOR ANALYSIS	ISSUED BY
11/15/62	1 x 8 oz. W.M. Bottle	J. P. Prader

UNRESTRICTED INFORMATION

CHARACTERISTIC	LIMITS	METHOD (J.F.Q.)
Appearance	Black powder or Granules	16-A
Assay	97.0%, min.	22-570
Sulfates	0.05%, max.	120-570
Ferrous Salts	Trace, max.	S-570-1
Solubility:		112-B
10:10 of Water	Complete to slightly turbid, max.	
10:10 of Formula 30 Alcohol	Complete to slightly turbid, max.	

MONS 095283

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
826.38	Chlorine Gas
PLANT	USING DEPTS.
Anniston	04, 05, 27
DATE EFFECTIVE	SUPPLIER
8-31-66	See below

APPROVALS

PRODUCTION	RESEARCH
R. G. Moody /s/R. G. Moody - 8/11/66	L. R. Stark /s/L. R. Stark - 8/29/66
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
H. L. Phillips /s/H. L. Phillips - 8/15/66	O. E. Dolin /s/O. E. Dolin - 8/10/66

GENERAL DESCRIPTION

Greenish yellow gas

CHEMICAL FORMULA

Cl_2
Mol. wt. 70.91

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
Sept. 11, 1957	GLC sampling container	J. A. Liddle - 5/16/66

CHARACTERISTIC	LIMITS	Typical Values	METHOD
UNRESTRICTED INFORMATION			
Material Supplied by Diamond Alkali Company			
Assay, (Vol. %)	99.4 Min.		113
Residual Gases (Vol. %)	0.5 Max.		113
Moisture, ppm	40 Max.		
Non-volatile Matter, ppm	40 Max.		
Material Supplied by Depts. 56, 57 (Anniston)			
Assay (Vol. %)		85.5 - 93.5	113
H_2 (Vol. %)		1.5 - 2.5 (3 Max.)	113
CO_2 (Vol. %)		0.5 - 1.0	113
CO (Vol. %)		Nil	113
N_2 (Vol. %)		3.0 - 8.0	113
O_2 (Vol. %)		1.2 - 3.0	113

MONS 095284

(R) Routine Analysis, All others Analyzed by request only.

WBR-31d

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERIAL
38690	Lime Hydrate
PLANT	USING DEPTS.
Anniston	27, 28
DATE EFFECTIVE	SUPPLIER
7-20-66	Alabaster Lime Co.

APPROVALS

PRODUCTION	RESEARCH
R. G. Moody /s/R. G. Moody 6/9/66	N. F. Mueller /s/N. F. Mueller 7/13/66
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
H. L. Phillips /s/H. L. Phillips 6/10/66	O. E. Dolin /s/O. E. Dolin 6/8/66

GENERAL DESCRIPTION

White fine powder

CHEMICAL FORMULA

$Ca(OH)_2$

Mol. wt. = 74.10

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
Sept. 11, 1957	2 - 1 lb. cans	J. A. Liddle - 5/18/66

CHARACTERISTIC	LIMITS	METHOD
	Typical Analysis	
UNRESTRICTED INFORMATION		
CaO (%)	73.00	
MgO (%)	1.20	
SiO_2 (%)	0.36	
Al_2O_3 (%)	0.60	
Fe_2O_3 (%)	0.30	
Ignition Loss (%)	24.04	
$Ca(OH)_2$ (% Calculated)	97.50(1)	
Screen Analysis		
Through 200 mesh (%)	99.8	120
Through 325 mesh (%)	94.5	

Basis of Specifications: Suppliers typical analysis of product (5/23/66)

(1) $Ca(OH)_2$ % = 100% - [MgO(%) + SiO_2 (%) + Al_2O_3 (%) + Fe_2O_3 (%)]

MONS 095285

(R) Routine Analysis. All others Analysed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERIAL
19400	Attapulgus Earth - RVM Grade
PLANT	USING DEPTS.
Anniston	27-L
DATE EFFECTIVE	SUPPLIER
7-20-66	Minerals & Chemicals Philipp Corp.

APPROVALS

PRODUCTION	RESEARCH
R. G. Moody /s/R. G. Moody 6/9/66	N. F. Mueller /s/N. F. Mueller 7/13/66
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
H. L. Phillips /s/H. L. Phillips 6/10/66	O. E. Dolin /s/O. E. Dolin 6/8/66

GENERAL DESCRIPTION

Impure Hydrous Magnesium Aluminus Silicate

CHEMICAL FORMULA

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
Sept. 9, 1957	1 lb. can	H. A. Liddle - 5/25/66

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
Screen Analysis Through 200 Mesh (%)	95 Min.	
Loss on Ignition (% @ 950°C)	15 Max.	
Typical Analysis from Supplier (May 23, 1966)		
SiO_2 (%)	67.5	
Al_2O_3 (%)	12.8	
Fe_2O_3 (%)	4.5	
MgO (%)	11.1	
CaO (%)	1.6	
K_2O (%)	0.8	
Na_2O (%)	0.1	
TiO_2 (%)	0.6	
Other (%)	1.0	

MONS 095286

(R) Routine Analysis, All others Analyzed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
60200	Trichlorobenzene
PLANT	USING DEPTS.
WGK	246
DATE EFFECTIVE	SUPPLIER
JUN 3 1965	Hooker

APPROVALS

PRODUCTION	RESEARCH
P. E. Heisler	R. W. Bucknell
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
G. C. Kamenko	W. J. Gresham

GENERAL DESCRIPTION: TCB

CHEMICAL FORMULA:

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
8-29-63	3 x 5 pt amber bottles	M. D. Dealy

	CHARACTERISTIC	LIMITS	METHOD
	UNRESTRICTED INFORMATION		
	Color	APHA 15, max.	10105
(R)	Condition	Clear	10105
(R)	Sp. Gr. @15.5/15.5°C	1.460 - 1.477	10114
	Acidity (mg. KOH/g.)	0.014, max.	10109
	Moisture (H_2O)	75 ppm., max.	10620
(R)	Refractive Index @25°C	1.5680 - 1.5715	10125
	Free Chlorides	0.10 ppm., max.	10118
	Crystallizing Point	10°C, max.	10107
	Dielectric Strength @25°C	30 KV, min.	11605
	Distillation Range (ASTM D-850, Modified) (corrected for Stem and Barometric Pressure)		10108
	First Drop	205°C, min.	
	5% by volume	210°C, min.	
	50% by volume	213°C, min.	
	90% by volume	215°C, min.	
	Corrosion Test, 6 hrs at 210°C with bright aluminum foil		10126
	Change in weight of Al	0.0%	
	Color	APHA 200, max.	
	Condition	Clear	
	Acidity (mg. KOH/g.)	0.014, max.	
	Free Chlorides	0.10 ppm., max.	
	Monsanto Stability Test	0.5 ppm., max.*	11503
	Hydrolysis Stability Test	0.5 ppm., max.	12672
	Fire Point	None to boiling point	10123
	Fixed Chlorine	58.5%, min.	10088

*Every other car can be over 0.5 ppm but never over 1.0 ppm. Values over 0.5 ppm to be cleared in advance.

Certificate of Analysis required on the above properties - To be mailed to the Chief Chemist, W. G. Krummrich Plant the same day shipment is made.

MONS 095287

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL (GLYCIDAL PHENOL ETHER)
36100	Phenoxypropene Oxide
PLANT	USING DEPTS.
Anniston	28
DATE EFFECTIVE	SUPPLIER
8-28-67	Shell Chemical Co.

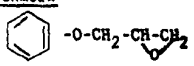
APPROVALS

PRODUCTION	RESEARCH
H. L. Williams /s/ H. L. Williams	L. R. Stark /s/ L. R. Stark
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
H. L. Phillips /s/ H. L. Phillips	G. W. Miller /s/ G. W. Miller

GENERAL DESCRIPTION

$C_9H_{10}O_2$

CHEMICAL FORMULA



Mol. wt. 150.17

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
New	1 x 16 oz. W.M. bottle	J. A. Liddle 8-14-67

CHARACTERISTIC	LIMITS	METHOD
UNCLASSIFIED INFORMATION		
(R) Specific Gravity @ 20/20°C	1.105 - 1.115	052
(R) Total Chlorine	0.2% Max.	
(R) Inorganic (free) Chlorides, ppm	40 Max.	014
Purity (based on Epoxide content percent by weight)	98%	
Color, APHA	30 Max.	
Impurity A (as determined by Anniston GLC method)	0.05 Max.	
General Information (from supplier's technical bulletin)		
Boiling Point @ 760 mm	245°C	
Melting Point	3.5°C	
Flash Point	175°F	
Refractive Index @ 20°C	1.5314	
Viscosity @ 20°C	7.05 CS	
@ 30°C	4.88 CS	

MONS 095288

(R) Routine Analysis. All others Analyzed by request only.

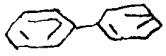
IN 84 MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT CODE: 826.01
METHOD IDENTITY:
SALES CODE: 135-000-03-09

Issued 5/5/61
Anniston Plant
By W. W. Klemmer
Revised to add k values 11-18-65

PRODUCT (Trade name): Biphenyl — TYPE: Technical
PRODUCT (Chemical name): Biphenyl — TYPE: Technical
CHEMICAL FORMULA



$C_{12}H_{10}$

MOL WT.: 154.20
USE: Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
7/8/65		1 x 16 oz. can

Characteristics	Limits	Typical Values
Routine tests		
Appearance	White to pale yellow crystals	White crystals
Crystallizing Point, °C	68.7 Min.	68.8
Color, Gardner	6 Max.	1.5
General Test (determined only on request)		
Biphenyl, %		99.9
Napthalene, %		0.001
3 - Methylbiphenyl, %		0.05
4 - Methylbiphenyl, %		0.02
Unknown, %		<0.03
Distillation Range, °C 1st drop - Dry Point	Must include 255°C	

This specification for internal use only

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION

W.G.K. and Anniston
FINISHED PRODUCT
SPECIFICATION

PRODUCT CODE: 81102 | DEPT: 243 WGK 27 Ann.
USE: Sales
SALES CODE: 1040-260-04/11/16-03/09

Issued 1/10/64
W. G. Krumbhaar Plt
By M. D. Deck

PRODUCT (Trade Name) TYPE
Aroclor 1248 Electrical Grade (Pyranol 1498)

PRODUCT (Chemical Name) TYPE

Corrected 2-16-64

CHEMICAL FORMULA

Mixture

MOL WT.

SUPERSEDES SPECS OF: 2-12-60

SAMPLE FOR ANALYSIS: 3 x 5 pt. Amber Bottles

UNRESTRICTED INFORMATION

Routine Tests	Specification	Method No' WGK	Annist
Color, APHA	50, max.	10,084	14-44-
Condition	Clear	10,084	G-1
Sp. Gr. @65/15.5°C	1.405 - 1.415	10,114	14-49-
Acidity (mg. KOH/gm.)	0.010, max.	10,087	14-42-
Moisture	35 ppm., max.	10,620	14-53-
Refractive Index @25°C	1.6285 - 1.6305	10,125	14-34-
Inorganics (Free) Chlorides	0.05 ppm., max.	T-2092	014
Pour Point	0°C, max.	10,115	14-27-5
Dielectric Constant (1000 cycles @100°C)	4.5 - 4.7	11,608	14-36-5
Resistivity (500 VDC @100°C 0.1" GAP) as OHM-CM x 10^9	500, min.	11,607	14-35-5
Hydrolysis Stability Test (as Chlorides)	1.0 ppm., max.	T-2087	010
Monsanto Stability Test (as Chlorides)	0.5 ppm., max.	11,503	14-61-57
Distillation Range (ASTM D-20, Corrected)		10,117	14-31-54
10% Distilled by Wt.	345°C, min.		
90% Distilled by Wt.	385°C, max,		

General Tests (made only on request)

Sulfates	None	12,169	- - -
Dielectric Strength @25°C	35 KV, min.	11,605	043
Fire Point	None to boiling point	10,123	14-23-54
Viscosity @54.4°C (SUS)	73 - 80	10,086	14-4-52
Corrosion Test (6 hrs. @210°C with bright aluminum foil)		10,126	14-55-57
Change in wt. of Al (%)	0.0		

(over)

MONS 095290

A. C. Color, APHA	60, max.	10,084 14-44-54
A. C. Condition	Clear	10,084 G-1
A. C. Acidity (mg. KOH/g.)	0.010, max.	10,087 14-42-54
A. C. Inorganic (Free) Chlorides	0.05 ppm., max.	10,118 010

GENERAL INFORMATION

Specific Heat @25°C	0.27
Fixed Chlorine	47.5 - 48.5%

Monsanto
ORGANIC CHEMICALS DIVISION

ANNISTON AND KRUMMRICH
FINISHED PRODUCT
SPECIFICATION

PRODUCT: Aroclor 1260 Electrical Grade			DEPT. Ann WGK -
PLANT: Anniston Krummrich	PRODUCT CODE: 82614 - Ann. 81105 - WGK		USE: Sales
DATE EFFECTIVE: 5/25/66	SALES CODE: 1040-290-04/11/16-03-09		

APPROVALS

PRODUCTION: P. E. Heisler	RESEARCH: W. D. Robinson
SALES: W. E. Ault	QUALITY CONTROL (Chief Chemist): O. E. Dolin W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

MOL WT.

SUPERSEDES SPECS. OF: 2/7/62	SAMPLE FOR ANALYSIS: 3 x 5 pt. Amber Bottles	ISSUED BY: J. P. Prader

CHARACTERISTIC		LIMITS	WGK.	Anniston
	UNRESTRICTED INFORMATION			
(R)	Color (APHA)	150, max.	10084	14-44-54
(R)	Condition	Clear	10084	--------
(R)	Specific Gravity @ 90.15.5°C.	1.555 - 1.566	10114	14-49-54
(R)	Acidity (mg KOH/g.)	0.014, max.	10087	14-42-54
(R)	Moisture (H_2O) (ppm)	35, max.	10620	14-53-54
(R)	Viscosity @ 210°F. (SUS)	72 - 78	10086	14- 4-52
(R)	Refractive Index @ 25°C.	1.6455 - 1.6470	10125	14-34-54
(R)	Inorganic (Free) Chlorides	0.05 ppm, max.	10118	14-48-57
(R)	Pour Point (°C)	25 - 34	10115	14-47-54
(R)	Distillation Range (ASTM D-20, Corrected)		10117	14-31-54
	10% Distilled by wt.	385 - 398°C.		
	50% Distilled by wt.	390 - 404°C.		
	90% Distilled by wt.	400 - 420°C.		
(R)	Corrosion Test (6 hrs. @ 210°C. with bright aluminum foil)		10126	14-55-57
(R)	Change in weight of Al (@)	0.0		
(R)	A.C. Color (APHA)	150, max.	10084	14-44-54
(R)	A.C. Condition	Clear	10084	--------
(R)	A.C. Acidity (mg. KOH/g.)	0.014, max.	10087	14-42-54
(R)	A.C. Inorganic (Free) Chlorides	0.05 ppm, max.	10118	14-48-57

(Over)

UNRESTRICTED INFORMATION (Cont'd.)

Characteristic	Limits	Method	
		WGK	Anniston
Flash Point	None	10123	14-28-54
Fire Point	None	10123	14-28-54
Fixed Chlorine (%)	59.5 - 60.5	10088	14-13-54
(R) Dielectric Strength @ 50°C.	30 K.V., min.	11605	---------
(R) Resistivity (500 VDC @ 100°C. 0.1" gap as ohm-cm x 10^9)	500, min.	11607	14-35-57
(R) Dielectric Constant 1000 cycles, 100°C.	3.6 - 3.8	11607	14-36-45

RESTRICTED INFORMATION

(R) Monsanto Stability Test (As Chlorides in ppm.)	0.7, max.	11503	14-61-57

GENERAL INFORMATION

(R) Evaporation Loss after 6 hrs. @ 100°C. (%)	0.2, max.	10116	12-32-54
(R) Stability test - after 30 days at 100°C. in glass sealed in air.	No liberation of chlorine or chlorides	------	--------

Monsanto
ORGANIC CHEMICALS DIVISION
ANNISTON, ALA. AND KRUMMRICH

FINISHED PRODUCT SPECIFICATION

PRODUCT		DEPT.
Aroclor 1242, Electrical Grade		246 27 1
PLANT	PRODUCT CODE	USE
WGK, Ann.	81101 - 82612	Sales
DATE EFFECTIVE	SALES CODE	
6/17/66	1040-240-04/11/16-03/09	

APPROVALS

PRODUCTION: P. E. Heisler	RESEARCH: R. W. Bucknell
SALES: W. E. Ault	QUALITY CONTROL (Chief Chemist): O. E. Dolin / W. J. Gresham

GENERAL DESCRIPTION — CHEMICAL FORMULA

Also known as Pyranol 1499

MOL WT.

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
4-13-62	3 x 5 pt. Amber Bottles	J. P. Prader

CHARACTERISTIC	LIMITS	METHOD: WGK	METHOD: Anniston
UNRESTRICTED INFORMATION			
(R) Color (APHA)	40, max.	10084	14-44-54
(R) Condition	Clear	10084	G-1
(R) Sp. Gr. @ 25/15.5°C.	1.381 - 1.392	10114	14-49-54
(R) Acidity (mg. KOH/g.)	0.010, max.	10087	14-42-54
(R) Moisture (H_2O)	35 ppm., max.	10620	14-53-54
(R) Refractive Index @25°C.	1.6240 - 1.6260	10125	14-34-54
(R) Inorganic (Free) Chlorides	0.05 ppm., max.	12741	014
(R) Pour Point	-14°C. or Lower	10115	14-47-54
(R) Dielectric Constant (1000 cycles @ 100°C.)	4.70 - 4.90	11608	14-36-54
(R) Resistivity (500 VDC @ 100°C. @ 1" Gap) As OHM-CM x 10^9	500, min.	11607	14-35-57
(R) Hydrolysis Stability Test (As Chlorides)	0.5 ppm, max.	12672	010
(R) Monsanto Stability Test (As Chlorides)	0.4 ppm., max.	11503	14-61-57
(R) Distillation Range (ASTM D-20, Corrected)		10117	14-31-54
10% Distilled by wt.	325°C., min.		
90% Distilled by wt.	360°C., max.		
Sulfates	None	12169	------
Dielectric Strength @ 25°C.	35 KV, min.	11605	043
Flash Point (°F)	338 - 392	10123	14-28-54
Fire Point	None	10123	14-28-54
Corrosion Test (6 hrs. @ 210°C. with bright aluminum foil)		10126	14-55-57
Change in wt. of AL %	0.0		
A.C. Color (APHA)	60, max.	10084	14-44-54
A.C. Condition	Clear	10084	G-1
A.C. Acidity (mg. KOH/g.)	0.010, max.	10087	14-42-54
A.C. Inorganic (Free) Chlorides	0.05 ppm., max.	12741	014
Visc. @ 100°F. (SUS)	82 - 92	10086	14- 4-52

(Over)

MONS 095294

(R) Routine Analysis - All others Analyzed by request Only

Monsanto
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT		DEPT.
Aroclor 1254 Electrical Grade		246 - 2
PLANT	PRODUCT CODE	USE
WGK and Ann.	81103 82613	Sales
DATE EFFECTIVE	SALES CODE	
JUN 28 1965	1040-280-04/11/16-03/09	

APPROVALS

PRODUCTION	RESEARCH
P. E. Heisler	R. W. Bucknell
SALES	QUALITY CONTROL (Chief Chemist)
W. E. Ault	W. J. Gresham, O. E. Dolin

GENERAL DESCRIPTION — CHEMICAL FORMULA

Also called Pyranol 1476

MOL WT.

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
4-13-62	3 x 5 pt. Amber Bottles	M. D. Dealy

UNRESTRICTED INFORMATION

	CHARACTERISTIC	LIMITS	WGK METHOD	Ann. METHOD
(R)	Color, APHA	50, max.	10084	14-44-54
(R)	Condition	Clear	10084	G-1
(R)	Sp. Gr. @65/15.5°C	1.495 - 1.505	10114	14-49-54
(R)	Acidity (mg. KOH/gm.)	0.010, max.	10087	14-42-54
(R)	Moisture (H_2O)	35 ppm, max.	10620	14-53-54
(R)	Refractive Index @25°C	1.6370 - 1.6390	10125	14-34-54
(R)	Inorganic (Free) Chlorides	0.05 ppm., max.	T-2092	014
(R)	Pour Point	7 - 12°C	10115	14-27-54
(R)	Dielectric Constant (1000 cycles @100°C)	4.15 - 435	11608	14-36-54
(R)	Resistivity (500 VDC @100°C 0.1" GAP) as OHM-CM x 10⁹	500, min.	11607	14-35-57
(R)	Hydrolysis Stability Test (as Chlorides)	1.0 ppm., max.	T-2087	010
(R)	Monsanto Stability Test (as Chlorides)	0.5 ppm., max.	11503	14-61-57
(R)	Distillation Range (ASTM D-20, Corrected)		10117	14-31-54
	10% Distilled by Wt.	366 - 378°C		
	50% Distilled by Wt.	371 - 383°C		
	90% Distilled by Wt.	379 - 394°C		
	Sulfates	None	12169	
	Dielectric Strength @25°C	35KV, min.	11605	043
	Flash Point	None	10123	14-28-54
	Fire Point	None	10123	14-28-54
	Visc. @210°F (SUS)	44-48	10086	14-4-52
	Corrosion Test (6 hrs @210°C with bright aluminum foil)		10126	14-55-57
	Change in wt. of Al (%)	0.0		
	A. C. Color, APHA	60, max.	10084	14-44-54
	A. C. Condition	Clear	10084	G-1
	A. C. Acidity (mg.KOH/g.)	0.010, max.	10087	14-42-54
	A. C. Inorganic (Free) Chlorides	0.05 ppm., max.	10118	010

MONS 095295

(R) Routine Analysis. All others Analyzed by request Only

Monsanto
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT		DEPT.
Inerteen 54201-KA		28
PLANT	PRODUCT CODE	USE
Anniston	826.81	Sales
DATE EFFECTIVE	SALES CODE	
5-3-67	1050-210-16-009	

APPROVALS

PRODUCTION	RESEARCH
R. G. Moody /s/ R. G. Moody 2/24/67	L. R. Stark /s/ L. R. Stark 5/1/67
SALES	QUALITY CONTROL (Chief Chemist)
W. E. Ault /s/ W. E. Ault 4/27/67	G. W. Miller /s/ G. W. Miller 2/21/67

GENERAL DESCRIPTION

Mixture - 69.9% Aroclor 1254
29.9% Trichlorobenzene
0.2% Phenoxypropene Oxide

CHEMICAL FORMULA

MOL WT.

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
Tentative 7-29-65	3 x 5 pt. Amber bottle	J. A. Liddle 2/21/67

UNRESTRICTED INFORMATION

CHARACTERISTIC	LIMITS	METHOD
(R) Color, APHA	50 Max.	046
(R) Condition	Clear	G-1
(R) Specific Gravity @ 15.5/15.5°C	1.518 - 1.528	052
(R) Acidity, Mg KOH/gm	0.014 Max.	050
(R) Inorganic Chloride, ppm	0.05 Max.	045
(R) Viscosity @ 100°F, SUS	58 - 62	049
(R) Resistivity @ 100°C, ohm-cm	500 x 10^9 Min.	121
(R) Dielectric Strength @ 25°C, KV	35 Min.	043
(R) Refractive Index @ 25°C	1.6153 - 1.6173	048
(R) Power Factor @ 100°C, 1 KC, %	0.50 Max.	121
@ 100°C, 60 cycles, %	5.0 Max.	
(R) Distillation		
1st Drop	200°C Min.	
Below 270°C	35% Max.	
90% by wt.	379 - 394°C	
(R) Pour Point	-30°C Max.	053
(R) Moisture (H_2O) ppm	30 Max.	047
(R) Fire Point	None to Boiling	114
(R) Phenoxypropene Oxide, %	0.18 - 22	11865 (WGK)
(R) Corrosion Test - 6 hrs. @ 210°C with bright aluminum foil change in weight of aluminum	0.0%	136
(R) After Corrosion Test		
Color, APHA	200 Max.	046
Condition	Clear	G-1
Acidity, Mg KOH/gm	0.014 Max.	050
Inorganic Chloride, ppm	2.0 Max.	014

MONS 095296

(R) Routine Analysis, All others Analyzed by request Only

Monsanto
ORGANIC CHEMICALS DIVISION
Anniston & Krummrich
FINISHED PRODUCT SPECIFICATION

PRODUCT: Transformer Inerteen PPO		DEPT. 246
PLANT: WGK Anniston	PRODUCT CODE: 81092 82649	USE: SALES
DATE EFFECTIVE: 4/18/66	SALES CODE: 1050-200-16-03/09	

APPROVALS

PRODUCTION: P. E. Heisler	RESEARCH: M. E. Gibbs
SALES: W. E. Ault	QUALITY CONTROL (Chief Chemist): W. J. Gresham — O. E. Dolin

GENERAL DESCRIPTION: Mixture - 60% Aroclor 1260: 40% Trichlorobenzene
Plus 0.20% Phenoxypropene Oxide
(Westinghouse Inerteen 7336-9)

CHEMICAL FORMULA:

MOL WT.

SUPERSEDES SPECS. OF: 6/23/65	SAMPLE FOR ANALYSIS: 3 x 5 pt. Amber Bottles	ISSUED BY: J. P. Prader

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
(R) Color	APHA 150, max.	10,084
(R) Condition	Clear	10,084
(R) Sp. Gr. @ 15.5/15.5°C.	1.560 - 1.568	10,114
(R) Acidity (mg. KOH/g.)	0.014, max.	10,087
(R) Moisture (H_2O)	30 ppm, max.	10,620
(R) Refractive Index @ 25°C.	1.6137 - 1.6147	10,125
(R) Inorganic (Free) Chlorides	0.10 ppm., max.	10,118
(R) Dielectric Strength @ 25°C.	35 KV, min.	11,605
(R) Resistivity @ 100°C. 500 VDC and 0.1" gap	100×10^9 ohm/cm, min.	11,607
(R) Viscosity @ 100°F.	52 - 56 SUS	10,086
(R) Pour Point	-32°C., max.	10,115
(R) Distillation Range: (ASTM D-20, Corrected)		10,117
First Drop	200°C., min.	
Below 270°C., by wt.	40%, max.	
90% by wt.	395 - 415°C.	
(R) Corrosion Test, 6 hrs. @ 210°C. with bright Aluminum Foil		10,126
(R) Change in weight of Al.	0.0%	
AC Color	APHA 200, max.	10,084
AC Condition	Clear	10,084
AC Acidity (mg. KOH/g.)	0.014, max.	10,087
AC Inorganic (Free) Chlorides	2.0 ppm, max.	10,118
(R) Phenoxypropene Oxide	0.18 - 0.22%	11,865
(R) Fire Point	None to Boiling Point	10,123
(R) Dielectric Constant 1000 cycles, 100°C.	3.7 - 4.0	10,608
Fixed Chlorine	59.1%, min.	10,088
Arc Formed Combustible Gases	1.0%, max.	10,711
Electrical Stability (aged 96 hrs. @ 100°C, covered jar, 15% air)		10,121
Decrease in Resistivity	10%, max.	

MONS 095297

RESTRICTED INFORMATION			
(R)	Power Factor @ 60 cycles and 100°C.	5.0%, max. (over)	11,608

(R) Routine

GENERAL INFORMATION

Dielectric Constant 1000 cycles, 25°C.	4.0 - 4.3
Analysis of each 10 ml. fraction of a fractional distillation Chlorine/Hydrogen Ratio	1, min.
Arc Formed Combustible Gases	1.5%, max.

MONS 095298

Monsanto

FROM (NAME & LOCATION) General Offices - St. Louis

March 18, 1968

SUBJECT Project Loss Prevention Report 68-13

REFERENCE CEA 1668

TO : A. J. Heinze
R. S. Yates

cc R. E. Howard
Y. Terajima
S&PP Project File
S&PP Location File

COPIES TO THOSE PRESENT

R. J. Green
A. J. Heinze
W. B. Howard
R. M. Kountz
R. S. Yates

DATE AND PLACE OF MEETING March 14 and 15, 1968, Creve Coeur, Missouri.

CEA 1668 Biphenyl and Aroclor, MMK Plant, Yakkaichi, Japan.

This project is for design of a plant to make 5M ppy biphenyl and 10M ppy Aroclors. The process design package is being developed by CED; mechanical design, purchasing, and construction will be handled by MMK. Currently it is planned to build the Aroclors unit first and to build the biphenyl plant some 18 months later. The capital for the total project is approximately $1.7M.

PERSONNEL

1. Access to Benzene Distillation Structure The present plan is to provide access to the benzene distillation structure's two levels by means of a single ladder to each level. These are not normally classed as operating levels. It was recommended that the project manager check this planning with Mr. George Gorbell. The same recommendation also applies to the access to the platform for the biphenyl distillation area.

2. Personnel Protection at Furnace Preheater and Retention Tank

It is recommended that personnel protection, probably railing, be installed at both ends of the furnace preheater and also at the retention tank.

PROCESS

1. Control House Location — The plant layout drawing was actually a study drawing rather than a final layout design. However, it is recommended that, on even the study drawing, the control house not be shown enclosed in a U formed by operating units and tank storage.

2. Protection of Oil-Fired Pyrolysis Furnace — The pyrolysis furnace will be protected in the same manner as will be that planned for the Newport biphenyl project.

3. Inert-Gas Purge — Carbon dioxide will be provided at the unit. It is recommended that a provision be made in the operating procedure for inert-gas purge of equipment, including the fractionating column, before start-up in order to avoid a benzene-air interface.

4. Overpressure Protection of Fuel Oil Pumps — It was agreed that PVC 104 will be moved to just downstream of the fuel oil pumps (positive displacement) to protect the pump as well as the other equipment protected by the PVC.

5. Protection on Chlorinator — The chlorination of biphenyl is highly exothermic. Dual alarms will be provided by two TRA's on the chlorinator.

6. Protection Against Chlorine-Hydrocarbons Explosion — The literature on the process indicates that biphenyl can burn in chlorine. The flash point of biphenyl in chlorine is not known at present. It is recommended that the flash point of biphenyl be determined in chlorine in order to determine whether protection is needed to prevent the addition of chlorine to the chlorinator in the absence of ferric chloride catalyst.

7. Aroclor Furnace Controls — It is recommended that the Aroclor furnace controls (for oil firing) be checked with Mr. George Ostroot.

8. Temperature Alarm on Process Stream Out of Aroclor Furnace — It was agreed that a temperature alarm needs to be installed in the discharge line from the furnace. It appeared that this alarm can be added to the temperature recorder already provided.

FIRE PROTECTION — The following fire protection recommendations are the same as those made for Biphenyl and Aroclor plants in the United States:

1. Provide automatic water spray protection throughout the Benzene Stripping Column structure and over the equipment containing benzene in the Benzene Recovery Area. These systems should be hydraulically designed for a density of 0.35 gpm per sq. ft.

2. Connect the proposed automatic water spray systems to the existing water supplies and provided post indicator type control valve located 40 ft. from the area being protected.

3. Provide sufficient two-way hydrants to allow available hose streams at any point in the area where fire may occur. Extend the existing mains to supply water to the proposed hydrants.

4. Fireproof the skirt of Benzene Column #1-111 for a two-hour fire rating.

5. Provide a dike around Benzene Tank #1-101 and Acetone Tank #1-103 with a capacity equal to the larger tank.

6. Provide an ordinary hazard, dry pipe, automatic sprinkler system in the Warehouse and Drumming Building.

   It is understood that drumming of Aroclors in metal drums and storage of Aroclors in metal drums on wood pallets (3 high) will be done in this building. The principal reason for automatic sprinklers is to protect against a fire in the wood pallets that could expose the drummed Aroclors.

LOSS EXPOSURE The probable maximum loss was estimated to occur from the explosion of the biphenyl chlorinator. The cost for replacement of the chlorinator and repair of auxiliary equipment was estimated to be $20M. The downtime for replacement was estimated to be one month. On this basis the business interruption loss would be approximately $100M.

It is noted that this will also be the probable maximum loss after the biphenyl unit goes into operation. That is, the probable maximum loss for any reasonably foreseeable incident in the biphenyl unit will be less than the losses outlined just above.

LOSS PREVENTION REVIEW BY PLANT The S&PP Branch of CED does not plan a complete review of design prints for safety provisions. S&PP requests plant personnel, including plant safety personnel, to follow the development of design prints and to check th progress of construction with particular emphasis on personnel safety and loss prevention.

LOSS PREVENTION GUIDE Attached to A. J. Heinze and R. S. Yates' copy of this report is a copy of Form G-1425, Loss Prevention Review Guide, which can be used as a safety check list for this project.

SAFETY MANAGER R. J. Green will be Safety Manager for this project. Please contact him for any subsequent safety meetings that are needed.

R. J. Green

R. J. Green

W. B. Howard

W. B. Howard

bjr
Attachment

Central Engineering Department September 15, 1965

LOSS PREVENTION REVIEW GUIDES

Safety and Property Protection

MONSANTO

MONS 095303

Reference: CED Procedure 602 Loss Prevention Review

Form G 1425 Copies available from Office Supplies Dept.

SECTION I - LOSS PREVENTION REVIEW GUIDE

PERSONNEL SAFETY REVIEW

A. Project Site Location

1. What consideration has been given to exposure to or from the neighborhood from fire, explosion, noise, air and stream pollution, and exposure to or from existing operations?

2. How many means of access are provided for the site to facilitate entrance by emergency vehicles?

3. What access roads to the site are exposed to railroad blockage, or highway congestion and other hazards? What are plans to minimize and control these hazards?

4. What sharp curves exist in access roads? How much are curves banked? Has the need for barricades been considered?

B. Building and Structures

1. What standards are being followed in the design of stairways, platforms, ramps and fixed ladders?

2. Are sufficient general exit facilities and escape routes available? Will alternate means of escape from roofs be provided?

3. What lighting is provided?

4. How are doors and windows hung to avoid projecting into or blocking walkways and exits?

5. How is the structural steel grounded?

MONS 095304

C. Operating Areas

1. How are equipment, steam, water, air and electric outlets arranged to keep aisles and operating floor areas clear of hoses and cables?

2. What consideration was given to ventilation of hazardous fumes, vapors, dust and excessive heat?

3. What space is provided for the temporary storage of raw materials at process points, and temporary space for finished products?

4. Where is laminated safety glass used for windows of control rooms exposed to potential explosion hazards? What alternate escape routes to safe locations are provided?

5. What arrangements are planned for pressure relief venting of area, if applicable?

6. How much clearance and what platforms are provided around equipment for conducting maintenance operations safely?

7. How are nozzles and manholes sized and located with consideration for safe clean out, maintenance operations and emergency removal of people from vessels?

8. How is protection provided against contact with hot surfaces?

9. What is the head clearance in walkway and working areas?

10. What consideration is being given to the proper guarding of exposed moving parts of power-driven equipment before operation start-up? What are the specifications for guards? Where are guards located? What type of guards are they?

11. What provision is made for interlocking covers and guards with operating mechanism?

12. Are manually-operated valves, switches and other controls readily accessible to the operator from a safe location?

13. Where do vents discharge? What potential dangers of injury to personnel or damage to property do they present?

14. Where are hoists being installed? What type? What safety features are incorporated in the design?

15. How are elevator shaftway door interlocks and gate contacts designed? How will bi-parting doors be operated? What provision has been made for safety astragals?

16. How much material handling is to be done by mechanical means and how much by manual handling?

17. Where are Monsanto standard emergency showers and eye baths provided?

18. What provision has been made for a safe storage and dispensing location for flammable liquid drums?

19. What platforms and working areas are there where the operation is of a nature which requires at least two exits for speedy exit from the area in an emergency?

20. Where excessively noisy operations are concerned, what measures are contemplated to reduce the noise level to a safe range?

21. How are supervisors' offices located relative to the operations? What provision is made for emergency exits?

22. What positive electrical power disconnects are being installed for purposes of lockout?

Yard

1. How are roadways laid out with consideration for the safe movement of pedestrians, vehicles and emergency equipment?

2. Where railroad car pullers are to be installed, how are the control stations fully protected against broken cable whiplash? How will operator be protected from being caught between cable or rope and capstan or cable drum?

3. How are flammable liquid tank car and tank truck loading and unloading docks grounded?

4. What means are provided on loading platforms for access to work areas of tank cars and trucks?

5. What handrail protection is being provided?

6. How is safe access provided to tops of storage tanks on which persons go for contents measurement and vent maintenance?

7. Is there sufficient head clearance and good visibility in walking and working areas?

8. What yard lighting is provided?

SECTION II - LOSS PREVENTION REVIEW GUIDE

PROCESS SAFETY REVIEW CHECK LIST

Note: Consider the check list in terms not only of steady state operation, but also start-up, shut-down, and upsets of all conceivable types.

A. Materials

1. What process materials are unstable or spontaneously ignitable?

2. What data is available on amount and rate of heat evolution during decomposition of any material in the process?

3. What precautions are necessary for flammable materials?

4. What flammable dust hazards exist?

5. What materials are highly toxic?

6. What has been done to assure that materials of construction are compatible with the chemical process materials that are involved?

7. What maintenance control is necessary to assure replacement of proper materials, e.g., to avoid excessive corrosion, to avoid producing hazardous compounds with reactants?

8. What changes have occurred in composition of raw materials and what resulting changes in process?

9. What is done to assure sufficient control of raw material identification and quality?

10. What hazards can be created by failure of supply of one or more raw materials?

11. What assurance is there of adequate raw material supply?

12. What hazards can occur as a result of loss of gas for purging, blanketing, or inerting? How certain is gas supply?

13. What precautions need to be considered relative to stability of all materials in storage?

14. What fire extinguishing agents are compatible with process materials?

15. What fire emergency equipment and procedures are being provided?

B. Reactions

1. How are potentially hazardous reactions isolated?

2. What process variables could, or do, approach limiting conditions for hazard?

3. What unwanted hazardous reactions can be developed through unlikely flow or process conditions or through contamination?

MONS 095310

4. What combustible mixtures can occur within equipment?

5. What precautions are taken for processes operating near or inside the flammable limits?

6. What are process margins of safety for all reactants and intermediates?

7. What reaction rate data are available on the normal, or abnormally possible, reactions?

8. How much heat must be removed for normal, or abnormally possible, exothermic reactions?

9. How thoroughly is chemistry of the process known? (See NFPA "Manual of Hazardous Chemical Reactions")

10. What foreign materials can contaminate the process and create hazards?

11. What provision is made for rapid disposal of reactants if required by plant emergency?

12. What provisions are made for handling impending runaways and for short-stopping an existing runaway?

13. How fully is the chemistry of all desired and undesired reactions known?

14. What hazardous reactions could develop as a result of mechanical equipment (pump, agitator, etc.) failure?

15. What hazardous process conditions can result from gradual or sudden blockage in equipment?

16. What raw materials or process materials can be adversely affected by extreme weather conditions?

17. What process changes have been made since the previous process safety review?

C. Equipment

1. In view of process changes since the last process safety review, how was adequate size of equipment assured?

2. Are any venting systems manifolded and, if so, what hazards can result?

3. What procedure is there for assuring adequate liquid level in liquid seals?

4. What is the potential for external fire which may create hazardous internal process conditions?

5. Is explosion suppression equipment needed to stop an explosion once started?

6. Where are flame arresters and detonation arresters needed?

7. In confined areas, how is open fired equipment protected from spills.

8. What safety control is maintained over storage areas?

9. In the case of equipment made of glass or other fragile material, can a more durable material be used? If not, is the fragile material adequately protected to minimize breakage? What is the hazard resulting from breakage?

10. What emergency valves and switches cannot be reached readily?

11. When was pertinent equipment, especially process vessels, last checked for pressure rating?

12. What hazards are introduced by failure of agitators?

13. What plugging of lines can occur and what are the hazards?

14. What provisions are needed for complete drainage of equipment for safety in maintenance?

15. How was adequacy of ventilation determined?

16. What provisions have been made for dissipation of static electricity to avoid sparking?

17. What requirements are there for concrete bulkheads, or barricades to isolate highly sensitive equipment and protect adjacent areas from disruption of operations?

D. Instrumentation Control

1. What hazards will develop if all types of motive power used in instrumentation should fail nearly simultaneously?

2. If all instruments fail simultaneously, is the collective operation still fail-safe?

3. What provision is made for process safety when an instrument, instrumental in process safety as well as in process control, is taken out of service for maintenance? When such an instrument goes through a dead time period for standardization or when, for some other reason, the instrument reading is not available?

4. What has been done to minimize response time lag in instruments directly or indirectly significant to process safety?

5. Has the process safety function of instrumentation been considered integrally with the process control function throughout plant design?

6. What are effects of extremes of atmospheric humidity and temperature instrumentation?

7. What gages, meters, recorders cannot be read easily? What modifications are being made to cope with or solve this problem?

8. What is being done to verify that instrument packages are properly installed? Grounded? Proper design for the environment?

9. What procedures have been established to testing and proving instrument functions?

10. What periodic testing to check performance and potential malfunction is scheduled?

E. Operations

1. When was the written operating procedure last reviewed and revised?

2. How are new operating personnel trained on initial operation and experienced operating personnel kept up to date, on plant operating procedures, especially for start-up, shutdown, upsets and emergencies?

3. What plant revisions have been made since the last process safety review?

4. What special clean-up requirements are there before start-up and how are these checked?

5. What emergency valves and switches cannot be reached readily? What procedures are there to cope with these situations?

6. What safety precautions are needed in loading liquids into, or withdrawing them from, tanks? Has possibility of static electricity creation been adequately taken care of?

7. What process hazards are introduced by routine maintenance procedures?

8. What evaluation has been made of the hazards of sewered materials during normal and abnormal operation?

9. How dependable are supplies of inerting gas and how easily can supplies to individual units be interrupted?

10. What safety margins have been narrowed by revisions of design or construction in efforts to de-bottleneck operations, reduce cost, increase capacity, or improve quality?

11. What provisions does the operating manual have for coverage of start-up, shut-down, upsets, and emergencies?

12. What economic evaluation has dictated the choice on process between a batch process and a continuous one?

F. Malfunctions

1. What hazards are created by the loss of each feed, and by simultaneous loss of two or more feeds?

2. What hazards result from loss of each utility, and from simultaneous loss of two or more utilities?

3. What is the severest credible incident, i.e., the worst conceivable combination of reasonable malfunctions, which can occur?

4. What is the potential for spills and what hazards would result from them?

G. Location and Plot Plan

1. Has equipment been adequately spaced and located to permit anticipated maintenance during operation without danger to the process?

2. In the event of the foreseeable types of spills, what dangers will there be to the community?

3. What hazards are there from materials dumped into sewers of neighboring areas?

4. What public liability risks from spray, fumes, mists, noise, etc. exist, and how have they been controlled or minimized?

SECTION III. - LOSS PREVENTION REVIEW GUIDE

ELECTRICAL SAFETY REVIEW CHECK LIST

A. Design

1. How completely does the electrical system parallel the process?

   a) What faults in one part of the plant will affect operation of other independent parts of the plant?

   b) How are instruments for a plant protected from faults or other voltage disturbances?

2. How are interlocks and shutdown devices made fail safe?

   a) What is the need for each interlock and shutdown used?

   b) How have interactions and complications been minimized?

   c) How do we insure continued use of protective devices?

   d) What requirements or standards were used for the hardware that has been selected?

3. How has the area NEC classification been established and hardware and techniques selected?

   a) What process details effect the classification, group, and division?

   b) What "UL approved" hardware is unavailable for this job? Does this require testing.

   c) Are any new techniques being applied on this job?

4. Is the electrical system simple in schematic and physical layout so that it can be operated in a straight forward manner? (This minimizes human error in switching for isolation and load transfer.)

5. What electrical equipment can be taken out of service for preventive maintenance without interrupting production? How?

6. How is the electrical system instrumented so that equipment operation can be monitored? Will this eliminate down time due to equipment failures caused by unknown overloading?

7. What are the overload and short circuit protective devices?

   a) Are they located in circuits for optimum isolation of faults?

   b) What is the interrupting capacity?

   c) How are they coordinated?

   d) What instructions are furnished for field testing on installation and for later during life of equipment?

8. What bonding and grounding is provided?

   a) How does it protect static buildup?

   b) How does it provide lightning protection?

   c) How does it provide for personnel protection from power system faults?

9. Check Lighting.

   a) Adequacy for safe normal operation?

   b) Adequacy for normal running maintenance?

   c) Adequacy for escape lighting during power failure?

10. How is tankage grounding coordinated with cathodic protectic

11. What is the probability of accessability during mishaps of power disconnects, starters, etc.?

12. What communication is provided to operate a complex safely? (Telephones, radios, signals, alarms, etc.)?

13. What are spacings and clearances for normal traffic maintenance, and for fire fighting?

14. What provisions are needed for interlocks and what is the schedule for checking operability of interlocks?

15. Where sequence controllers are used, is there an automatic check, together with alarms, at key steps after the controller has called for a change, and is there a check together with alarms at key steps before the next sequence changes?

BOILER AND MACHINERY CHECK LIST

A. Boilers

1. Safety Valves

a) How are long and large vent lines supported?

b) What drain connections are provided?

c) Is first drum valve set to relieve boiler working pressure?

d) Is the last drum valve set to pop at or below 103% of boiler working pressure.

2. Blow-off piping

Have you used steel piping of next higher gauge than required for boiler pressure, avoided sharp radius ells, and sloped all lines and drained all low points in the lines?

3. Feedwater Piping

Is the bypass around feedwater regulator accessible from the operating level and located where the drum level gage glass can be seen?

4. Steam Outlet Piping

a) Are there separate non-return and header stop valves where 2 or more boilers discharge into the same piping system?

b) Is there a visible free blow and drain in piping between non-return and header stop valves?

c) Are there condensate drain provisions for all sections of piping?

d) Is there adequate piping expansion flexibility? How is piping supported?

5. Drum Water Level - Attended Operation

a) Are there both high and low water alarms?

b) Is there a low water cut-off of gas or oil burners? (If drop or loss of plant steam pressure does not jeopardize process safety)

c) Is gage glass visible from feedwater regulator bypass valve?

d) Is remote drum level gage independent of drum level controls?

6. Drum Water Level - Unattended Operation

a) Where and how are high and low boiler water levels monitored?

b) Are two independent low water level switches interlocked with gas or oil burner safety shut-off valves?

7. Gas burner control and piping - General

a) What type of plug cocks have been provided for manual shut-off service?

b) Is there in-line strainer in gas line ahead of all regulating and safety shut-off valves?

c) How do you provide for stable gas pressure regulation at all loads? This may require a small regulator in parallel with the full-sized regulator for start-up or low fire service.

d) Is there a double safety shut-off and vent valve arrangement? What type of reset is there for each valve?

e) What type of automatic fuel-air ratio control is used?

f) Is there separate pressure regulation of pilot gas?

g) Is safety control circuit DC, or 120v AC with the safety controls in the ungrounded circuit.

h) How do you insure positive, tamper-proof time period to provide minimum of 6 air changes in combustion chamber before light-off. Air flow rate during purge should be at least 70% of maximum capacity.

i) What controls are provided to prevent burner firing rate from being reduced below minimum stable flame.

j) What controls or interlocks are installed to prevent burner light-off when insufficient combustion air flow is present.

k) What interlock is there to assure low-fire burner light-off?

l) What method is provided for monitoring of main burner flame for alarm and/or automatic burner shut-off in event flame failure?

8. Additional gas burner controls and interlocks for unattended operation:
   a) How is monitoring of main burner flame accomplished?
   b) What other interlocks for safety shut-down of burner?
      1) High gas pressure?
      2) Low gas pressure?
      3) Low combustion air flame?
      4) Low boiler water (double switches)?
   c) Is there flame scanner response line of 2-4 seconds?
   d) Is there tamper-proof programmed light-off sequence to purge, light and prove pilot, light and prove main flame, post purge?
   e) How have you set up positioning fuel-air ratio controls?
   f) Are you using a self-checking feature for flame scanner and flame scanner relay circuitry?

9. What provision is made in the oil burner controls and piping for each of the following items?
   a) oil line strainer
   b) oil pressure control
   c) heater for heavy oil
   d) single safety shut-off valve
   e) start-up recirculating line for heavy oil
   f) positive fuel-air ratio control
   g) low oil pressure alarm or interlock
   h) low oil temperature alarm or interlock for heavy oil
   i) low atomizing steam pressure alarm or interlock
   j) positive purge cycle and low fire start controls
   k) interrupted pilot

10. Additional oil burner controls and interlocks for unattended operation:
    a) Are interrupted and proved pilot and monitoring of main oil burner flame with interlock to close safety shut-off valve during flame failure provided?

b) What other interlocks for safety shut-down of burners are used for:
1) Low oil temperature - for heavy oils

2) Low oil pressure

3) Low combustion air flow

4) Low atomizing steam pressure

5) Low boiler water (double switches)

c) How do you provide a tamper-proof programmed light-off sequence?

d) Do you use positioning fuel-air ratio controls?

II. Piping and Valves

1. How were piping systems analyzed for stresses and movement due to thermal expansion?

2. How are piping systems supported and guided?

3. How are piping systems provided for anti-freezing protection, particularly cold water lines, instrument connections and lines in dead-end service such as piping at standby pumps?

4. What provisions for flushing out all piping during start-up?

5. Are any cast iron valves being installed in strain piping?

6. Are any non-rising stem valves being installed?

7. Are double block and bleed valves used on emergency interconnections where possible cross-contamination is undesirable?

8. How are controllers and control valves reached for maintenance?

9. How are by-pass valves readily reached for operation?

10. Are any mechanical spray steam desuperheaters used?

11. Are all control valves reviewed for safe action in event of power or instrument air failure?

12. What provisions are made for testing and maintaining primary elements of alarm and interlock instrumentation without shutting down processes?

13. What provisions for draining and trapping steam piping are provided?

C. Pressure and Vacuum Relief

1. What provision is there for flame arresters on discharge of relief valves or rupture disks on pressurized vessels?

2. What provisions are there for removal, inspection, and replacement of relief valves and rupture disks, and what scheduling procedure?

3. What need is there for emergency relief devices: Breather vents, relief valves, rupture disks, and liquid seals? What are the bases for sizing these?

4. Where rupture disks are used to prevent explosion damage, how are they sized relative to vessel capacity and design?

5. Where rupture disks have delivery lines to or from the disks, what has been done to assure adequate line size relative to desired relieving dynamics? To prevent whipping of discharge end of line?

6. How are discharges from vents, relief valves, rupture disks, and flares located to avoid hazard to equipment and personnel?

7. What equipment, operating under pressure, or capable of having internal pressures developed by process malfunction, is not protected by relief devices and why not?

8. How have you supported discharge piping independently of relief valves. Make piping as short as possible and with minimum changes in direction?

9. Did you provide drain connections in discharge piping of relief valves where condensate could collect?

10. Are relief valves provided on discharge side of positive displacement pumps; between positive displacement compressor and block valves; between back-pressure turbine exhaust flanges and block valves?

11. Where rupture disks are in series with relief valves to prevent corrosion on valve or leakage of toxic material, install rupture disk next to the vessel and monitor section of pipe between disk and relief valve with pressure gage and pressure bleed-off line. Have any rupture disks been installed on discharge side of relief valve?

12. What provisions for keeping piping to relief valves and vacuum breakers at proper temperature to prevent accumulation of solids from interfering with action of safety device are provided?

D. Machinery

1. Are adequate piping supports and flexibility provided to keep forces on machinery due to thermal expansion of piping within acceptable limits.

2. What is separation of critical and operating speeds?

3. Are check valves adequate and fast acting to prevent reverse flow and reverse rotation of pumps, compressors and drivers?

4. Are adequate service factors on speed changing gears in shock service provided?

5. Are there full-flow filters in lube-oil systems serving aluminum bearings?

6. Are there provisions for draining and trapping steam turbine inlet and exhaust lines?

7. Are there separate visible-flow drain lines from all steam turbine drain points?

8. Are driven machines capable of withstanding tripping speed of turbine drivers?

9. Are non-lubricated construction or non-flammable synthetic lubricants used for air compressors with discharge pressures of greater than 75 psig to guard against explosion?

10. What provisions are made for emergency lubrication of critical machinery during operation and during emergency shutdowns?

11. Are provisions made for spare machines or critical spare parts for critical machines?

12. Are there provisions for operation or safe shut-down during power failures?

13. Are vibration switches on alarm or on interlock for cooling tower fans provided?

SECTION V - LOSS PREVENTION REVIEW GUIDE

FIRE PROTECTION CHECK LIST

1. If the building has enclosed walls and the construction or occupancy has combustibles, what kind of automatic sprinklers (wet or dry pipe systems) are provided?

2. If the building has open walls and the construction or occupancy has combustibles, how much water spray protection (HADS or pilot head heat actuating systems) been provided?

3. What existing hydrants serve the project? What additional ones are to be provided?

4. What fixed or portable monitor nozzles (on hydrants or separate) are provided for coverage for manufacturing facilities or storage facilities in open areas (not within open or closed wall buildings)?

5. How have the underground fire mains been extended or looped to supply additional sprinkler systems, hydrants and monitor nozzles? Dead ends should be avoided. What sectional control valves have been provided?

6. How many small hose standpipes are provided inside of buildings?

7. What type, size, location and number of fire extinguishers are needed?

8. What flammable liquid storage tank protection has been provided? Foam? Dykes?

9. Where have total flooding or local-application carbon dioxide systems been provided?

10. Where is load bearing structural steel exposed to flammable liquids or combustible gases so that fireproofing is needed?

11. How has adequate drainage been provided to carry spilled flammable liquids and water used for fire fighting away from buildings storage tanks, and process equipment?

12. What protection has been provided for dust hazards?

13. What is the capacity of fire water supplies? What is the maximum fire water demand?

14. How long will supplies meet this maximum demand?

15. What is the spacing of flammable liquid storage tanks?

16. What is the estimated probable maximum loss (PML)? Has Form #G1324 been completed?

17. What is the approximate "hold-up" of flammable liquids in the manufacturing equipment broken down by flash points?

18. What attention has been given to protection of process equipment from external fire?

19. Are liquid inventory tanks near or under the ground instead of elevated?

20. Is the area pad or flooring designed to conduct spill liquid away from process equipment? What facilities are provided for drainage?

21. How have major storage tanks or vessels been located to minimize hazard to process equipment in the event of rupture or burning?

PROCESS DESCRIPTION

MMK BIPHENYL AND AROCLOR MANUFACTURING FACILITIES

PROJECT CEA-1668

BIPHENYL PLANT

T A B L E O F C O N T E N T S

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Section A Synopsis of Biphenyl Process

Benzene is converted into biphenyl, terphenyl, higher polyphenyls, tars, carbon and hydrogen by raising its temperature above 650°C and holding the reaction mass until part of the benzene has reacted. This reaction is carried out continuously in a burner tubuler-type radiant furnace in which the benzene is passed through 3½" Incoloy 800 radiant tubes. Acetone is added to the feed to allow the reaction to proceed at a faster rate.

Fresh benzene and recycled benzene is fed to the furnace via the convection section near the roof of the unit where the liquid feed is vaporized. The vapors from the furnace convection section are then raised in temperature by passing them thru the shell side of the furnace preheater. This step recovers waste heat from the hot product gases from the furnace radiant section by passing these gases thru the tube side of the preheater unit.

The preheated benzene vapors from the shell side outlet of the preheater are returned to the furnace where they are further heated in the radiant section to the reaction temperature. These hot product gases, after giving up some sensible heat by passing thru the tubes of the preheater, are then routed thru the tubes of the benzene stripper reboiler and then pass into the column.

In the stripping column, the unreacted benzene is distilled overhead and the net crude biphenyl bottoms stream is pumped to storage. The column bottoms are pumped thru the shell side of the column reboiler in which heat is exchanged with the hot product gases in the tubes to generate sufficient stripping vapors in the bottom of the column to produce essentially benzene-free polyphenyls from the column.

The overhead benzene vapors are condensed in a vertical, water-cooled condenser and the condensed benzene liquid then flows to a combination reflux and furnace feed tank. Make-up (fresh) benzene from storage is pumped into this tank on liquid level control. Part of the benzene from this tank is pumped back to the column as reflux on flow control. The furnace is also fed with fresh plus recycle benzene from this tank, also on flow control.

The hydrogen and other gases from the furnace operation pass continuously from the reflux/furnace feed tank to a vertical receiver equipped with a Brink demister element. Saturated, mist-free gas then passes to a two-stage reciprocating compressor where the gas is compressed and subsequently cooled to recover benzene. This recovered liquid benzene is intermittently pumped back to the reflux/furnace feed tank. The compressed gas is vented to the atmosphere.

Section A - Con't

Crude biphenyl from storage is pumped on flow control to a 20-tray fractionating column where biphenyl is separated from terphenyls and higher polyphenyls. This column operates continuously in conjunction with an oil-fired reboiler furnace. The total biphenyl overhead stream is condensed in a vertical condenser and then flows to the column reflux receiver. Part of the biphenyl product from this receiver is pumped back to the column as reflux and the net biphenyl liquid from the receiver is pumped to storage. The net column bottoms stream (Santowax C) is pumped on level control from the column to a residue receiver prior to being drummed for disposal.

Section B Pyrolysis Furnace - Benzene Column

1. Pyrolysis Furnace Facilities

Following is a brief description of the major equipment items in the pyrolysis furnace area:

a) Pyrolysis furnace, Item 1-105: The pyrolysis furnace is a box-type tubular furnace consisting of convection and radiant sections and is equipped with pressure atomized type burners, arranged in 3 rows of on each side of the unit. The furnace is fired with a low-sulfur distillate fuel oil and is supplied combustion air at the burners by a forced draft fan (Item 1-109).

b) Furnace preheater, Item 1-108: The preheater is made up of two Brown Fintube multi-bare tube hairpin sections. Each 4" chrome-moly shell contains 7 - 20 ft. Hastelloy X bare tubes giving a total heat transfer area of 148 $ft.^2$ The tubes are constructed with wrapped spiral spacers which function as baffles. Fluid routing at the preheater is hot product gases parallel flow thru the tubes and convection section vapors series flow thru the shells.

c) Retention tank, Item 1-110: The retention tank, located at the outlet of the furnace radiant section, is a 1'-6" O.D. by 5'-7" high Incoloy 800 vessel with a volume of approximately 66 gallons. This vessel provides additional residence time for the reaction gases from the furnace to attain the desired benzene conversion.

d) Combustion air blower, Item 1-109: The air blower is a centrifugal type unit designed for a volumetric capacity of 1500 CFM and a static head of 42" W.C. The blower is located near the furnace and supplies the combustion air requirements for the unit.

Section B - Con't

2. Benzene Column Facilities

Following is a brief description of the major equipment items in the benzene column area:

a) Column reboiler, Item 1-110: The column reboiler is a 170 ft^2 floating head unit mounted at grade adjacent to the column. This unit is provided to generate sufficient stripping vapors in the column bottom section to produce an essentially benzene-free crude biphenyl bottoms product.

b) Benzene stripping column, Item 1-111: The benzene column is 3'-3" diameter by 31'-0" high steel fractionating column containing 12 valve-type trays. This column operates continuously in conjunction with the pyrolysis furnace system.

c) Column condenser, Item 1-114: The column condenser is a 1019 ft^2 fixed tube sheet, vertical unit in which condensation and sub-cooling of benzene occurs inside the tubes. This condenser is mounted over the reflux/furnace feed tank for gravity drainage of liquid benzene into this vessel.

d) Column reflux and furnace feed tank, Item 1-115: The reflux/furnace feed tank is a 1250 gallon, vertical, steel vessel which is equipped with a water boot in the bottom head.

e) Crude biphenyl storage tanks, Items 1-118 and 1-119 The crude biphenyl storage tanks are 3,000 gallon, vertical, insulated, steel storage tanks. These tanks are equipped with panel mounted, N_2 purged level indicators and steam coils.

3. Initial Furnace Dry-Out Operation

The initial heating of the furnace must be gradual to assure slow drying of the furnace refractory. This dry-out procedure should be followed initially and also after the furnace has been down for extended periods of time.

a) Prior to lighting off the furnace for the first time, it is assumed that the utility (and process) lines have been pressure tested, that all instrumentation has been checked out and that the furnace system is ready for operation. It is further assumed that the furnace box has been purged with steam and/or air and has been found negative to MSA or equivalent explosion meter before any attempt is made to light the first burner. The furnace box must be negative to an explosion meter when the lighting torch is first inserted.

Section B - Con't

The following procedure is recommended for initial furnace operation:

a) Admit steam to each burner throughout the entire furnace. This is done by opening the 3/4" block valves in the 90# steam line and opening the 3/8" cock at the steam inlet to each burner. (Steam is admitted at this point to make sure that the unlighted burner tips stay "cool" in the presence of other lighted burners).

b) Start the air blower and set the auto/manual switch on FIC-3 to manual to allow air to flow to the burners in the bottom row of the furnace. Regulate the air manifold pressure to about W.C. by throttling FV-3 control valve.

c) Make certain that the 3/8" oil inlet cock to each burner is closed.

d) Set the auto/manual switch on F_rICA-3.1 to manual.

e) Set the auto/manual switch on TRCA-2 to manual and regulate the output air signal so that when FIC-3 is switched to automatic, the air flow to the burners will be continued smoothly.

f) Switch FIC-3 to automatic control and use TRCA-2 for final adjustment of air flow to the bottom row of burners at about W.C. on the bottom row air manifold.

g) Start fuel oil pump 1-145 (or 1-146). With the F_rICA-3.1 control valve closed and all the individual burner oil inlet cocks closed, the oil flow will be diverted back to the storage tank (Item 1-144) via pressure regulator PCV-104.

h) With the F_rICA-3.1 auto/manual switch on manual, set the pointer so that an output results which will just start to open the control valve.

(Note: To implement the startup which requires oil flow through the F_rICA-3.1 control valve without allowing flow to the burners, provide a 40-50 gallon barrel under the 1/2" drain line from the oil manifold).

With fuel oil now being pumped into the drain barrel, adjust the pointer to attain about PSIG on the oil manifold. This is equivalent to about Btu/Hr. firing rate per burner for the GPH oil nozzle.

Section B - Con't

i) Insert the acetylene torch adjusted for strong flame in the lighting hole of one of the burners in the bottom row.

j) Close the inlet steam cock of the burner selected and slowly open the burner oil cock. After a burner is lighted, the oil cock must be fully open. This cock is not intended to be used for throttling.

(Note: For design operation of the furnace, the required oil flow to each burner will be equivalent to about Btu/Hr. which will require an oil manifold pressure of about PSIG. The combustion air manifold pressure required for these conditions is about W.C. It is important to adjust the air manifold pressure on lighting to a value to give an oil-rich (air deficient) fuel mixture at the burner. For lighting the burners at an oil manifold pressure of PSIG, the air manifold pressure should be about W.C.

k) If a burner does not light in about 10 seconds, the oil to that burner should be cut off and the furnace purged out. Burners which are operating will provide a partial purge in removing an explosive mixture. If there is any question regarding hazard in lighting after admitting unburned fuel, an MSA test should be made.

l) Burners should be held on the rich side until about 30% of the total burners are operating. The burners should be adjusted (pressure and oil/air ratio) so that the burner cup is not radiant but rather is generating a long flame which crawls up the furnace wall.

m) The temperature increase in the top of the radiant section should not exceed 50°F per hour and should be in general accord with the general Selas procedure for drying-out refractory on furnace walls. (a copy of this procedure is attached as Appendix A).

n) It is suggested that non-adjacent burners be lighted first in the bottom row, adding burners later in the top row and in the middle row.

o) After 50 to 60% of the burners are in operation, switch the auto/manual selector on FICA-3.1 to automatic to maintain the setting selected to result in the desired burner operation.

Section B - Con't

p) The procedure for burner operation in the top zone of the unit is essentially the same as described above for the bottom zone except that the air flow controller (FIC-5) is not indexed from a temperature controller.

In operation, this difference will require that the set point on FIC-5 be controlled manually during startup, and not pneumatically from a TRC.

q) With all the burners in the bottom row in operation the high select relay on F$_r$ICA-3.1 can now be set. Adjust the air supply to the high select relay in the F$_r$ICA-3.1 output to a value which will provide a minimum oil pressure of about PSIG.

r) For proper operation, the stack damper should be adjusted to provide a draft of about 0.1" W.C. at the top of the radiant section at all times.

It is also important that a nominal draft be maintained at the arch which will represent the minimum draft point in the furnace structure.

When lighted burners are shut down to light new ones, immediately start the steam purge to the shut-off burner to avoid possible nozzle coking. As burners are required, a scattered pattern should be used in the lower rows progressing upward as required.

During the drying operation, periodic observations must be made and steps taken to prevent surface thermal shock or steaming within the refractories which may spall or crack them.

4. Initial Conditions of Startup

At this point, it is assumed that the furnace initial dry-out phase has been completed, that the furnace and benzene column systems have been leak checked and pressure tested and are ready for initial startup.

The following items should now be checked prior to initial startup:

a) Furnace

Check to see that all burner cocks on each of the burners are closed.

Section B - Con't

b) Utilities

Check the CO_2 system to assure 200 PSIG supply header pressure using PI-18, located on the furnace control wall. Also check to assure a minimum of 1000 lbs. (Approximately 8600 SCF) of CO_2 available for purging.

Check that cooling tower water is flowing through the shell side of benzene condenser 1-114.

Make sure adequate instrument air pressure is available at the unit.

c) Fuel Oil

Check fuel oil tank 1-144 to assure an adequate supply of oil is available.

Check operability of fuel oil by-pass pressure control valve PCV-104 by starting pump 1-145 or 1-146 with the fuel feed lines to the furnace blocked off. When the set point of this valve has been reached, oil should pass through the opening valve as observed in the sight glass just downstream of the valve. (This system was used during initial furnace dry-out operations).

d) Instrumentation

Set the furnace shutdown interlock switch to by-pass location. This allows startup by taking the benzene high-low flow (FRCA-1), the fuel oil low pressure (PA-14) and combustion air low pressure (PA-11) functions temporarily out of the system.

See Section B (Page 4) for the settings on the burner control instruments.

Set the system back pressure controller PRCA-26 to zero; this will open valve PV-26. Make sure PV-26 bypass valve (X96-2") is closed.

Set the fuel oil flow controllers to each zone (F_ICA-3.1 and F_IC-5.1) to zero and close the block valves at the automatic valves.

Make sure all valves on pressure gauges are open and set approximately 25 SCFH (max.) CO_2 flow on the purge meters to PI-22, 23, 32 and 35.

Section B - Con't

Set the benzene column overhead temperature controller TRCA-28 to control on the 12th tray of the column (TR 40-point 9) at 80°C to control reflux flow rate. Set column reflux controller FRC-27 on cascade and automatic control.

e) Raw Materials

Check the benzene storage tank (item 1-101) to make sure an adequate volume of on-specification benzene is in storage. Line up valves from benzene storage pump 1-102 to reflux/furnace feed tank.

Check the acetone storage and feed tank (1-103) to assure the tank is essentially full of on-specification acetone prior to startup.

f) Reflux/Furnace Feed Tank

Initially fill this tank with approximately 850 gallons of benzene from storage. Check operation of tank level controller LICA-30 during initial filling step. Vent tank via vent line with flame arrestor during filling.

Check the water boot interface level indicator LI-52 and manually drain water from the boot as required.

Initially align valves in the off-gas line from the tank to vent hydrogen to the atmosphere. (Make sure the block valve in the gas line to benzene receiver 1-122 is closed).

g) Stripping Column

Charge tank 1-118 (or 1-119) with about 300 gallons of liquid biphenyl.

Pump product biphenyl from the crude storage tanks into the sump of the stripping column. This can be done using pump 1-120 and the 1" rerun feed line to the column.

5. Initial Startup

a) Open the damper in the pyrolysis furnace stack and purge the fire box with air via blower 1-109. The purge volume should be about 5 times the volume of the box. Check the box with an explosion meter to make sure the box is safe to light the burners.

Section B - Con't

b) Align valves to allow CO_2 to be purged through the furnace tubes at the rate of 125 SCFM to be observed on meter F1-9. This is done to displace air from the furnace tubes, preheater and column system prior to feeding benzene.

c) With the furnace access doors closed, the furnace is now ready for initial operation. Align valves for the lighting of the 2nd and 4th burner on the bottom row on each side of the furnace. Manifold (oil and air) pressure readings and general operator experience are used during this initial lighting operation. Start oil pump 1-145 (or 1-146) and air blower 1-109 and light off the above mentioned burners. (Refer to Section 3 above for burner lighting procedure).

d) Start pump 1-112 (or 1-113) and begin circulating biphenyl liquid through the shell side of the column reboiler.

e) The furnace box temperature, as observed on the multi-point recorder TR-10 points 2 through 7, is now raised to about 450°C, lighting more burners as required. This temperature is attained over a 4-hour period.

f) After the furnace box temperature has reached 500°C, start CO_2 flowing through the furnace tubes and open the manual valves (along the furnace control wall) in the benzene feed line to the furnace. Start the reflux/furnace feed pump 1-116 (or 1-117) and manually set 5 GPM benzene feed rate on flow controller FRCA-1. Light additional burners to maintain 500°C on TRCA-2.

(Note: After 18 to 20 of the burners have been lighted, the oil flow controllers can be switched from manual to automatic control).

g) Continue venting CO_2 to the atmosphere via the off-gas line vent from the reflux/furnace feed tank and slowly decrease the flow of CO_2 to the system as the benzene flow rate is increased. During this adjustment maintain temperature on TRCA-2 at 500°C.

h) The benzene is now switched to automatic control and raised to 8 GPM. Additional burners are lighted as required to maintain 500°C temperature on TRCA-2.

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i) Gradually increase the benzene flow rate to 15 GPM while maintaining the process vapors outlet temperature at 500°C. When all the burners have been lighted, heat input to the furnace is increased by increasing the oil and air flow rates to the burners.

j) When the benzene feed rate exceeds 12 HPM and the combustion air rate exceeds about 45,000 SCFH (total), the shutdown interlock switch can be changed from the by-pass to control location.

k) Adjust the benzene flow rate to 21 GPM (design rate) and adjust the pressure at PI-22 initially to about 90 PSIG by throttling the PRCA-26 control valve. The rate of pressure increase should be about 10 PSI per hour.

l) Switch furnace temperature controller TRCA-2 to automatic cascade control.

m) The unit will be initially operated under these conditions for a period sufficient to test the unit and train the operators.

n) The furnace outlet (control) temperature is now raised to approximately 715°C (8.5% net conversion) at a rate not to exceed 100°C per hour.

o) When the furnace outlet temperature reaches 650°C biphenyl formation starts to occur. Start acetone charge pump 1-104 and feed acetone into the system at a rate of 9 lbs/hour (approx. 1.4 GPH).

p) As biphenyl is formed and flows to the benzene column, the net bottoms liquid (crude biphenyl) volume will increase. Check the column sump level (LRCA-31 and LI-31) and open the block valves at automatic valve LV-31 to allow column liquid level control.

q) As part of the benzene flowing into the furnace tubes is converted to biphenyl, make-up benzene will be pumped into the reflux/furnace feed tank on liquid level control, LICA-30.

r) To minimize benzene losses in the off-gas from the reflux/furnace feed tank (now being vented to atmosphere), the hydrogen compressor is now started up. (See Section C for compressor operating procedures).

s) To prevent vaporization of crude biphenyl in the column reboiler (1-110) adjust the 4" globe valve on the reboiler outlet line to give a pressure of 10 PSIG as read on PG 33.

Section B - Con't

6. Normal Operations

This unit is completely automatic and only routine observations need be made to be sure that all controls are functioning as intended. These checks are as follows:

a) Observe the water level in the reflux/furnace feed tank by periodically draining water through the bottom nozzle. Check the water level interface indicator, LI-52, to be sure it is operating properly. It is located at the tank.

b) Observe the level controller (LICA-30) on the reflux/ furnace feed tank and be sure it is operating properly.

c) Observe the tubes in the furnace through the peepholes to check for leaks and hot spots. Check the temperature throughout the furnace.

d) Observe the furnace stack. If black smoke is noted, a tube is probably leaking and the unit should be shut down.

e) Check the liquid level in the bottom of the benzene column (LRCA-31).

f) Check the temperature of vapors leaving the vaporizer section of the furnace (TR-10, point 15). If the vapor temperature is not 175°C, light off the top row of burners on each side and adjust the oil and air flow to the burners until this temperature is obtained.

g) Check for leaks throughout the system.

h) Adjust the outlet water from the condenser (1-114) to maintain sufficient water pressure in the remainder of the plant. The maximum exit water temperature is 49°C (TG-42). The feed water pressure should be about 41 PSIG.

i) Check the furnace inlet pressure (PRA-4) and the shell and tube side pressure drop at the preheater, 1-108. Also check PI-22.

7. Control and Alarm System

Alarms are provided on all critical control points. They are as follows:

a) High and low liquid level alarms are provided on the reflux/furnace feed tank (LICA-30).

b) High and low flow alarms are provided on the benzene flow controller (FRCA-1) in the furnace feed line.

Section B - Con't

c) The fuel oil and air to the furnace are provided with low pressure alarms (PA-14 and PA-11 respectively).

d) A high temperature alarm is provided on the furnace process outlet temperature controller TRCA-2.

e) Fuel oil feed to the furnace will alarm on low flow rates to both zones of the furnace (F_rICA-3.1 and F_rICA-5.1.

f) The pressure recorder (PRA-4) on the benzene feed to the furnace inlet is alarmed for high and low pressures.

g) Pressure controller (PRCA-26) on the stripping column feed line is alarmed for high pressure.

h) High and low level alarms are on the benzene column level controller, LRCA-31. High level alarms are provided on the crude biphenyl storage tanks (LIA-57.1 and 57.2).

i) All pumps sound an alarm when they kick off.

j) An automatic shutdown system is provided to initiate an emergency shutdown of benzene, fuel oil and combustion air feeds for any of the following abnormal conditions:

Power Failure

In the event of a power failure, the following sequence of events will occur to initiate the interlock circuit and shut the unit down.

(1) The fuel oil solenoid valves will close, stopping all air and oil feed to the furnace burners. (The oil pump and air blower will also stop).

(2) The air system to the benzene flow control valve will be vented to the air, thus closing the valve.

(3) The 110 volt power supply for the interlock circuit and instrument control panel originate at the same source. Therefore, a local power failure on the circuit will initiate an emergency shutdown.

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Low Fuel Oil Pressure

A pressure switch (PA-14) is installed to initiate a furnace shutdown if the fuel oil pressure drops to 100 PSIG.

Low Combustion Air Pressure

A pressure switch (PA-11) is installed in the air line from blower 1-109 to initiate a furnace shutdown if the air pressure drops to 15" W.C.

High and Low Benzene Flow

Abnormal high and low benzene flow rates will initiate an emergency interlock shutdown of the pyrolysis unit. The interlock system will be set for a normal range of 10 to 30 GPM of benzene flow.

High Stack Temperatures

Excessive heat input causing high stack temperatures in the furnace will shut the system down (TIA-6). The setting for the interlock shutdown will initially be set for 1,000°F.

Section C Hydrogen Compressor System

1. Compressor Facilities

Following is a brief description of the major equipment items in the benzene recovery (hydrogen compressor) area.

a) Benzene receiver, item 1-122: The benzene receiver is a 200 gallon, vertical steel vessel provided to serve both as an inlet knock-out drum and recovered benzene receiver. This tank is equipped with a stainless steel/glass fiber Brink mist eliminator to assure mist-free gas to the compressor inlet. This vessel operates intermittently with benzene return pump 1-123 on pump-out.

b) Hydrogen compressor, item 1-124: The hydrogen compressor is an Ingersoll-Rand 2-stage reciprocating type ESH unit driven by a 30 HP motor. The compressor runs at 600 RPM and discharges the rated benzene-saturated hydrogen flow of 70 ICFM at 300 PSIG.

Section C - Con't

The compressor is provided with cylinder force-feed lubrication system with low oil pressure shutdown switch. The compressor cylinders are cooled with tempered water. The machine is also provided with manual inlet valve unloaders for capacity control.

c) Compressor inter-and-aftercooler, items 1-125 and 1-127: The inter-and-aftercoolers are Brown Fin-tube double pipe fin tube units in which the gas is cooled (and benzene condenses) in the annulus and tower water flows inside the tube.

d) Compressor receivers, items 1-126 and 1-128: These receivers are both 125 gallon, vertical steel vessels which function as condensed (recovered) benzene receivers. These vessels are equipped with automatic level controllers (LCA-71 on 1st stage receiver and LCA-81 on 2nd stage receiver) and benzene is continuously pressured back to the benzene receiver prior to transfer to the reflux/furnace feed tank.

2. Pre Startup and Check-Out

Prior to initial startup on hydrogen, it is assumed that the following activities have been accomplished:

Pre Air Test activities - Prior to making the initial air test, the compressor suction piping must be checked to make sure it is free of weld metal, slag, dirt and scale and that the suction line temporary filter (special fitting 511) is in place. Also it is advisable to brush the inside walls (and then flush) of receivers 1-122, 126 and 128 to remove dirt and loose scale. The suction and discharge piping must be "gas tested" and any leaks detected and eliminated prior to admitting H_2 into the system.

Air test and mechanical check-out. The machine must initially be operated on air to check "run-out", alignment and proper lubrication. The following steps should be followed for the initial air test:

a) Insert a line blind on the 3" gas inlet nozzle on the side of the benzene receiver.

b) Remove the 10" hand hole cover on the top head of the benzene receiver to serve as the air source.

c) Start the flow of cooling water to heat exchangers 1-125 and 1-127.

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d) To operate the machine (initially) fully unloaded, adjust the handwheels of the four 1st stage unloaders and the two 2nd stage unloaders to the downward position. The machine will now run fully unloaded.

e) Operate the cylinder jacket tempered water system and adjust the water temperature to 40°C. Start the water circulating through the cylinder jackets. Set TIC-114 at 40°C.

f) Start the compressor and run fully unloaded for at least one hour. Initial mechanical inspections are made during this period.

g) Initially set back pressure controller PRCA-87 at 150 PSIG. Close block valves around suction pressure control valve (PV-86) as this system will not be used for the air test.

h) After the 1-hour unloaded compressor run, fully load the machine by reversing the procedure outlined in step (d) above. The unit is now compressing atmospheric air (from 1-122) and discharging it to the atmosphere via pressure valve PV-87.

(Note: Moisture from the air will be condensed and collected in items 1-126 and 128 after the machine is loaded. Align block valves around the level control valves on the bottom piping from these drums such that this water will be blown back to 1-122. Check the level gauge on this vessel and periodically drain as required).

i) Run the compressor fully loaded at 150 PSIG for about 2 hours. At the end of this period, increase the setting of PRCA-87 to 300 PSIG. Run machine at 300 PSIG for approximately 4 hours.

j) After this 4-hour run, shut down the machine. Replace the 10" hand hole cover on the head of 1-122 and remove the temporary line filter (special fitting 511).

k) During the air test, check the operability of the frame oil system low pressure shutdown. Also, adjust the cylinder lubricator system for 6-8 drops per minute to the 1st stage cylinder and 3-4 drops per minute to the 2nd stage cylinder.

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1) Refer to Sections 2A-1 through 2A-19 of the I-R instruction booklet for mechanical checks that need to be made.

3. Compressor Purging

Prior to initial startup of the compressor with hydrogen, the system must be purged with an inert gas (N_2) to a safe oxygen level. Also, before a maintenance inspection of the machine, the compressor system should be purged with N_2 prior to opening any part of the machine or associated equipment. A N_2 purge line is tied into the compressor suction line (gas outlet line from 1-122) which is to be used for inerting the system.

The following procedure can be used for N_2 purging:

a) Install a line blind at the 3" gas inlet flange on 1-122.

b) Make sure all vessel drain valves are closed.

c) Open the 3/4" block valves in the N_2 line and slowly pressure up the compressor system to about 30 PSIG.

(Note: Install a temporary pressure gauge on the 1/2" drain connection on 1-122 level gauge LG-64 to check the N_2 pressure in this part of the system). Check this pressure at various points in the piping (PG-68, 73, 79 and 96).

d) Slowly depressure the system to about 1 PSIG via pressure valve PV-87.

e) Again repressure the system to 30 PSIG N_2 pressure and depressure to 1 PSIG (as above) for the second time.

f) Sample the gas mixture at the high pressure vent (1/2" valve between PV-87 and upstream block valve) and analyze. When this analysis indicates 4% O_2 or less, the system is safe to admit hydrogen.

(Note: During this purging step, be sure to bleed off N_2 from the drain valves at vessels 1-122, 126 and 128 for several minutes).

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4. Initial Hydrogen Startup

The following procedure should be followed for initial start-up on hydrogen.

a) Check that cooling tower water is flowing to exchangers 1-125 and 1-127.

b) Start tempered tower water flowing through the cylinder jackets and set TIC-114 at 40°C.

c) Set PC-85 at 1.75 PSIG. Set PRCA-86 at 1 PSIG and PRCA-87 at 300 PSIG, all on automatic control.

d) Make sure valves and piping are aligned to blow recovered benzene back to 1-122.

e) Start the steam purge to the hydrogen vent line. Throttle the block valve just ahead of the steam orifice such that PG-84 reads about 9 PSIG. This corresponds to a steam rate of approximately 70 lbs/hour.

f) Make sure the lubricator oil levels are adequate.

g) Fill the 1-122 receiver with benzene to a point that the demister drain leg is submerged.

h) Adjust the handwheels of the four 1st stage inlet valve unloaders and the two 2nd stage unloaders to the downward position (unloaded).

(Note: At this point it is assumed that the pyrolysis-benzene column system is in operation and that gas is being vented from the reflux/furnace feed tank to the atmosphere via the 2" vent line containing the flame arrestor).

i) Open the 3" block valve in the gas line to item 1-122 and close the vent line block valve.

j) Start the compressor (unloaded). The machine will be up to speed in a few seconds.

k) After the machine is up to speed, fully load the unit by adjusting the handwheels of all six unloading valves to the raised position.

l) The machine is now compressing hydrogen to 300 PSIG from a controlled inlet pressure of 1 PSIG and condensed benzene is accumulating in receivers 1-126 and 1-128. Take a complete set of temperature, pressure and level readings.

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5. Compressor System Instrumentation and Controls

Following is a description of the instrumentation, controls and automatic shutdown features on the compressor system:

a) Pressure Controllers

Suction pressure is controlled with pressure controllers PRCA-86, located on the control room panel. The machine suction pressure is sensed at a point on the first stage suction line and the suction pressure (normally 1.0 PSIG) is controlled by recycling 300 PSIG gas from the discharge line, taking the pressure drop across PV-86 control valve.

Discharge pressure is controlled with pressure controller PRCA-87, also located on the control room panel. High pressure gas (300 PSIG) is expanded across PV-87 control valve and is vented to the atmosphere.

Overpressure can occur in the system caused by a compressor failure. Should the system pressure begin to rise, pressure controller PC-85 control valve will open and vent gas to the atmosphere.

b) Automatic Shutdown Devices

The following instruments are provided with alarms and cause the compressor to shut down:

Low suction pressure.

High liquid level in benzene receiver 1-122.

High liquid level in first stage receiver 1-126.

Frame lubrication system low pressure.

c) Benzene receivers - liquid level controls

Automatic liquid level controllers are provided on first and second stage receivers (LCA-71 and 81). These controllers function to maintain a specified level in these vessels by blowing recovered benzene back to item 1-122. High and low level alarms are provided on these tanks to detect instrument malfunction.

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Liquid benzene is pumped from 1-122 intermittently back to the reflux/furnace feed tank via controller LC-63. Level switch LC-63.2 automatically starts pump 1-123 and when the level in 1-122 drops to the LC-63.3 position, the pump automatically stops. Low level alarmLA-63.4 is provided at the bottom nozzle of 1-122.

d) Gas high temperature alarms

Temperature switches (TAH-90 and TAH-91 respectively) are installed in the gas discharge lines from each stage which will activate the annunciator panel alarm should the exit gas temperature reach the preset value of 190°C.

6. Normal Operation

The operation of the compressor system is completely automatic and normally requires periodic monitoring as follows:

a) Check benzene level in items 1-122, 1-126 and 1-128 every 4 hours.

b) Check ΔP across item 1-122 every 8 hours. (dP1-93)

c) Check pressure on items 1-122, 1-126 and 1-128 every 4 hours.

d) Check the following temperatures & pressures every 4 hours:

Cylinder discharge pressures (PG-68 and PG-96).
Item 1-122 gas outlet - TR 40, point 11.
Items 1-126 (TG 72) and 1-128 (TG 78) gas outlet.
Hydrogen from compressor cylinders. (TG 67 and TG 75).
Jacket water supply, TG 83.
Jacket water discharge, TG 82.

e) Maintain jacket water temperature (TIC 114) 10°C above item 1-124 inlet temperature.

f) Check out alarms and automatic shutdown devices once a month.

Section D Biphenyl Refining System

Crude biphenyl from storage (tanks 1-118 and 1-119) is continuously pumped to a valve-type tray fractionating column where product biphenyl is taken overhead, cooled and then is directed to storage. Santowax C bottoms from the column is pumped to a receiver prior to being drummed for storage.

Overhead product quality is controlled by reflux flow controller FRC-153 which is reset by temperature controller TRC-154. Heat required to generate stripping vapors at the bottom of the column is provided by the column reboiler furnace, item 1-132. Santowax C liquid level is maintained in the column sump via level controller LRCA-152.

1. Biphenyl Refining Facilities

Following is a brief description of the major equipment items in the biphenyl refining area:

a) Biphenyl refining column, item 1-129: The biphenyl refining column is a 1'-6" dia. x 39' high, steel, insulated column containing 20 fractionating trays. The column operates essentially at atmospheric pressure and continuously produces product biphenyl as overhead product at a rate equivalent to 5.5M lbs/year.

b) Column reflux drum, item 1-135: The column reflux drum is a 200 gallon, vertical, insulated, steel leg-supported drum containing a level controller and internal steam coil.

c) Refining still furnace, item 1-132: The refining column reboiler is an oil-fired, vertical, cylindrical heater containing a cross-flow convection section and helical-coil radiant section. This unit is designed for a heat absorption rate of 400MBTU/hour and will be located adjacent to the refining column.

d) Column condenser, item 1-133: The column condenser is a 50 $ft.^2$ vertical unit mounted above the reflux drum. This condenser is cooled with tempered water from the controlled-temperature warm water circulating loop.

e) Column residue receiver, item 1-138: The residue receiver is a 500 gallon, vertical, insulated, steel leg-supported drum containing a steam-purged level indicator and steam coils (internal and external). Santowax C residue is intermittently drummed from this vessel for disposal.

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f) Refined biphenyl storage tank, item 1-137: The refined biphenyl storage tank is a 10,000 gallon, vertical flat bottom, steel tank equipped with a steam jacketed conservation vent valve and internal steam coil.

g) Column tempered water system: The column tempered water system consists of a 125 gallon water tank (item 1-139), a 46 ft.^{2} U-tube cooler (item 1-140) and water circulating pumps (items 1-141 and 1-142). This system provides temperature-controlled warm water to the column overhead condenser 1-133.

2. Initial Startup Check-out

Before initial operation of the system, preliminary checks must be made of valves and instruments to see that they are in the proper position for initial startup.

a) Preliminary check of the column feed system

Steam tracing should be on all lines in the feed system and on the column at least 30 minutes before crude biphenyl is charged.

The 1/2" by-pass valve around the feed control valve (FV-139) should be closed. The 1" block valves on either side of the feed control valve should be open.

The 1" feed valve to tray 14 should be open.

The feed flow controller (FRC-139) in the control room should be set in the manual position and the flow control valve should be closed.

b) Preliminary check of bottoms circulating system

Steam tracing should be on at least 30 minutes before crude biphenyl is charged.

The inlet valve to the circulating pump (item 1-130 and 1-131) should be closed at this time but will be opened later. The valve in the discharge line from this pump should be open.

The 3/4" block valves to and from the bottoms sample chamber should be closed.

The furnace coil drain valve (if any) should be closed.

Drain valve in the column bottoms line should be closed.

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The globe valve in the return line to the column sump should be open.

The 3/4" by-pass valve around the sump level control valve (LV-152) should be closed. The 3/4" block valves on either side should be open.

Valves in the bottoms discharge line should be set so that the column bottoms are recycled back to the crude biphenyl tanks (items 1-118, 1-119).

The column level controller, LRCA-152 should be set in the manual position and the control valve should be fully shut.

The column temperature controller (ΔTRCA-141) should be set in the manual position and the control valve (in the fuel oil line to the furnace) should be fully shut.

c) Preliminary check of the product cooling and collection system.

The 3/4" block valve in the reflux line should be open. Reflux flow control valve FV-153 should be open.

Valves in the product discharge line from pump 1-136 should be set so that the product is recycled to crude biphenyl storage tanks (items 1-118, 1-119) on initial startup.

Biphenyl sample valve in the feed line to reflux tank 1-135 should be shut.

Steam tracing should be turned on all product lines.

(Note: During the initial startup, no product will be put into the Biphenyl storage tank until the column is at equilibrium and is producing specification material. This is being done to prevent contamination of the Biphenyl storage tank with off-grade product.

At this point, the tempered water system should be started up and the circulating water temperature should be adjusted to 186°F via TIC-115. Warm water should be started through the Column Condenser (item 1-133).

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3. Initial Startup

For initial startup, it is assumed that the column is empty with no liquid either on the trays or in the sump.

The column is now ready to be charged with crude biphenyl, and this should be done in the following manner.

a) Start the feed:

With the feed control valve closed, start the feed pump (item 1-120 or 1-121) in the crude biphenyl storage area.

Slowly open the feed valve to let approximately 2 GPM of feed into the column. This is done by manually adjusting the output air from the feed flow controller (FRC-139) to the feed control valve. 2 GPM should be indicated on the controller.

Check the FRC-153 reflux control valve to see that it is fully open.

b) Start the heat-up:

When the column level controller LRCA-152 indicates 50%; open the inlet valve to the circulating pump (item 1-130) and start the pump. Check for leaks.

As soon as the column sump level reaches 50%, manually close the feed control valves to stop the feed pump.

Start up the refining still furnace (item 1-132) as per vendor operating instructions for lighting the burner, etc.

Manually adjust the Δ TRCA-141 temperature control valve so as to raise the temperature of the circulating bottoms stream to 260°C as read on TR 170, point 1.

Again slowly open the feed valve, and feed 2 GPM until the column level again reaches 50%.

Manually adjust the TV-141 temperature control valve and raise the temperature of the circulating bottoms stream to 300°C.

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Check the temperature at the top of the column (TR-170, point 7). As soon as it reaches 255°C, visually check the sight glass on the reflux drum (item 1-135) to see that there are no leaks or plugs.

Manually adjust the TV-141 temperature control valve and raise the temperature of the circulating bottoms stream to 360°C.

c) Set for normal operation:

Set the column level controller (LRCA-152) to 50% and switch to automatic control.

Set the overhead temperature controller TRC-154 at 255°C to control reflux flow. Set reflux flow controller FRC-153 on cascade and automatic control.

Set the feed flow controller (FRC-139) to about 1.7 GPM (Design feed rate) and switch to automatic.

Set the reflux drum level controller (LICA-158) to 50% and set on automatic control.

Manually adjust the temperature of the circulating bottoms stream to 390°C. This is the liquid temperature in the column sump. Set temperature controller ΔTRCA-141 to automatic with the set point at about 12°C. This will control the reboiler furnace outlet temperature to approximately 402°C.

Watch the multipoint, TR-170. As soon as the column reaches equilibrium, the column temperature points will level out. At this time samples of the bottoms and product should be taken, analyzed and corrections made as follows:

1. Bottoms-too high in biphenyl concentration correction - Increase the temperature controller set point by 3°C, allow the column to reach equilibrium and resample.

   (Note: A good check point for biphenyl quality is column top temperature. Normally this will run 255°C.

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2. Product - Too high Santowax C concentration - low C.P. correction - Increase reflux and resample.

Note: The column is now making specification overhead product and is at equilibrium. Both product and bottoms can be switched to storage. This should be done as follows.

Reset the valves in the product line so that the product enters the biphenyl storage tank (item 1-137). The valve to the crude biphenyl storage tank (item 1-118, 1-119) should be closed. Reset the valves in the bottoms line so that the Santowax C is directed to storage. The valve in the line to the crude biphenyl storage tanks should be closed.

The refining system is now operating at normal conditions.

4. Normal Operations

The operation of the refining system is completely automatic and normally requires periodic monitoring as follows:

a) Adjust the feed to the desired flow rate and adjust the reflux flow as necessary to maintain quality product.

b) Check levels in the column (LRCA-152) and reflux drum (LICA-158) every 4 hours.

c) Check ΔP across reboiler furnace coils every 8 hours.

d) Monitor overall column system temperatures (TR-170) every 4 hours.

e) Check tempered water system to make sure temperature control system is operating properly.

f) Control temperature of biphenyl condensate from the condenser (1-133) at about 90-95°C by adjusting the temperated water temperature accordingly.

5. Normal Shutdown

a) Short Term Shutdown

A short term shutdown of the column system is accomplished by putting the column on total reflux. This should be done in the following manner:

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Set the reflux controller, FRC-153, to manual and adjust the output air to the control valve so that the valve is fully open. Close the block valves around LV-158 control valve.

Close the feed valve by adjusting the feed controller, FRC-139, to 0. Stop the feed pump (item 1-120 or 1-121). The column is now on total reflux.

b) Long Term Shutdown

Accomplish a short term shutdown.

Shut down the furnace in the manner prescribed in the operating instructions for the furnace (item 1-132).

Set the valves in the bottoms pump-out line so that the bottoms can be pumped back to the crude biphenyl storage tanks (items 1-118, 1-119).

Set the column level controller in the manual position and slowly open the level control valve.

Listen to the bottoms circulating pump (item 1-130 or 131) as soon as it starts cavitating, shut down the pump.

Close the LRCA-152 level control valve.

Place a drum under the furnace coil drain valve and drain the coil.

Drain the impeller housing of the bottoms circulating pump (item 1-130 or 131).

Drain the product line and shut down the tempered water system.

Blow out the bottoms line to the Santowax C receiver, 1-138.

(Note: Leave steam on all tracing and jackets in the still area.

PROCEDURE FOR DRYING-OUT REFRACTORY ON FURNACE WALLS

The refractory of a new furnace, or a furnace which has been idle for a long period, should be dried out slowly so that the expansion is gradual and uniform. In this way, the entrapped water will vaporize slowly rather than boil violently, spalling the brickwork.

If a new furnace is idle during the cold season, measures should be taken to keep the furnace warm; either by lighting a few burners or by steam circulation through the coils, if conditions permit. The refractory which may have absorbed moisture should never be exposed to the atmospheric temperature below 32°F.

In new furnaces, drying-out can be started two days after the refractory lining has been finished and cured.

Where pilots are supplied with the furnace, and time is available, the pilots can be lighted and left burning for 24 hours before lighting the main burners. This can serve as a preliminary to drying-out and will help dry-out the burner cups.

SUGGESTED HEATING RATES FOR VARIOUS WALL THICKNESS

A. Overall Refractory Thickness-Up to 9"

Increase at 50°F/hour to about 400° to 500°

Hold for 8 hours at 500° maximum

Increase at 50°F/hour to 1000°F

Hold for 8 hours at 1000°F maximum

Increase at 100°F/hour to operating temperatures

B. Overall Refractory Thickness - more than 9"

Increase at 50°F/hour to about 400° to 500°

Hold for 12 hours at 400° to 500°F maximum

Increase at 50°F/hour to 1000°F

Hold for 12 hours at 1000°F maximum

Increase at 100°F/hour to operating temperatures

This is an approximate guide only. During the drying-out operation, periodic observations must be made and precautions taken to prevent surface thermal shock or steaming within the refractories which may spall or crack them. Additional holding-time at existing temperature should be held whenever moisture is evident.

Avoid any spot concentration of heat by spreading out and staggering the heat input. For inspirating type burners, set the fuel pressure in the range of 5 to 7 psig which will insure stable burner operation while putting the maximum number of burners in operation and insuring a more uniform heat distribution.

Use a rich air-gas mixture to give a soft flame at the burners by restricting the air flow. Care should be taken to insure that the longer than normal flame does not come in contact with the process tubes.

Temperature should be measured close to the top of the radiant box in the flue-gas stream. A temporary thermocouple can be inserted through the lighting hole of a burner which is not in service.

The lighting of burners should start with the bottom rows, progressing upward as more burners are required.

Although less than half of the burners are likely to be used for drying-out, all the burners should be tested at this time for operability. That is, during the drying out period burners should be turned off in certain rows and other burners lighted in the same row so that at the end of the operation each burner shall have been operated for at least several hours.

If the furnace is not started up immediately after it has been dried-out, it is recommended that the temperature of the furnace be maintained at about 500°F.

During the drying out phase, it is recommended that steam is flowing through the furnace tubes. Prior to lighting the first burner, align the 2" valves in the steam line to the inlet of the convection section and allow steam to purge through the furnace tubes. This steam flow should continue throughout the duration of the drying out step.

PROCESS DESCRIPTION

MMK BIPHENYL AND AROCLOR MANUFACTURING FACILITIES

PROJECT CEA 1668

AROCLOR PLANT

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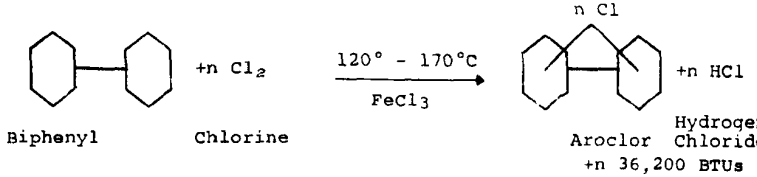
Section A Synopsis of Aroclor Process

Aroclor is a trade name used to designate the chlorination products of biphenyl, biphenyl high boilers or mixtures of the two. Liquid aroclors are produced by chlorinating biphenyl to 21, 32, 42, 48, 54, 60 and 62% chlorine content. Chlorination above 63% yields a solid product at normal temperatures. Liquid aroclors are identified by a four digit number. The last two digits represent the chlorine content (percentage by weight) and the first two numbers represent the Aroclor condition. If the first digit is "1" it signifies that the Aroclor has been made from biphenyl. If the second digit is "1" the product has not been distilled, if it is "2" the product is a distilled grade. As an example, Aroclor 1242 is a distilled chlorinated biphenyl containing 42% chlorine.

The first process step in the manufacture of Aroclors is a chlorination step. Biphenyl and chlorine gas in the presence of ferric chloride catalyst are reacted to form a chlorinated aromatic compound. Hydrogen chloride gas is liberated during the reaction and either recovered or drowned in a scrubber and sewered. The main reaction is a simple replacement of hydrogen by chlorine but the product of the chlorination contains trace amounts of halogen-containing by-products which are less stable than the direct ring-substitution products. Many isomers are formed.

A schematic representation of biphenyl chlorination is as follows:

Taking molecular weights: Biphenyl = 154 Aroclor = 254.1 to 395.6
Chlorine = 71
Hydrogen chloride = 36.5 n from 3.05 to 6.75



The second process step is aeration. Dry air is blown through the crude product for two hours to remove trace amounts of HCl and other impurities.

The third process step is a flash distillation. This is carried out under vacuum with a vacuum controller preset to give 40mm Hg absolute at the still pot. Heat is supplied through means of an external oil fired heater. Distillation must be carried out in the presence of lime. The residue product which contains lime, ferric chloride, and tars is generally drawn off to drums and may be sold as "Montars".

The fourth process step is to treat the distilled aroclor with Porocel, an alumina oxide product. The Porocel absorbs moisture and chloride ions to improve the electrical properties of the Aroclor.

The fifth process step is blending of Aroclor 1260 or 1254 with trichlorobenzene and adding glycidyl phenyl ether (phenoxyl propene oxide) which is a chloride scavenger. Trichlorobenzene increases the fluidity of the Aroclors without seriously harming the electrical properties.

Section B Chlorination

I. Biphenyl Feed Tank

A. Startup and Normal Operation

1. A fixed charge of 6400 lbs. of biphenyl is added to the biphenyl feed tank for all batches.

2. Charging Procedure:

   a) Check that steam is on the heating coil and that the temperature controller is operational.

   b) Add biphenyl. The tank overflow nozzle to Item 1-137 is set at 785 gallons or 6400 lbs. which is the set point.

B. Control and Alarm System

1. Steam is controlled by means of a temperature controller on the coil of the tank - Set at 100°C.

2. Reference is made to Drawing I-9, Loop No. TIC-202.

C. Emergency Shutdown

1. Shut down agitator.

2. Shut down feed.

D. Scheduled Shutdown

1. Examine coil and agitator to determine if any corrosion has taken place.

E. Miscellaneous

1. An agitator is required in the mix tank if flakes are melted and/or ferric chloride is added to this tank.

II. Chlorinator, Chlorinator Cooler, Chlorinator Circulating Pump

A. Startup and Normal Operation

1. Design Considerations:

a) Charges to the chlorinator are based on the batch system at WGK's Department 246. The design criteria for this unit is, 1) superficial vapor velocity is equal to the superficial vapor velocity in the WGK unit and, 2) liquid level over the sparger is equal to or greater than that in the WGK chlorinator.

b) A fixed charge of biphenyl is added to the chlorinator in order to prevent mistakes in charging. This charge must be in the chlorinator at the start of chlorination.

c) Manufacture of the liquid Aroclors is essentially the same. Batch data are listed below:

Type of Aroclor	1142	1148	1154	1160
Biphenyl charge	6450#	6450#	6450#	6450#
Chlorine charge(1)	9489#	12220#	15350#	19150#
Chlorination time	6 hrs.	7½ hrs.	9 hrs.	11 hrs.

(1) 99% available chlorine

Type of Aroclor	1142	1148	1154	1160
Crude Specific Gravity Hi	1.352	1.415	1.505	1.566
Crude Specific Gravity Low Temp.	1.341 65°C	1.405 65°C	1.495 65°C	1.555 90°C
Wt. of Crude Batch	11312#	12713#	14142#	16200#
Crude Density Temp.	10.5#/gal. 150°C	11.1#/gal. 150°C	11.8#/gal. 150°C	12.5#/gal 150°C
Batch volume	1080 gal.	1145 gal.	1200 gal.	1295 gal.
Theoretical Molecular Weight	257.7	292.6	326.6	369.5

d) The heat removal system is based on a heat evolution rate of 510 Btus/hour per pound of chlorine added. Normal evolution at the maximum chlorine input rate of 2015 pounds/hour of chlorine is 916000 Btu/hour with allowances being made for some radiation losses. Design rating of the external air cooled heat exchanger is 1,150,000 Btu/hour. The addition of a two speed motor to the recommended unit will give a turn down of 3 to 1 representing at the lower speed on input of 670 pounds/hour of chlorine. Batch circulation rate is 250 gallons/minute which allows for a complete batch turnover in 4.3 to 5.2 minutes.

A manual-automatic selector switch is installed on the panelboard to control the fan and select the motor speed.

e) The flow of chlorine to the chlorinator is controlled by a flow controller which allows manual adjustment of chlorine feed rate with automatic control. The input rate is varied through three levels during the chlorination period to keep entrainment losses to a minimum.

f) Chlorination temperature is held constant by a temperature controller which controls the opening of a shutter assembly located above the air-cooled exchanger.

g) An entrainment separator, Item 1-205, is installed at the top of the chlorinator to prevent aroclor from being lost from the chlorinator. The mist eliminator as proposed will eliminate 99% of all particles 3 microns and smaller. Vapor will pass through the unit.

h) Sparger design is based on a chlorine velocity of 50 feet per second, a chlorine rate of 2015 lbs/hour with a 1/4 inch hole size.

2. Chlorinator Operation

a) Charging Biphenyl

1. Close valve under chlorinator.

2. Check that chlorinator circulating pump is off and that discharge valve to blow tank is closed.

3. Check that 3" valve in the chlorinator vent line at top of chlorinator is open.

4. Check that 4" valve on the mist eliminator line is open.

5. Open charge line between biphenyl measuring tank and chlorinator. When charging is complete check that the biphenyl tank is empty.

6. Close valves between measuring tank and chlorinator. At this time open the 4" cap on the chlorinator and add one bottle of ferric chloride to the chlorinator (3 pounds).

7. Close the 3" valve in the chlorinator vent line.

b) Operation of Chlorinator

1. Check that cooler fan is off. Note the selector switch could be in the automatic position.

2. Align chlorinator valves so that the chlorinator pump will circulate from the bottom of the chlorinator to the top through the circulating pump and heat exchanger.

3. Turn the circulating pump on.

4. Set the chlorine integrator on the panelboard to a value corresponding to 80% chlorination. Set points for 80% chlorination for different aroclors are listed below:

   Basis: 6450# Biphenyl Charge

Aroclor	Set Point
1142	7500
1148	9700
1154	12300
1160	15300

5. Set the chlorine recorder controller at 1010 pounds/hour and put in automatic position.

6. Set temperature recorder controller at 150°C and put in automatic position. Normal batch temperature is 150°C. Manual position switch for 1200 RPM or 600 RPM fan speed depending on weather conditions. Fan will turn on when circulating temperature reaches 150°C.

7. Push chlorine start button.

8. Open hand valve on the chlorine line to the chlorinator.

9. The temperature in the chlorinator will rise to 120°C within 30 minutes after the flow is started.

   Take a specific gravity reading after two hours to check for a short charge batch. Gravity for all batches should be less than 1.200 at 65°C after two hours. If gravity is above 1.200 take frequent gravity readings to prevent over-chlorination.

10. All chlorinator batches should have the chlorine feed rate stepped up manually, but controlled automatically in the following time sequence:

First two hours	1010 lbs/hour
Next one hour	1335 lbs/hour
Continue to finish	2015 lbs/hour

11. When the batch is 80% finished the light on the annunciator panel will go on and the horn will blow.

12. Take a gravity at this point.

13. Continue to take gravities, as necessary, until the batch is at the desired finished gravity.

    Batches should be finished as follows:

Type of Aroclor	Hrs. of Chlorination Time	Batch to be Finished at Specific Grav.	Temperature °C
1142	6	1.341 to 1.352	65
1148	7½	1.405 to 1.415	65
1154	9	1.495 to 1.505	65
1160	11	1.555 to 1.566	90

14. When the batch has been chlorinated to the desired gravity:

    a) Shut off the chlorine hand valve.

    b) Open vent valve, close the off-gas valve.

    c) Turn fan off at the selector switch at the control board.

    d) Pump batch to blow tank. Make sure the chlorinator is empty by observing the level in the blow tank.

    e) While the batch is being transferred, stroke the chlorine flow controller and temperature controller by moving the selector switch on the instrument receivers to manual and turning the set points to maximum range than to zero.

    f) When the chlorinator is empty close pump away valves and promptly recharge with biphenyl.

B. Control and Alarm System

1. This is covered in detail on Instrument Schematic Drawing I-9 which covers the following loops:

   FRC-206 Chlorine flow to chlorinator
   TRCA-211 Temperature control, chlorinator cooler

   and Drawing I-13 which covers the following loop:

   PR-316 Chlorine pressure to chlorinator

2. Controls and alarms are set up as follows:

   a) Low temperature alarm for chlorination batch, 120°C, TRCA-211. The fan will shut off at 130°C and will not start again until the temperature reaches 150°C.

   b) High temperature alarm for chlorination batch, 175°C, TRA-200, Point 5.

   c) High temperature alarm for chlorination batch, 175°C, TRA-200, Point 4. If this temperature is reached then the flow of chlorine is shut off to the chlorinator. A stop may be required on the chlorine inlet valve if a sudden shut-off will affect the chlorine plant.

   d) If the circulating pump motor stops for any reason the chlorine inlet valve is closed. Pump shutdown is alarmed.

   e) An alarm will sound when a set amount of chlorine is added to the chlorinator.

C. Emergency Shutdown

1. Shut off the flow of chlorine to the chlorinator.

2. Shut down the circulating pump.

D. Scheduled Shutdown

1. Examine sparger to determine erosion or corrosion of inlet holes.

2. Examine brinks element to make certain seal pipe is intact.

III. Aeration

A. Startup and Normal Operation

1. Design Considerations:

   a) Product in the blow tank is blown with air from an external blower or other air source to remove HCl from the crude aroclor.

   b) Blowing operation may reduce the acid number, and/or moisture content.

   c) External coils are installed on the side of the tank with a steam regulator to control the product temperature.

   d) Sparger design is based on an air velocity of 50 feet per second, an air rate of 450 pounds/hour with a 1/4 inch hole size.

   e) HCl fumes are drowned in a water-gas scrubber of the Venturi type. The scrubber is based on 50 pounds of water required to drown 1 pound of HCl gas. Heat of solution of HCl gas is 865 Btu/pound.

2. Aeration Tank Operation

   a) Operation of Air Blower

      1. Start air blower and adjust outlet pressure to 10 psig.

   b) Operation of Drowning Jet

      1. Open water valve to scrubber.

      2. Adjust water valve to give a pressure gauge reading of 60 psig.

   c) Operation of Aeration Tank

      1. Energize the plate coil heating system. This is normally left on at all times.

      2. Open valve to sparger at top of tank and observe pressure gauge. A constant vibration of the pointer indicates that the aroclor is being properly blown.