

✓ Biphentyl, std. n. fg. proc.

*✓ Dept. 20
ANNISTON File 8
Folder #2*

MONSANTO CHEMICAL COMPANY
ANNISTON, ALABAMA

STANDARD MANUFACTURING PROCESS

FOR

BIPHENTYL

Date: September 17, 1957

Prepared by: R. L. Hedworth ✓
Supervisor: Jack Clegorn
Production Supt: J. E. Crouch

Copy No. 5

DEFENDANT'S
EXHIBIT

9

8-31-04 949

*Reproduced by L.H. Wfg. Proc. Control
Anniston Plant by L.J. Anderson*

DSW 048614

MWV 018257

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S/ Safety Committee 8/14/64

EXPERIMENTAL COMPANY
ANNISTON, ALABAMA

27 Jul 64

ITERATIVE AMENDMENT 1

To

BIPHENYL STANDARD MANUFACTURING PROCESS

Of

17 Sep 57

TITLE: Test Acetone as Promoter in Biphenyl Tubular Unit.

OBJECTIVE: This amendment provides for a six month test of the use of Acetone as a promoter in the Biphenyl Tubular Unit process.

PRESENT PRACTICE:

Isopropanol is presently used as the promoter to help convert benzene to Biphenyl.

PROPOSED CHANGE:

Acetone would be substituted for Isopropanol. Initial test would be with gram lot quantities using a statistically designed test to compare promoting characteristics of Acetone versus Isopropanol. Long term test will be carried out when short term test confirms previous findings.

JUSTIFICATION:

Of 24 promoters tried by Research, Acetone is the best with Isopropanol second best. Under the existing furnace conditions Isopropanol pyrolyzes to form Acetone. Research data indicates a 10% increase in conversion using Acetone instead of Isopropanol.

Cost differential amounts to \$5000 per year B.T. savings if Acetone is used.

DSW 048616

MWV 018259

27 Jul 64

REVIEW OF DATA:

1. TSD Progress Report 3701298, No. 1, 30 Jun 64 - Analysis of Statistically designed test data shows Isopropanol increases its effect on conversion with increasing temperature. Pyrolysis of IPA forms Acetone at furnace conditions which is a better promoter than IPA.

2. Benzene Pyrolysis in a Laboratory Tubular Reactor - Report No. 2772, 5 Apr 61, by Z. Borsch, A. Z. Lipman and W. D. Robinson.

Nine promoters tried with Acetone best and Isopropanol second best.

3. Biphenyl, Tubular Process, Report No. 2491, 23 May 50, by J. E. Saunders and R. J. Slocumbe.

Acetone found to be effective promoter.

4. Diphenyl from Benzene, Final Report 117-881, 5 Oct 35 by C. Conover and A. E. Huff.

Of 17 promoters tried, Acetone best and Isopropanol second best.

SAFETY:

No additional precautions are needed for using Acetone as the promoter.

The short term drum testing will provide the proper grounding accessories to assure safe handling of the Acetone.

Existing spec on Acetone shows less than 0.002% Acetic Acid which should not increase corrosion. Water content is about 0.2% which should not appreciably increase Acetic Acid formation. Long term test will check this.

QUALITY:

The use of Acetone should have no effect on the quality of Biphenyl-Santovan produced.

EXTENT OF DEMONSTRATION:

The test period will be six months.

Originated by:

V. R. Haupt

DSW 048617

MWV 018260

Revisive Amendment 2
Biphenyl Manufacturing Process

- 3 -

27 Jul 64

APPROVAL:

Department Supervisor:

B. O. Severson
B. O. Severson

8/7/64
Date

TSD Superintendent:

L. C. Fahrmeister
L. C. Fahrmeister

8-7-64
Date

Production Superintendent:

R. G. Murphy
R. G. Murphy

8/7/64
Date

Plant Manager:

J. R. McClain
J. R. McClain

8/10/64
Date

Research Group Leader:

S/ R. W. Bucknell
R. W. Bucknell

8/17/64
Date

Research Manager:

S/ R. L. Hubbard
R. L. Hubbard

8/14/64
Date

Tech. Production Manager:

S/ J. A. Mullendore
J. A. Mullendore

8/17/64
Date

DSW 048618

MWV 018261

MEMORANDUM

Inter Office Correspondence

From Location: Anniston, Alabama

Date : 27 Jul 64

Subject : EXTENSION OF TESTING PERIOD

Reference : TENTATIVE AMENDMENT NO. D TO THE
STANDARD MANUFACTURING PRACTICES FOR
BIPHENYL (Sept. 17, 1957) Issued
29 Jan 64

TO : J. R. McClain
R. C. Moody
L. C. Talmaster
J. O. Severson
D. Danks - G. O.
D. E. Reamer - G. O.
M. L. Hubbard - So. 2nd
R. W. Bucknall - So. 2nd
Central Technical File - So. 2nd

Testing of Semi-Continuous Distillation of Crude Biphenyl is incomplete. See Crude Biphenyl Distillation Report No. 570:26:4 dated 1 Jul 64 for outline of the testing program. An extension of the normal six months acceptance or rejection period for Tentative Amendment D is requested. Should you have any objection, please let it be known by 7 Aug 64 as this is the end of the six month period.

[Signature]
J. R. Haupt

VRL:hd

DSW 048619

MWV 018262

Distribution:
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SAFETY REVIEW COMMITTEE

S/ H.L.H. 2-5-64

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CONFIDENTIAL

MORRANTO CHEMICAL COMPANY
AHLSTON, ALABAMA

29 Jan 64

TENTATIVE AMENDMENT D

To

BIPHENYL STANDARD MANUFACTURING PROCESS

OF

17 Sep 57

RETAIN IN
FILES

☐ 6
Nos.
☐ 2

TITLE:

TESTING OF SEMI-CONTINUOUS DISTILLATION OF CRUDE BIPHENYL.

OBJECTIVE:

This amendment cancels Tentative Amendment C and provides for a six month test of semi-continuous distillation of crude Biphenyl to yield specification grade Biphenyl and Biphenyl "free" Santowax C.

PRESENT PRACTICE:

Crude Biphenyl was formerly purified by batch distillation. Tentative Amendment C allowed the testing of continuous distillation. Continuous distillation did not yield Biphenyl "free" Santowax C and, therefore, a semi-continuous distillation of Biphenyl is carried out at present.

SUGGESTED CHANGE:

The stills will be fed on the eighth tray from the top of the column continuously until the still pot fills. Then a tails fraction will be removed leaving Biphenyl "free" Santowax C to be transferred for further purification.

JUSTIFICATION:

Semi-continuous distillation increases production capacity and yields a higher purity Biphenyl than that of batch distillation.

SOURCE OF DATA:

See TSD Progress Report 370:26, No. 2, entitled "Continuous Distillation of Crude Biphenyl" for details on semi-continuous distillation.

DSW 048620

MWV 018263

Tentative Amendment D
Biphenyl Manufacturing Process

- 2 -

29 Jan 64

PRECAUTIONS:

No additional precautions are needed for semi-continuous distillation above that of batch distillation.

QUALITY:

Semi-continuous distilled Biphenyl has a higher crystallising point and a lower color (Gardner scale).

EXTENT OF DEMONSTRATION:

The test period will be six months.

Originated by

W. R. Haupt

APPROVAL:

Department Supervisor:

B. O. Severson

2/3/64
Date

Production Supervisor:

R. G. Moody

2/3/64
Date

Plant Manager:

J. R. McClain

2/3/64
Date

Research Manager:

S/ H. L. Hubbard
H. L. Hubbard

2/6/64
Date

Technical Production Manager:

S/ D. B. Hosmer
D. B. Hosmer

2/11/64
Date

Research Group Leader:

S/ R. W. Bucknell
R. W. Bucknell

2/7/64
Date

DSW 048621

MWV 018264

MONSANTO CHEMICAL COMPANY

Inter-Office Correspondence

FROM LOCATION : Anniston, Alabama CC:

DATE : March 18, 1963

SUBJECT : Extension of Testing Period

REFERENCE : TENTATIVE AMENDMENT NO. C TO THE
STANDARD MANUFACTURING PROCESS FOR BIPHENYL
(September 17, 1957) Amendment C issued 10/12/62

TO : J. R. McClain
2. G. Moody
J. C. Larkin
E. O. Sorenson
J. F. Scickley - G. O.
D. B. Gosmar - G. O.
H. L. Hubbard - So. 2nd
W. D. Robinson - So. 2nd
Central Technical File - So. 2nd - *Russell*

Keep This Copy for

Delays in installation and production requirements have caused a delay in the completion of the Continuous Biphenyl Distillation Study. Those tests made to date have been highly successful, but have been hampered by continued mechanical problems. An extension of the normal six months acceptance or rejection period for Tentative Amendment C is requested. Should you have any objection, please let it be known by April 15th. This date is the end of this six month period.

[Signature]
W. R. Haupt

VRE:kd

DSW 048622

IN 10

MWV 018265

CONFIDENTIAL

SAFETY REVIEW COMMITTEE

Master copy signed by

Safety Committee

10/22/62

cc: J. R. McClain
R. C. Moody
J. C. Larkin
B. O. Severson
V. R. Haupt
D. B. Homer - G. O.
H. L. Hubbard - So. 2nd
W. D. Robinson - So. 2nd
Central Technical File - So. 2nd
J. F. Stickley - G. O.

MONSANTO CHEMICAL COMPANY
ANNISTON, ALABAMA

October 12, 1962

TENTATIVE AMENDMENT NO. C

TO THE STANDARD MANUFACTURING PROCESS

FOR

BIPHENYL

September 17, 1957

TITLE: TESTING OF CONTINUOUS DISTILLATION OF CRUDE BIPHENYL.

OBJECTIVE: This amendment covers the testing of continuous distillation of crude Biphenyl to yield specification grade Biphenyl and Biphenyl "free" Santowax C.

PRESENT PRACTICE:

Crude Biphenyl is presently purified by means of a batch distillation. Four fractions are removed; benzene, heads (benzene and Biphenyl), Biphenyl, tails (Biphenyl and Terphenyls), and Santowax C as residue. The heads and tails fractions are recycled from batch to batch.

TEST:

The test will be carried out in the two existing distillation columns. The first column will separate benzene as overheads and Biphenyl, Santowax C as bottoms. The second column will produce Biphenyl overhead and Santowax C as bottoms. The specific goals of this test are:

1. To prove the feasibility of producing specification grade Biphenyl on a continuous basis.
2. To prove the feasibility of producing Biphenyl "free" (less than 1%) Santowax C on a continuous basis.

Specific details of the test may be found in a memorandum entitled "Operating Instructions for Biphenyl Continuous Distillation Test", authored by V. R. Haupt.

DSW 048623

MWV 018266

Tentative Amendment No. C
To The Standard Mfg. Process
For Biphenyl

- 2 -

October 12, 1962

The test period will be less than one week and any off-grade Biphenyl and Santowax C will be reworked to prevent contamination of related products.

It is requested that this amendment be approved permitting this study.

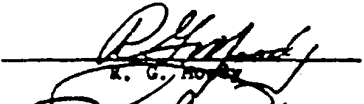

V. R. Haupt

APPROVALS: Department Supervisor:


S. O. Severson

15 Oct 62
Date

Production Superintendent:


K. G. Hovde

10/15/62
Date

Research Group Leader:


W. D. Robinson

10/15/62
Date

Asst. Director of Research:

Master copy

S/H. L. Hubbard

H. L. Hubbard

10/22/62

Date

DSW 048624

MWV 018267

WATER_PCB-SD0000051633

STANDARD MANUFACTURING PROCESS
FOR
BIPHENYL

TABLE OF CONTENTS

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DSW 048625

MWV 018268

SECTION A

SYNOPSIS OF PROCESS

Biphenyl is produced by the pyrolysis of Benzene. The process used in the manufacture of Biphenyl is commonly known as the Lead Pot Process. The equipment consists of a vaporizer coil, three stainless steel lead filled gas fired pots and two fractionating columns, all in series. Benzol is continuously vaporized in the vaporizer coil and heated to the pyrolysis temperature as it is bubbled through the molten lead in the three pots. The unreacted Benzol and the reaction products, Biphenyl, Terphenyl and Hydrogen, pass through two orifice plates and into the stripping column. The superheat in the gas drives the unreacted Benzol up the column, with the Hydrogen, where it is condensed and returned to the feed tank. The Hydrogen, saturated with Benzol, is cooled and compressed to remove the remainder of the Benzol. This Benzol is recycled through the process with fresh Benzol. The underflow, flows by gravity to a crude Biphenyl storage tank.

The units are operated between 10 and 30% conversion by regulating the converter temperature between 750° and 810°C. The higher temperatures giving the higher conversion.

The pot pressures range from 60 to 100 psig on the No. 1 Preheater, to 30 to 70 psig on the converter. The higher pressures are encountered when the units are nearly plugged.

The crude Biphenyl, which is approximately 75% Biphenyl, 15% Terphenyl, and 10% Benzol is charged batch-wise to a fractional distillation unit and the Biphenyl is distilled off. Heat for the distillation is provided by circulating the charge through a coil in a gas fired furnace. Four distillation cuts are made: (1) a Benzol cut, to recover the Benzol that was left in the underflow from the first column, (2) a heads cut which is a mixture of Benzol and Biphenyl, (3) a Biphenyl cut and, (4) a tails cut which is a mixture of Biphenyl and Terphenyl.

The heads and tails cut are recycled to the next distillation charge.

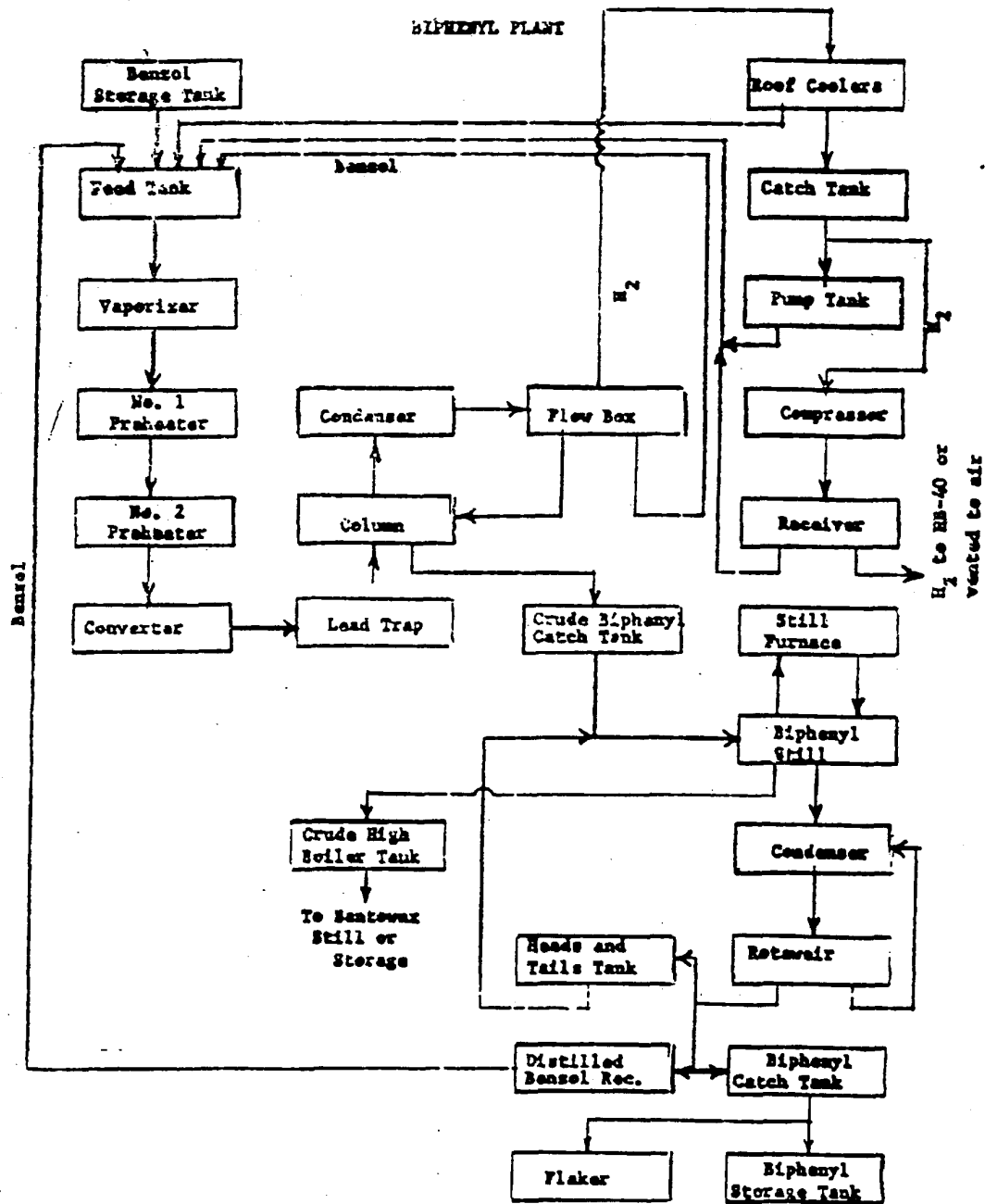
The still residue, Santowax C, consists essentially of Ortho, Meta, and Para Terphenyls and certain higher boiling compounds. It is pumped to storage for use in the Santowax R process.

DSW 048626

MWV 018269

FLOW SHEET OF MATERIALS

BIPHENYL PLANT



DSW 048627

MWV 018270

SECTION C

EQUIPMENT

There are 12 Biphenyl units of approximately equal capacity. Each unit consists of a pipe coil vaporizer located in the flue gas exhaust stack, three stainless steel pots partially filled with molten lead and suspended in a firebrick setting for burning natural gas, and a fractionating column with a condenser and split flow box. The pots are 24" O.D. x 72" long, with a dished bottom.

The units are numbered 2 through 13, there having been a No. 1 unit at one time. The present number of units were erected in five installments, Nos. 2 and 3 having been erected at different times, Nos. 4 and 5 together, Nos. 6, 7, 8, 9 and 10 together, and Nos. 11, 12 and 13 together. These groups are all slightly different in some details. There are, however, only two fundamentally different types of units. Nos. 4 and 5 differing from the rest in that one of the preheater pots is replaced by a shell and tube heat exchanger. Although the heat exchanger effected a gas savings as predicted, it proved impractical due to carbon and tars fouling the tubes, and the idea was abandoned on the subsequent units.

A larger converter pot (sojourn pot) has been installed on No. 11 unit to provide additional sojourn time. This additional time results in a lower temperature for a given conversion as compared to the other units. The heat input to the unit remains at a maximum and this extra heat is utilized by the addition of more Benzol feed to the unit.

The reduced pyrolysis temperatures made possible by the larger pot result in:

1. A 10-15% production increase,
2. Reduction in gas consumption per pound of Biphenyl produced,
3. Reduction of pot failures and maintenance cost,
4. Reduced carbon formation, thus permitting better operating efficiency of the unit,
5. Reduced lead carry over has been observed on the unit equipped with the sojourn pot.

Drawings which describe the equipment used in this process are listed in Phosphate Division Engineering Department Report No. 117.

DSW 048628

MWV 018271

SECTION D

RAW MATERIALS

The raw materials listed below are used in the production of Biphenyl.
The following pages are specification sheets for these raw materials.

Benzol

Lead

Isopropyl Alcohol

DSW 048629

MWV 018272

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
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 W. B. Dunlap
W. B. Dunlap

VOL. WT.
78.108

SUPERSEDES SPEC. OF
App. 1952

SAMPLE FOR ANALYSIS
2 x 16 oz. NN Bottles

UNCLASSIFIED SPECIFICATIONS

GENERAL TESTS

ASTM METHODS

Appearance	Clear liquid with no free water or suspended matter	-----
Acidity	No free acid	D-847
Acid wash test	Barrett 2 max	D-848
Color	APHA 25 max	D-853
Crystallizing point	5.30°C min. wet basis 5.39°C min. dry basis	D-852
Distilling range: 100% over in	1°C	D-850
Range to include	80.1°C	(Using Bunsen Burner)
Specific gravity: 60/60°F	0.8820 to 0.8860	
25/25°C	0.8740 to 0.8780	
Copper corrosion	Negative	D-849
sulphur compounds	Free of H ₂ S and SO ₂	D-853
Total sulfur	0.020% Max.	D-90

DSW 048630

MWV 018273

☐ IN 11 **MONSANTO CHEMICAL COMPANY**
ORGANIC CHEMICALS DIVISION
ANNISTON PLANT
**RAW MATERIAL
SPECIFICATION**

PRODUCT CODE

35400

METHOD IDENTIFY

SUPPLIER

National Lead Co.

Issued Sept. 11, 1957

STANDARDS DEPT.
Anniston, Ala. Plant

MATERIAL

Lead

CHEMICAL FORMULA

Pb

By W. B. Dunlap
W. B. Dunlap

MOL. WT.

207.21

SUPERSEDES SPECS. OF

New

SAMPLE FOR ANALYSIS

UNCLASSIFIED SPECIFICATION

<u>General Test</u>	<u>Imp. Anal. Res.</u>
Ag (%)	0.01
Cu (%)	0.06
Bi (%)	None
Zn (%)	0.0001
Fe (%)	0.0001
As, Sn, Sb (%)	0.0002
Cd (%)	0.0002
Co, Ni (%)	0.0046
Assay (% Pb by difference)	99.92

Basis of Specification: Suppliers typical analysis of product (5/25/56)

DSW 048631

MWV 018274

IN 11 MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
ANNISTON, ALABAMA PLANT
RAW MATERIAL
SPECIFICATION

MATERIAL ISOPROPANOL, 99%

CHEMICAL FORMULA

PRODUCT CODE

10950

METHOD IDENTITY

SUPPLIER

CARBIDE & CARBON

Issued Sept. 11, 1957

STANDARDS DEPT.
Anniston, Ala. Plant

W. B. Dunlap
W. B. Dunlap

MOL. WT.

SUPERSEDES SPEC. OF

SAMPLE FOR ANALYSIS

1 x 16 oz. Narrow mouth bottle

UNCLASSIFIED SPECIFICATIONS

General Tests

Specific Gravity (20/20°C)
Distillation (760 m.m.)

Isopropanol (%)

Acidity (%)
(as Acid no.)

Dilution Test

Water (%)

Non-volatile (g/100 ml)

Color (APHA)

Odor

Suspended matter

Flash point (°F)

Specifications

0.7862 - 0.7876

shall be entirely distilled within
1°C range which shall include
82.3°C

99.5 min.

0.002 max. as acetic acid

0.019 max.

Clear

0.5 max.

0.002 max.

10 max.

Non-residual

Substantially free

70

Specification furnished by Supplier May 18, 1956

DSW 048632

MWV 018275

SECTION II

PROCESS IN DETAIL

In production

Biphenyl has been manufactured by the lead pot process since 1923 at Anniston. The process is essentially the same as when it was first developed. The only variation being the use of a conversion promoter and a higher operating pressure.

Process investigation has pointed out many of the variables which affect the production of Biphenyl and the by-product Santowax. For instance, it has been determined that a balance between Biphenyl and Santowax production can be maintained over a wide range of requirements due to the relationship between the percent conversion and the relative amount of Biphenyl to Santowax produced. That relationship being:

$$1.058 (x) (\text{Percent Conversion}) = \text{Percent Santowax (Terphenyl)} \\ \text{in the crude Biphenyl}$$

The 1.058 is referred to as the sigma (Σ) factor. This sigma factor remains relatively constant over the operating range of the process, consequently, it was possible to develop the Biphenyl operations chart which shows the relationship between Biphenyl production, Santowax production, the percent conversion, and the Benzene feed rate.

Another important development in the manufacture of Biphenyl was establishment of the relation between the percent conversion and the sojourn time at different operating temperatures. This relation brought about an evaluation of optimum sojourn time which resulted in a larger converter pot, commonly known as the sojourn pot, (now being demonstrated) and a higher operating pressure. Both have permitted increased feed rates and lower operating temperatures for a given conversion rate.

The actual relation between temperature, conversion and sojourn time has not been determined when a conversion promoter is used. The sojourn time, temperature, percent conversion chart is presented to pictorially show the general relation of these factors.

To summarize - three factors affect the amount of Benzene that is converted per pass through the unit, they are: (1) temperature, (2) sojourn time and, (3) the conversion promoter. The relative amount of Biphenyl production to Santowax production can be changed by changing the percent conversion. That is--at high conversion rates more pounds of Santowax are produced per pound of Biphenyl than at some lower conversion rate. The conversion rate is controlled by changing the temperature within the pots.

DSW 048633

MWV 018276

From Benzol is added continually to the feed tank from the Benzol storage tank. Isopropyl Alcohol is added to the Benzol feed in amounts varying between zero and 0.5% to promote the conversion (see comments). Normally 0.15% is added to the Benzol as it is pumped through automatic control valves to the individual units. The feed then passes through a coil in the bottom of the column where the liquid is preheated, and to a coil in the flue gas exhaust stack where the liquid Benzol is vaporized. The Benzol vapor is bubbled through molten lead in the two preheater and converter pots where it is heated to the pyrolysis reaction temperature. After leaving the converter, the Benzol and conversion products pass through a lead trap for removing entrained lead and then enter the bottom of the fractionating column. The temperature in the converter pot is usually controlled between 750 - 810°C. The temperature needed for the desired conversion changes as the pots begin to plug with carbon due to the higher pressures and resulting longer sojourn time in the pots.

Every 30 to 90 days the carbon is physically cleaned from the distributor and vapor lines. (See attached table for feed rates, reflux, temperatures.)

The super-heat in the vapors from the converter is utilized in a fractionating column to effect a partial separation of the Benzol from the conversion products. The column top is maintained at a temperature of 100°C (at higher temperatures some Biphenyl begins to come over) by refluxing the condensed Benzol vapors back to the top plate. The units are usually operated with a 60-70% reflux, with 90% being set as a maximum. If 90% reflux does not cool the vapors, leaving the top plate of the column below 100°C, then it is necessary to reduce the feed. The conversion product and some Benzol is condensed in the column and allowed to overflow from a carbon trap on the bottom of the column to a crude Biphenyl catch tank for storage prior to batch distillation. The overhead vapors from the column are cooled by a condenser and the liquid Benzol drained to a rotavair for splitting the flow into reflux and recycle Benzol. The recycle Benzol flows to the Benzol feed tank by gravity.

The by-product Hydrogen, saturated with Benzol vapor flows from the condenser and rotavair, is collected in a common header at a pressure of approximately 3 psig and passes through a series of coolers where some of the Benzol is condensed. This Benzol runs by gravity back to the feed tank. From the coolers the saturated Hydrogen gas is passed to a collecting tank in which more of the Benzol condenses out and drains to the Benzol pump tank. This pump tank is located in the compressor room, and periodically the Benzol is pumped to the Benzol feed tank.

The Hydrogen from the collecting tank is compressed by three 2-stage compressors, in parallel, to between 250-350 psig. The system includes an intercooler and aftercooler for condensing the Benzol vapors. This liquid Benzol is collected in pressure receivers after each stage. The Benzol is periodically blown by the Hydrogen pressure over into the pump tank for return to the Benzol feed tank. The Hydrogen is used in the manufacture of MB-40, MB-20 or vented to the atmosphere.

DSW 048634

MWV 018277

Process

E-3

In the second type of unit (Nos. 4 and 5) the flow of material is essentially the same as that described above except the vaporized Benzol feed emerging from the vaporiser coil passes through the shell side of a heat exchanger in lieu of one of the preheaters. The conversion products and Benzol from the converter pot are passed through the tubes of the heat exchanger, counter-current to the Benzol feed, thus transferring sensible heat from the converter gases to preheat the feed. The conversion products and vapors from the converter pot then pass on to a column, as is the case with the other units.

The underflow from the column is collected by gravity in two crude Biphenyl catch tanks. The piping to the tanks is steam jacketed to prevent the crude Biphenyl, which is approximately 75% Biphenyl, 15% Terphenyl and 10% Benzol, from freezing and has a liquid trap to prevent vapors from entering the catch tanks from the column. The tanks are vented into the Hydrogen gas header to equalize the pressure and prevent loss of Benzol. The crude Biphenyl is pumped directly to one of the two batch Biphenyl stills.

The two batch stills each consist of a horizontal, cylindrical tank surmounted by a fractionating column, a submerged pump and a gas fired coil heater. The stills are operated at atmospheric pressure. The pump circulates the still charge from the tank, through the coil heater and back to the tank. The rate of distillation of the crude Biphenyl is controlled by the combustion rate of natural gas in the gas fired heater, which is controlled by an adjustable automatic temperature recorder and controller to heat the crude Biphenyl sufficiently to give a temperature rise of 15-25°C between the inlet and outlet of the coil.

The vapors from the still are separated in a 20 plate fractionating column. The first cut from the column is Benzene. This cut is taken at a top plate vapor temperature of 80°C and at a reflux rate of 20% return and 80% forward flow. The Benzene flows by gravity to a catch tank for subsequent recycling through the Biphenyl units. The next fraction, distilling above 80°C, is taken until the freezing point of the distillate reaches 68.6°C. This cut is called the head cut and consists of a mixture of Benzol and Biphenyl. It is made with 50% of the material being returned to the column as reflux. The Biphenyl cut is taken after the freezing point of the distillate reaches 68.6°C and is continued until the pot temperature reaches 350°C. This cut is made with a 20% reflux. The tail cut, at 15% reflux follows the Biphenyl cut and is continued until the distillate on the top plate reaches a temperature of 300°C. This cut consists of a mixture of Biphenyl and Terphenyl. Occasionally it is necessary to make a cut of material boiling between Benzol and Biphenyl which accumulates in the system. This material, usually about 20 to 30 gallons, is collected in a drum and discarded.

Water is circulated through the condenser at the top of the fractionating column during the distillation and is cut back at the end of a distillation in order to keep the condenser from freezing.

DSW 048635

MWV 018278

The Santowax C, remaining in the still as residue consisting essentially of Ortho, Meta, and Para Terphenyls and certain higher boiling compounds, is pumped to the Santowax C catch tank and then to the Santowax still, Santowax C storage tank, or into drums for storage.

Biphenyl is stored for internal use or flaked for sale. The temperature of the storage tank should not exceed 90°C as higher temperatures will cause discoloration.

A conventional water cooled drum flaker is used for flaking Biphenyl. No temperature controls are necessary. The flaked Biphenyl is packaged in paper bags or fiber drums.

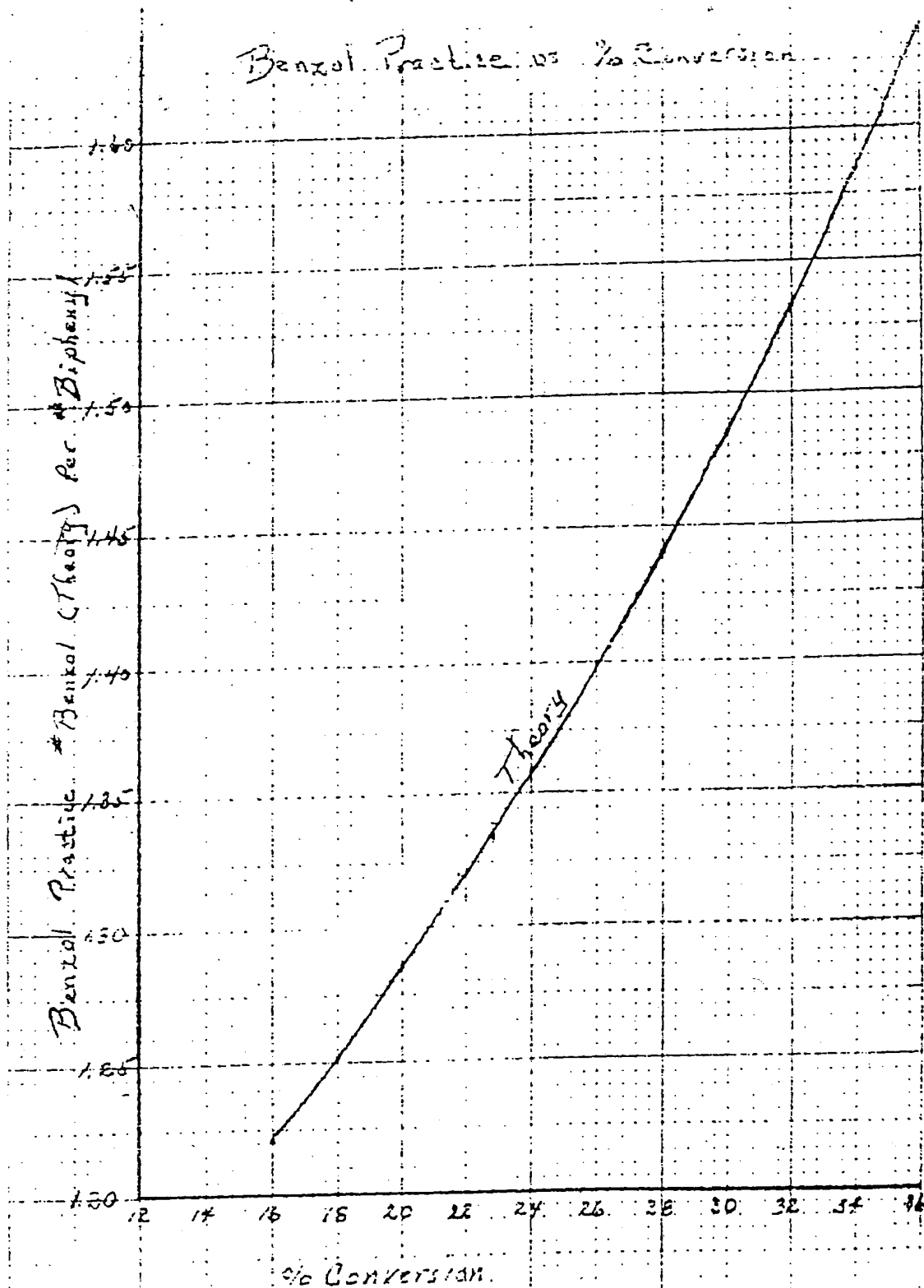
Charts

- Fig. 1 - Shows the theoretical Benzol practice at various percent conversions.
- Fig. 2 - Shows the monthly Biphenyl and Santowax production rates at various percent conversions between 2,000 and 3,600 gallons per hour of Benzene feed.
- Fig. 3 - Shows the pounds of Hydrogen produced per pound of Biphenyl at various percent conversions.
- Fig. 4 - Shows the pounds of Santowax "C" produced per pound of Biphenyl at various percent conversions.
- Fig. 5 - Shows the percent conversion that can be expected at different reaction temperatures and various sojourn times.
- Fig. 6 - Shows monthly Biphenyl production at various percent conversions between 2,000 and 3,600 gallons per hour of Benzene feed.
- Fig. 7 - Shows the conversion being obtained on the units as indicated by the specific gravity of the condensed vapors passing to the column.

DSW 048636

MWV 018279

Benzol Practice vs % Conversion



Actual Benzol Practices

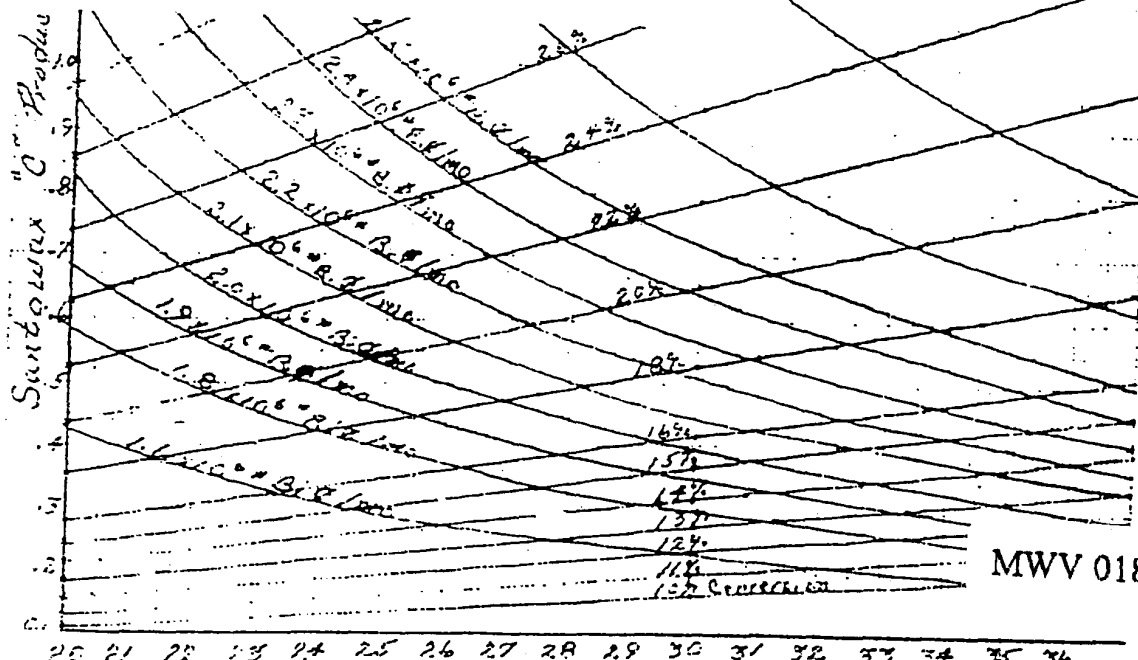
R. L. H. H. H.
2-2-52

DSW 048637

MWV 018280

WATER_PCB-SD0000051646

16	15	14	13	12	11	10	9	8	7	6	5	4	3	2	1
16	15	14	13	12	11	10	9	8	7	6	5	4	3	2	1



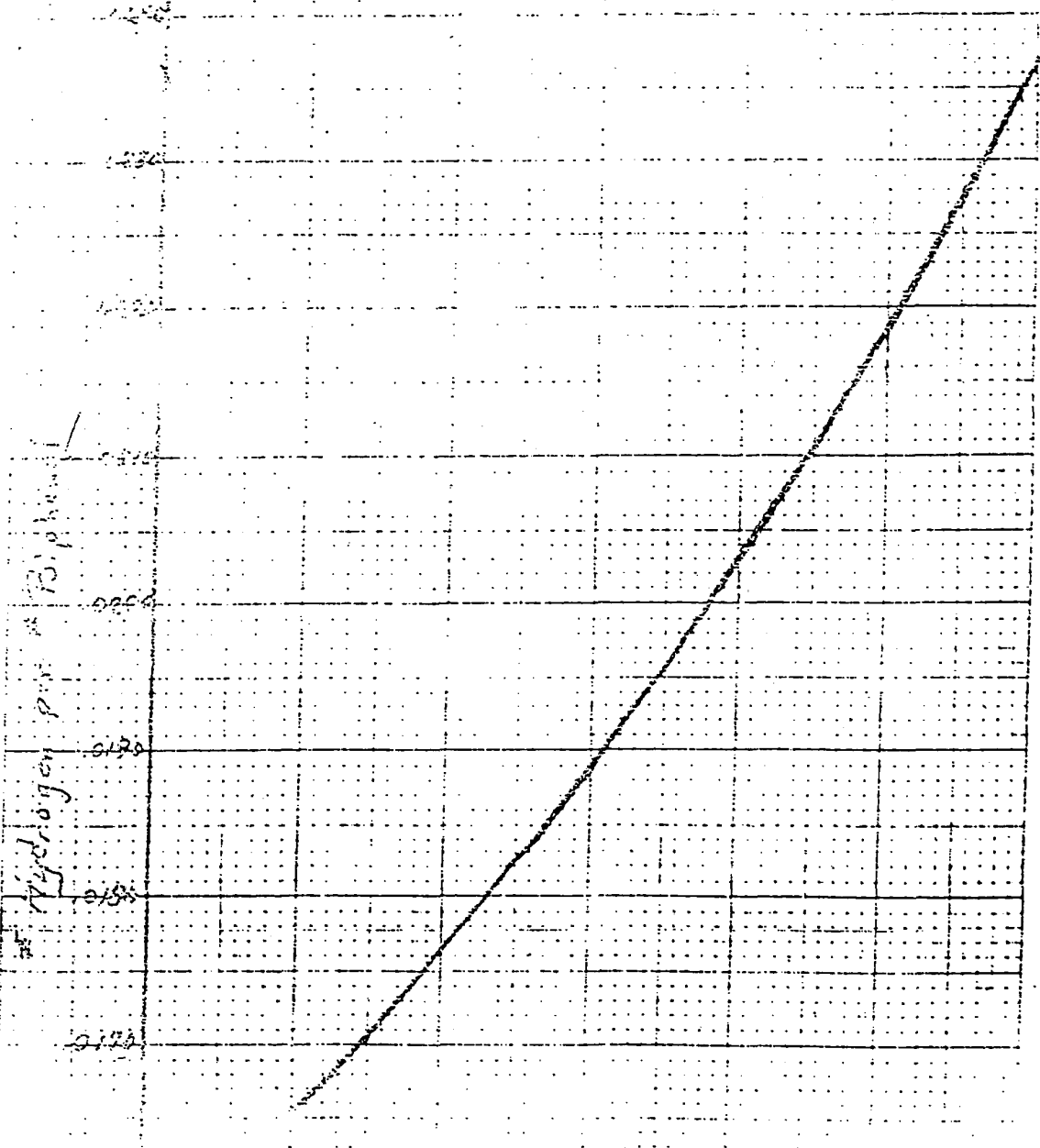
MWV 018281

Revised Feat. 6.14 x 100 (0.14 x 100 = 14.0%)

K. H. Johnson
7-18-52

DSW 048638

Hydrogen peroxide
X. Conversion



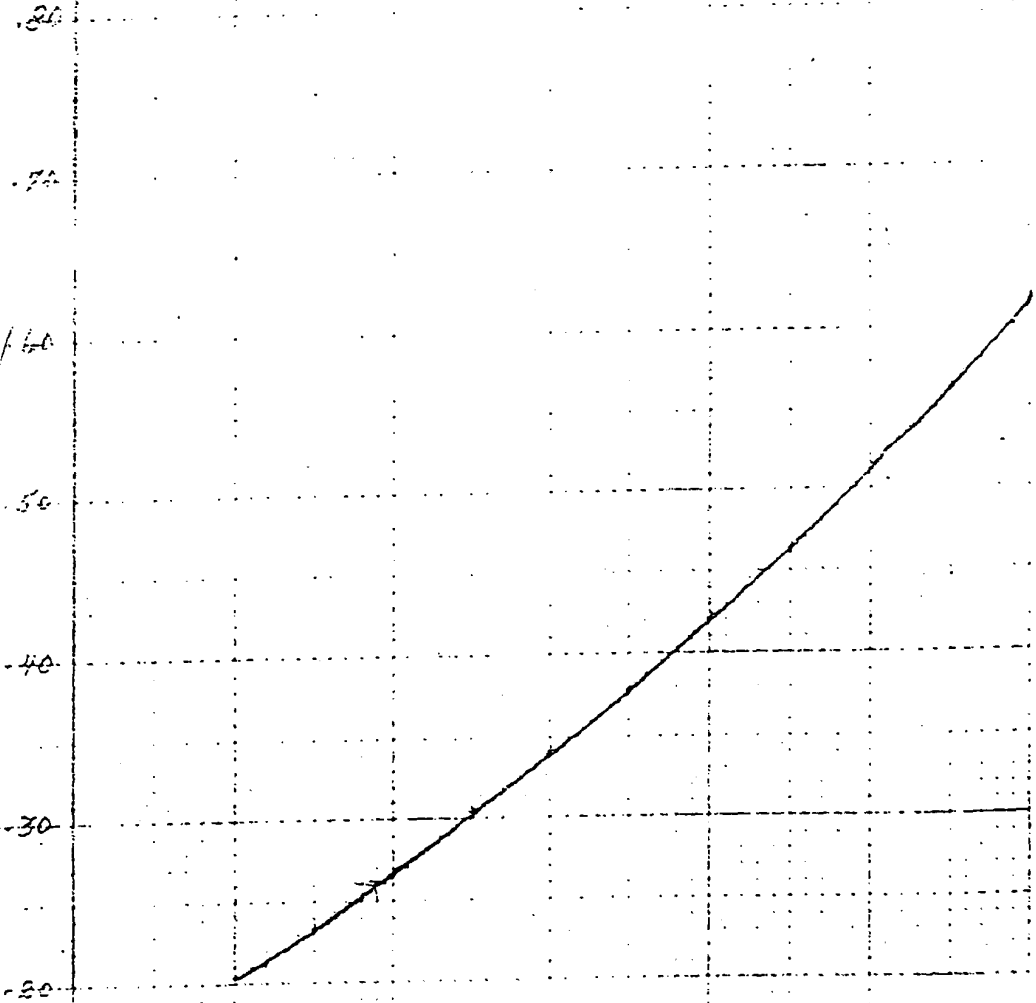
MWV 018282

DSW 048639

46 CONVERSION

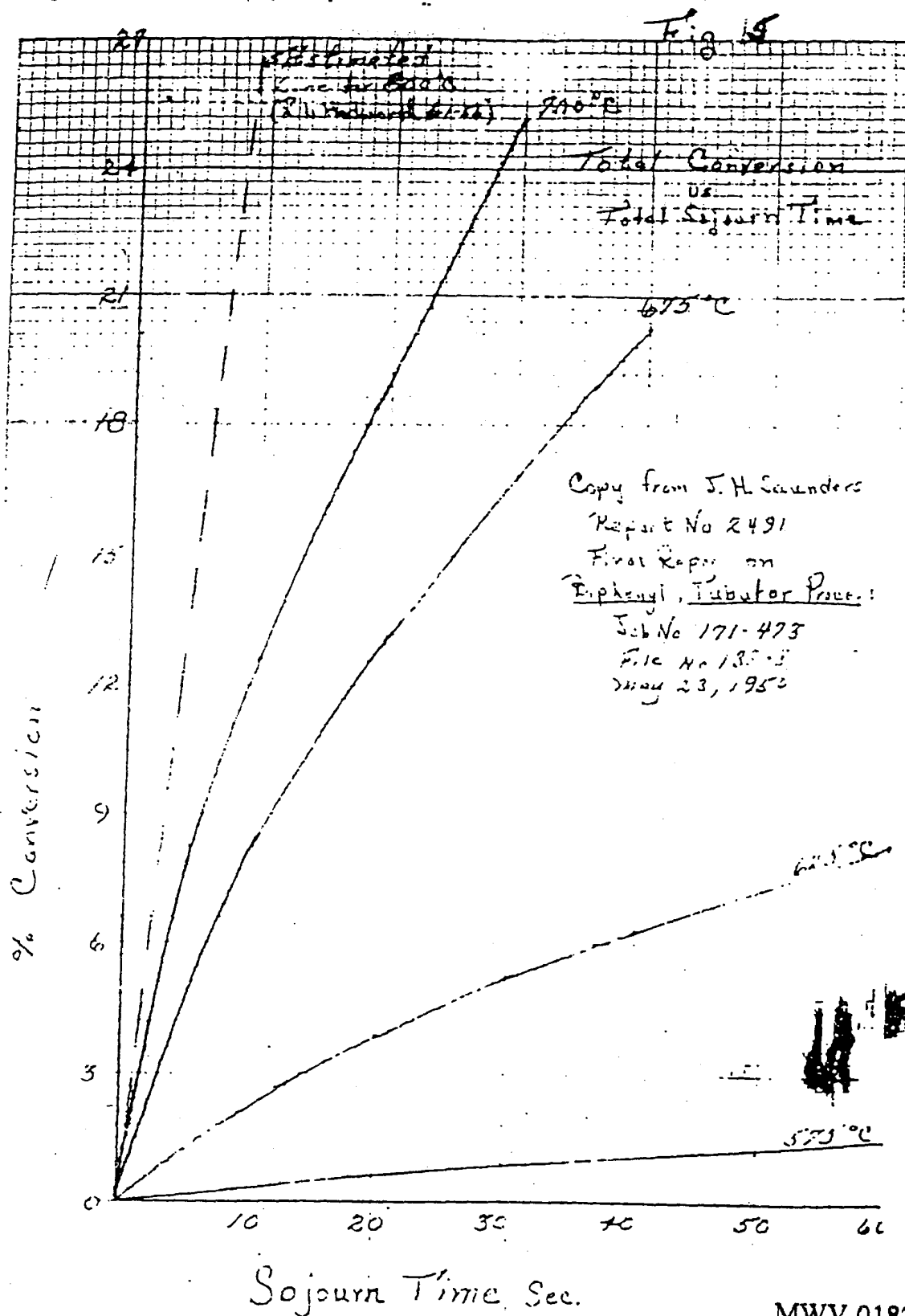
140 10 DIEZIGEN MAPLE PAPER
10 x 10 PER

Sankuash C. per h Biph. yf!



MWV 018283

DSW 048640

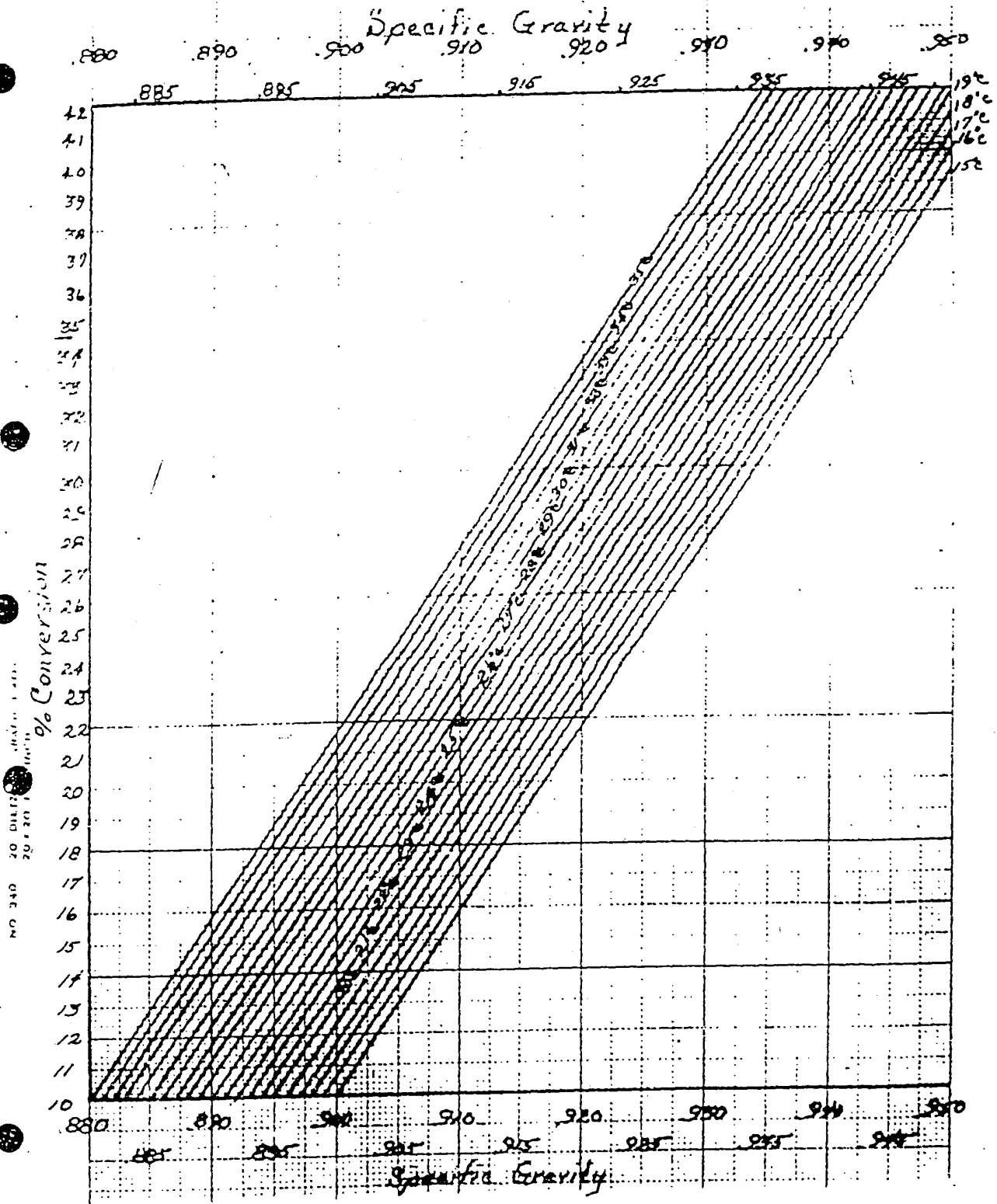


MWV 018284

DSW 048641

1870

Fig 7



DSW 048643

MWV 018286

TYPICAL BIPHENYL UNIT OPERATIONS

8:00 AM 4-9-57

0.15% Isopropanol

Unit No.	Feed Rate gal./hr.	Reflux Column Temp. °C	No. 1 P.H.		No. 2 P.H.		Conversion		Goose-neck Press.	Blue Gas Temp. °C	Cond. Water Out °C	Sump Line Temp. °C	Vaporizer Coil Outlet °C	Feed Benz. Pressure	% Conv. at No. 1 Midnight	Benzol Runback Temp. °C																																																																																																						
			Temp.	Press.	Temp.	Press.	Temp.	Press.																																																																																																														
11	310	55	542	78	660	70	742	44	52	625	51	190	405	130	46	46																																																																																																						
2	180	72	425	68	592	45	742	28	25	X	X	175	X	92	66	66																																																																																																						
3	210	60	500	60	652	44	770	32	24	X	32	180	425*	100	14.2	26																																																																																																						
4			Unit Down - Plugged																																																																																																																			
5	175	55	-	-	588	35	760	28	15	350	20*	180	140	63	18.8	32																																																																																																						
6	230	70	446	70	656	57	760	42	48*	285	25	240	X	102	17.8	35																																																																																																						
7	215	68	402	80	660	60	740	60	40	302	39	206	170	102	15.4	20																																																																																																						
8	250	71	440	77	660	65	760	45	38	400	32	240	X	102	16.6	42																																																																																																						
9	225	90	420	70	652	54	750	48	30	X	30	218	160	100	16.5	62																																																																																																						
10	250	60	403	65	646	50	780	38	X	435	38	212	X	82	19.0	20																																																																																																						
12	245	90	500	68	700	48	772	56	36	285	X	217	280	98	17.8	32																																																																																																						
13	180	90	550	50	702	40	782	30	27	240	30	212	245	77	16.5	38																																																																																																						
Total 2,470																																																																																																																						
No. 11 Unit Read 23% Conversion by Instrument Benzene line pressure 168 psig Cooling Water Temp. 22°C.																																																																																																																						
<table border="0"> <tr> <td colspan="2">Hydrogen</td><td>Out</td><td>Water</td><td>Out.</td><td colspan="2">Hydrogen In Roof Cooler</td><td colspan="10"></td></tr> <tr> <td>1</td><td>280</td><td>24</td><td></td><td></td><td>1 & 2</td><td></td><td colspan="10"></td></tr> <tr> <td>2</td><td>36</td><td>-</td><td></td><td></td><td>3 & 4</td><td></td><td colspan="10"></td></tr> <tr> <td>3</td><td>24</td><td>18</td><td></td><td></td><td>X Not Recorded</td><td></td><td colspan="10"></td></tr> <tr> <td>4</td><td>-</td><td>24</td><td></td><td></td><td>* Probably wrong readings</td><td></td><td colspan="10"></td></tr> <tr> <td colspan="2"></td><td></td><td></td><td></td><td>- Not recorded</td><td></td><td colspan="10"></td></tr> </table>																	Hydrogen		Out	Water	Out.	Hydrogen In Roof Cooler												1	280	24			1 & 2												2	36	-			3 & 4												3	24	18			X Not Recorded												4	-	24			* Probably wrong readings																	- Not recorded											
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4	-	24			* Probably wrong readings																																																																																																																	
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DSW 048644

MWV 018287

SECTION V

COMMENTS ON THE PROCESS

It has been established that a higher conversion can be obtained at a given temperature with all fresh Benzene feed than with all recycled Benzene feed. The fresh Benzene has been postulated to contain a conversion promoter which is used up as the Benzene is passed through the units, accounting for the lower conversion with recycled Benzene.

To eliminate the fluctuating conversion a level control has been provided in the feed tanks to continuously add fresh Benzene to the recycle in the feed tanks.

Conversion Promoter

The actual mechanism by which Isopropyl Alcohol promotes conversion is not known. It is believed to sensitize the Benzene to the formation of Biphenyl and Terphenyl by increasing the rate of free phenyl radical formation.

Isopropyl Alcohol is only one of a number of products which sensitize the Benzene. Isopropyl Alcohol was chosen because of economics. Other sensitizers are listed in Research Report No. 117-881 by Huff and Conover, dated October 5, 1935.

Lead Levels

The lead is adjusted to a 25" outage in the No. 1 Preheater, a 30" outage in the No. 2 Preheater, and a 35" outage in the converter whenever these pots are opened for inspection, cleaning or repairs. These outages have been found to be optimum with respect to lead carryover and heating of the Benzene feed.

Lead Dross

Occasionally lead dross will contaminate the lead and raise the melting temperature. When this contamination occurs the lead in the top of the pot is dipped out and fresh lead added.

Water in Benzene Feed

Water is drained from the Benzene feed tanks about every two hours. If this water is permitted to enter the units, it will cause a sudden surge in pressure and rupture the pot flange gaskets.

Excessive Temperatures

Higher than normal temperature sometimes results from a thermocouple failure. When this condition is encountered carbon is formed. This carbon plugs the distributor and pipe lines.

DSW 048645

MWV 018288

Safety

Benzene, the major raw material handled in the Biphenyl Department, is a flammable liquid and its vapors form explosive mixtures with air. It presents a fire hazard unless proper care is taken in storage and handling.

It possesses a high degree of toxicity. Continued inhalation of low concentrations may cause serious, even fatal blood changes. The liquid, after prolonged contact, may cause defatting of the skin resulting in dermatitis or secondary infection.

Benzene can be handled, shipped, and stored safely, provided the personnel handling the product recognize the hazards and are thoroughly familiar with the necessary precautions.

Some of the principle precautions are:

- (1) Keep away from sparks or open flame.
- (2) Provide adequate ventilation.
- (3) Ground all containers and equipment before discharge to reduce danger from static electricity.
- (4) Avoid inhalation of Benzene vapor.
- (5) Avoid skin contact with Benzene.

Hydrogen is a highly inflammable gas which forms explosive mixtures with air. In areas where Hydrogen is handled or stored, every effort should be made to prevent sparks or fires of any kind.

Biphenyl and Santowax (Terphenyl) - Both Biphenyl and Santowax have a very low order of toxicity. These products are not to be taken internally or maintained in contact with the skin, eyes or other body parts.

Biphenyl has a flash and fire point of 106°C and 124°C respectively.

DSW 048646

MWV 018289

SECTION C

PROCESS CONTROL PROCEDURE

Samples are taken from Benzol tank cars before the cars are unloaded for a freezing point determination. Freezing point on the Benzol must be 5.3°C minimum.

Samples of the gas leaving the Biphenyl converters are taken daily and the conversion rate determined by specific gravity. The percent conversion is regulated to produce the required amounts of both Biphenyl and Santowax. (See Chart Fig. 7)

The percent conversion is regulated by changing the converter pot temperature. The converter pot is usually operated at a temperature of approximately 780°C to give a conversion of 15-17%. The temperature required to get this conversion changes as the pots begin to plug with carbon, causing increased sojourn time due to the increased pressure. No. 1 and No. 2 preheaters are usually operated at approximately 490°C and 670°C respectively.

When the pressure on No. 1 preheater pot reaches 100 lbs. the unit is shut down and cleaned out. At this point the pressure on No. 2 preheater will be approximately 80 lbs. and on the converter approximately 60 lbs.

The units are usually operated with a 60-70% reflux, with 90% being set as a maximum. The reflux cools the vapors leaving the top plate below 100°C. If 90% reflux does not cool the top plate vapors below 100°C, then it is necessary to cut the feed.

Biphenyl freezing points are used as a control point for the Biphenyl distillation cut. The freezing point is to be 68.6°C minimum before this cut is taken.

Typical batch data sheets are attached.

DSW 048647

MWV 018290

MONSANTO CHEMICAL COMPANY
ANNISTON, ALABAMA, PLANT

Biphenyl Oper

FOR 24 HOURS BEGINNING 7:00 A. M. 8-13

19 57

No. 1 S

OPERATOR **R.H.B.**

TIME	FEED RATE G. P. H.	FLOW BOX SET-0	TEMPERATURE, DEGREES CENT.					PRESSURE POUNDS					
			COLUMN	BUMP LINE	CONV'R LEAD	NO. 2 P.H. LEAD	NO. 1 P.H. LEAD	LINE	FEED	NO. 1 PREH.	NO. 2 PREH.	CONV. NECR	H ₂ 1-2
4	215	85-13	78	198	743	680	493	164	93	67	57	42 27	68
9			78	200	743	680	493						
10			80	173	740	678	493	165	94	65	55	40 25	71
11			80	183	742	678	498						
12			82	178	743	680	496	164	96	66	59	44 26	72
1			84	188	743	678	497						
2			84	182	746	680	500	162	96	66	57	42 26	73
3													

REMARKS: Benzol runback temp. #2-78, #3-80, #4-64, #5-45, #6-49, #7-48, #9-72, #10-44, #12-74.

No. 2 S

OPERATOR **Truitt**

4	215	85-15	90	205	740	675	492	164	96	66	58	42 25	74
5			88	182	740	675	495						
6			88	198	740	672	482	164	95	66	58	42 25	72
7			80	198	732	662	468						
8			78	190	730	660	468	164	96	66	58	42 25	72
9			Busy										
10			75	189	735	668	475	165	92	68	58	42 25	72
11													

REMARKS:

No. 3 S

OPERATOR **Norton**

12	215	85-15	75	190	738	665	470	165	94	68	57	42 25	70
1													
2			77	198	738	662	468	165	95	68	57	42 25	70
3			77	193	738	660	468						
4			78	185	740	668	475	165	95	68	57	43 26	70
5			78	182	740	668	475						
6			77	186	740	668	478	165	95	68	58	43 26	70
7													

REMARKS:

MWV 018291

DSW 048648

nyl Operating Record

1957

UNIT NO. 9

No. 1 SHIFT

CHIEF OPERATOR I. C. R.

HYDROGEN COOLER TEMPERATURES DEG. C.												SUMP. TANKS MEASUREMENT		HYDR'N COMPR. SEAL
GOOSE NECK	H ₂ IN		H ₂ OUT				WATER IN	WATER OUT						
	1-2	3-4	1	2	3	4		1	2	3	4			
27	68	65	34	43	17	32	26	30	30	30	30	3'10"	1'3"	OK
25	71	70	38	46	22	35	28	32	32	32	32	chg. steel 1'6"		OK
28	72	71	40	46	28	36	29	34	34	34	34	1'0"	2'4"	OK
26	73	71	40	45	26	36	30	34	34	34	34	1'0"	2'10"	OK

CONVERSION SAMPLE

TIME	12:00
CONV'R. TEMP.	745
NO. 2 P.H. TEMP.	680
SP. GRAVITY	892
AT TEMP., C.	31
CONVERSION	16.3

FLUE GAS	OK
----------	----

44, #12-74, #13-68

Water press. 60 lbs.

No. 2 SHIFT

CHIEF OPERATOR McCullion

25	74	72	41	47	26	37	30	34	34	34	34	Mty	3.6	OK
35	72	72	38	46	24	36	29	34	34	34	34	Mty	4.2	OK
25	72	68	35	44	21	32	26	30	30	30	30	1.0	Mty	OK
25	72	67	34	43	20	21	25	31	30	29	29	1.3	Mty	OK

TIME	
CONV'R. TEMP.	
NO. 2 P.H. TEMP.	
SP. GRAVITY	
AT TEMP., C.	
CONVERSION	

FLUE GAS	
----------	--

No. 3 SHIFT

CHIEF OPERATOR Adcock

25	70	68	34	44	18	32	24	30	28	28	28	2-0	1-3	OK
25	70	66	32	44	18	30	24	29	29	28	28	2-9	1-3	OK
26	70	66	34	44	20	32	24	30	30	28	28	3-5	1-3	OK
26	70	68	34	44	20	32	26	30	30	29	29	4-0	1-3	OK

TIME	12:00
CONV'R. TEMP.	738
NO. 2 P.H. TEMP.	665
SP. GRAVITY	0888
AT TEMP., C.	32
CONVERSION	14-0

FLUE GAS	
----------	--

Raised conv. 20750

MWV 018292

DSW 048649

BIPHENYL DISTILLATION RECORD

Distillation No. 169 Date Started 5-1, 19 54
 Operator Started C.L.M. Shift 2
 Still No. 1 Operator Finish C.S. Shift 1
 Started Charging Still at 5:30 P.M.

Charge:

H&T Tank; inches before 34 inches after 0 Gallons Charged 408
 No. 1 S. T. inches before 51 inches after 0 Gallons Charged 3485
 No. 2 S. T. inches before _____ inches after _____ Gallons Charged _____
 No. 3 S. T. inches before 0 inches after _____ Gallons Charged _____
 No. 4 S. T. inches before _____ inches after _____ Gallons Charged _____
 Benzol Still inches before _____ inches after _____ Gallons Charged _____
 Total Gallons Charged 3893

Circulating at 6:30 P.M.; Fire on at 6:35 P.M.

Flow Box Settings:

1. On <u>20</u> at <u>5:30 P.M.</u>	5. On <u>3</u> at <u>7:45 A.M.</u>
2. On <u>5</u> at <u>6:35 P.M.</u>	6. On _____ at _____
3. On <u>10</u> at <u>11:20 P.M.</u>	7. On _____ at _____
4. On <u>4</u> at <u>1:00 A.M.</u>	8. On _____ at _____

Benzol: Ran 22 " or 506 gal

Heads: Started over at 11:20 P.M. Finished at 1:00 A.M.
 Ran 24" " or 2314 lbs.

Tails: Started over at 7:45 A.M. Finished at 8:30 A.M.
 Ran 12 " or 1,157 lbs.

Biphenyl: Total in Distillation 76 " or 16,796 lbs.

High Boiler: Gross Weight _____ lbs. No. of Drums _____
 Net 6,000 lbs.
 Tare Weight _____ lbs.

MWV 018293

DSW 048650

SECTION R

STANDARD COST SHEET

DSW 048651

MWV 018294

MONSANTO CHEMICAL COMPANY

PRODUCTION COST STANDARDS

Anniston

PLANT

PRODUCT.....Biphenyl

DEPT. NO. 20 82601

EFFECTIVE DATE July 1, 1957

YIELD ON.....% CAPACITY 2,100,000 lbs.

INVENTORY STANDARD 2,100,000 lbs.

YIELD ON.....% COST UNIT 100 lbs.

INVENTORY COST \$7.211

THEORY	QUANTITY PER UNIT	PRICE PER LB. OR GAL	UNIT COST
10950 Isopropyl Alcohol	650	06231	040
14700 Benzol	1331770	05250	7 023
38400 Lead	200	18100	036
RAW MATERIAL WAREHOUSING & HANDLING			
GROSS TOTAL MATERIALS			7 099
CREDITS			
82601 Hydrogen	510	10000	051
82601 Santowax C	20 670	01120	1 472
TOTAL CREDITS			1 523
NET TOTAL MATERIALS			5 576

MONTHLY PRODUCTION BRACKETS

	1,050,000 lbs.		1,575,000 lbs.		2,100,000 lbs.			
	UNIT COST	AMOUNT	UNIT COST	AMOUNT	UNIT COST	AMOUNT	UNIT COST	AMOUNT
MATERIALS	5 576	58548	5 576	87822	5 576	117096		
STEAM	068	714	068	1071	068	1428		
ELECTRICITY	049	515	049	772	049	1029		
COMPRESSED AIR								
WATER PURCHASED	011	116	011	173	011	231		
WATER PLANT	083	872	083	1307	083	1743		
FUEL GAS, COAL, OIL	328	3439	328	5158	328	6878		
REFRIGERATION & ICE								
LABOR	225	2365	265	4183	199	4183		
SUPERVISION	058	610	039	610	029	610		
S. S. TAX & COMP. INS. PENS.	031	327	033	527	025	527		
REPAIRS-EQUIPMENT	429	4500	406	6400	381	8000		
REPAIRS-BUILDING	004	44	003	44	002	44		
SUPPLIES	034	360	033	520	034	720		
LABORATORY	007	72	007	104	007	144		
CLOTHING & LAUNDRY	001	10	001	15	001	20		
Tech. Service	041	430	027	430	020	430		
TOTAL DIRECT CONV.	1 369	14374	1 353	1314	1 237	25987		
DEPRECIATION	178	1869	119	1869	089	1869		
FACTORY INDIRECT EXPENSE	618	6488	412	6488	309	6488		
TOTAL INDIRECT CONV.	796	8357	531	8357	398	8357		
TOTAL PRODUCTION COST	7 741	81279	7 460	117493	7 211	151440		
OPERATING PERIOD	30 DAYS 3 SHIFTS		30 DAYS 3 SHIFTS		30 DAYS 3 SHIFTS		DAYS	SHIFTS
LABOR	953.0	%	1680.2	@	1680.2	@		
STEAM	M LBS	1050		1575		2100		
ELECTRICITY	KWH	420		630		840		
COMPRESSED AIR	CF	13 25		19687		26250		
WATER PURCH.	MG	126		189		252		
WATER PLANT	CE	4673		7009		9345		
LABORATORY	Units	144		208		288		

MWV 018295

DSW 048652

SECTION I

FINISHED GOODS SPECIFICATIONS

The following page is a specification sheet for finished goods.

MWV 018296

DSW 048653

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION

PRODUCT CODE

28

METHOD IDENTITY

--

SALES CODE

1315-000-T-09

Issued July 8, 1957

STANDARDS DEPT.
Anniston, Ala. Plant

FINISHED PRODUCT SPECIFICATION

PRODUCT (Trade name)

BIPHENYL

TYPE

TECHNICAL

PRODUCT (Chemical name)

BIPHENYL

TYPE

TECHNICAL

CHEMICAL FORMULA

$C_{12}H_{10}$

By W. E. Doolan
W. E. Doolan

MOLE WT.

154.15

USE

WGR, Anniston, Sales

SUPERSEDES SPEC OF

7/16/45

SPECIAL No. 10

--

SAMPLE FOR ANALYSIS

Pint can molten, 2-pound bag flaked

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Specification

Test No.

Appearance

Colorless to pale yellow
crystallized solid or flakes

--

Freezing Point ($^{\circ}C$)

68.7 min.

13-10-54

*Color (Gardner 1953)

6 max.

13-11-57

Distillation Range (1)

2.5 max.

13-12-54

($^{\circ}C$, Corrected

1st drop - dry)

NOTE: (1) Distillation must include $255^{\circ}C$ within the range.

*Gardner 1953 Liquid Color Standards

Using Fluorescent Lighted Comparator, Cat. No. 430

or using Gardner-Hellige Comparator, Cat. No. 440-605V

Gardner Laboratory, Inc.

Bethesda 14, Maryland

MWV 018297

DSW 048654