MONSANTO COMPANY

ORGANIC CHEMICALS DIVISION

STANDARD MANUFACTURING PROCESS FOR AROCLORS - DEPARTMENT 246

Date: July 20, 1964

Author: D. E. Harris

Dept. 246 - Process Description

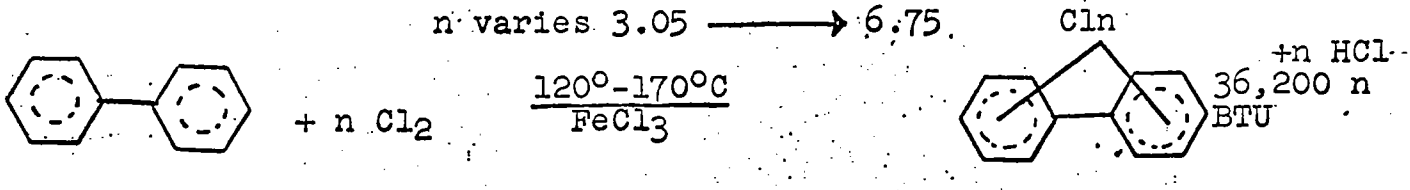
AROCLOR

SECTION I

SYNOPSIS OF PROCESS

Aroclor is a trade name used to designate 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 four digit numbers. The last two digit numbers represent the chlorine content (percentage by weight), and the first two numbers represent the Aroclor condition (crude or distilled). Crude Aroclors start with the prefix 11 whereas distilled Aroclors start with the prefix 12. For example, Aroclor 1242 is a distilled chlorinated biphenyl containing 42% chlorine.

The first process step in the manufacture of Aroclors is a chlorination reaction. Biphenyl (Ø-Ø) and chlorine (Cl_2) gas in the presence of a Ferric Chloride ($FeCl_3$) catalyst are reacted to form a chlorinated aromatic compound. Hydrogen chloride (HCl) gas is liberated during this reaction and piped to the muriatic acid department. The exact chemistry of biphenyl chlorination is unknown. Many isomers are formed. A schematic representation of biphenyl chlorination is as follows:



BIPHENYL	CHLORINE	AROCLOR	HYDROGEN CHLORIDE
M.W. = 154	M.W. = 71	M.W.=(254.1-395.6)	M.W.=36.5

DSW 200812

Dept. 246 - Process Description

AROCLOR

SECTION I

SYNOPSIS OF PROCESS (Cont'd.)

The second process step is the aeration and distillation of the crude Aroclor. Dry air is blown through the crude Aroclor for a period of two hours or more. Aeration removes most of the free hydrogen chloride. Distillation of crude Aroclors is done under vacuum automatically controlled with the controller pre-set between 30-50 mm Hg. ags. to obtain the best performance. Distillation of crude Aroclor separates residue, lime, ferric chloride, and tars from the finished product.

The third process step includes blending, filtration, and storage of Aroclors. Approximately 0.1 - 0.2% by weight of Attapulgus earth is added to the distilled Aroclor. The mixture is agitated and filtered to the finished product tank. The purpose of the Attapulgus earth is to absorb moisture and chloride ion to improve the electrical properties of the Aroclor. During the filtration of distilled Aroclor, 2 - 16 oz. samples of the finished product are taken to the laboratory for control analyses. If the control analyses pass specifications, the batch is transferred to a storage tank.

This process description will describe the production of Aroclors 1242, 1248, 1254, 1260, 1254 xylene mix, and 1262.

DSW 200813

Dept. 246 - Process Description

AROCLOR

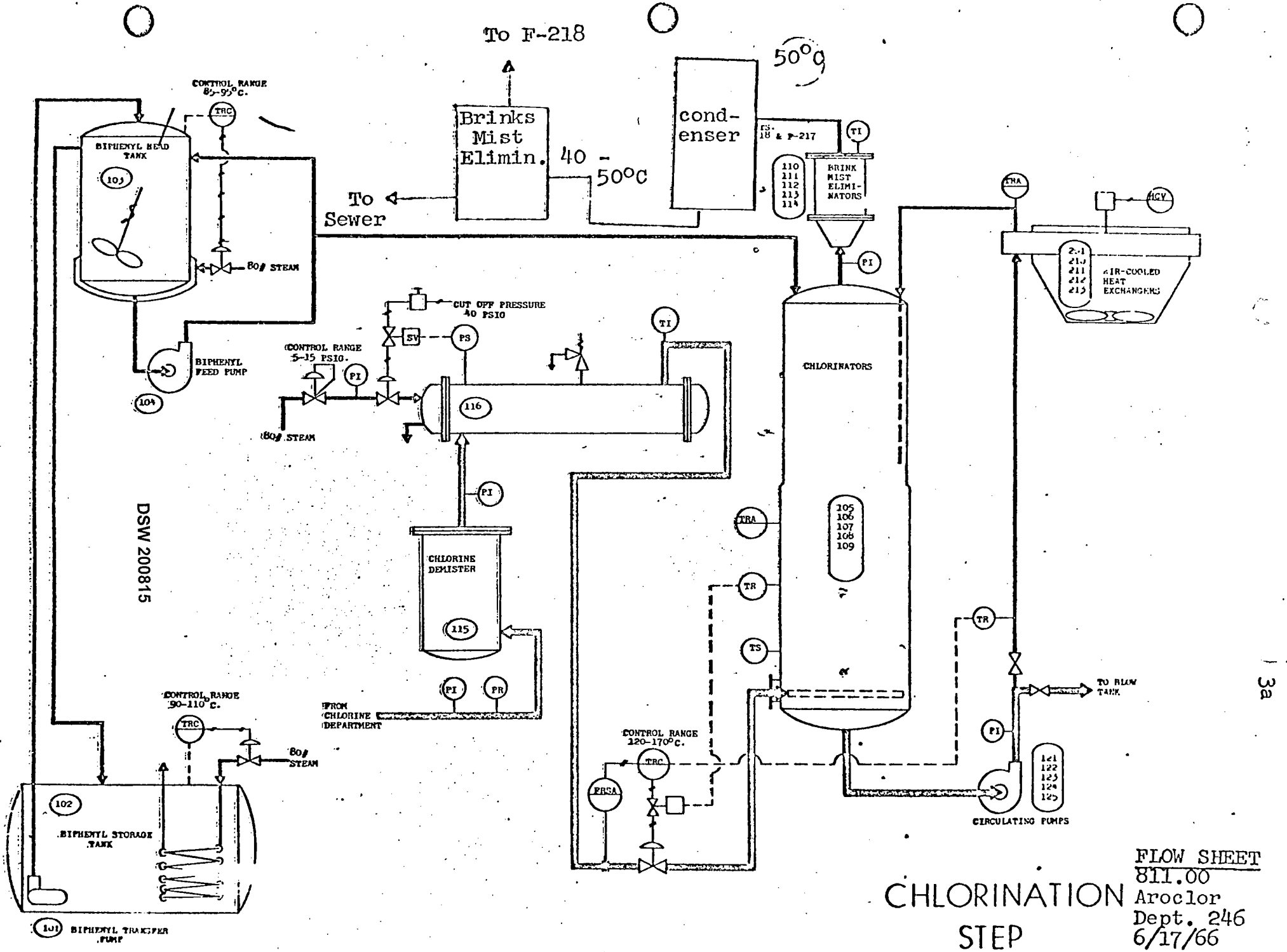
SECTION II

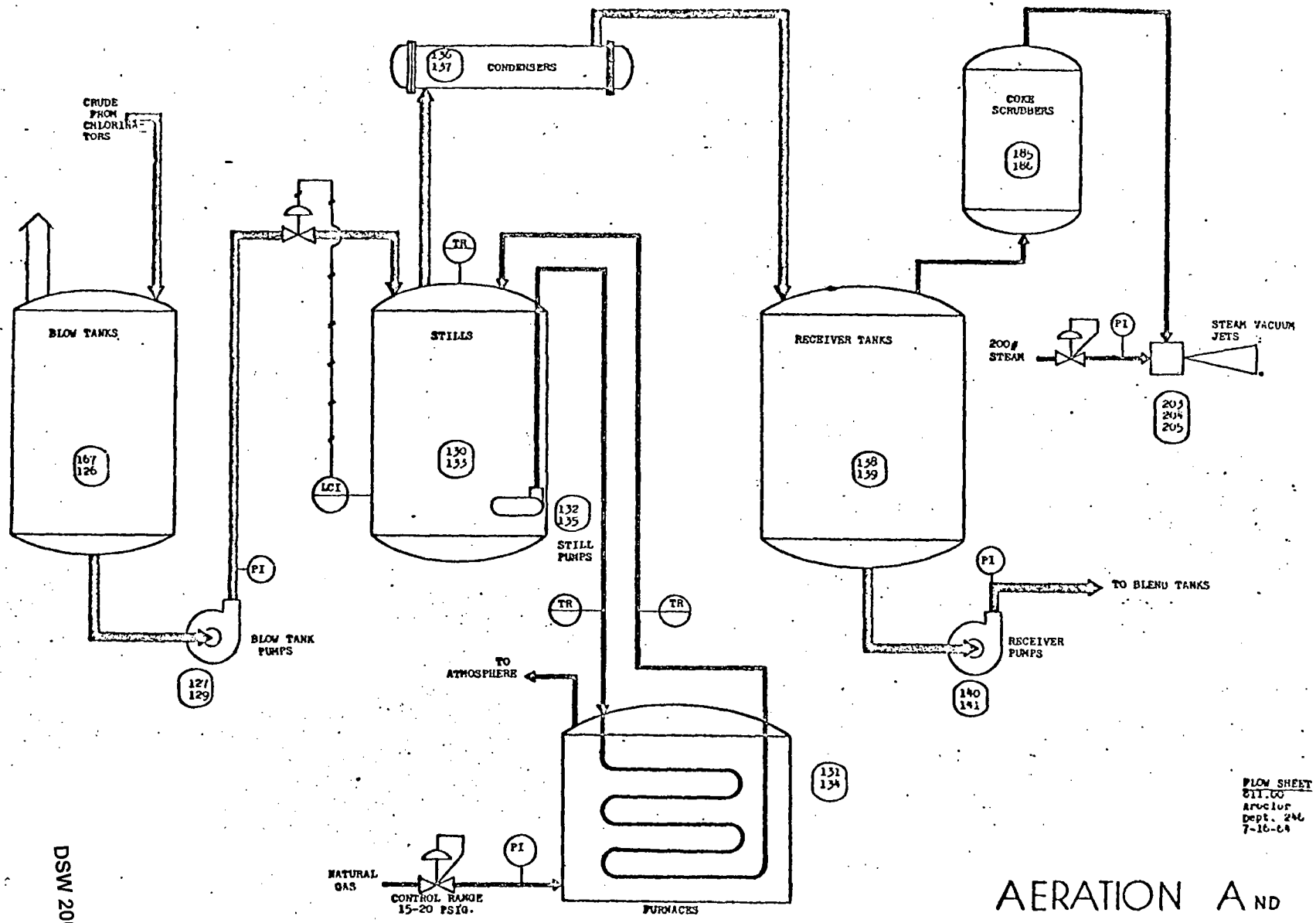
FLOW SHEETS

This section contains the following flow sheets.

1. Complete process flow sheet.
2. Chlorination Reaction Step
3. Aeration and Distillation
4. Blending, Filtration and Storage.

DSW 200814



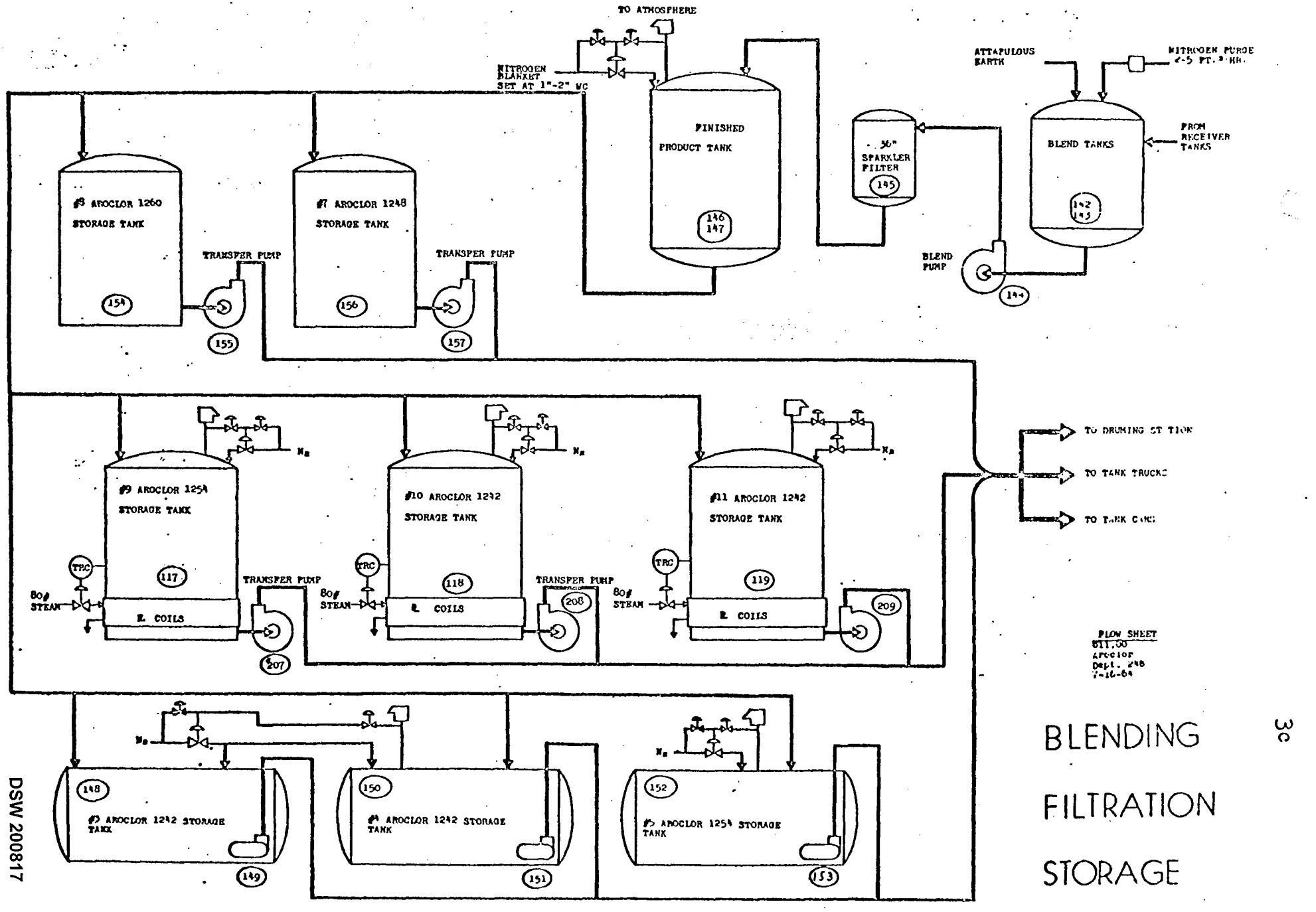


FLOW SHEET
611.00
Arochlor
Dept. 246
7-16-69

AERATION AND DISTILLATION

DSW 200816

3b



DSW 200817

STLCOPCB4058881

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
W.G.KRUMMRICH PLANT
RAW MATERIAL
SPECIFICATION

MATERIAL CODE
82601

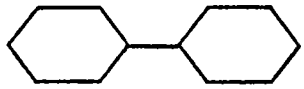
USING DEPTS.
246

SUPPLIER
ANNISTON PLANT

Issued DEC 8 1964
W. G. Krummrich Plant
by M. D. Daly

MATERIAL
DIPHENYL (BIPHENYL)

CHEMICAL FORMULA



MOL WT. 154.20

SUPERSEDES SPECS OF
4/6/51

SAMPLE FOR ANALYSIS
2 x 16 oz. W.M.Bottles

UNRESTRICTED INFORMATION

Routine Tests	Specification	Method Number
Appearance and Color	Light Yellow Cryst. Solid	10,252
Crystallizing Point	68.7°C, min.	10,298
GENERAL INFORMATION		
Distillation Range:		
First Drop to	2.5°C, max.	10,299
Dry Point	Must include 255°C within Range	

DSW 200818

Monsanto
ORGANIC CHEMICALS DIVISION
W. G. Krummrich Plant
RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERI
33500	Ferric Chloride Anhydride
PLANT: WGK	USING DEPTS.: 233
DATE EFFECTIVE: JAN 7 1966	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 SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
11/15/62	1 x 8 oz. W.M. Bottle	J. P. Prader

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
		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.	

DSW 200819

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

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE: 38800	MATERIAL: Lime, Hydrated
PLANT: WGK	USING DEPTS.: 222, 246
DATE EFFECTIVE: 7/21/66	SUPPLIER: Mississippi Lime Co.

APPROVALS

PRODUCTION: P. E. Heisler	RESEARCH: M. E. Gibbs
PROCUREMENT: C. A. Pratte	QUALITY CONTROL (Chief Chemist): W. J. Gresham

GENERAL DESCRIPTION — CHEMICAL FORMULA

$Ca(OH)_2$

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
6/20/63	1 x 16 oz. W.M. Bottle	

CHARACTERISTIC	LIMITS	METHOD
Appearance	Fine white or slightly gray powder, free from lumps or gritty material	10,188
Assay ($Ca(OH)_2$)	92.00%, min.	10,191
Carbonates as ($CaCO_3$)	3.00%, max.	10,193
Magnesium	0.50%, max.	10,192
Sulfates (as SO_3)	0.30%, max.	10,189

Supplier's certificate of analysis to be sent on day of shipment to:

Chief Chemist, Monsanto Co., Monsanto, Ill.

DSW 200820

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

Monsanto

ORGANIC CHEMICALS DIVISION
W. G. Krummrich Plant
RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATE
19409	Attapulgus Earth 200/UP
PLANT	**USING DEPTS.**
WGK	246
DATE EFFECTIVE	**SUPPLIER**
1-21-66	Mineral & Chemical Phillips Corp.

APPROVALS

PRODUCTION	**RESEARCH**
P. E. Heisler	W. D. Robinson
PROCUREMENT	**QUALITY CONTROL** *(Chief Chemist)*
C. A. Pratte	W. J. Gresham

GENERAL DESCRIPTION — **CHEMICAL FORMULA**

Clay

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
11/15/62	2 x 16 oz. W.M. Bottles	J. P. Prader

CHARACTERISTIC	LIMITS	METHOD

UNRESTRICTED INFORMATION

(R)	Volatile Matter (at 950°C.)	6 - 8%
(R)	Mesh Test:	
	Through USS 325	90%, min.

This material is not accepted without the supplier's analysis of the above properties.

DSW 200821

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

WGK - 314

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
43099	Nitrogen Gas
PLANT	USING DEPTS.
WGK	See Below (3)
DATE EFFECTIVE	SUPPLIER
FEB 18 1965	Air Reduction Sales Co.,

APPROVALS

PRODUCTION	RESEARCH
J.M.C., P.E.H., W.B.P., C.H.S.	R.W.B., H.W.K., M.A.T., W.D.R., J.A.S. W.F.S.
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
C.H.C.	W.J.G. C.H.R. J.S.M.

GENERAL DESCRIPTION — CHEMICAL FORMULA

Grade - O. P. Special Nitrogen

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
New	---	M. D. Dealy

CHARACTERISTIC	LIMITS	METHOD
Assay (1)	99.9%, min.	(2)
Oxygen	0.1%, max.	
Hydrogen	0%	
DewPoint	-76°F.	

Typical Analysis

Nitrogen	99.78%
Argon	0.12%
Oxygen	0.1%
Carbon Dioxide	<.00005%
Acetylene	<.00005%

(1) Nitrogen plus inert rare gases
(2) Any analyses to be obtained from supplier on request.
(3) B-215, C-216, 244, 246, 247, E-254, A-255, 256, 267, 270.

DSW 200822

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

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATER
64005	5° Xylene
PLANT: WGK	USING DEPTS.: A-255
DATE EFFECTIVE: 6/21/66	SUPPLIER: Chem Tech

APPROVALS

PRODUCTION: J.E. Smith	RESEARCH: C. Eaker (Nitro)
PROCUREMENT: A. H. Smith	QUALITY CONTROL (Chief Chemist): W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

$C_6H_4(CH_3)_2$

mol.wt. 106.16

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
7/12/63	1 x 16 oz.	J. P. Prader

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
(R) Appearance	Clear Colorless liquid	--------
(R) Sp. Gr. @15.5/15.5°C.	0.860 - 0.870	T-2516
(R) Distillation Range		T-2518
1st Drop	137°C., min.	
Dry Point	143°C., max.	
100% over in	5°C., max.	
Acidity	No free acid	-------
Sulfur compounds	Free of H_2S and SO_2	-------

Supplier's certificate of analysis to be sent on day of shipment to:

Chief Chemist, Monsanto, Company, Monsanto, Ill.

DSW 200823

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

Monsanto
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT		DEPT.
CHLORINE, Liqu		232
PLANT	PRODUCT CODE	USE
WGK	81059	Sales and Internal
DATE EFFECTIVE	SALES CODE	
JUN 29 1966	3060-000-06-03	

APPROVALS

PRODUCTION	RESEARCH
T. W. Dalton	N. F. Mueller
SALES	QUALITY CONTROL (Chief Chemist)
W. E. Ault	W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

MOL WT. 70.914

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
6/24/63	1 x 40 ml. Beaker 3/4 full	J. P. Prader

CHARACTERISTIC		LIMITS	METHOD
UNRESTRICTED INFORMATION			
(R)	Appearance	Clear heavy liquid	10,748
(R)	Color	Amber to greenish yellow	10,748
(R)	Moisture	.015%, max.	12,793
(R)	NVM	.01%, max.	12,793
Iron as:			10,688
	Fe.	*Report %	
	$FeCl_3.6H_2O$	Report %	

*When used in manufacture of ACl products, the specification on Iron as Fe is 20 ppm max. (H. V. Moss)

For use in Depts. 264, 258, 238, 243, 244 and 251.

DSW 200824

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

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
Krummrich and Anniston
FINISHED PRODUCT SPECIFICATION

PRODUCT CODE: 81101-82612
DEPT. OR JOB: WGK-246-Ann.-7
USE: Sales
SALES CODE: 1040-240-04/11/16-03/09

Issued
W. G. Krummrich Plant
By M D Sealy

PRODUCT (Trade Name): Aroclor 1242
TYPE: Non-Electrical Grade
PRODUCT (Chemical Name):
TYPE:

CHEMICAL FORMULA: Mixture

MOL WT.:

SUPERSEDES SPECS OF: 5-4-61
SAMPLE FOR ANALYSIS: 3 x 16 oz. W. M. Bottles

UNRESTRICTED INFORMATION

Routine Tests	Specification	WGK	Anniston Method No.
Condition	Clear	10084	G-1
Color, APHA	100 max.	10084	046
Sp. Gr. (25/15.5°C)	1.381 - 1.392	10114	052
Acidity (mg. KOH/g.)	0.010, max.	10087	050
Moisture (H_2O) (ppm.)	50, max.	10620	047
General Tests			
Distillation Range (ASTM D-20 Corrected)		10117	14-31-54
10% Distilled by wt.	325°C, min.		
90% " " "	360°C, max.		
Viscosity @100°F (SUS)	82-92	10086	049
Flash Point (°F)	338 - 392	10123	14-28-54
Fire Point	None	10123	14-28-54

DSW 200825

IN 84 (REV. 12/83)

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
Krummrich and Anniston
FINISHED PRODUCT
SPECIFICATION

PRODUCT CODE	DEPT. OR JOB
81103 - 82613	WGK-246-Ann.27

USE: Sales

SALES CODE: 1040-280-04/11/16-03/09

Issued 7/15/64
W. G. Krummrich Plant
By W D Scully

PRODUCT (Trade Name): Aroclor 1254
TYPE: Non-Electrical Grade

PRODUCT (Chemical Name):
TYPE:

CHEMICAL FORMULA: Mixture

MOL WT.:

SUPERSEDES SPECS OF: 5-4-61

SAMPLE FOR ANALYSIS: 3 x 16 oz. W. M. Bottles

UNRESTRICTED INFORMATION

Routine Tests	Specification	WGK Method No.	Anniston No.
Color, APHA	100, max.	10084	046
Sp. Gr. @65/15.5°C	1.495 - 1.505	10114	052
Acidity (mg. KOH/g.)	0.010, max.	10087	050
Moisture (H_2O) (ppm.)	50, max.	10620	047
General Tests			
Flash Point	None	10123	14-28-54
Viscosity @210°F (SUS)	44-48	10086	049

DSW 200826

MONSANTO COMPANY

ORGANIC CHEMICALS DIVISION

ANNISTON, ALABAMA

STANDARD MANUFACTURING PROCESS FOR BIPHENYL

Date: November, 1965

Author: B. O. Severson

STANDARD MANUFACTURING PROCESS

BIPHENYL

SECTION I - SYNOPSIS OF PROCESS

PART I - TUBULAR UNIT AREA

Biphenyl is manufactured in two general equipment areas: the pyrolysis area known as the tubular unit area, and the final product distribution and draw-off area known as the still area.

The manufacture of Biphenyl starts in a pyrolysis process in which benzene, under conditions for controlled rearranging, is converted into a polyphenyl mixture. This polyphenyl mixture which contains Biphenyl, three Terphenyl Isomers, Tri-phenylene, Quatraphenyls, higher polyphenyls, tars, carbon, and hydrogen, is made by the continuous pyrolysis of benzene in a direct fired tubular furnace. The product split is controlled by varying conditions of benzene feed, converter pressure, furnace gradient temperature, converter exit temperature, promoter, and per cent of recycle polyphenyl added to the benzene feed stream. The process is operated at conditions to provide the production demand split between Biphenyl and the Terphenyls under conditions that minimize quatraphenyl and coke lay-down, and increases on-stream time, tube preheater life, furnace efficiency, gas usage, and external retention tank life.

Each of the two furnaces and external piping is divided into two independent process streams. In effect, there are two tubular converters per furnace. To these furnace-converters a continuous stream of fresh and recycle benzene plus promoter is fed. The benzene feed first enters the vaporizer section in the top of the furnace. The vaporized benzene near 250°C. leaves the furnace and passes through the shells of a series of external preheaters. These preheaters increase the productivity of the furnace and lower the gas consumption by recovering heat from the polyphenyl-benzene product stream as it leaves the furnace through the tube side of these preheaters. The preheated benzene vapors, now raised to near 500°C. where cracking is initiated, are returned to the converter section of the furnace. The preheaters and associated external tankage is the most sensitive area for coke deposit and metal failure in the system. Operating techniques and conditions that reduce these problems are a part of the process.

The preheated vapors are returned to the top of the cracking or radiant section of the Selas furnace where, in 14 - 5"-40' Incoloy 800 tubes, the temperature is raised by the 88 burner radiant gas-fired furnace to an exit bulk temperature of from 700 to 750°C.

Synopsis of Process
Tubular Unit Area

Net conversion to polyphenyl is normally controlled between 5% and 20%. The profile temperature of the product stream is regulated by the firing conditions of each of four rows of burners with the objective of controlling the conversion rate through the converter (radiant) section.

The benzene-polyphenyl stream exits from the furnace into a partially insulated unbaffled sojourn pot which provides additional external cracking time for increased furnace productivity. The product stream flows through a carbon grinding tank and then passes into the tube side of the Hastelloy X bundles in the external preheaters. The benzene-polyphenyl stream temperature is reduced to near 360°C. as it raises the uncracked benzene in the shell side from 200°C. to near 500°C.

The benzene-polyphenyl stream leaves the furnace area and enters the adjacent stripping area where the stream is separated into a benzene-free polyphenyl bottoms and a recovered overhead benzene stream. The sequence of steps in the stripping operation starts with the hot benzene-polyphenyl stream passing through the shell side of a reboiler where it uses up most of its remaining super heat to the column bottoms which are circulated on the tube side of the reboiler. The product stream continues from the reboiler to the 4th tray of the 12-plate column. The 12 Glitsch Ballast tray column utilizes the super heat delivered at the reboiler to strip unreacted benzene which is condensed by passing through the tube side of a recovered water cooled condenser. The condensed benzene, 80-95% of the feed to the column, is collected in a reflux tank. From here the recycle benzene is returned by level control pumping to the furnace feed tank where it, with make-up benzene, is recycled back to the pyrolysis furnace. A benzene reflux stream is used to control the column temperature so that the desired amount of polyphenyl is maintained in the benzene overhead cut. The benzene-free polyphenyl still bottoms, held near 270°C., are pumped continuously to one or two 15,000 gallon crude polyphenyl storage tanks. These tanks also serve as feed tanks for the two still Biphenyl recovery system.

The benzene saturated by-product hydrogen, which is split off during the pyrolysis reaction, flows from the benzene reflux tank to the hydrogen compressor area. Here the gas is compressed in a two-stage reciprocating compressor to 300 psig. The compressor system serves two purposes; the primary being to recover benzene and return it to the feed tank, and the secondary to feed the by-product hydrogen into the natural gas stream which is firing the furnace.

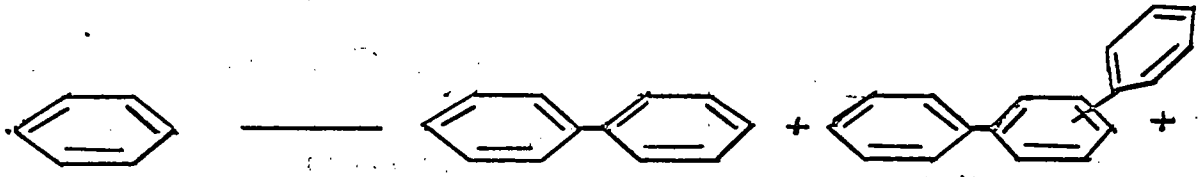
Synopsis of Process
Tubular Unit Area

The cooling water required for the benzene condenser, hydrogen compressor area condensers, and the air compressor condensers, is supplied by a two section Fluor cooling tower. Here the water is chemically treated to maintain the required pH chlorine concentration and chromate concentration.

The equipment in the benzene feed area, furnace area, stripping area, polyphenyl storage area, hydrogen compresser area, and the water recovery system, are all controlled by one operator from a central control room. The control room area includes the motor control section, air compressor section with back-up CO_2, and the instrument panel board and the laboratory area.

In summary, benzene under controlled temperature, pressure, and flow, is cracked into Biphenyl and Terphenyls in a direct fired tubular reactor. The polyphenyls formed are separated from the excess benzene in a stripping column. From the column, benzene-free crude polyphenyl is delivered to the distillation area where Technical Biphenyl is recovered and crude Terphenyls are passed on to the Terphenyl distillation area.

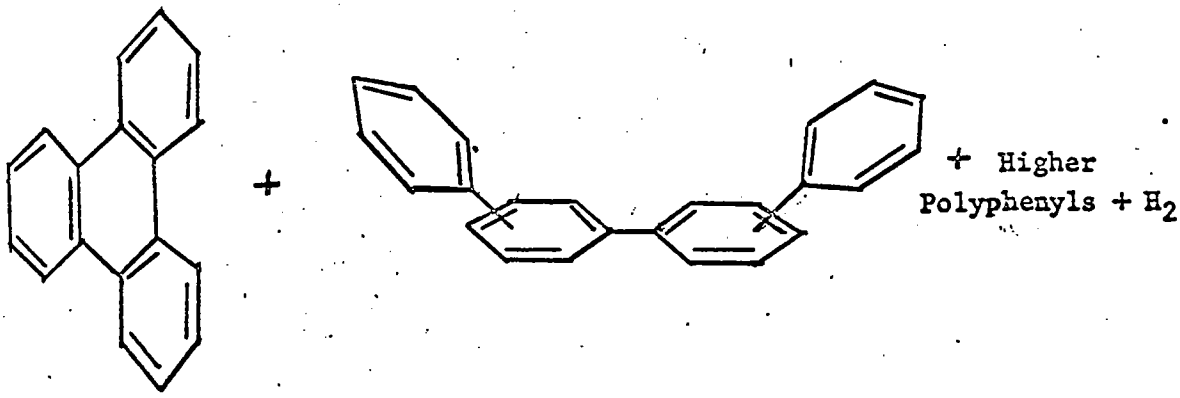
The equation for the reaction is:



78.1
Benzene

154.2
Biphenyl

230.3
Terphenyls



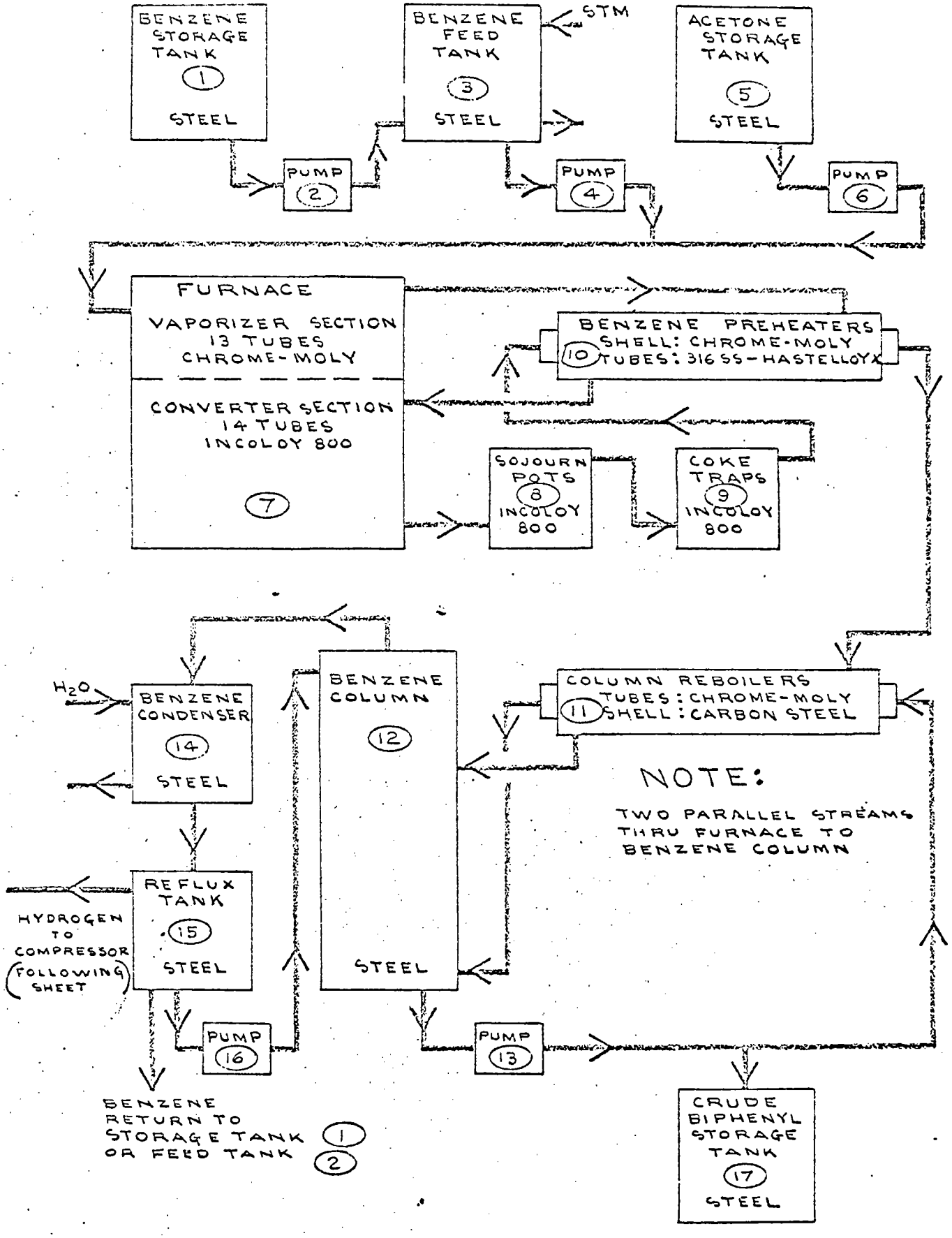
+ Higher Polyphenyls + H_2

228.3
Triphenylene

306.4
Quatraphenyls

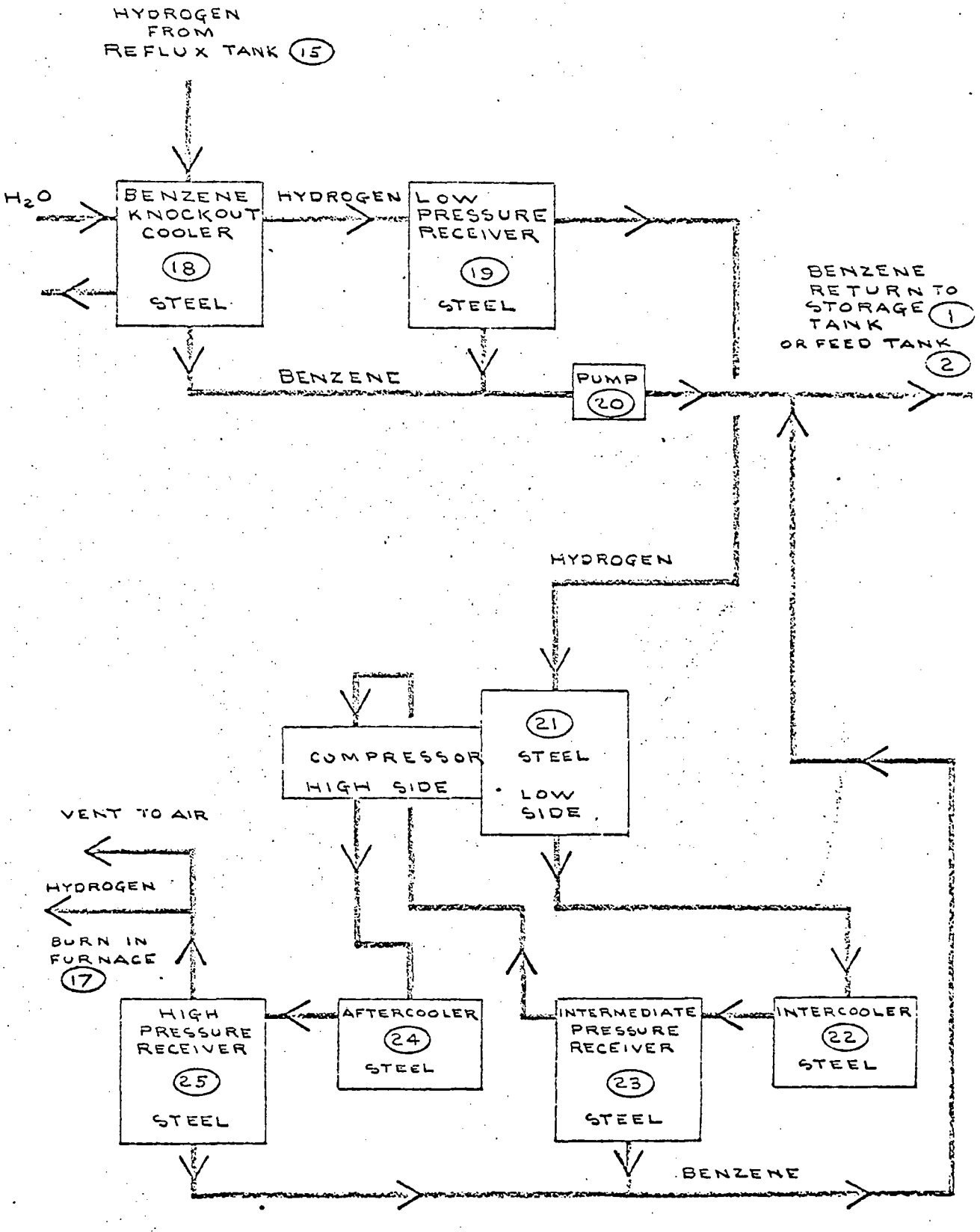
DSW 200830

BIPHENYL PROCESS FLOW DIAGRAM



DSW 200831

BIPHENYL PROCESS (CONTINUED)
HYDROGEN AND BENZENE RECOVERY FLOW DIAGRAM



DSW 200832

SECTION III. PROCESS IN DETAIL

The process for producing a Biphenyl-Terphenyl mixture by pyrolysis of benzene will be described in this section under the following headings:

A. Process Control

B. Critical Process

C. Benzene Receiving and Feed Operation

D. Furnace Operation

E. Benzene Stripping Operation

F. Hydrogen Compressor Operation

G. Recovered Water System

H. Control Room Operations

A. Process Control

The various control loops of this system have the ultimate function of controlling the output product stream at a predetermined Conversion. The net Conversion sets the ratio of Biphenyl to Terphenyl. The production rate of these two products is fixed by setting a net Conversion and a flow rate.

The benzene feed control system maintains a fixed level of make-up and recycle benzene in a feed tank. The benzene furnace feed from this tank is mixed with a metered amount of acetone promoter and fed to the furnace at a set pressure and flow rate.

The furnace control loops consists of the exit product stream temperature cascaded with the natural gas feed to the radiant burners, and an independent back pressure control loop. The gas delivered to the burners is varied to hold the exit temperature constant and the back pressure holds a steady converter outlet pressure.

The benzene stripping column is refluxed controlled by the 10th plate temperature. The stripping column conditions determine the percentage polyphenyl in the benzene recycle stream which, in turn, effects the ratio of Biphenyl and Terphenyl in the product stream. The level controls for the column sump and for the benzene reflux receiver are used to deliver a uniform feed of recycled benzene to the furnace feed tank, and benzene free crude polyphenyl to the two product receivers.

Shutdown protection is provided in the event of high or low benzol flows, high furnace stack temperature, high and low natural gas pressure and flow. Two annunciator alarm systems warn of out of normal limit conditions.

The hydrogen compressor system operation is controlled by a pressure-compressor unloader loop on the suction side of the compressors. Shutdown loops are provided for low suction pressure and high receiver levels to protect the system.

The recovered water system is controlled by a pressure-temperature loop that operates the two discharge pumps and two tower fan systems at the cooling tower. Pressure controls on the instrument air system operate the two instrument air compressors and a back-up CO_2 system.

B. Critical Process Variables

Any specific Conversion can be obtained by a great variety of different sets of conditions. The route makes no difference to the composition of the product. However, the effects of this different route on process efficiency and production costs vary widely. The selection of the proper range of this critical variable is important to proper operation.

There are five critical process variables in the polyphenyl production system which determine the percent of benzene that is converted to polyphenyl. This percentage is referred to as Conversion, and as this percent Conversion increases, the ratio of Biphenyl to higher polyphenyls decrease. Thus, as Conversion is raised when the demand for Biphenyl increases, the production of Terphenyls and Quatraphenyls increase at a faster rate than Biphenyl.

Two of the critical variables are: (1) benzene flow rate, which is referred to gallons per minute per pass, and (2) converter pressure as measured at the converter (radiant section) outlet. These two variables determine residence time and gas concentration. The residence time, the average time for a unit of benzene to pass through the converter section of the furnace, controls the conversion level. That is, while holding the other variables constant, Conversion will increase with increasing residence time. The normal range of benzene flow rates for current operations are from 35 to 50 gpm per pass. The normal pressure range for current operations is between 60 and 100 psig. The selection of these two variables is a compromise. Lowest unit gas costs can be obtained at the low benzene flows, however, low flame can cause excessive tube support temperatures and carbon laydown in the furnace and external piping. High benzene flows favor reduced Terphenyl production, best furnace life conditions at a higher gas and recovered water usage. The upper pressure is limited by the thickness and age of the tubing and cast return bends, and at the lower pressure, level is limited by the reduced productivity of the furnace.

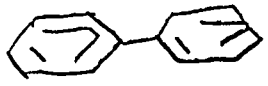
IN 54 MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION

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 ty. 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	

DSW 200835

This specification for internal use only

IN 100

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.

DSW 200836

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
ANNISTON, ALABAMA PLANT

RAW MATERIAL SPECIFICATION

PRODUCT CODE: 10950

METHOD IDENTITY:

SUPPLIER: CARBIDE & CARBON

Issued Sept. 11, 1957

STANDARDS DEPT.
Anniston, Ala. Plant

MATERIAL: ISOPROPANOL, 99%

CHEMICAL FORMULA:

By /s/W. B. Dunlap
W. B. Dunlap

MOL. WT.:

SUPERSEDES SPECS. OF:

SAMPLE FOR ANALYSIS: 1 x 16 oz. Narrow mouth bottle

UNCLASSIFIED SPECIFICATIONS

General Tests	Specifications
Specific Gravity (20/20°C)	0.7862 - 0.7876
Distillation (760 m.m.)	Shall be entirely distilled within 1°C range which shall include 82.3°C
Isopropanol (%)	99.5 min.
Acidity (%) (as Acid no.)	0.002 max. as acetic acid 0.019 max.
Dilution Test	Clear
Water (%)	0.5 max.
Non-Volatile (g/100 ml)	0.002 max.
Color (APHA)	10 max.
Odor	Non-residual
Suspended matter	Substantially free
Flash point (°F)	70

Specification furnished by Supplier May 18, 1956

DSW 200837

IN 55

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVIS.
ANNISTON, ALABAMA PLANT

RAW MATERIAL SPECIFICATION

PRODUCT CODE: 14700

METHOD IDENTITY:

SUPPLIER:

Issued Sept. 11, 1957

STANDARDS DEPT.
Anniston, Ala. Plant

MATERIAL: BENZENE - NITRATION GRADE (ASTM D-835)

CHEMICAL FORMULA:

By /s/W. B. Dunlap
W. B. Dunlap
Revised 11-18-65
To include typical values

MOL. WT.:

SUPERSEDES SPECS. OF: App. 1952

SAMPLE FOR ANALYSIS: 2 x 16 oz. NN Bottles

Characteristics	Limits	Typical Values
Appearance	Clear liquid with no free water or suspended matter	Clear
Acidity	No free acid	---
Acid wash test	Barrett 2 max.	---
Color	APHA 25 max.	5
Crystallizing point	5.30°C. min. wet basis	---
	5.39°C. min. dry basis	5.42
Distilling range:		
100% over in	1°C.	---
Range to include	80.1°C.	---
Specific gravity:		
60/60°F.	0.8820 to 0.8860	---
25/25°C.	0.8740 to 0.8780	---
Copper corrosion	Negative	---
Sulphur compounds	Free of H_2S and SO_2	---
Total Sulfur, ppm	200 Max.	1
Total Chlorine, ppm	---	1

DSW 200838

IN 64 MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT CODE - -
Dept. 120
METHOD IDENTITY - - -
SALES CODE - - -

Issued 11-18-65
Anniston Plant
By O. E. Dolin

PRODUCT (Trade name): Hydrogen
TYPE: By-product

PRODUCT (Chemical name): Hydrogen
TYPE: By-product

CHEMICAL FORMULA: Mixture

MOL WT. - - -

USE: Internal

typical values

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
2-11-60		100 cc Gas Burette

General Tests (made only on request)

Characteristics	Typical Value
Hydrogen (H_2), mole %	86.12
Methane (CH_4), mole %	5.83
Ethane (C_2H_6), mole %	0.90
Ethylene (C_2H_4), mole %	0.55
Propane (C_3H_8), mole %	0.08
Propylene (C_3H_6), mole %	0.17
Butanes (C_4H_n), mole %	0.02
Carbon dioxide (CO_2), mole %	0.33
Nitrogen and carbon monoxide (N_2&CO), mole %	2.16
Benzene (C_6H_6), mole %	3.84

DSW 200839

This specification is for internal use only

STANDARD MANUFACTURING PROCESS

BIPHENYL

APPENDIX K - BACKGROUND OF PROCESS AND ANNUAL REMARKS

Large scale commercial production of Biphenyl became feasible with the invention of the lead-pot process by Federal Phosphorus Company in 1927. These researchers recognized that the essential elements of successful conversion of benzol to Biphenyl were (1) preheat the vapors almost to reaction temperature, (2) quickly raise the vapor to reaction temperature, (3) remove and cool vapors immediately below reaction temperature. This was early accomplished by passing preheated benzol vapors through a bath of molten lead. Eventually 13 lead pot units were built at the Anniston Plant in 5 different expansions, with only minor changes in design. A significant improvement was made in 1954, with installation of a larger converter pot in No. 11 Biphenyl unit, giving longer sojourn time with consequent greater capacity, lower temperature, less carbon and longer life.

A significant improvement in the basic process resulted from use of isopropanol promoter in 1948, giving the same conversion at lower temperature, thus increasing equipment life. Use of promoter was discontinued due to corrosion of auxiliary equipment, then permanently restored in 1954. In 1965, acetone replaced isopropanol, giving higher conversions at lower raw material cost.

Research in 1927 had indicated that brief sojourn of benzol vapors in a heated tube would provide an alternate commercial process. Further research, particularly in 1950 and 1960, established the basis for a tubular reactor. The first tubular unit was installed at Anniston in 1961, the second unit in 1964. The second unit featured 4 additional tubes in the converter section to provide longer sojourn. The carbon trap was relocated to catch carbon dislodged from exterior piping. Preheaters were redesigned to increase vapor velocities on the shell side, and reduce carbon formation. These features successfully reduced maintenance and increased conversion. The benzene stripping column integrated with each furnace so completely removes benzol that a head cut is no longer required on the Biphenyl stills.

The original Biphenyl still was a direct-fired pot surmounted by a packed fractionating column. It was replaced in 1940 with a 20 tray bubble cap column and gas fired coil heater, and within the next two years this was duplicated to provide the present two stills. Originally operated as batch stills, both were revised in 1963 for semicontinuous operation with a boil-down every 24 hours, an improvement derived from complete removal of benzol in the stripping columns.

Biphenyl was marketed originally as a solid in 250 pound drums. In early 1930's the present flaker was installed. At present, Biphenyl leaves the plant as solid in drums and tank cars, and as flaked in 100 pound bags.

Liquid Hydrocarbons

3/1/67

	Anniston	UGK	
Forecast – 1971 – Total	46 m (+30% = 60 m)	43 m (+30% = 56 m)	
1242	31 m (+30% = 40 m)	23 m (+30% = 30 m)	
Others	20 m	26 m	
Capacity – Now – Total	44 – 48 m	30 m	
– Probable – Total	54 m	30 m → 40 m	w/ $70,000 expenditure } TSD estimates
		40 m → 45 m	w/ $120,000 " }
– 1242 – Now	28 m	} Depends on mix.	
– Others – Now	20 m		
– proposed			
1242	28 m	40 – 45 m	
Others	20 m +	30 m –	

DSW 200841

STLCOPCB4058905