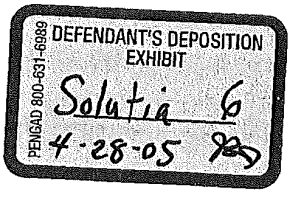
PROCESS FOR THE MANUFACTURE OF

DIPHENYL & SANTOWAX

Compiled by E. Mather, November 1950



Distribution List:

Mr. E. A. O'Neal, Jr., London
Dr. W. H. Garrett, London
Dr. W. D. Scott, London
Mr. J. S. Brough, London
Mr. D. W. Thrift, London
Ruabon Research Files (Mr.W.H.Ritchie)
Mr. G. V. Taylor, Newport (2)
Mr. P. J. C. Haywood, Newport
(Analytical section only of the report)
Mr. C. B. Durgin, Anniston
Mr. A. M. Ellenburg, Anniston
Mr. J. W. Pennington of M.C.L.,
(at Anniston, November 1950)
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St. Louis M.C.L. file (E.Mather)

DSW 462055

INTRODUCTORY NOTE

Preliminary information was sent from Anniston to Mr. E. A. O'Neal, Jr., in London, June 1946.

Mr. D. S. Haverercroft and the writer paid a brief visit to Anniston in September 1947 to collect information needed for evaluation and design work in London, and they produced the report "Notes on the Process for the Manufacture of Diphenyl & Santowax at Anniston", September 1947.

A set of Anniston drawings, out of over 350 drawings on file, was sent to London shortly afterwards, the selection being such as would give full constructional details relating to Anniston's preferred designs.

Since then there has been a great deal of correspondence between M.C.L. and Anniston; Mr. H. L. Barden visited Anniston in August 1950 to review the process from the angle of production work rather than design work, (see the relevant chapter of his "Report on a Visit to U.S.A.", August 1950), and the present writer visited Anniston November 6-10, 1950, mainly to deal with certain questions raised by the M.C.L. mechanical engineers.

This present report is intended to condense the information, collected as above, into a form convenient for use within Monsanto Chemicals Limited, particularly in production work. It is hoped that it will be of use in the final stages of the construction of the Newport plant, and that it will form the basis of an operating manual for use at Newport, to be supplemented and kept up to date as new information comes forward.

EARLIER REPORTS

In a letter to Mr. D. S. Havercroft, Sept.11/47, Mr. M. Buis listed the following six reports which were already in London files:

German Chemical Industry Report Series
German Production of Diphenyl, Terphenyls and Chlorinated Derivatives, by Wayne E. Alexander, H.C.C., St.Louis, Oct.4.1946.

Extract from German Report, C.I.O.S. XXIX-14 pp.56-57. Diphenyl.

Design Data 4th Diphenyl Unit, D.A.Roper, with Study of Diphenyl Costs, E.H.Buford, 8.9.39.

Final Report on Plant Tests on No.6 Diphenyl Unit - Part 1. Job No.171-41 File No.138, 17th Sept.1941. R.A.Ewing.

Design Data for Diphenyl Still, D.A.Roper, Sept.1939.

Report No.1924 Final Report on Separation of Diphenyl High Boiler into fractions or known mixtures Part III - Developing Plant Processes for Production of Santowaxes R.C.M.P. & MF. Job No.171-61, File No.218-B. Prepared by A.M.Ellenburg.
"Equipment Design for Santowax R Plant, May 20/43", Job 1-352-Report #31 by G.R.Shockley, accompanied this report.

In September 1947, D. S. Havercroft & E. Mather wrote a report "Notes on the Process for the Manufacture of Diphenyl & Santowax at Anniston."

On Sept.25/47 copy #11 of Anniston report:

1640, Final Report on Study of the Diphenyl Process Part II. A Review of the Process. Job 171-41, File 138, F.E.Hubbard, Anniston, Oct.15/41,

was handed to Mr. Havercroft for London, also a copy of

Addendum to 1712, Final Report on Study of Diphenyl Process, Part III, Continuous Let-down Procedure, Job 171-41, File 138. A. M. Ellenburg, Anniston, Jan.31/45.

Earlier Reports, contd

On Oct.13/47, microfilm copies of the following two reports were sent to Mr. W. M. Cooper, London:

1642. Final Report on Diphenyl Operations, J.E.Moose, October 1941.

1712. Final Report on Study of Diphenyl Process, Part III, Hubbard, Fenn & Miles, Anniston, Oct.19/42.

On Mar.11/48, a copy of report 1937: "Highly Chlorinated Quaterphenyls" was sent from St.Louis to Ruabon.

Mr. Ellenburg, January 20/48, reviewed the following reports:

214 "Benzol Losses in Waste Hydrogen from Diphenyl Plant", J.L.Howerton, Sept.22/33.

445 "Recommendations for the Operation of the Crude Diphenyl Still", J.N.Carothers, June 23/33.

948 "Investigation of Benzol Losses --- at the Diphenyl Plant", Morton Harris, Aug.21/35.

Mr. Ellenburg, Apr.28/48, cited the following reports on the German tube process:

(1) I.G.Farbenindustrie at Leverkusen Germany, July 25/45, A Visit by Turnock & Lowdermilk, PB 6687(1946).

(2) B.I.O.S. Final Report #893, Item #1, (1947) Fay & Richards.

A copy of Anniston report 2211 "Interim Report on Alloy Testing", 171-429, W.W.Wade, Apr.22/48, discussing the corrosion - erosion of the lead pots by the vapour blast, was sent to London (Mr. Havercroft), July 1/48.

A microfilm copy of the Organic Division report "Diphenyl from Benzene", 117-881, Oct.5/35, on the effects of impurities in benzene, was sent to Mr. Thrift of M.C.L., May 9/49. The process description in this report is said to have been based largely on that of the report "Design Data for 4th Diphenyl Unit", by D.A.Roper.

OUTLINE OF PROCESS AS WORKED AT AMNISTON, 1950

Benzene vapour is passed continuously through molten lead at a high temperature, causing some 16% to 20% of the benzene to be converted, in a single pass through 3 lead baths in series, into a mixture of diphenyl, terphenyl, etc, with the evolution of hydrogen. The resulting mixture of hydrogen with the reaction products and the unchanged benzene is passed up a fractionating column, which has a condenser and reflux split box, so that the sensible heat of the vapours is used to give a rough separation of the organic components of the mixture.

The fractionation system yields a forward flow of liquid benzene, and an underflow of a mixture of benzene, diphenyl, terphenyl, etc., while the hydrogen passes forward, saturated with benzene. This hydrogen stream is cooled and compressed for the recovery of more benzene. The column underflow is fractionally distilled batchwise at atmospheric pressure to yield distillates of benzene and of saleable diphenyl, and of course, intermediate fractions which are recycled to the still.

The still bottoms, containing the higher boiling components of the mixture, are called crude Santowax.

Some of the crude Santowax is distilled "straight-over", under vacuum, to yield Santowax R, and some is put to permanent storage because the supply exceeds the demand.

Crude Santowax has also been distilled under vacuum, with fractionation, giving ortho terphenyl of about 94% purity. Centrifuging of the distillate, or better, fractional distillation, gives a better product.

The m-p fraction from distillation, has been allowed to crystallize in pans, slurried with water at about 85°C, centrifugated and washed in the centrifuge with hot water. The centrifuge cake is then about 95% para terphenyl. The oil from the centrifuge liquors, separated and dried, is called "Santowax M".

See Anniston report #1924, Jan.8/45.

See page 50 below for a note on the flaking of Santowaxes.

All the recovered benzene is recycled and there is a practically continuous make-up of fresh material. This is believed to ensure the presence of a certain catalyst which is present in the raw benzene, but eliminated by the processing. (See under "Comments on Process", page 111 below).

NOTE ON CHOICE OF PROCESS

See the Anniston Report 1640, Oct.15/41, F.E.Hubbard, also Mr. A. M. Ellenburg's letter of April 28/49, citing B.I.O.S. etc. reports on the "tube" process.

The research departments at Ruabon and Anniston agree in believing the tube process may be superior to the lead pot process. Some pilot plant work has been done in this direction at Anniston.

See page 116 below for the choice of the method of recovering benzene from the offgas.

OUTLETS FOR PRODUCTS

At Anniston some of the diphenyl is flaked for sale, but by far the greater part is used either at Anniston or at Monsanto, Illinois, for the manufacture of Aroclors. Monsanto Chemicals Limited do not expect to sell diphenyl. A little also is used at Monsanto, Ill., for the manufacture of o. & p. amino diphenyls.

A mixture of crude Santowax and diphenyl may be chlorinated to give the Aroclor 2565 used as a roofing compound.

Santowax R is used as the starting material for Aroclors 5442 & 5460, also for the hydrogenated product HB-40, which is used as an "extender" in plasticizers, for example in Vinyl film. The Santowax R has some little application in high temperature greases for valves.

Monsanto Limited have given some consideration to the possibility of usefully disposing of still residues. Dr. Scott, January 29/48, considered that the residues would not be suitable for making firelighters. Dr. Hardy, March 4/48, passed on Mr. Ellenburg's review of the possibility of chlorinating the residues to about 60-65% chlorine content to make a cable joint-box composition. Mr. Ellenburg thinks that chlorination at 250-280°C. to 35-40% would be about the best procedure, but warns us that the residues are not of constant composition.

It was thought at one time that M.C.L. markets would call for a higher ratio of high boilers to diphenyl than is normally obtained in the Anniston process, and some thought was given to the possibility of shipping some of the Anniston excess stock of high boilers to Britain, and calculations of the British import duty were made. Mr. J. V. Head's note of March 2/48, however, indicates it is now improbable that such shipments will be necessary. Reference is made to a memorandum by Dr. W. D. Scott of Ruabon, dated June 20/47, on the long term development of outlets for diphenyl.

In the distillation of crude Santowax to give Santowax R, about 15% of the weight of the crude is left as a residue, and some of this is sold as "Montar 9".

Some of the hydrogen after cooling and compression, as outlined above, is used for the hydrogenation of Santowax R to give HB-40. Any excess of hydrogen is vented to atmosphere after being compressed and cooled. At one time Anniston put the excess hydrogen into the fuel gas for the furnaces, but the consequent changes in the air requirement of the burners were troublesome.

SCALE OF WORKING

About 950,000# diphenyl per month, and 80,000# Santowax R per month, were produced at Anniston during the first nine months of 1947.

In September 1949, nine units were in operation, each rated at about 4000# diphenyl per day.

In November 1950, there were twelve units, numbered 2-13, in existence, the original No.1 having been taken out.

Newport needs were estimated by Mr. Cooper, Oct.1/47, to be 1,000,000 lb diphenyl & 240,000 lb Santowax per year.

The annual sales of the ortho terphenyl have averaged only around 4000 lb, and those of "Santowax M" around 10,000 lb. There has been very little sale for the para isomer.

PATENT POSITION

See Anniston Research Department, Report No. 1540, page 6.

PROPERTIES OF MATERIALS CONCERNED

1. Benzene

M.Wt.	78
M.Pt.	5.3°C
B.Pt.	80°C
Specific Gravity	0.883 to 0.886 15.5/15.5°C
Specific Heat:	
solid	$\frac{0.375}{0°C}$
liquid	$\frac{0.389}{5°C}$
	$\frac{0.39}{25°C}$ (I.C.T.)
	$\frac{0.463}{80°C}$
Vapour Cp	$\frac{0.3756}{100°C}$
	$\frac{0.5556}{800°C}$

Formulae for Sp.Ht. at constant pressure:-

Cp = 0.258 + (0.000875 x °C) Perry 1st Edn.
= 0.266 + (0.0006732 x °C) Ind.Eng.Ch., July 1935.

Latent Heat, B.t.U./lb:	
fusion	54.5
evaporation	169.
Flash Point °F.:	
closed Cleveland	12
Explosive Limits in air:	
Lower %	1.4
Upper %	8
Auto-ignition temperature	1076°F.

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Vapour Pressures:

Constants in V.Pr. Formula Log_{10} pr. m.m. Hg = $\frac{-52.23A}{\text{Temp.abs.}}$ + B

Range °C	A	B
-58 to -30	42.904	9.551
-30 to + 5	44.222	9.846
0 to 42	34.172	7.9622
42 to 100	33.295	7.6546

[illegible] Ang 9/39.

Properties of Materials Concerned, contd

1. Benzene (contd)

Thermal Conductivity, (liquid & gas) See sheet 3, Design Data for Units 4 & 5, D. A. Roper, for curves.

2. Diphenyl

See "Description of Process used in Dept.246", Sept.27/46, Lyles, Sofranko & Becker, pages 49 & 81.

See also "Design Data for 4th Diphenyl Unit," Aug.9/39.

Specific Gravity: 0.992 @ 75°C/4°C.

The Swann Chemical Co. bulletin of 1930 gave thermal data on diphenyl.

Viscosity of Diphenyl (from Anniston files)

°F	Centipoises
160	1.439
170	1.309
180	1.208
190	1.114
200	1.034
220	0.901
250	0.752
300	0.574
350	0.459
400	0.374
450	0.313
482	0.283

Specific Heat (from Anniston files)

°F	B.t.U./lb
200	0.416
300	0.470
400	0.547
500	0.616
600	0.658
700	0.679
800	0.686
900	0.690
1000	0.696

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Properties of Materials Concerned, contd

2. Diphenyl (contd)

Another record gives the following values for liquid diphenyl:

°C	Sp.Heat cals./gram
100	0.360
150	0.380
200	0.412
250	0.460
300	0.518
350	0.575
400	0.610
450	0.625

Vapour Cp = $\frac{0.460}{250^{\circ}C}$ $\frac{0.625}{450^{\circ}C}$ (D.A.Roper, Design Data for Units 4 & 5)

Density of Liquid Diphenyl (from Anniston files)

°C	grams./cc
75	0.9885
100	0.9695
150	0.9294
200	0.8885
250	0.8470
300	0.8010
350	0.7510
400	0.6918
450	0.6180

Heat of Vaporization:

65.4 g.cal/g @ 258.5°C.

Properties of Materials Concerned, contd

3. Santowax R

contains about 1% diphenyl
10% ortho terphenyl
46% meta terphenyl
21% para terphenyl
22% triorthophenylene, quaterphenyls etc.
(Anniston Final Report 1942)

Upper boil point	137-143°C.	Changed Sept.23/47) —	137-145°C
Lower boil point	45-60°C.		45-63°C

The density of the Santowax may be assumed to be the same in both the crude and distilled form. Viscosity also is the same.

Specific Gravity of Crude Santowax:

140°C	1.026
175	1.000
215	0.98
300	0.964 (est. from curve of other points).

Viscosity of Santowax R

155°C	32.4 Saybolt Universal Seconds
225	28.5 " "

Molten Santowax flows about like water at 25°C.
(A.M.Ellenburg's letter Jan.6/49).

Specific Heats (estimated)

Solid	0.4
Liquid	0.5

Latent Heats (estimated)

Fusion	63 B.t.U./lb
Vaporisation	140 B.t.U./lb

Boiling Range: 360-450°C.

Flash Point: Cl. open cup 191°C.

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Flame Point: Cl. open cup 238°C.

Another account gives Flash Point 190°C; Flame point 220°C.

Properties of Materials Concerned, contd

3. Santowax R (contd)

Values assumed in the design of the Santowax still at Anniston:

Sp.Heat	-	0.5
Heat of Vaporization	-	140 B.t.U./lb
Viscosity at 250°C	-	5 centipoises *
Viscosity at 350°C	-	2 centipoises *

* (Mr. Ellenburg said these values were too high).

4. Terphenyls

	"Hold Point"		B.Pt. @ 30 mm
Crude ortho (Santowax O)	53-55°C		205°C
Crude meta (Santowax M)	83-85°C	mixture	240°C
Crude para (Santowax P)	209-213°C		
M - p eutectic	84.8°C		
Ternary eutectic	about 74°C.		

See also Monsanto Technical Bulletin #P-103, Jan.4/47 issue, for the properties of the separated crude isomers sold.

HEAT BALANCE

The heat balance is discussed in the Anniston Design Data, 4th Unit, by D. A. Roper, Aug.9/39, page 6.

The heat of reaction of benzene to diphenyl is given as 59.29 lb°C per lb at 740°C.

Trans.Am.Chem.Eng. V, 41, p.144, (1938), gives 61 lb°C per lb.

This is calculated to be less than 2% of the heat needed in a 3-pot unit.

SAFETY CONSIDERATIONS

(From Anniston report 1642 of October 1941, section "Health & Fire Hazards,", and from discussions at Anniston in 1947 and 1950).

Health

It is obviously advisable for the operators to minimise their contact with benzene vapours and with the vapour from the hot lead, but there is no history of trouble from either of these in the Diphenyl plant. We are warned that lead vapours may be evolved during the burning out of lead traps and carbon traps.

When lower grade benzene was used for making diphenyl in 1930-31, the operators in the Aroclor department suffered from skin eruptions (acne), thought possibly to be due to chlorine derivatives from styrene in the diphenyl. The disappearance of the trouble coincided with the change to better benzene. For a time the Aroclor operators had to bathe on leaving work and a change of work clothing was provided, but this practice was discontinued.

See notes on pages 20 & 82 below relating to the general ventilation of the building.

Fire

The conversion units contain hydrogen and organic vapours at high pressures and temperatures so any failure of gaskets or of the units themselves may lead to the escape of a cloud of inflammable vapour, near to the pot furnaces. The amount of organic material present in the conversion unit at any one moment is, however, quite small, and the flow of benzene to the units can be shut off from outside the building.

In the early days at Anniston, fires were not infrequent, but there has been none of consequence in recent years. Emphasis is laid on good workmanship in the construction and maintenance of the units.

There were twelve conversion units at Anniston in November 1950, with their columns and other auxiliary equipment, two of the units being in one room, four in another, and six in a third room. The three rooms are separated by fire walls with communicating doors, some of them automatically closing, but held open by chains having a fusible link. There are two

Safety Considerations, contd

Fire, contd

diphenyl stills, each in its own room, these two rooms having connecting doors into one another, but not into the conversion rooms. The still rooms contain the receivers for the finished diphenyl, the heads and tails fractions, and the still bottoms, but the benzene receiver is in a pit, behind a fire wall outside the building. The crude diphenyl from the convertor columns flows to two receivers which are outdoors, alongside the benzene receiver.

The main store tanks for fresh benzene are in a fenced area, well away from any manufacturing buildings, and the benzene feed tank and pumps are in a smaller fenced area, somewhat nearer to the diphenyl plant.

Benzene vessels are systematically grounded (earthed). See the note on page 83 below on the objection to internal steam coils in the benzene tanks.

The department is well supplied with extinguishers, both CO_2 and CCl_4 types, and there is a permanently piped Foamite connection to the main benzene vessels. There is a drum of CCl_4 on a stillage on the ground floor of the conversion room for recharging extinguishers, and all the appliances are subject to routine inspection.

Cloths soaked in CCl_4 are used to stop off pipe openings etc. during repair work and the units may be filled with CO_2 during welding repairs.

The convertor rooms are not treated as having a dangerous atmosphere. It is not at all unusual to have escaping vapours burning round the cover of the convertor pot (especially now some of the convertors are being run at over 20 psig). If vapour leaks from the pot flange, it may not ignite, especially if the seal between pot and furnace is good, but it is better to ignite the vapour rather than to have diphenyl subliming about the building, and lodging on roof members, etc. Welding is done in the building, and of course, some of the vapour pipes are red hot. Naked flames are used to deal with blocked pipes in the conversion system.

The diphenyl still rooms do, of course, contain large amounts of combustible material because of the batch nature of the operation. The heating coil furnaces for the two stills are side by side in a room of their own, cut off from all other rooms.

Those of the still receivers which are indoors have vent pipes, carried out through the roof of the building, and the outdoor

Safety Considerations, contd

Fire, contd

benzene receiver has its vent pipe carried well up into the air. Each still body has a bursting disc in a pipe leading up through the roof, (see page 96 below).

Anniston are a little restricted in doing welding repairs to their Santowax distillation system because of its proximity to the HB-40 plant, which uses hydrogen under high pressure.

The receivers for the hydrogen at each stage of compression have Al-Pb bursting discs, in pipes venting above the roof of the compressor room.

Care is needed, of course, in restarting the gas-fired furnaces. There is a powerful draught up the furnaces and the pilot jets may easily be blown out.

Mr. Ellenburg, Aug.18/48, stated that Class I, Group D motors are specified for pumping diphenyl and Santowax. He also stated that the bottoms from the diphenyl stills, if discharged at around 400°C, may take fire. At one time it was the practice to purge the stills with CO_2 before recharging, but this is not now thought necessary, and all that is done is to allow the stills to cool to about 200°C or lower before recharging. When the crude diphenyl, hot from the store tanks, is pumped into the hot still, there may be a great rush of vapour through the condensing system.

Since the introduction of the restriction orifices after the convertor pots, these pots have been running at about 20 psig, so that there is a greater risk of blowing out the gasket on the convertor cover. Some of the convertors have been fitted with a loose sheet iron ring fence about 6" deep, round the cover joint, to deflect any possible burst of flame.

Fire Fighting Equipment

Mr. Harden, August 1950, reported as follows:

> "Ample CO_2 and foam extinguishers are provided. Anniston has two standard foam extinguishers on each floor of the convertor section and several in the distillation section. On the ground floor, available for use throughout the diphenyl aroclor shed was a large hand-cart foam appliance. The benzene tanks are permanently piped, to top of each tank and into the sump, for foam which is supplied from a central point".

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Safety Considerations, contd

Fire Fighting Equipment, contd

Each convertor unit has about a 56 lb cylinder of CO_2 permanently connected to a valve leading into the benzene feed line where it runs below the operating floor before rising to the top of the vapourizer. These cylinders are supported against the stanchions on the ground floor. No provision is made for warming them. A drum of CCl_4 is kept on a stillage on the ground floor for easy recharging of the extinguishers. There are numerous safety showers, each with the usual yellow painted ring on the floor below them.

Hydrogen

The conversion units contain hydrogen, and so do the crude diphenyl store tanks, because they are vented into the hydrogen main. These vent pipes from the crude diphenyl tank to the hydrogen main are usually very hot. Air must of course be purged from the system before any hydrogen is taken to the compressors, and usually CO_2 is used for this purpose (400 cu.ft. for a convertor unit), also to replace hydrogen before a unit is opened for repairs. There is no harm in allowing the CO_2 to go through to the hydrogen compressors, in fact there is the advantage of recovering its benzene content. It is no longer thought necessary to do oxygen determinations on the hydrogen stream. The crude diphenyl receivers are vented into the hydrogen collecting system, and there is a risk that if they are pumped empty, the hydrogen may escape into the diphenyl still system.

Welfare

As already mentioned, only ordinary common sense care is taken against poisoning by lead or benzene, and no special clothing is supplied beyond face masks and leather gloves for men dealing with molten lead.

Simple "firescreens" of sheet iron on pipe frames are used to shield the man and the equipment during the collection of the samples for the % conversion test. See page 75 below.

Mr. Harden reported, August 1950:

> "High temperatures are fairly general throughout the department particularly above the convertor. All floors are of the vertical strip lattice construction to improve ventilation, and large portable

Safety Considerations, contd

Welfare, contd

> "Man-cooling fans are used to blow up through these floors whenever a man has to work upstairs. Normally all operations are controlled from the ground floor, all possible controls and instruments being located there. The writer regards attentions to this detail as essential if conscientious maintenance of equipment is to be obtained."

Repair and maintenance work on the conversion units at Anniston is made more difficult by the radiation of heat from the surrounding units, especially in the room which contains six units. The lead traps, in particular, offer a big radiating surface.

The diphenyl building with good ventilators in the roof, is very high, and the open-work steel floors permit a strong updraught of fresh air to flow through the rooms from the ground floor, which in every case is completely open on the east side.

When molten lead is being baled about on the operating floor, the ground floor below is roped off. See page 39 below.

SAMPLES SENT FROM ANNISTON TO MONSANTO CHEMICALS LIMITED

The following samples were sent from Anniston, Nov.13/47:

G-4070	Benzol
G-4071	Crude diphenyl
G-4072	Finished diphenyl
G-4073	Santowax R
G-4074	Still bottoms from diphenyl still
G-4075	Still bottoms from Santowax still (also 10 lb Montar No.9 requested June 22/48),

but only the first two reached Ruabon, so the last four were repeated January 25/49, to London,(and were received there Feb.21/49).

The following samples also were sent to M.C.L., on the dates stated:

June 22/48	Santowax R
Nov./49	Benzene as used at Anniston.

CORROSION

Benzene and Diphenyl under the working conditions are not corrosive, but it has been found advisable to use stainless steel for the lead pots and connecting vapour pipes.

Anniston Interim report 2211, dated April 22/48, describes pot failures by "washing erosion" of the welds and the pot bottoms, failure of the welds, external erosion by lead, and by other causes, and also recommends the use of 310 stainless welding rods, or of 309 rods with subsequent annealing of the pots at 2050°F. Copy #11 of this report was sent to Mr. D. S. Havercroft in London, July 1/48.

See page 57 below for a discussion of the harmful effects which foreign elements in the lead might have on the stainless steel of the pots.

In January 1949 it was reported the Anniston experience up to that date showed 309 type stainless steel to give better service than 316 type.

The Texas City Research Report No.52 "Special Report on Lead Pot Corrosion", E.Fitzgerald, Feb.17/50, (copy seen at Anniston, Nov./50), deals with the corrosion of lead pots used to crack propane at 800°C to ethylene (formerly for pyrolysis of ethylbenzene to styrene). It reviews the information available up to April 1947. Thermowells had suffered badly, and the pot bodies became pitted around the level of the lead surface and became eroded where there was turbulence of the lead. Laboratory tests showed that lead oxide was highly corrosive to the stainless steel used.

Samples of different stainless steels immersed in the lead pots gave inconclusive results, but Hastelloy and Hascrome appeared to have high resistance. Pure nickel, monel, and nickel-chromium alloys with 80% nickel dissolved in the lead in a few hours. W.W.Wade, Anniston, Apr.22/48, reported poor resistance with 68 nickel - 18 chromium alloy.

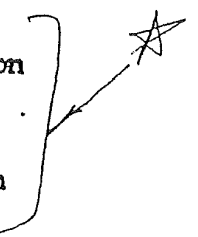
Carburation of the metal was suspected, with the formation of brittle coating easily removed by the pounding action of the lead.

A patent search revealed that the resistance of high chrome alloys was known.

The report ended with a recommendation to run all the pots at a low rate to diminish turbulence rather than to run a few pots at high rate. It was recommended too to put carbon in the pots at the outset, to reduce lead oxides, but this recommendation has not been adopted at Anniston.

Corrosion, contd

Lead is known to escape sometimes through the cover joints on the pots at Anniston. It may form lead oxide outside the pot, and this lead oxide may attack the outer surface of the pot. There is no point in trying to detect lead in the stack gases because there is always some lead lying about in the furnaces.



GASKETS

Anniston report 1642, Oct.15/41, recommends Garlock 721 gaskets for temperatures up to 500°C, and Goetz corrugated steel-asbestos gaskets above 500°C.

For the cover of the second preheater a 5/16" round soft copper gasket is used with an asbestos gasket inside it. This double gasket originated at Texas City. but the asbestos ring there is outside the copper. For the cover of the convertor a gasket of 1" braided asbestos rope is used, (see pages 42 & 87 below), but if the distortion of the flanges could be prevented, the 5/16" copper gasket would be better.

Copper and its alloys have given poor service in the hydrogen system, apparently because of H_2S. Copper gaskets in the compressors have completely failed, and Babbitt metal has been proposed.

FLOW SHEET

The attached flow sheets of materials, pages 26 & 27, are taken from Anniston Research Department, Report No. 1642, October 15, 1941.

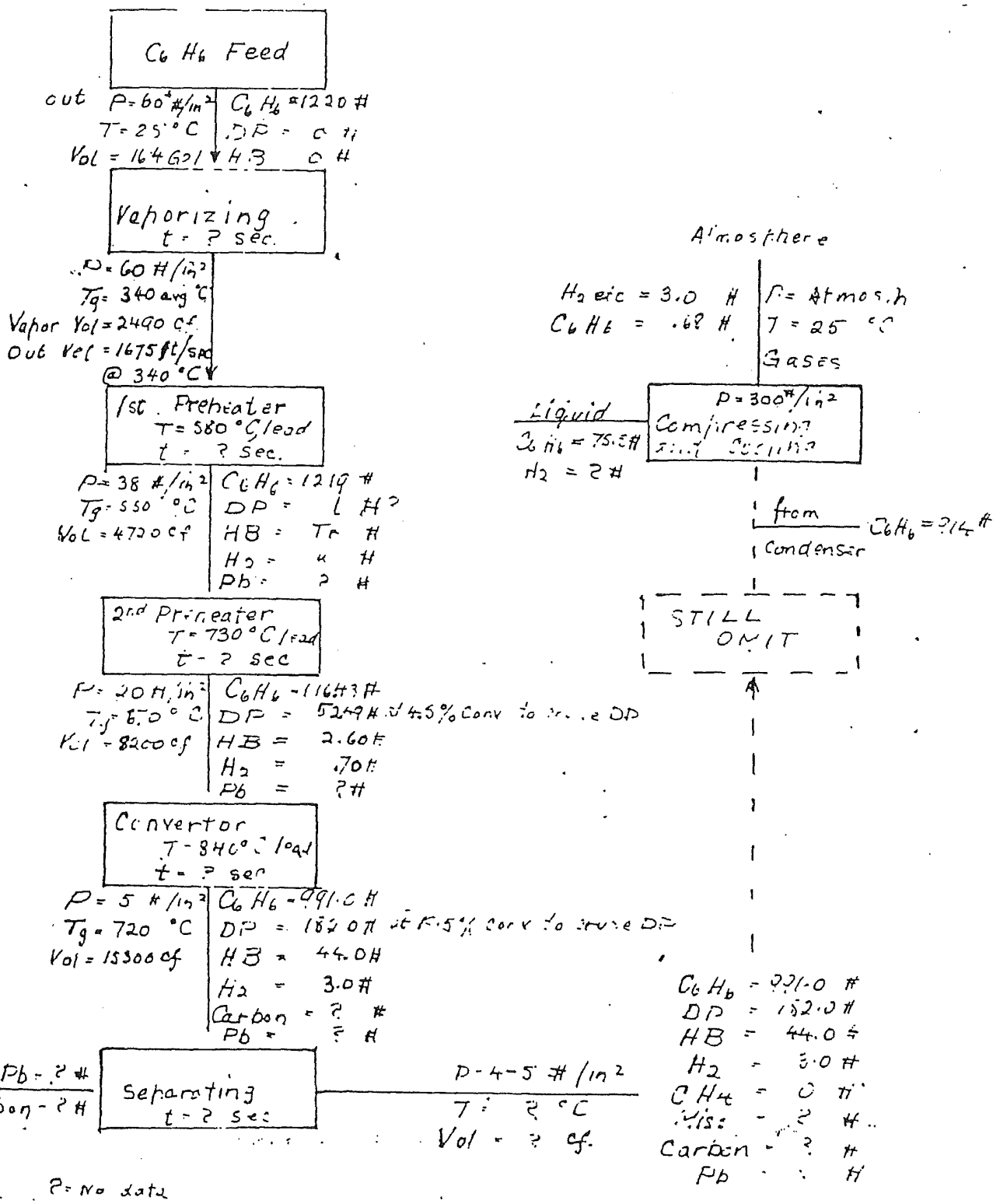
The same report gives the following data:-

0.1796 U.S.Gall benzene consumed per 1 lb distilled diphenyl produced (compare cost records given on pages 118- below, of the present report).

The "shut-down" time on the conversion systems averaged 1.6% over a period of 8 months.

See also page 28.

Diphenyl Process Flow Sheet

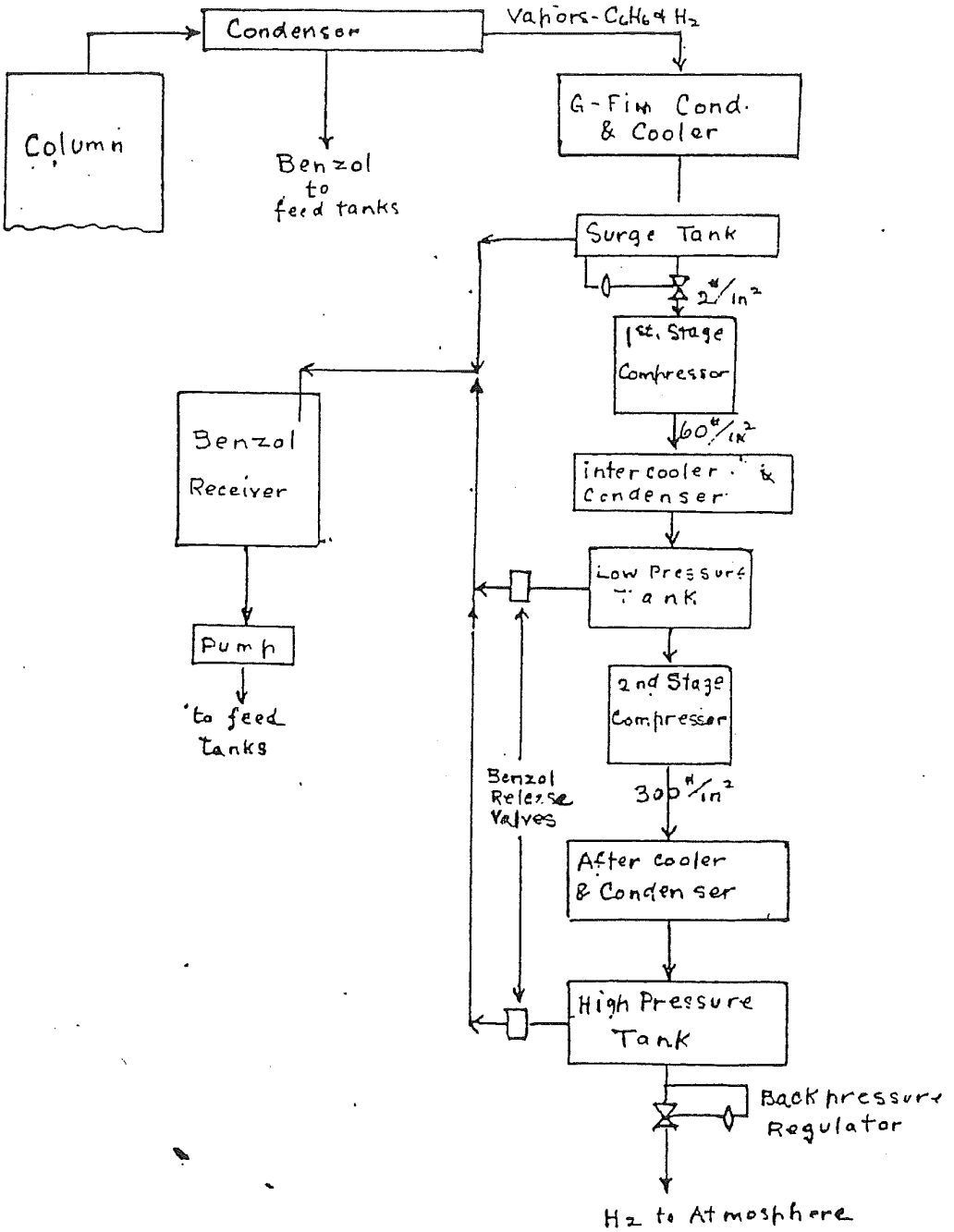


? = No data

g = gas

DSW 462081

Flow Sheet for handling off Gases from Column.



DSW 462082

COMPOSITION OF SOME INTERMEDIATE LIQUORS

	% Benzene	% Toluene	% Styrene	% Diphenyl	% Naphthalene	% Diphenyl benzenes o	m	p	% Tri ortho phenylene	% High Boiling Mtls	% Unidentified
ıde phenyl	4-8									20	
ɜads" or yrene action	20	0.5	20	35	1						19
ly about 220 lb of this fraction produced in each convertor per year. See also pages 48, & 116 below.											
phenyl ıll ttoms 930				0.9		4.3	58.8	29.5	1.1	5.4	
phenyl ıll ttoms 941				trace		6-10	53-68	15-30	1-3	2-4	

Some of these values will naturally vary a little, depending on the exact mode of operating the plant units.

DSW 462083

PROCESS IN DETAIL

Benzene Handling

Benzene is received at the plant in tank cars and is unloaded "over the top" by pump to 5 large horizontal storage tanks (#1) situated some 300' from the diphenyl building. Water is used to prime the pump, and of course it must be separated afterwards by draining from the store tanks, or from the recycle tank.

From the Storage Tanks the benzene is pumped by (#2) to (#3) Recycle Feed Tank. Here recycled material is constantly made up with fresh benzene. This is necessary in view of the fact that a catalyst, apparently present in the fresh benzene, is removed in one pass through the convertors. (See under "Comments on Process," pages III & II2 below). A float within the Recycle Feed Tank automatically operates the pump, item #2. There are also two floats within the tank for high and low level alarms. There is a history of a little trouble due to the sticking of those various floats; see notes on item #3, page 84 below.

The recycle feed tank now in use is a horizontal cylindrical vessel with bulged ends, and the benzene outlet line to the pumps, item #4, is taken from the bottom by a jacketed line (jacket, however, is not used), cemented through the containing wall which surrounds the tank. This take-off line rises a few inches inside the tank to trap any water and solid matter, and the tank has a sump, with a drain valve for tapping off the water, etc. Regular and frequent operation of this valve is a very important part of the operation of the plant because a shot of water, pumped through to the vapouriser, may flash off almost explosively, and might break some joint in the pressure system. Water gets in from its use, in priming the overhead suction lines from tank cars, and may get in from perforation of the tubes of any of the condensers.

The following streams of benzene enter the recycle feed tank:

(1) A continuous stream of condensate from the convertor condensers, item #11,
(2) An intermittent stream from the receivers, item #27, of the hydrogen cooling and compression system. This stream comes at about hourly intervals, under hand control of the pump, item #28.
(3) The benzene distillate from the benzene receiver, item #21, of the diphenyl still. This distillate comes forward in batches at Anniston, via the pump, item #21a, but Mr. Ellenburg's letter, Jan.20/48, indicated that a similar procedure at Newport might cause too great a

Process in Detail, contd

Benzene Handling, contd

variation in the ratio of fresh benzene to recycled benzene, because the flow of fresh benzene would be very much smaller at Newport than at Anniston, but the diphenyl still batches probably not much smaller.

Mr. Ellenburg suggests interposing a buffer tank between the receiver, item #21, and the recycle feed tank, item #3, so that the stream of recycled material from #21 can be smoothed out.

At Anniston, the operator waits until the float alarm on item #3 is "ringing on low" before pumping in benzene from the diphenyl distillation on the hydrogen compression, then he stops when the alarm "rings on high".

There is a case on record in which the recycle feed tank was overpumped, and the excess benzene backed up the distillate line and into the convertor reflux split boxes of the conversion units, and escaped down the columns into the crude diphenyl store tank. This overpumping was detected by the fall in temperature of the column underflow streams.

There are two (2-stage Gould) (#4) centrifugal pumps for feeding benzol at approximately 100# pressure from the recycle feed tank to the conversion units, and one or both may be in operation depending on the number of units operating at the time. There is a Fisher strainer before the pumps, and there are two filter pots, after the pumps, in the Control room - only one in operation at a time, to allow for cleaning.

These two filter pots are vertical cylindrical vessels about 6" I.D. by 9" deep with bolted-on covers, fit of course for working pressures around 100 psig. They have baskets inside to support cotton filtercloth, but in November/50 the cloth had rotted and had not been replaced. It is thought that glass cloth might serve. There are Nordstrom 3-way cocks on the inlet and outlet lines for easy switching of the pots, and there is a pressure gauge on each line, to indicate the filter resistance. The main purpose of filtration is to protect the flow controllers. Solid matter carrying forward to the vapourizer would be simply carried through in the vapour stream, and probably end in the lead trap or the carbon trap of the convertor. It is said that a little dust (from the pots) comes forward in the recycled benzene, and there is pipe scale from the benzene lines.

Process in Detail, contd

Benzene Handling, contd

The filter pots deliver to a header, serving a row of 13 Foxboro recording flow controllers, one for each unit, plus one spare, the piping being so arranged that the spare controller can be switched in to replace any of the others. These controllers are in a special room which is entered from outdoors and which has a communicating door to the main control room, otherwise it is completely shut off from the rest of the plant.

Apart from the benzene flow controllers this room contains only the meters for the fuel gas to the furnaces, (and a controller for water to the HCl absorber for the Aroclor plant).

Mr. J. C. Clegorn, the department manager, mentioned that Rotameters were formerly used, set in a glass box draining to outdoors, to take away any benzene leakage. There was a certain advantage in having the visible flow, and he would like sight boxes (fit for 120 psig) in the present flow lines.

The benzene delivery lines from the controllers are cemented through the wall between this room and the convertor rooms, at about the level of the convertor pot lids, a separate line to each conversion unit. Each line dips to a stop valve within easy reach of the ground floor, inside the convertor room, and then rises to the top of the vapourizer unit. Immediately after the stop valve there is an inlet for CO_2 from a cylinder chained to one of the stanchions of the building. No provision is made for warming the CO_2 cylinders, though the ground floor, being completely open to the air on one side, is not very warm.

The normal flow of benzene is 170 USG per conversion unit per hour.

Process in Detail, contd

Conversion

There were 12 conversion units, item #5, at Anniston in November 1950, two of them (units 4 & 5) being somewhat different from the rest in that they had a tubular preheater, then two lead pots, whereas the other ten units each had the preferred normal arrangement of three lead pots and no tubular preheater. (See page 85 below for the reasons for choosing the three pot arrangement).

Each of the 10 normal units consists of a (#6A) Vapourizer, (#5A, 5B, & 5C), three lead pots in series, (called respectively 1st preheater, 2nd preheater, and convertor), (#7), lead trap, (#8) fractionating column, with (#11) condenser and (#12) reflux split box. The underflow from the column passes through item (#9), carbon trap, and then by gravity through steam-jacketed lines to the crude diphenyl receivers, item (#10). The vapourizer consists of a coil (formerly two coils in parallel) within a stack through which the exhaust gases from the pot furnaces are passed countercurrent to the benzene flow. There is also (#6B), another stack for the purpose of by-passing the furnace gases, if necessary, away from the vapourizer coil. The temperature of the vapour leaving the vapourizer is about 250°C, but see below, pages 33,34. If the vapourizer has two coils in parallel, an attempt must be made to share the flow between them by manipulation of the valves at the exits from the individual coils. See the notes below, under item #6A, page 91, relating to the possible loss of benzene vapour by perforation of a vapourizer coil.

Each conversion pot is of stainless steel about 22-3/4" I.D. by about 6'-0" deep, and contains molten lead. The following figures are given for the depth of lead in each pot:

Depth of Lead:	Anniston report 1642, Oct.15/41,	H.L.Harden, Aug/50. (confirmed Nov/50)
1st Preheater pot	4'-0"	2'-1" dry dip
2nd Preheater pot	4'-0"	2'-6" dry dip
Convertor pot	3'-6"	2'-11" dry dip
Height of Distributor inlet above bottom of pot given as	10"	out of 5'-11-3/8" internal depth

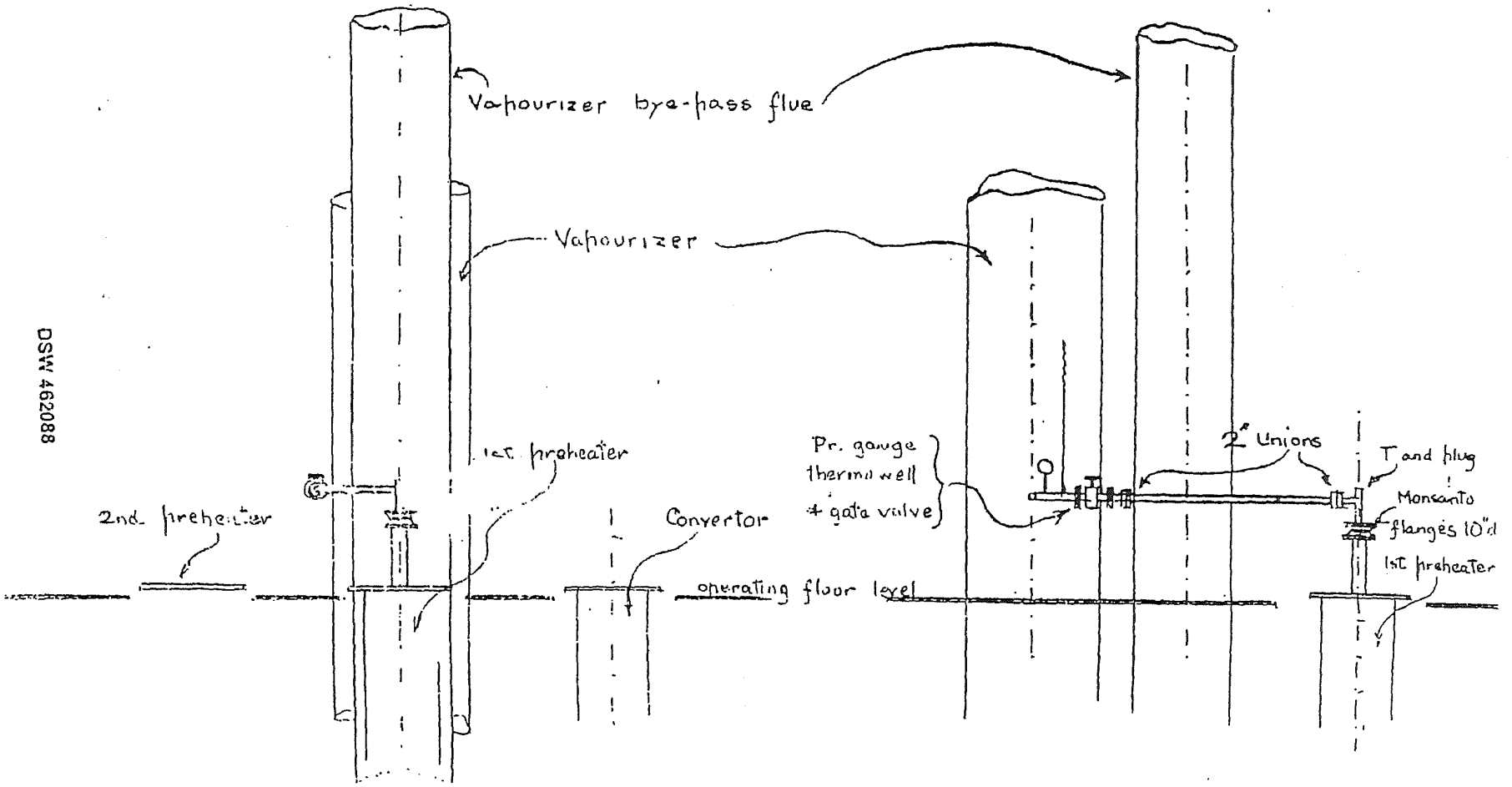
There is little entrainment from the first preheater to the second, and not much from the second preheater to the convertor, but considerable entrainment from the convertor pot to the lead trap, so the depths of lead are liable to vary a little. If it is not desired to open a pot, the loss of lead may be made up with molten lead poured in through the neck which carries the thermo well.

CONNECTION FROM VAPOURIZER TO FIRST LEAD POT

Scale $\frac{1}{4}$" = 1'-0"

EM Nov. 25. 50

All other piping omitted



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Process in Detail, contd

Conversion, contd

Each pot cover carries (1) a dip pipe to carry the vapours down into the molten lead, (2) a thermo well, and (3), a gas outlet. There are horizontal baffles or splash plates to diminish the entrainment of lead. The first and second pre-heaters also have a connection to an equalizing line with a stop valve. These equalizing lines connect into the bottoms of the convertor columns, item #8, and are normally closed. They provide a convenient location for pressure gauges (on branches below the stop valves), and could be used to by-pass a pot in emergency, and they can be used to diminish the chance of forcing lead back upstream during an interruption of the benzene flow.

The lead pots hang, by their rims, in a row, each in a gas-fired, fire-brick lined furnace, the first preheater being between the other two pots.

Anniston report 1642 of Oct.15/50 gives the following approximate figures for temperatures in the conversion system:

Vapouriser (Double coil type)	Benzene in 25°C	out 340°C	(137° reported Aug/47
	Flue gas in 700°C	out 190°C	
1st Preheater	Combustion gases	650-700°C	
	Lead temperature	570-600°C	(380°C, Aug/47).
	Vapour exit	550-580°C	
2nd Preheater	Lead temperature	720-740°C	
	Vapour exit	650-670°C	
Convertor	Lead temp. controlled at 840 ±5°C. (but see "Comments on Process", pages 111- below)		
	Vapour exit	710-720°C.	
Column	Top	80°C - sometimes higher--up to 97°C	
	Bottom	135-250°C - normally about 200°C	

The furnace casings had a temperature of about 100°C as judged by hand, November 1950.

Process in Detail, contd

Conversion, contd

Mr. Harden reported the following temperatures in August 1950:

Unit No.	6	7	8	9	10	11	12	13
Vaporiser	150-200	320	180	150	120	300	350	310
No.1 Preheat	480	530	450	495	480	560	570	540
No.2 Preheat	720	730	750	750	740	740	740	745
Converter	745	785	815	775	800	820	790	825
Top Column	85	90	83	86	80	85	90	80
Flue gas Inlet	All couples U/S : -Sheaths burnt out							
Flue gas Outlet	175	235	322	316	230	190	250	200
Benzene flow	170 U.S.G. per hour							

Earlier records are given in a copy of the plant record sheet page 51 below.

There is a Multipoint temperature recorder for each conversion unit and a recorder-controller for the conversion pot temperature. A sample strip of the multipoint chart was sent to Mr.W.M.Cooper. Oct.31/47.

The pressure drop through the double coil vapourizer, at 170 USG benzene/hour, was 8 - 10 psi.

Pressure of vapour entering 1st preheater	50-60 psig
Pressure of vapour leaving 1st preheater	38-45 psig
Pressure of vapour leaving 2nd preheater	15-25 psig
Pressure of vapour leaving convertor	4-7 psig

The pressures vary a little as the depth of lead changes by entrainment of lead from the pot, and as carbon builds up in the gas passage. The gauges waver a little because of the bubbling and swinging of the lead in the pots.

Carbon builds up around the baffles and in the pot exit lines during operation, causing a slow increase in back pressure. This increases the retention time of the vapours in the system, and causes the conversion reaction to continue a little beyond the point thought best, so the operator lowers his pot temperatures a little to compensate. See Effect of Variables, page 114 below. Recently, the back pressure on some of the units has been purposely increased by the insertion of orifice plates in the lines from the convertors to the columns, in the hope of

Process in Detail, contd

Conversion, contd

attaining the same degree of reaction with usefully lower pot temperatures, so that the pots would last longer. (The vapours on these units can be heard roaring into the lead trap). This is experimental, and experience is showing that the increased pressure on the convertor gasket may cause troubles which outweigh the possible advantages. Mr. Ellenburg's letter of Dec.14/49 states that two orifice plates of stainless steel 1/8" thick are used, one with a 1.31" orifice between lead trap and vapour pipe and the other with a 1.14" orifice, between vapour pipe and column. This arrangement gives the required pressure drop without using too small an orifice. The pressure gauge on the vapour pipe thus reads the pressure between the two orifices.

Mr. Ellenburg gives the following comparison of conditions with and without the orifice plates:

	Gauge Pressures	
Location of pressure gauge	No.10 unit old conditions	No.12 unit with orifice plates
After #1 preheater	29 psig	46 psig
After #2 preheater	18	33
After Convertor	--	21
After 1st orifice plate	--	13
After 2nd orifice plate (column)	3	3

Convertor temperatures in the "pressurized" pot were around 785-790°C for 17.5% conversion. The lower temperatures may save a little fuel gas as well as lengthening the life of the pots, and in the early runs, the deposition of carbon in the gas passages appeared to have been greatly diminished.

In November 1950 pressures up to 20 psig were observed after the first orifice plate.

The following operating conditions were reported in September 1947:

> "The benzene pumps deliver at pressures of up to 100 psig, and the benzene reaches the first lead pot at about 50-60 psig (fall in pressure through the control instruments), the second pot at about 38-45 psig, and the third pot at about 15-25 psig, and leaves the third pot at about 4-7 psig, the drop in pressure in each pot being due mainly to the hydrostatic head of molten lead."

Process in Detail, contd

Conversion, contd

Natural gas is used to heat the convertors and stills and there is a separate gas meter for each unit. These meters are installed in the room mentioned above, page 31, which also contains the benzene flow controllers. There is a stop cock on the gas main immediately inside the room, with a long handle by which it could be operated from outdoors. The main then has a branch to each unit, each branch having its own stop valve, then a pressure reducer. The line then branches again, one branch serving the burners on the #1 preheater, under purely manual control, and serving the pilot burners on all the units, and the other branch serving the control valves for No.2 preheater and the convertor. (See page 107 below).

The flue gases from the #2 preheater and from the convertor go downwards to cross flues leading to the bottom of the setting of #1 preheater, so that that unit gets a good deal of its heat from the flue gases. The #1 preheater setting has gas burners of its own to supply any additional heat needed and for use in starting up from cold.

The temperatures of the lead in the two preheater pots are controlled by hand operation of the gas valves, but there is a high temperature alarm which would cut off the gas on #2 if the temperature should run away, say, by leakage of benzene vapour into the furnace. See also under instrumentation, page 107 below.

Typical operation records are given on page 51 below.

Process in Detail, contd

Note on Starting a Conversion Unit

Mr. H. L. Harden, August 1950, reported as follows:

"The start-up was witnessed of a diphenyl unit from cold, after cleaning and minor repairs. The following instructions apply to an Anniston unit using natural gas. Other fuels may necessitate slight modifications.

"Put on one burner of each unit. Throttle back until warm enough to keep alight. The use of the pilot light will assist in keeping the burners alight. Flue gas is to go up the by-pass stack to avoid burning-up the vaporiser coil. All vents on the convertor should be open in the early stages of warming up. As the setting warms up the other burners are lit one by one, till all are alight and burning steadily. Burners are adjusted (by hand or automatically by controller) to obtain the desired temperatures. Only the convertor temperature appears critical. When temperatures are approximately correct, start CO_2 through vaporiser, the preheat vessels and convertor to the column and out by the column vent. Divert flue gas from by-pass to vapouriser to warm up the latter, and close vents on the preheaters. Pressure on the preheaters should then build up and enable leaks to be discovered. Development of c.20 psig on No.1 and c. 10 psig on No.2 takes about $\frac{1}{4}$ - $\frac{1}{2}$ bottle of CO_2 and it is assumed that the pressure build-up is a sufficient guarantee that the air has been adequately purged. There should of course be no pressure to speak of in the convertor if the column is properly vented. Then, provided temperatures (including vaporiser) are about right, cut off CO_2 cut in benzol feed at a low rate (60 USG per hour). As soon as the lead trap begins to smoke, indicating that benzol feed has reached it, close the column vent, put water on the condenser and open valves to reflux split box, benzol run-back and biphenyl line to crude tank. The latter line must be hot before starting until sufficient feed of crude diphenyl is available to prevent the line from making up.

"Cautiously increase the benzene feed by approximately 10 USG per hour every half-hour or so. The governing factors in effecting this increase are maintenance of preheat temperature (which tends to fall as the benzene feed increases) and the avoidance of high convertor temperatures.

"The preheater must on no account be allowed to cool to anything approaching the melting point of the lead. Maximum temperature of the convertor should be 830-840°C (800°C if operating under pressure). The benzene feed should also be checked frequently until confidence is established because any mechanical or electrical failure here will cause a suck back of lead into the vaporiser coil."

Process in Detail, contd

Note on Stopping a Conversion Unit

It is of course important to prevent ingress of air to the units and to take care that the lead is not forced backwards through the system by allowing the pressure to fall at the inlet end of the system. When the gas burners have been cut off, the benzene feed is stopped, and CO_2 is forced into the benzene inlet line to the vapourizer until liquid ceases to condense in the converter condenser. A full bottle (about 56 lb) of CO_2 is used. During this period the uncondensed gas stream, consisting of hydrogen and some CO_2, will have been going forward to the hydrogen compression system, to prevent the loss of its content of benzene, and the CO_2-hydrogen mixture may be vented to waste after compression and cooling. When the distillation ceases, the system is closed off from the hydrogen collecting header, and from the crude diphenyl tank, and the run-back line to the benzene recycle feed tank, and vented through the pipe which passes from the top of the reflux split box to a point above the roof.

The conversion unit may then be opened up for inspection and repair.

Process in Detail, contd

Note on Emptying a Lead Pot

When a pot has to be emptied, the usual shut-down routine is applied, the system allowed to cool a little, the pipe joints and cover joint broken, the space on the ground floor below roped off for safety, then the cover, with its dip-pipe and thermo well hoisted out by means of a stirrup bolted to the central flange. The lead in the pot is, of course, still molten, and a little drips off the dip-pipe. The cover is laid on the floor in a convenient place. The surface of the lead in the pot is usually covered with floating solid debris, and this is dredged out by means of the long-handled perforated ladle (see sketch #1) and knocked out in the wall of the box frame on the furnace cover. It is convenient to remove the lead trap, to make room to work, and in most cases the trap will contain lead which has to be melted out as described later, page 92 and will contain solid carbon, iron oxide, etc.

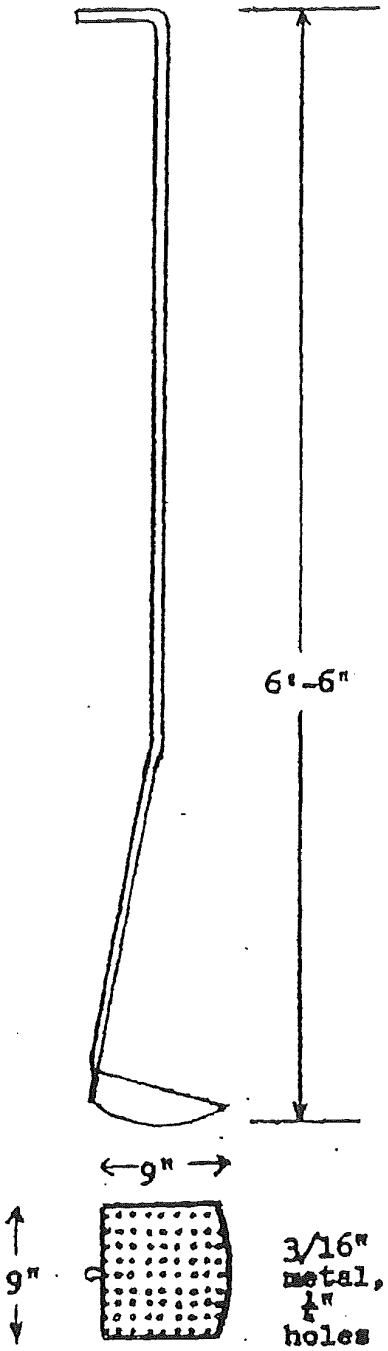
The lead molds are made of channel iron, about 2" deep overall and up to 36" long, 6" wide, with sloping ends welded in. Ten of these molds are then placed, five on each side, near the top of the pot, and two coloured men take turns to dip out the molten lead by means of the unperforated ladles, (see sketch 2) and pour it carefully into each mold in turn until all are full. This is rather hot work, especially in Summer, and the ladles of lead are rather heavy, but the job is not too bad. The operator uses the edge of the pot as a fulcrum to lift the ladle, and steps backwards a little, dragging the ladle upwards until he can get a sufficient purchase on the handle to lift the ladle over to the row of molds. He wears a face mask, and leather gloves, but no other protective clothing. The lead may spit a little if there is moisture in the molds, and casual passers-by have to take care not to step into the molds. The ladle handle is occasionally wiped with a wet rag to cool it.

When ten molds are full, the two men take a breather for about 15 minutes until the lead has frozen over (tested by poking with a rod), then the molds are dragged a few yards away, and inverted to dump the ingots. Double handled tongs are used. The molds are then refilled in exactly the same way, and so on until the pot is practically empty, about 60 ingots in all.

The pot is then hoisted out of the setting by means of a sling chain, bolted to the companion flange, care being taken that the bolts holding the halves of the flange are still effective, and the pot is run along until it comes over the hinged part of the floor, so that it can be lowered to the ground floor. Care is taken to close the hinged part immediately the operation is ended.

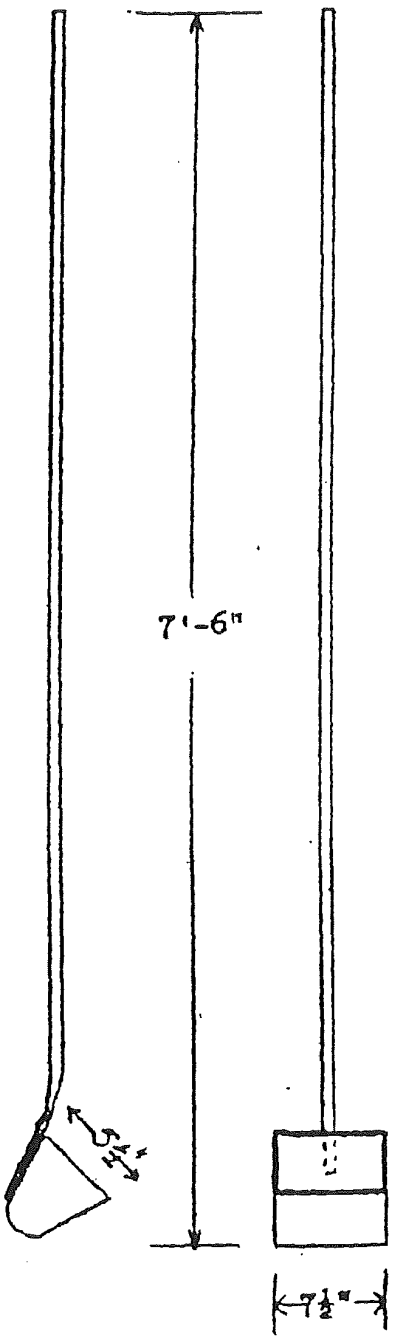
Handles

of 1" piping



Sketch

No.1



Sketch

No.2

DSW 462096

Process in Detail, contd

Note on Emptying a Lead Pot, contd

Sometimes a pot is moved with the lead still in it, for example, if the distributor assembly will not break loose because of carbon incrustation, or if a sound pot is to be moved from one setting to another, but such cases are quite rare. The lead would be allowed to solidify before the pot was moved. The usual procedure is to bale out the lead as described above.

The lead ingots usually leave the molds quite easily, and they are stacked near the unit until the new pot is in place, then they are packed, on end, in the pot, and melted down, and any lead needed for make-up is added.

The cover with the dip pipe and thermo well can, of course, be lowered into position only while the lead is molten.

Process in Detail, contd

Note on the Maintenance of the Lead Pots

(See also the notes on item #5, pages 121 below).

The practice sheets, pages 120 & 121 below, give some figures for the frequency of repairs.

The first preheater with its welded-on cover, low vapour velocities and relatively low temperature can run for years without attention. The second preheater, and more particularly, the convertor, suffer from deposition of carbon in the vapour passages of the cover, damage to the dip pipe, thermowell, and baffles by high temperature and by the mechanical and possibly corrosive effect of the lead, failure of the pot wall, usually by corrosion-erosion from inside, at or below the level of the lead, cracking of the weld between the pot and its rim (this weld carries the weight of the pot and contents and there is continued gentle bumping of the lead and vibration of the pot), failure of the cover gasket or warping of the cover flanges, and from entrainment of lead in the vapour stream. If lead comes in contact with the outside of the pot, from a gasket leak or from a perforation of the pot wall, it may cause marked erosion, apparently from the solvent effect of lead oxide on the stainless steel, but this is not a major cause of failure.

If there is a partial blockage in the vapour outlet, it may show up as a darker patch on the glowing pipe. The pipe leaving the convertor needs cleaning about each 3 weeks and that from the second preheater about once every 8 weeks. Carbon may also build up inside the cover, round the vapour exit, or in the orifices of the baffle plates around the distributor. The baffle plates slowly sag, apparently purely from high temperature. A convertor pot usually runs 5 to 9 weeks before needing overhaul.

The carbon may be burnt out of a pipe by heating with an oxy-acetylene torch, then turning off the acetylene, but care must be taken not to overheat the metal when the carbon burns in the oxygen stream, and the welder should take care not to breathe too much of the vapour.

There has been some discussion of possible overheating of the pot walls above the level of the lead, but apparently the splashing of the lead on to the walls does a little cooling, and of course the furnace draught is downwards in the cases of the second preheater and the convertor.

Mr. Harden, August 1950, suggested to Anniston that a stream of CO_2, passed through the hot convertor, might remove the carbon deposit by converting it to carbon monoxide. Anniston have

Process in Detail, contd

Note on the Maintenance of the Lead Pots, contd

made a few trials of this, by passing two cylinders full of CO_2 through a unit once a week, and though the unit finally choked with carbon, it was believed that the choking had been delayed by the use of CO_2.

Although the pressure inside the convertor is relatively low, the joint of the convertor cover gives a great deal of trouble, because the temperature is near the yield point of the metal. The edges of the cover gradually bend downwards and the edges of the companion (backing) flange under the pot lid bend upwards until they meet, and the gasket no longer holds. The gaskets on the convertors are made up from round braided asbestos rope 1" in diameter. See page 87 below.

Leakage of lead from a defective pot will probably be detected first as a drip of lead from the furnace casing. (Note that it may drip from the wrong casing by penetration through cracks in the furnace setting, and care should be taken to examine the undersides of all three pots before deciding which actually is leaking). The pressure gauges on the pots may help to indicate which pot is losing its lead. Leakage of vapour into the furnaces may show up as black smoke in the stack, or as an increase in furnace temperature. See also the stack test discussed under item #6A, page 91 below.

If a pot is sound in some parts and defective in others, it may be made good by welding, for example, a new bottom may be welded in. A pot which has seen service in the #3 position may be shifted to the lighter duty of #1 position and so on.

See notes under "Corrosion", page 22 above, for an account of the corrosion of the pots.

Process in Detail, contd

The mixture of vapours and hydrogen leaving the convertor pot passes through the centrifugal lead trap, item #7, where most of the entrained lead separates, and into the bottom of the fractionating column, item #8.

The underflow of the column leaves via (#9) a carbon trap, through steam jacketed lines to one of (#10) two sump tanks. These tanks are vented to the "off-gas" line from the top of the columns.

At the top of the column there is a (#11) Condenser and (#12) reflux split box, with an equalising vapour line between the two.

The reflux split box is adjusted to give a benzene forward flow containing at the most a few parts percent of diphenyl, and to give an underflow of crude diphenyl containing as low a benzene content as can be attained. The Anniston report 1642, Oct.15/41, states that the crude diphenyl flow contains about 4 to 8% of benzene. Diphenyl still records seen in November 1950 indicated that about 17% of the crude diphenyl was recovered as benzene fraction. See page 53 below.

Mr. A. M. Ellenburg, Nov.12/47, wrote:

> " The reflux flow is used to control the temperature of the underflow from the column at about 200°C. The reflux ratio varies according to the temperature of the condensed benzene. -------(which) varies according to the length of time the condenser has been in service. As the film builds up on the water side of the condenser, the cooling efficiency drops off, and more of the hot reflux must be used to obtain the same amount of cooling. These ratios vary from 70/30 to 90/10------".

The vapour line from the convertor to the bottom of the column has a branch for sampling the vapours for the purpose of making the conversion test, (see below under "Conversion Test", page 75,) and another branch carrying a pressure gauge. The conversion test is performed once a day on each unit, usually in the morning shift.

Process in Detail, contd

Treatment of Off-gas

The off-gas from the convertor columns, consists of hydrogen, saturated with benzene, and it goes via (#25), a finned cooler with water jacket, to (#26) a surge tank, from which it goes to either of two compressors (#29).

The compressors work in two stages, 60 psig and 300 psig., and the compressed gas from the first stage goes downwards through a vertical interstage cooler (#30) to a low pressure tank (#31) where condensed benzene is deposited, then the hydrogen goes on to the second stage of compression. After the second stage the gas goes downwards through the second cooler (#32) into the high pressure tank (#33) where it deposits more benzene.

The surge tank (#26) has a 55" water seal to a hooded vent pipe through the roof of the compressor room, and there is an automatic control instrument which maintains the pressure in the surge tank between 11 and 16 oz. per sq.in. above atmospheric, by reacting on the unloading valves of the compressors.

The benzene which collects in the surge tank gravitates to the benzene receivers (#27) and that which collected in the pressure was formerly discharged by float traps, also into (#27), but the trap seatings failed by corrosion, see page 103 below, and the transferrence is now done at about hourly intervals by means of hand-operated valves. The pressure vessels have gauge glasses, and the discharge lines have sight glasses. Corrosion of copper and alloys in the hydrogen compression system is rather marked and is attributed to the effect of H_2S. See page 101 below.

Oxygen determinations were made on the hydrogen at first, but are no longer thought necessary. After any shut-down, the hydrogen is vented to atmosphere and the system purged with 400 cu.ft. of CO_2.

At Plant "B" there is a hot wire combustible gas recorder permanently installed in the hydrogen stream going from the chlorine cells to compressors for the hydrogenation plant, wired up to stop the compressors if more than a trace of oxygen is present in the hydrogen stream.

Mr. Ellesburg, June 30/48, wrote: DSW 462101

> " -----As you know, our Worthington compressor has two stages and operates at 150 psig and 300 psig at the discharge of the low and high stage respectively. These pressures are not critical and the choice of pressures depends upon the size of the cylinder chamber as well as the amount of gas to be handled---".

See under item #29 below (page 101) for his further remarks.

Process in Detail, contd

Diphenyl Distillation

Crude diphenyl is pumped through steam traced lines from the sump tanks to either of two horizontal diphenyl batch stills (#13). Each still is fitted with (#13B), an external gas-heated coil, on the side of a fire wall remote from the still, through which the charge is circulated by means of (#12A), a submerged centrifugal pump. The "spread" or temperature difference between inlet and outlet of the coil is automatically controlled through the gas supply to the burners, and is automatically recorded. The controller is set for a spread of 20°C, but when seen in November 1950 it was running at about 16° to 18° spread, i.e. the heat input of the burners was falling short of the control limit. See under "Instrumentation", page 108 below, for details of the control instruments.

A typical still charge (Sept/47) was 3400 USG crude diphenyl plus 450 USG recyled "heads & tails" fraction, and the still cycle took about 18 hours.

The vapours pass to (#14) a 20 plate column with (#15) condenser, and (#16) reflux box working at atmospheric pressure. Cuts are taken based on F.P. determination, (but see Mr. Barden's comments below).

The reflux is changed during the distillation and the following is typical of operation (in September/47):

	Reflux	/	Product
At Start	100		0
Benzene Fraction (up to 80°C at top of column)	25		75
Heads Fraction	50		50
Diphenyl Fraction	30		70
Tails Fraction	20		80

Mr. Harden, August 1950, however, reported:

"The total reflux period seems to be practically confined to the still charging operation. The still and column are usually still hot from the preceding batch and the incoming charge starts distilling immediately. Total reflux operation at this stage rapidly cools the column. It can happen that this initial distillation is too rapid for the split box to deal with it. In such a case it is usual to relieve the box by sending a portion to the heads and tails receiver for return to the still. It is essential to avoid sending any of this initial distillate to the benzene recovery system since it is liable to be contaminated with diphenyl and so lead to excess production of high boilers. The charge used at the time of the visit was 3800 USG of crude diphenyl and heads and

Process in Detail, contd

Diphenyl Distillation, contd

"tails from previous run (about 4,200 USG total).

"Some excess pressure is apt to develop at start of distillation, presumably by plugging of condenser by condensate. If this is observed, a vent is available for temporary relief. This pressure build-up was not apparent to the writer but the operator claimed that he could tell by the flow in the split box.

"Circulation through the coil is started before lighting burners. As the system is usually hot, the burners can be put full on quite quickly. The burners are controlled by the "spread" between inlet and outlet temperatures. This is normally set to 20°C, but is reduced to 10-15°C when distilling heads to minimise carry over of good diphenyl.

"Officially, the change-over from heads to good diphenyl should be based on Cr.Pt. (68.4°C). In practice it is usual to change-over arbitrarily after one hour's distillation because the time spent in testing samples means unnecessary diversion of product to heads for redistillation. The arbitrary time factor obviously depends on distillation rate and may not apply on another installation.

"Similarly, the tails fraction is cut out as soon as temperature (see specimen chart) indicates need, as delay in f.p. determinations results in contamination of good diphenyl. Furthermore, it has been found that f.pt. can be O.K., but boiling range out of specification if the cutting out of tails is delayed.

"General rules:-

Use top of column temperature to cut into heads.
Use time and coil inlet temperature to cut into diphenyl.
Use coil inlet temperature (345-355) to cut into tails.
Use top of column temperature to cut off tails."

The fore run is benzene, which is run off to (#21) a small buffer tank (situated near the sump tanks), from which it is periodically pumped to the recycle feed tank.

Process in Detail, contd

Diphenyl Distillation, contd

Each still has three other receivers:

(#18) Heads and Tails, which are pumped back into the still, by means of a submerged pump, for the next batch.

(#19) Diphenyl Receiver. From here the finished product may be pumped by means of a submerged pump to storage tanks, to the Aroclor plant, to tank cars, or to a small flaker.

(#20) Bottoms Receiver. Crude Santowax is pumped here at the end of the distillation, using the submerged pump of the still. This material may be pumped to the Santowax still or drawn off into drums for permanent storage. Excess material may be pumped into a pond.

The distillate flows through (#17) a small open box from which it may be sampled and diverted to either the diphenyl or the "Heads and Tails" receiver. The benzene fore runnings by-pass this box.

Each of these three receivers is fitted with a steam coil and submerged pump.

A typical operating record is given on page 52 below, and a summary of records on page 53 . This record includes figures for the reflux ratios used.

A sample chart from the Multipoint temperature recorder was sent to Mr. W. M. Cooper, in London, Oct.31/47.

Anniston recycle the cooling water in their condenser, at atmospheric pressure, cooling it in a separate heat exchanger. This is to prevent scaling of the condenser by impurities from the pond water. They rely simply on hand control of the cooling water to the heat exchanger to give sufficient condensation without causing freezing of diphenyl in the condenser. The temperature of the recycled water need not ordinarily be above the freezing point of diphenyl, but it is allowed to rise to 80-85°C during the collection of the "tails" fraction. Instrumented control of the cooling water was abandoned in favor of hand control.

The benzene distillate is examined before being pumped to the recycle feed tank, to make sure it does not contain more than 2-3% at most of diphenyl. The operators judge by a greenish colour which appears if there is much diphenyl, and by evaporating a few drops between the fingers, but a freezing point test would be the safer criterion. The diphenyl may have flashed over at the start of the distillation, as described on page 45 above, or may have come over through inefficient fraction (insufficient reflux).

Process in Detail, contd

Diphenyl Distillation, contd

The build up, in the heads fraction, of materials other than benzene and diphenyl is very slow (only about 0.065% on the diphenyl according to Anniston report 1642), but as the fraction is recycled to one batch after another, styrene and other contaminants do slowly accumulate in it, (see under "Flow Sheet", page 28 above, for typical values; see also page 116 below), and the cut between benzene and diphenyl becomes less sharp. Occasionally therefore a heads fraction "cut" from about 90°C to 200°C, amounting to about 10 gallons, is side-tracked, allowed to cool to deposit diphenyl, which is drained off and returned to the system, and the liquid contaminants discarded. Mr. Ellenburg, Jan.14/49, in answer to a suggestion from London, agreed that the heads fraction might be accumulated until there was enough to charge a still, instead of being recycled each time, but he advised against that course on the grounds that a larger store tank would be needed, and that the Anniston method is generally more convenient.

Any impurities boiling just above diphenyl will go into the tails fraction, but this fraction is simply recycled so that the impurities finally go out in the crude Santowax.

Mr. Ellenburg advises against handling or shipping diphenyl in drums because of the difficulty of re-melting. Anniston flake any diphenyl not handled in tank cars or by pipe line. London, Feb/49, did not expect to sell any diphenyl. The diphenyl still bottoms (Crude Santowax) are kept molten in steam-heated tanks if they are to be processed further. The excess production of bottoms at Anniston is put into old drums, and more of it has been pumped away into an excavation. If the stored material is needed again, it is put into a melting pot and then moved by pumping. At one time when smaller (direct fired) stills were used, Anniston purged the diphenyl still system with CO_2 between batches, but discontinued this as being unnecessary with the present stills. The still is simply allowed to cool a little after being discharged, then a little care taken in pumping in the new (hot) charge of crude diphenyl. Still bottoms, discharged at about 400°C have been known to take fire. The circulation through the heating coil is not started until the still has settled down after the charging.

Process in Detail, contd

Distillation of Santowax

This is carried out under about 50 mm of Hg absolute pressure at the receiver, using a DeVine Vacuum Pump (#40). The present equipment was originally set up for the separation of crude Santowax into Santowax O, M and P, which are crude ortho, meta, and para isomers of terphenyl. For Santowax R only straight over distillation is required, with a single receiver (#39) instead of the series of receivers installed in the present plant.

The still charge is received at about 150°C in one of two Charging Tanks (#35A and #35B). These are interconnected at a low level and really constitute one vessel. They contain heating coils and one has a submerged pump (#34D), by which liquor is circulated through (#34C) an external preheater coil on the side of a firewall remote from the tanks and still. The coil is heated by gas burners and is supppprted in a steel flue with firebrick lining. The material is preheated to 230°C. The same pump is used to transfer the hot liquor to the (#35) still which has a similar heating arrangement. The still capacity is 6-7000# of crude. The still has a 10" vapour outlet (and explosion disc) to (#36), 12 plate column. This column has (#37), a condenser consisting of a single jacketed tube in the form of a zig-zag. (See pages 104 & 105 below). This condenser was used in place of the original shell and tube condenser because the latter often became blocked with frozen material. There is a reflux split box (#38), but reflux is used only in the (rough) separation of crude Santowax into the o, m & p isomers.

The Santowax R distillate is run from the receiver (#39) to the buffer vessel (#39B) and pumped from there, by (#39A) to the Aroclor plant, the Santowax R flaker, or to the HB-40 plant, as required. The still bottoms are sold as Montar 9, any excess production being dumped.

Dr. Hardy reported, Oct.17/47:

> "A total forward flow is maintained on the split flow box throughout the Santowax R distillation. This is necessary in order to get over some of the higher boiling constituents which boil above the terphenyls. These materials must be present to bring the hold point of the Santowax within our specifications.
>
> "Our original Santowax still had no reflux at all and the new still (1943) is supposed to duplicate the product of the old still".

Process in Detail, contd

Distillation of Santowax, contd

Distilled Santowax is a reddish brown liquid crystallising to a yellow solid at room temperatures.

The distillation is controlled by "Hold point" determinations -- there are two such points and the specification for the product requires:

Upper hold point 137-143°C (changed Sept.23/47 to 137-145°C)

Lower hold point 45-60°C (changed Sept.23/47 to 45-63°C)

A sample strip of temperature recorder chart for various points on the Santowax still system was sent to Mr. Cooper, Oct.31/47.

On January 6/49, Mr. A. M. Ellenburg wrote:

"At one time, distilled Santowax was allowed to solidify in drums, and shipped, but it was noticed that the product tended to segregate as it solidified. As a result, the outer layer of the drums contained the high melting portion of the Santowax R, and the center of the drum was made up of the lower melting fractions.

"In order to obtain a uniform product, so that a whole drum would not have to be melted to get out 10 pounds, the idea of flaking the Santowax was proposed. A chill wheel is used to cool the material, and a blade scrapes the material off the rolls, from which it falls into bags or lined barrels. (Note, however, that Santowax has two "hold" points in its crystallization curve, see page 13 above, and gives first a plastic mass at about 140°C, then a waxy solid at about 50°C, a fact which affects the flaking process. E.M.)

"The lumpy material results because the Santowax is not chilled sufficiently before placing in the bags, and a slight fusion of the flakes results. However, the partial chilling is enough to give a uniform solid product. The crude Santowax could be handled a similar way if necessary, because its characteristics are similar to those of Santowax R. A big tank for storage of the molten crude is, of course, the easiest solution to a storage problem".

Anniston, Jan.14/49, pointed out that the Santowax flaking could be done on the same drum as the diphenyl flaking.

See also the note under Specifications & Test Methods, page 67 below.

MONSANTO CHEMICAL COMPANY
ANNISTON, ALABAMA, PLANT
FOR 24 HOURS BEGINNING 7:00 A. M. 1-9- 1977

Diphenyl Operating Record

UNIT NO. 2

No. 1 SHIFT

OPERATOR Castle, Bell, Dempsey, Simmons   CHIEF OPERATOR [illegible]

		Column	Sump Line	Conv'n Lead	No. 2 P.R. Lead	No. 3 P.R. Lead	Sump Tank Measurement	Hydr'n Compr Seal
170	21.7							
✓	✓	90	222	822	745	385	[illegible]	O.K.
✓	✓	90	200	822	[illegible]	[illegible]		
✓	✓	90	200	822	750	380	[illegible]	O.K.
✓	✓	90	200	825	750	[illegible]		
✓	✓	90	200	825	750	[illegible]		
✓	✓	90	200	827	750	[illegible]	1' ?	O.K.

CONVERSION SAMPLE

TIME	8:10
CONV'N TEMP.	825
NO. 2 P.R. TEMP.	745
SP GRAVITY	[illegible]
AT TEMP. C.	[illegible]
CONVERSION	[illegible]
FLUE GAS	

Run Back [illegible] ... Heat Flux [illegible]

No. 2 SHIFT

OPERATOR Beck, Vaughn, [illegible]   CHIEF OPERATOR [illegible] Shepherd

		Column	Sump Line	Conv'n Lead	No. 2 P.R. Lead	No. 3 P.R. Lead	Line	Feed	No. 1 Fresh	No. 2 Fresh	Water Out	Sump Tank Measurement	Hydr'n Compr Seal
170	21.7	90	200	825	750	385	90	45	41	16	26 26 22 26	1'5" ?	O.K.
✓	✓	90	200	828	725	390	✓						
✓	✓	90	200	828	750	395	✓						
✓	✓	90	200	828	725	390	✓			17		1'3" Empty	O.K.
✓	✓	90	204	827	750	392						2'6" ?	O.K.
✓	✓	90	200	827	750	390		✓					
✓	✓	90	200	827	750	392	90	45	42	16		Empty Empty	O.K.

No. 3 SHIFT

OPERATOR Adams, Mullinax, McClusky   CHIEF OPERATOR White (Flowers)

		Column	Sump Line	Conv'n Lead	No. 2 P.R. Lead	No. 3 P.R. Lead	Line	Feed	No. 1 Fresh	No. 2 Fresh	H2 In		H2 Out				Water Out				Sump Tank No. 1	No. 2	Hydr'n Compr Seal
170	21.7	90	200	828	750	390	90	46	41	17	51	67	24	24	24	26	27	27	27	27	1"	Empty	O.K.
✓	✓	90	200	828	750	390	✓	✓															
✓	✓	90	200	825	745	390																	O.K.
✓	✓	90	200	828	745	390	90	46	41	17	48	62	24	24	24	25	27	27	28	28	1'	?	✓
✓	✓	90	200	828	745	390																	O.K.
✓	✓	90	200	825	750	390	✓																
170	21.7	90	200	825	750	390	90	46	41	17	56	64	26	26	27	27	28	28	28	28	1'7"	?	O.K.

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[redacted]

[redacted]

Diphenyl Distilliation Record

Distilliation No. 755 Date Started 4-1 194 7

Operator Start RCL Shift 3

Still No. #2 Operator Finish P Shift 1

Started Charging Still at 11:15 P.M.

CHARGE:

H&T Tank; inched before 37 inches after 4 gallons charged 396

No. 1 S.T. inches before 39 inches after 0 gallons charged 3464

No. 2 S.TT inches before ____ inches after ____ gallons charged ____

No. 3 S.T. inches before 0 inches after 15 gallons charged ____

No. 4 S.T. inches before ____ inches after ____ gallons charged ____

Benzol Still " " ____ inches after ____ gallons charged ____

Total Gallons Charged 4060

Circulating at 12:[illegible] A.M.; Fire on at 12:5 A.M.

FIRING SETTINGS:

1. On 11C at [illegible] A.M.
2. On [illegible] at 12:[illegible] A.M.
3. On 5C at 3:15 A.M.
4. On 30 at 4:[illegible] A.M.
5. On 2C at 6:30 A.M.
6. On ____ at ____
7. On ____ at ____
8. On ____ at ____

BENZOL: Ran 15 " or 345 gal.

HEADS: Started over at 3:15 A.M. Finished at 4:[illegible] A.M.

Ran 18 " or 1735 lbs.

TAILS: Started over at 6:30 A.M. Finished at 7:00 A.M.

Ran 10 " or 1411 lbs.

DIPHENYL: Total in Distilliation 71 " of 5691 lbs.

HIGH BOILER: Gross Weight ____ lbs. No. of Drums ____

Net Tare Weight 117.5 lbs.

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Process in Detail, contd

Summary of Records of Diphenyl Still Working

(Taken November 1950 from the plant sheets as on the preceding page)

New Material to Still U.S.Gallon	Benzene Fraction off U.S.G.	Heads lb (8 lb/USG)	Tails lb	Diphenyl lb	Bottoms lb
3485	437	2024	964	18,785	4400
3394	598	1928	1157	17,459	5400
3394	667	2121	1157	15,470	4400
3485	506	2217	1446	17,680	5000
3869	690	2121	1157	17,238	4600
3394	690	2217	1253	17,017	4600
3485	874	2121	1350	16,133	--
3485	598	2024	1253	16,796	--
3576	391	2314	1157	15,249	--
3485	598	2314	1060	19,448	4800
3485	598	2410	1350	18,785	4200

SPECIFICATIONS AND TEST METHODS

Raw Materials

Benzene

In September 1947 Anniston were in a favourable position in respect of the quality of the benzene received. By a special arrangement with the suppliers in Birmingham, Alabama, they received material usually running 5.390C Cr.Pt., (on the sample "as is"), and at least up to "Nitration Grade" on all other points except possibly sulphur content. The arrangement was so good that Anniston were able to rely almost entirely on Birmingham control of the quality of the deliveries. The only specification was that implied in the description of the material as "Industrial Grade Benzene", and the deliveries were so much above specification that the specification was inoperative. Because of this position Anniston do not perform routine tests on benzene deliveries made to the diphenyl plant. There are only very few test reports on file, but the following which were available were believed to be representative of the material usually received:

Tank #	GATX 35461	GATX 21357	?	GATX 19403
Date	Oct.7/47	--	Aug/47	June 17/48
Fr.Pt.°C.	5.46	5.46	5.4	5.46
Sp.Gr.@ 60°F.	0.8845	0.8845	0.8825	0.886
Dist. range	--	--	0.9oC	1.°C (corr.)
1st drop	79.7	79.7	79.5	79.4
5%	80.2	80.2	80.2	80.1
50%	80.3	80.4	80.4	80.3
95%	80.4	80.5	80.4	80.4
dry	80.4	80.5	80.4	80.4
Colour	Water white	w.w.	--	w.w.
Wash test	#1	#1	--	#1
Total sulphur %	0.05	0.05	--	--
Acidity mg NaOH/g	--	--	--	0.0004
H_2O p.p.m.	--	--	--	700
Paraffins	--	--	--	nil

The freezing point method used was that of J.I.E.Ch.Vol.10, p.1010, (see page 56 below).

For the other tests, the latest A.S.T.M. methods were used.

In order to check the Anniston method of determination of the freezing point of benzene, against the Ruabon method, and to make sure that the Anniston material really had such a high test, by British test methods, a sample from rail tank GATX 21357, Nov/47, was sent to Ruabon. It was found to have a crystallizing point of 5.4°C by the standard Ruabon method.

Specifications & Test Methods, contd

Samples, representing material available in Britain, have been examined at Anniston:

	Sample NB279 Taken Jan.24/48	Sample 60768 Taken July 27/48
Fr.Pt.°C	5.16	5.35
Sp.Gr.@60°F	0.886	0.883
Dist. range		1.2 (corr.)
1st drop	79.6°C	71.4
5%	80.2	80.2
50%	80.3	80.2
95%	80.4	80.3
dry	80.6	80.6
Colour	w.w.	w.w.
Wash test	#9	#4
Acidity mg NaOH/g	0.0016	0.004
H_2O p.p.m.	458	621
Paraffins		nil

Anniston's comments on the British sample NB279 were that it was not up to the quality of the Anniston supply, it had a high acid wash colour, indicating incomplete acid treatment, and the presence of unsaturated or hydroxy compounds, but that it should give good diphenyl if the fractional distillation of the diphenyl were good enough. The sample left an odour of tar at the end of the distillation.

Anniston's comments on British sample 60768 were that its acid wash test was just at the limit of the Anniston specification (for "Industrial Benzene") again indicating incomplete acid treatment of the material. Material to this sample would produce good diphenyl, though a little extra attention to the diphenyl distillation might be needed.

The effects of impurities in the benzene were discussed in Anniston reports 1640, p.19, & 1642, pages 1 & 2. For a time no very high standard was set, but in recent years, trouble with the stability of Arcolors used for electrical work has renewed the interest in this subject. See pages 111- below.

In a "short form report", No.2152, dated November 17/47, Mr. A. M. Ellenburg of Anniston reviewed the quality requirements for benzene for making Biphenyl and Chlorbenzenes. He found the available evidence to be inconclusive, and recommended maintaining a limit of 5.0°C minimum for Diphenyl and 5.2°C for Chlorbenzenes.

Dr. E. E. Hardy, Dec.23/47, advised caution in accepting a lower standard, for benzene, which contained paraffins, would

Specifications & Test Methods, contd

yield styrene and apparently vinyl biphenyls. Here again the chlorine derivatives were blamed for skin trouble in the Aroclor plant. He pointed out that though the chlorinated products may be stabilized by being held at high temperatures, it is not certain that this process will go far enough.

On April 28/49, Mr. Ellenburg mentioned the beneficial effect which high temperatures in coke ovens are known to have on the purity of the benzene (freedom from paraffins).

See also the discussion of "Effects of Variation in Benzene Quality," page 111 below.

Test Methods for Benzene

Fr.Pt.

As mentioned above, Anniston use the method of Ind.Eng.Chem.10., p.1010 (20 cc sample in the "as is" condition, put in a test tube in an ice bath, and when the solid benzene begins to separate, the temperature will remain constant for a time. This temperature is taken as the solidifying point. Accurate ± 0.05°C.

Other Tests

Anniston use A.S.T.M. methods, but on Feb.15/49, Anniston reported that the British (N.B.C.) form of the acid wash test was simpler, especially in respect of the preparation of the standards. On applying the N.B.C. test to their own benzene, they found it to give a colour matched by only 0.1 g dichromate per liter of water as against the N.B.C. standard of 1.0 g. The same benzene gave colour #1 on the A.S.T.M. acid wash test.

Specifications & Test Methods, contd

Lead for the Pots

No specifications exist on lead used in the diphenyl lead convertor units other than that "chemical lead" is to be purchased. Apparently the effect which impurities in the lead may have on the stainless steel, is more feared than any effect on the conversion process itself.

Mr. W. W. Wade of Anniston have the following information:

"A typical analysis of chemical lead as supplied by the St.Joseph Lead Company, 250 Park Ave., New York, New York, was:

Pb:	99.9272%
Cu:	0.0608
As:	
Sb:	0.0001
Sn:	
Fe:	0.0001
Zn:	0.0001
Cd:	0.0003
Ni:	0.0048
Co:	
Ag:	0.0066
Bi:	nil

"Elements that should be particularly avoided, as observed in practice at Anniston, and as recommended by manufacturers of stainless steels, are, in order of importance: tin, zinc, bismuth, antimony, and cadmium. Tin, zinc, and bismuth should not exceed 0.0001% max., antimony and cadmium, 0.0005% max.

"Caution should be taken in plant operation to avoid contamination by any of the above elements."

Spectrographic analysis has been found useful for checking the purity of the lead for the convertors. We are warned to look out for impurities which may be picked up in any reused lead.

MCL have found a source of supply of lead apparently up to the Anniston standard except that the bismuth content is 0.001% and the lead percentage only 99.9955.

Anniston commented that they did not think 0.001% of bismuth would cause any serious trouble.

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Specifications and Test Methods, cont'd

Finished Products

Diphenyl (Technical) code 3980-500-74-09
(Sampled from the rail tank in the case of tank car sales) authorized July 16/45, supercedes Mar. 2/38.
Production Dept. No. 09

Appearance	yellow to pale amber solid,
Colour	equal to 2.0% aqueous $FeCl_3$ max.
Fr.Pt.	68.7° min.
Dist. range	2.5°C. max. including 255°C.

Freezing Point of Diphenyl

Standardised Anschutz thermometer 40-100°C. mounted vertically near shaft of a small electric stirrer. Bulb about 1/4" above stirrer blade, and 3/8" from the shaft. Magnifier clamped to thermometer.

Melt and mix the sample. Pour about 120 c.c. into 250 cc Griffin beaker making sure thermometer is not above 100°C. Set beaker under stirrer so blade is ½-1" above the beaker, and the thermometer immersed to 40°C. mark or more.

Run stirrer at moderate rate through test. When crystallization begins, the temperature will rise quickly about 0.4°C., then become constant for 2-5 min. before slowly falling. Highest reading taken avoiding parallax.

Dist. Range of Diphenyl

Plant "B" method slightly modified by R.Sudhoff, Mar.8/37.

200 cc Pyrex (Corning 1600) distilling flask. Neck wrapped with wet asbestos paper allowed to dry on. 200-275° range precision Taylor type thermo, engraved stem 0.2° division. 1° = 4 m.m. Both bulb and expansion bulb to be below the side tube. 6" auxiliary thermo. 0-150°, attached with bulb 2/3 way down the emergent stem. Cover both thermos

Specifications and Test Methods, cont'd

Finished Products, cont'd

with 3/4" glass tube. Correct for emergent stem, and barometric and thermometric error.

Condenser as for Barrett benzol distillation, i.e., 24" x ¼" glass tube @ 75° angle in copper trough. Water at 75-85°C. Auxiliary burner to prevent freezing at exit. Draft shield 5" I.D. x 6" high.

Flask on 6" x 6" piece of 1/4" transite board with 1-1/4' hole. Top of auxiliary bulb of thermometer level with bottom of side outlet. Cork stopper flask to condenser.

Weigh 100 g. sample into flask. Heat with Meker burner till ebullition starts. When vapour ring reaches into the neck, but not to the side arm, cool a little. Repeat 3 times to get thermometer and flask neck heated. Distil 2 drops/second into beaker on a balance.

Read:

1st drop	1%	3%	5%	50%	90%	95%	96%	97%	Dry
*				*					*

read auxiliary thermometer at *.

Record weight in receiver 1 minute after removal of flame.

Dry point: observe when last drop leaves bottom -- needs two observers.

(760 - Barom. reading) x 0.036 = T_1
(1st drop - average of auxiliary readings) x 0.000153 x no. of degrees above the bath = T_2.
Add T_1 and T_2 to observed readings.

Specifications and Test Methods, cont'd

Finished Products, cont'd

Diphenyl Colour

Melt 120 g diphenyl in low form Griffin 150 cc beaker. Compare @ about 80°C. with ferric chloride samples in bottles of same diameter as beaker.

Standards 25g. $FeCl_3 \cdot 6H_2O$ 0.625 g. $COCl_2 \cdot 6H_2O$ in 200 cc H_2O + 15 cc HCl (S.G. 1.18), make up to 250 cc.

Make Standards

½	10 cc stock per 200 cc
1	20
1½	30
2	40
3	60
4	80

Standard Nos. are % $FeCl_3 \cdot 6H_2O$.

Sample G-4072 of Finished Diphenyl was sent from Anniston to Ruabon, Oct.13/47.

Mr. Barden, August 1950, collected the following test results at Anniston:

"Typical Analyses:

Diphenyl

				Lot 246	247		
Date	31/7	25/7	20/7	18/7	18/7	12/5	(1950)
Colour	1.0	1.0	1.0	½	½	1.5	
F.Pt.	68.8	68.85	68.9	68.9	68.9	68.8+	
Dist. Range	0.6	0.7	.5	0.6	0.8	1.0	
1 drop	254.8	254.7	254.8	255.0	254.7	254.7	
1%	254.9	254.8	254.8	255.0	254.9	254.8	
3	254.9	254.9	254.9	255.1	255.0	254.9	
5	254.9	255.0	254.9	255.1	255.0	255.0	
10	255.0	255.0	255.0	255.1	255.1	255.0	
50	255.1	255.2	255.1	255.3	255.2	255.2	
90	255.1	255.2	255.2	255.3	255.3	255.2	
95	255.2	255.2	255.2	255.4	255.4	255.3	
96	255.2	255.3	255.2	255.5	255.4	255.4	
97	255.3	255.3	255.3	255.6	255.4	255.5	
dry	255.4	255.4	255.3	255.6	255.5	255.7	

Date	25/4	27/2	(1950)
Colour	1.5	1.0	
F.Pt.	68.8	68.8	
Dist. Range	1.2	2.5	
1 drop	254.8	254.9	
1	255.0	255.0	
3	255.0	255.0	
4	255.0	255.1	
10	255.1	255.2	
50	255.2	255.4	
90	255.4	255.6	
95	255.6	255.8	
96	255.6	256.0	
97	255.8	256.4	
dry	256.0	257.4	

Test results collected in 1947 are given on the next two pages, 62 & 63.

TECHNICAL DIPHENYL SHIPMENTS

Date	Car No.	Fr.Pt. °C.	Distillation 1st drop	1%	3%	5%	10%	50%	90%	95%	96%	97%	Dry Pt.	Colour	Dist. Range
Sep.3/47	MONX 659	68.9	255.1	255.2	.2	.2	.3	.5	.5	.6	.7	.8	255.9	1.0	.8
	MONX 504	68.9	255.1	255.1	.2	.2	.3	.5	.6	.7	.7	.9	256.1	1.0	1.0
Sep.7/47	GATX 37304	68.9	255.0	255.0	.1	.1	.2	.4	.4	.5	.6	.7	255.8	1.0	.8
Sep.10/47	MONX 147	68.9	255.1	255.2	.2	.2	.3	.4	.5	.6	.6	.7	255.8	1.0	.7
Sep.17/47	MONX 658	68.9	255.0	255.1	.2	.2	.2	.4	.5	.6	.7	.8	255.9	1.0	.9
Sep.19/47	MONX 651	68.9	254.9	255.0	.0	.1	.1	.3	.5	.5	.7	.8	255.9	1.0	1.0
	MONX 659	68.9	254.9	255.1	.1	.2	.3	.4	.5	.5	.5	.6	255.7	1.0	.8

FLAKED TECHNICAL DIPHENYL SHIPMENTS

Date	Lot	Fr.Pt. °C	Distillation 1st drop	1%	3%	5%	10%	50%	90%	95%	96%	97%	Dry Pt.	Colour	Dist. Range
Sep.12/47	185	68.9	255.2	.2	.2	.2	.2	.4	.5	.6	.7	.8	255.9	1.0	.7
	186	68.9	255.1	.1	.2	.2	.2	.4	.5	.6	.6	.7	255.7	1.0	.6
	187	68.9	255.2	.2	.3	.3	.3	.5	.6	.7	.7	.8	255.9	1.0	.7
	188	68.9	255.1	.2	.3	.3	.3	.5	.6	.7	.7	.8	255.9	1.0	.8
Sep.15/47	190	68.9	254.8	.8	.8	.9	.9	255.0	.2	.2	.4	.5	255.6	1.0	.8
Sep.24/47	191	69.0	254.9	255.1	.1	.1	.2	.3	.4	.5	.5	.5	255.6	0.5 brown	.7

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Specifications & Test Methods, contd

SANTOWAX R

The properties of Santowax R as given in "Monsanto Chemicals and Plastics" - 27th Edition - are typical analyses.

Tentative Sales Specification

Color	3.5 - 4.5 NPA
Solidification Hold Temperatures:	
Upper	137 - 143°C
Lower	45 - 69°C
Distillation Range:	At least 80% between 350 - 400°C (uncorr.) This property is not determined on every lot.

See also under "Properties of Materials", page 13 above.

Specifications and Test Methods (cont'd)

Analysis of Santowax R

DESCRIPTION

Santowax R is a yellow solid hydrocarbon mixture of micro-crystalline structure. The main constituents are ortho-terphenyl, meta-terphenyl with smaller quantities of diphenyl and tri-orthophenylene present.

The molten product is chilled on a flaking roll and bagged or put in paper lined barrels for shipment.

LABORATORY TESTS:

1. Color: 3.5 - 4.5 NPA

   Test Method: A portion of the sample is melted slowly over a low heat. Care should be taken not to overheat the sample. Some darkening due to decomposition is possible if the sample is heated above 250°C for any length of time. The molten sample is poured into a clean 15 mm test tube and the color is compared to the standard disc comparator of the National Petroleum Association.

2. Hold Points: Upper hold point 137-143°C
   Lower hold point 45°-60°C

   Changed Sept. 25/4 to: 137-145°
   45-63°

   Method: A 250 cc beaker is filled about three fourths filled with the flaked sample and placed on the hot plate to melt. After the molten sample has attained a temperature of 175°-200°C, the stirrer and thermometer are placed in the beaker. The material is allowed to cool while stirring until crystals appear in the liquid. Then the thermometer is watch closely to see if the temperature holds as the sample crystallizes. This temperature where the thermometer reads constant for a few minutes is known as the upper hold point.

   The stirrer is removed and the sample is now allowed to cool to about 80°C. The sample is then stirred by hand with the thermometer. The chilled material on the sides of the beaker must be intimately mixed with the molten sample in the middle at all times. The temperature at which the thermometer stops after it goes down and rises to 1° to 3° is called the lower hold point.

Specifications and Test Methods (cont'd)

Analysis of Santowax R - Page 2

Standard thermometers are used in this test. No correction is made for stem exposure.

3. Distillation Range: At least 80 per cent between 350-400°C (uncorr.)

Method: ASTM D-20 (modified)

The apparatus is designated in the ASTM method and consists of a flask, condenser tube, shield, receiver, and thermometer.

After assembly of the apparatus is complete, the sample is melted and stirred to insure proper blending.

One hundred grams of the sample is weighed into the flask, the flask is put in place and heat is applied so that the first drip comes over in from 5 to 15 minutes. The distillation shall be conducted at the rate of 50-70 drops per minute and the distillate collected in a weighed receiver.

The temperatures recorded shall be at the first drop and at each 10 degree interval between 350-400°C, the per cent distilled over shall also be noted.

This distillation range specification is subject to change as more history on this test of the product is obtained.

4. Sediment Test: There shall be no insoluble impurities in a molten sample of the product.

Method of Sampling: It will be very difficult to obtain a good sample for this test and, therefore, the best means of having a clean product is to eliminate the sources of impurities and keep a check on these sources.

For the above test, a 3 or 4 pound sample could be melted at hourly intervals to see if impurities were present.

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Specifications and Test Methods (cont'd)

Analysis of Santowax R - Page 3

For a fool-proof method, a mechanical means would be needed at the flaking rolls to remove the metal scraps which are the main contaminant at present. If the rolls were capable of cooling the product to 30°C, so that a cold, brittle product could be obtained, the metal could probably be removed by a vibrating screen of 4 mesh size. A magnet might also be capable of removing the metal scraps. At present the flaker is being resurfaced and a new blade is being installed to prevent the flakings from coming off the rolls.

Specifications and Test Methods, cont'd

Determination of Sulphur in Santowax

Tentative Method:

40 g. sample, 20 g. Nickel formate, 150 cc H.B. 40 (hydrogenated Santowax) in 1000 cc boiling flask. Heat @ rate of 30-40°C per minute to 285°C. Water of cryst. of formate comes out up to 200°. Formate begins to decompose around 240°, and rapidly @ 270-275°.

Heat to 285° and cool to 20-80°C.
Add 100 cc 1:1 HCl and some boiling chips.
Reflux till Ni has dissolved, (foams), with Meyer or similar apparatus attached to condenser, 50 cc ammoniacal $CdCl_2$ (1% of $CdCl_2$ $2\frac{1}{2}H_2O$) in the bulbs.

Wash CdS to beaker or flask.
Make acid to phenolphthalein with concentrated HCl, and dilute to about 250 cc.
Add 10 cc N/10 Iodine and 5 cc concentrated HCl added.
Leave 10 minutes.
Titrate iodine with N/10 thio using starch.
Calculate to % sulphur in Santowax.
Do blank.

Determination of Chlorine in Santowax

30 g. Santowax and 1 g. sodium in dry Ni crucible, 80-100 cc capacity.
Cover.
Heat over low flame, until Santowax has gone, then to dull red.
Remove cover and allow excess Na to burn under hood.
Cool, wash well with H_2O.
Make up to 250 cc.
Determine Cl gravimetrically in 100 cc aliquot.
Do blank by dropping Na into water.

Specifications and Test Methods, (

Santowax R Code G-1301 No Specification.

Date	Lot No.	Upper Hold Pt. °C.	Lower Hold Pt. °C.	Colour NPA
Sep.15/47	47	144.2	60.0	3.8
	48	144.2	61.0	3.8
Sep.22/47	50	142.0	60.0	3.6
	51	142.5	60.0	3.5
	52	142.0	56.0	3.5
	53	142.0	59.5	3.5

Sample G-4073 of Santowax R was sent from Anniston to Ruhben, Oct.13/47.

Specifications and Test Methods (cont'd)

Finished Products (cont'd)

Santowax, (crude individual isomers)

Only small amounts of the individual isomers have been made. Test methods used at Anniston are given below.

Analysis of Santowax O

DESCRIPTION:

This material consists of crystals coated with a eutectic oil. The color is pale yellow due mainly to the adhering oil. The product is about 97-98 per cent ortho terphenyl with traces of diphenyl and meta terphenyl which form the oil.

This product is distilled, mixed in the catch tank and run into drums to crystallize before shipment.

LABORATORY TESTS:

1. Color: 1.5 NPA

   Method: The sample is melted with precautions not to overheat and poured into test tube. The color is compared to the standard disc comparator of National Petroleum Association.

2. Hold Point: 53°C - 55°C.

   Method: A portion of the sample is melted to fill a 250 cc beaker one-half full. A stirrer and a thermometer (standardized) are inserted in the beaker and the material is allowed to cool while stirring slowly. When the temperature gets down to 55°C, add seed crystals slowly until sample begins to crystallize. Record highest temperature reached after thermometer starts to rise.

3. Distillation Range: 10°C maximum range. This range should include 335°C (corr)

   Method: ASTM D-20 (Modified)

   Same procedure as for Santowax R.

Specifications and Test Methods (cont'd)

Finished Products (cont'd)

Analysis of Santowax O - Page 2

The temperatures recorded should be at the first drop and at each 10 per cent of distillate up to 90. Then the temperature should be taken again at 96 per cent of distillate.

Necessary stem temperatures should be taken to correct for the emergent stem. Correction should also be made for the atmospheric pressure.

The distillation range test is subject to revision when more history on this test of the product has been obtained.

4. Sediment Test: No insoluble impurities present in the molten sample.

   Method of Sampling: The material shall be filtered through 100 mesh screen as it is loaded into drums. A sample of the molten product shall be inspected for solids. The drums also shall be inspected for cleanliness.

Specifications and Test Methods (contd)

Finished Products, contd

Analysis of Santowax "M"

This material is a yellow solid of crystalline structure. The purity is approximately 95 per cent meta terphenyl with para terphenyl making up the remainder. The present method of separation yeilds a meta-para eutectic at 3.3 per cent para which accounts for most of the para contaminants.

At present this product is crushed to lumps one inch or smaller, and shipped in paper bags or paper-lined barrels.

Laboratory Tests:

1. Color: 1.5 - 3.5 NPA

   Method: The sample is melted with care not to discolor by heat and poured into a 15-mm test tube. The color of the molten sample is compared to the standard NPA disc comparator.

2. First crystals: 90°C.

   Method: A 250 cc beaker is filled about two-thirds full of the molten sample and placed under a stirrer and a standard thermometer. The material is allowed to cool with stirring until the temperature reaches about 90°C. Seed crystals of the sample are added slowly as the cooling continues. The temperature at which new crystals come out of the solution is recorded as the "first crystals" point.

3. Hold Point: 83°-85°.

   Method: Stirring is continued in the above sample while the temperature goes down and then rises 1-3°C to the hold point. The highest temperature at which the temperature remains constant for a while is recorded.

Specifications and Test Methods (cont'd)

Finished Products (cont'd)

Analysis of Santowax "M"- Page 2

4. Sediment Test: No insoluble impurities are present in a molten sample of the product.

Method of Sampling: A sample of the crushed material as it is prepared for shipment shall be melted for this test. The product can be filtered before it is remolded after removing the water. Then it can be crushed to 1/2" size for shipment.

Under the present open air method of handling this material, the dirt is especially hard to keep out.

Specifications and Test Methods (cont'd)

Finished Products (cont'd)

Analysis of Santowax P

DESCRIPTION:

This material is a white solid which crystallizes in thick plates. The purity is 95-98 per cent with small amounts of meta terphenyl and tri-orthophenylene as the contaminants.

LABORATORY TESTS:

1. Color: 2.0 NPA

   Method: The sample is melted and poured into a 15-mm test tube. Of the standard disc comparator the color is judged with NPA standards.

2. Crystallization Point: 209-213°C

   Method: The cooling in this test is started in a mineral oil bath heated to 230°C. The sample is melted and poured into a large test tube. The test tube is placed in the mineral oil bath and cooling begins. The test tube contents are stirred as seed crystals of the sample are added. When new crystals appear in the solution the thermometer which is in the test tube is read. This temperature is the crystallization point of the para terphenyl.

3. Sediment Test: No insoluble impurities are found in molten sample of the product.

   Method of Sampling: A thief sample of each barrel of material shipped shall be examined for impurities.

   This material is also hard to keep clean under the present means of handling.

Control Tests

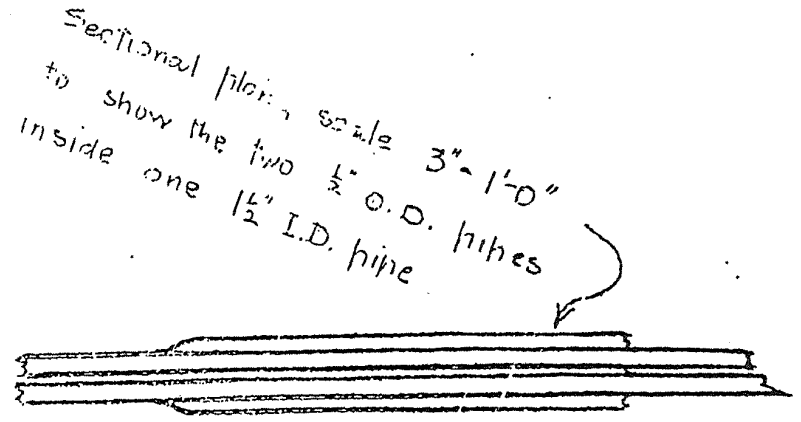
Conversion Stage : % Conversion

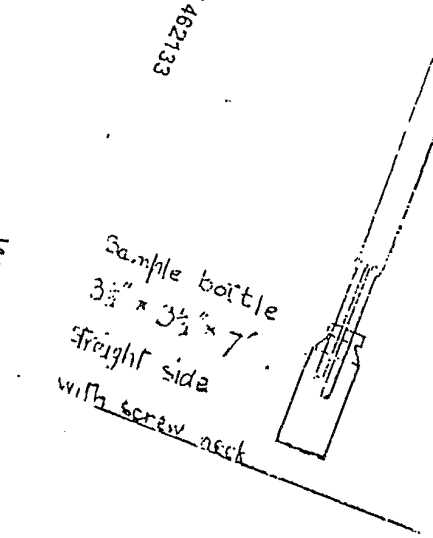
There is a connection from the pipe between the lead trap and the bottom of the column on the convertor, leading to a small, water-cooled condenser. Samples of the vapour may be drawn off at any time, condensed, and a careful measurement of the density of the condensate made, as a measure of the % conversion. The Armiston operator makes this test once a day on each unit, usually in the morning shift. Each unit has a sampling condenser, as shown on the attached sketch, permanently connected, and there is a steel sheet "fire screen" to protect the operator and the equipment from the radiation from the conversion unit (especially the lead trap).

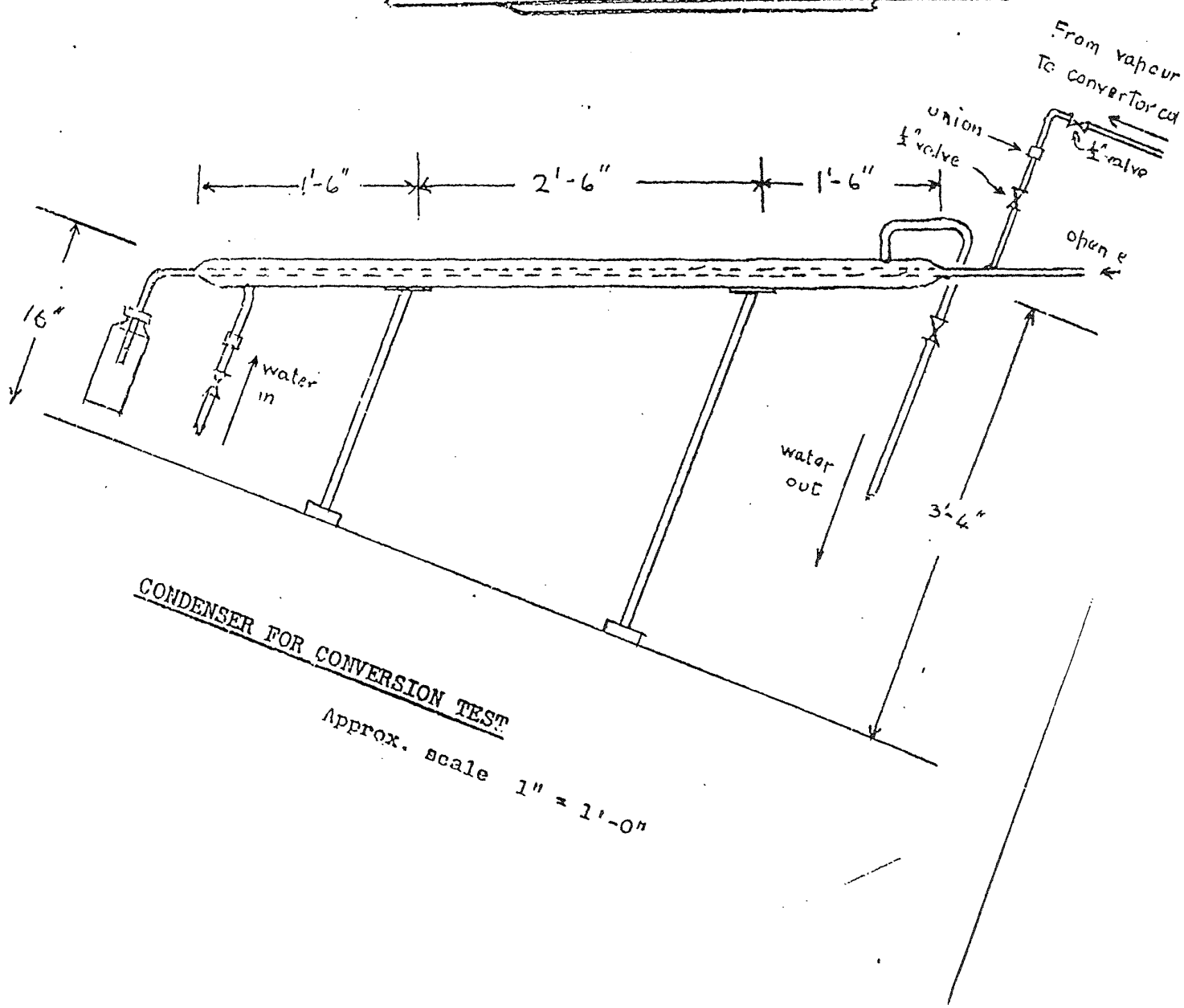
The operator starts the water flow through the condenser and opens the sampling valve enough to give a distillation rate of about 500 ml/hr as judged by eye. No purging with CO_2 is done. Some of the condensers have a screw cap fitting with the condensate and vent tube passing through, others simply have a two-holed rubber bung. In either case, the sample bottle must make a good vapour-tight connection. The use of spring clips to hold the bottle against the stopper has been suggested. The sample bottles are of glass, square, $3\frac{1}{2}$" x $3\frac{1}{2}$" x 7" on the straight side, with screw necks, and the operator carries 12 of them, one for each unit (clearly numbered) in a wooden rack, puts one on each unit, leaves them for about an hour, then turns off the vapour and water, removes the bottles, puts on their screw caps or blank corks, and carries the twelve of them to his test bench where he has a twelve-holed water bath, at about room temperature, and leaves them until their temperature is somewhere between 34° and 16°C.

A portion of each sample in turn is poured into a glass hydrometer jar and its temperature and density observed. The hydrometer is graduated 0.880 to 0.950 Sp.Gr. in 0.0005 divisions and should have been calibrated. The temperature should be determined with an accuracy of 0.2°C, and even at that the test is not highly accurate, but it is good enough for routine control.

The % conversion, corresponding to the density and temperature, is read from the attached nomograph. The samples do not crystallize, but care must, of course, be taken not to allow serious loss of benzene by evaporation during the sampling and testing. The samples are quite hot when first collected, and there is a distinct smell of benzene (and H_2S) from the vent of the test condenser while the sample is being taken. There is, of course, a slow stream of hydrogen escaping through the bottle.







CONDENSER FOR CONVERSION TEST

Approx. scale 1" = 1'-0"

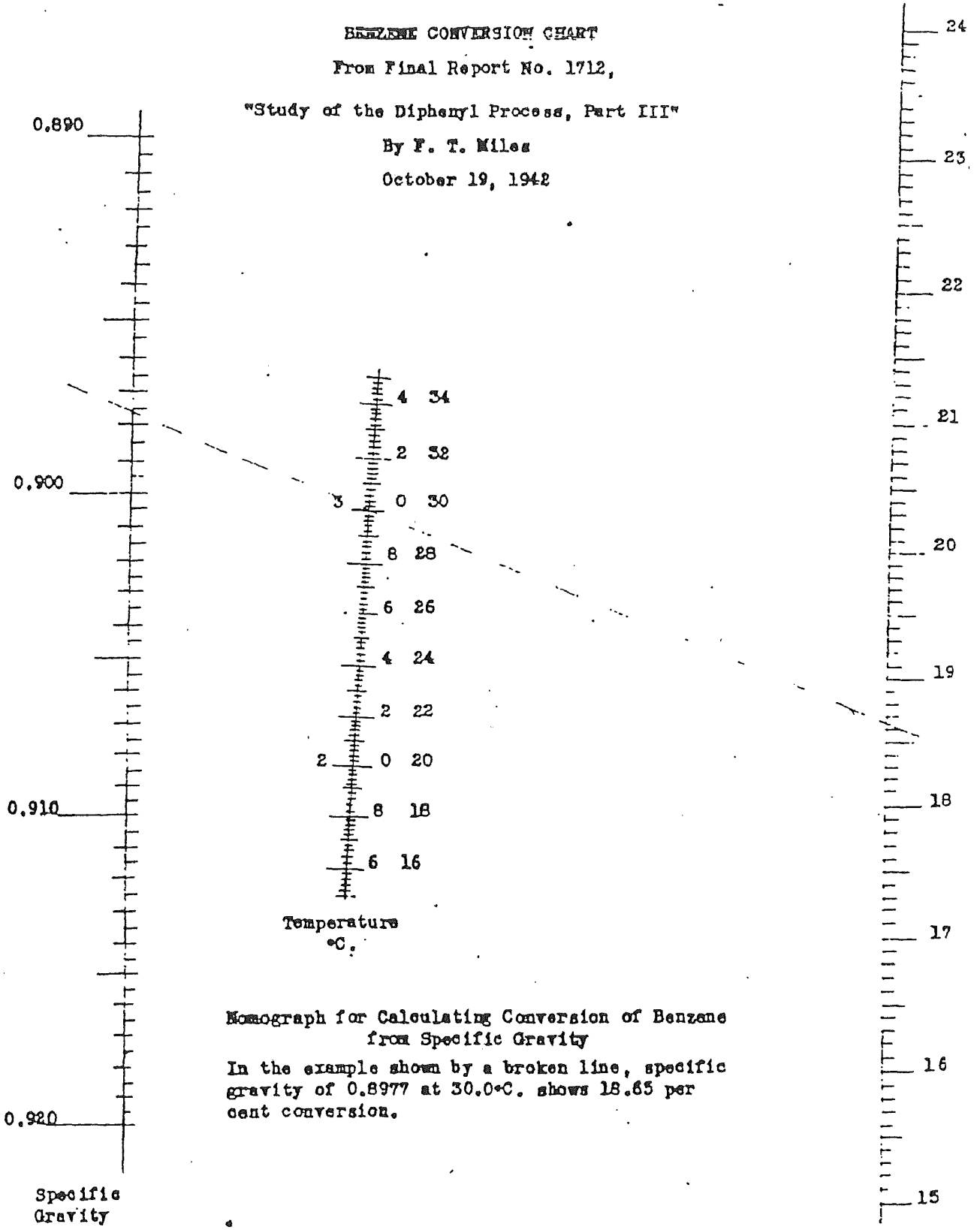
BENZENE CONVERSION CHART

From Final Report No. 1712,

"Study of the Diphenyl Process, Part III"

By F. T. Miles

October 19, 1942



Nomograph for Calculating Conversion of Benzene from Specific Gravity

In the example shown by a broken line, specific gravity of 0.8977 at 30.0°C. shows 18.65 per cent conversion.

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Control Tests contd

% Conversion, contd

This % conversion test is not done until a unit has settled down to steady running conditions. If the % conversion of any unit is distinctly different from 18%, the temperature of the conversion pot will be altered, 2 to 3°C at a time, to bring the conversion back to normal.

Analysis of Crude Diphenyl

Melt 100-150 g. in a 250 dist. flask with 10" column. Distil into weighed receiver to 275°C.

$$\% \text{ diphenyl} = \frac{\text{weight distillate} \times 100}{\text{weight sample}}$$

Test for Benzene Leaks into the Stack Gases

See page 91 below.

Distillation Stage

"Bold" points and crystallizing points are used in the control of the distillation of diphenyl and of Santowax, though in both cases Anniston run the stills largely by "dead reckoning", plus column top temperatures in the case of diphenyl, because of the time required to take the samples and make the test. (See page 46 above). The test bench in the department is, however, equipped with the mechanically stirred crystallizing point apparatus described under "Specifications & Test Methods" on page 58 above.

See sample G-4071 of Crude Diphenyl, sample G-4074 of Still Bottoms from the diphenyl still, and sample G-4075 of Still bottoms from the Santowax still, sent from Anniston to Ruabon, Oct.13/47. Samples G-4074 & G-4075 were lost, and repeat samples were sent Jan.25/49 to London.

Benzene recycled from the distillation stage should not contain more than a few parts per cent of diphenyl. The operator can judge by the colour of the benzene and by allowing a few drops to evaporate on his fingers, but a freezing point test would give a better criterion.

LIST OF DRAWINGS

There are over 350 drawings on file at Anniston, but copies only of those thought to be of interest to MCL have been sent to London at different times, as listed below.

Those marked "A" in column 3 were sent by Mr. Nagel of Anniston, to Mr.W.M.Cooper in London, by airmail, Oct.10/47, a second copy going by sea mail to Mr. M. Buis in London, Oct.13/47.

Drawing No.	Description	Date sent to M.C.L.	Sent to	Remarks
Conversion Unit				
AA-5581	Cast refractory tops for linings	A		
9C-5584-3	Cast steel door on shell of furnace	A		
9C-7817	Lead pot, flange, & thermowell	July 21/50	OWM	
9C-7845-5	Stacks on Vapourizer	A		
9C-7847-8	Pot #1, details, with later revisions	A		
9C-7937-4	Baffle for distributor	A & July 21/50	OWM	
AA-7955	Cast steel flange for distrib. pipe	A		
9C-8131-4	Details of column	A		
9C-8143-3	Traps for column	A		
9C-8147-3	Stainless piping for convertors	A		serves for references to 8147-1 & 8147-2
9C-8151	Brick lining, #6 unit	A		
9C-8153-3	Details of mounting of burners	A		
9C-8159-1	Manhole into stacks	A		
9C-8166-4	Pot cover	July 21/50	OWM	
9C-8166-2	Pot cover, Baffle locations, units 11, 12, & 13	A		

List of drawings, contd

Drawing No.	Description	Date sent to MCL	Sent to	Remarks
Conversion Unit, contd				
9C-8194	Layout of piping, diphenyl traps to sump tanks	A & Nov.13/50	DWT	see letter EM-DWT Nov.13/50 for amendments
9C-8205	Layout; benzene feed tanks & pumps	Nov.13/50	DWT	do
9C-8208-2	Layout; feed piping & meters	Nov.13/50	DWT	do
9C-9015	Layout units 11, 12, & 13	A		
9C-9019-1	Preheater pot	A		
9C-9019-2	Distributor	July 21/50	OWM	
9C-9022	Convertor shell, units 11,12,& 13	A		
9C-9024	Vapourizer coil details	A		
9C-9025	Vapourizer shell	A		
9C-9026	Vapourizer assembly	Apr.1/48	DSH	Revised to Nov.14/47
9C-9054	Gas piping to burners,& bill of materials	A		
9C-9055	Vertical condenser & split flow box	A		
9AA-9058	Expansion flange in steam jacketed piping	Aug.10/50	DWT	
9C-9069	Layout of benzene runback	Nov.13/50	DWT	see comment EM-DWT Nov.13/50
9C-9070	H_2 piping to coolers	Nov.13/50	DWT	
9C-9075	Benzene line, coolers to feed tanks	Nov.13/50	DWT	

List of drawings, contd

Drawing No.	Description	Date sent to MCL	Sent to	Remarks
No.2 Diphenyl Still				
9C-7870	Fractionating column	A		
9C-7871	Layout Diphenyl still #1	A		
9C-7872	Condenser	A		
9C-7873	Still	A		
9C-7875	Diagrammatic flow sheet. New still. Included in "Design Data for Diphenyl Still", Sept/38.	June/46		
9C-7877	Hiboiler tank	A		
9C-7886	Swivel split flow box	A		
9C-7888	Diphenyl still condenser details	July 29/48	DSH	
9C-8152	1500 gallon catch tank	A		
9C-8192	6000 gallon catch tank	A		
9C-8193-1	Layout of crude catch tanks	A		
9C-8210	Layout #2 still	A		
9C-8211	#2 Still heater shell	A		
9C-8212	#2 Still heater assembly	A		
9C-8213	#2 Still catch tanks	A		
9C-8214	#2 Still heads and tails tank	A		
9C-8215	#2 Still: piping from sump tank to still	A		
9C-8223	Rotaweir	A		
9C-8224	Rotaweir details	A		
9C-8254	Layout of piping, plan & elevation, Diphenyl still	A		

List of drawings, contd

Drawing No.	Description	Date sent to MCL	Sent to	Remarks
Hydrogen Compression				
9C-7360	Arrangements for burning H_2 (obsolete), not sent to MCL,			discussed in AME's letter June 30/48
9C-7366	H_2 compr; wiring & control	May 24/48	DSH	*
9C-8040	H_2 condenser piping	A		
9C-8165	wiring & piping diagram	A		See note on 9C-7366:
9C-8188	Layout & piping	A & Nov.13/50		See letter Nov.13/50 EM t DWT for amend-ments
9C-8279	Layout H_2 piping from units	A		
NYB-14836	Griscom Russel Company Condenser (Finned)	A		

* See AME's letter June 30/48 for corrections to this drawing, also E.G.Thoenas, May 21/48, explaining that it relates to one compressor, while 9C-8165 refers to 2 and 9C-9095 to 3.

9C-9095	Not sent to MCL			
Santowax Distillation				
9C-8551-1	High boiler distillation	June 1946	EAOM	
9C-8551-2	Elevation	A		
9C-8552	Highboiler distillation	June 1946	EAOM	
9C-8552-2	Pictorial Elevation	A		
General				
9C-7850 9C-7851	Diagrammatic Flow Sheets. Included in "Design Data, 4" Diphenyl Unit, "Aug.9/39	June 13/46		
9C-8140	#6 Unit. March 25/41	June 13/46	EAOM	

List of drawings, contd

Drawing No.	Description	Date sent to MCL	Sent to	Remarks
General, contd				
9C-8190-1	General layout, units 7,8,9,10	Nov.13/50		See letter KI to DWT Nov.1 1950 for amen ments
9C-8466	Flow Sheet - Converters	A		
9C-8550-2	Plan	A		
9C-9060	Instrumentation diagram, Diphenyl (electrical) Shows Benzene feed system	A & Nov.13/50	DWT	

GENERAL NOTES ON EQUIPMENT LIST

Reference should be made to Anniston drawings and design data for further details of the different pieces of equipment. The following list is intended only to give a general description of the equipment, and a few comments picked up during various visits to Anniston.

The equipment list is valid only for units other than #4 and #5.

The design was developed at Anniston and some modifications were introduced when units #4 and #5 were planned. These changes were found to be undesirable and the design of subsequent units, including #11, 12 and 13, (the last installed) followed the original pattern. See page 32 above and page 85 below.

All benzene lines are steam traced and lines carrying diphenyl or hi-boilers are steam jacketed, the jackets being liberally provided with expansion joints. (See Anniston drawing 9AA-9058). It is important to carry the steam jackets right up to the flanges.

The diphenyl conversion and distillation units were indoors, the store tanks and the Santowax distillation unit outdoors, with some shelter for the heating furnaces, vacuum pump and instrument panels.

See the notes on "Gaskets" page 24 above.

The conversion and still buildings are high, with effective roof ventilators, and are open completely on the East side, up to the level of the working floor. (Pot rim level). See also pages 16- above. The floors all are of vertical steel grating and Anniston are very emphatic about the advantages of this.

Flanges on the conversion system piping are of the standard ammonia pattern up to 3" pipe size, and are special cast steel tongue and grooved flanges of Monsanto design, above 3". Crane clamp gate valves up to 300°C, no valves above 300°C except cast steel gate valves on the diphenyl still.

Materials of construction for various items are detailed in Anniston Research Department, Report No. 1642.

See also pages 22 & 41 above and 86 & 88 below for notes on the choice and performance of different stainless steels.

Equipment List, contd

#1 Benzene Storage Tanks

Five horizontal steel tanks. See also pages 17 & 29 above.

#2 Pump for supplying #3

Controlled by float in #3 which cuts off the pump when the (5000 USG) tank, item 3, contains about 4,500 gallons, and re-starts it at about 4000 gallons.

#3 Recycle Feed Tank

See also page 29 above. Drawing 90-8205, but see amendments in letter EM - DWT, Nov.13/50.

The tank in use in November 1950 was a horizontal cylindrical steel vessel with bulged ends, about 7'-6" diameter by 13'-0" straight side. Capacity about 5000 USG. Set inside a concrete retaining wall outdoors at some distance from the flow control room. Pipes to and from the operating building run overhead under a steel sheet shield with a steam tracing line.

Fresh benzene in from #1 via #2 under float control; recovered benzene in continuously by gravity from the reflux split bores, item #12, of the conversion units, and semi-continuously by pump from the benzene receivers, item #27, of the hydrogen compression room. Benzene in (after examination), batchwise by pump from the benzene receiver, item #21, of the diphenyl still. This batchwise return is disliked, for Newport conditions because it would cause wide variations in the ratio of fresh to recovered benzene, and it has been proposed to interpose a buffer vessel so as to smooth out the flow.

Benzene out, by a bottom outlet with an "upstand" in the tank to retain water and solid matter, to the feed pump, item #4. The use of a bottom outlet helps to prevent air locking of the pumps. The line to these pumps is jacketed for steam, but it had no steam connections in November 1950. It is cemented through the retaining wall. Drain, from a sump at the lowest point of the tank for removal of water. This drain requires attention several times a day and should be conveniently located so that the operator can see when the water has all run out, and the benzene is coming. There is no special provision for sampling, though a sample might be drawn through the drain valve, or taken from one of the branches on the line from the feed pump, item #4.

Equipment List, contd

#3 Recycle Feed Tank, contd

Vented to the hydrogen system by means of a 1½" insulated line, (the tank contents are usually warm at Anniston, partly from the stream of recycled benzene.) There is a 1½" branch on the top of the tank, with a gate valve through which the tank might be gauged. This is very rarely done.

Float controller stopping the pump, item #2, when the tank contains 4500 USG, and restarting it when the level has fallen to 4000 USG. Floats for high and low level alarm. (There is no formal drawing of this arrangement). These various floats are now made of glass, stainless floats having failed, and stainless guide rods are recommended because sticking of the float may cause bad trouble. The domes over the float controllers are insulated and steam traced, but the tank itself is not insulated.

It is, of course, electrically earthed ("grounded").

Anniston experience is unfavourable to the use of internal steam coils in benzene tanks because of the risk of water getting into the benzene if the coil fails, and of benzene loss via the coil. Even if the coil is taken out over the top of the vessel, loss is possible by siphoning.

#4 Feed Pumps to the Conversion Units

Anniston report 1642 of Oct.15/41 describes the feed pumps as Wesco No.507 (one then for each unit) with hand-operated by-pass to set the delivery rate. Stainless shafts with "Ni Resist" impellors and liners. 1800 RPM.

In November 1950, one Gould centrifugal pump with an installed spare (and room for one more) was serving the whole 12 units through a row of flow controllers, one for each unit. It was of the 2-stage centrifugal type, 3400 RPM, (but this high speed is disliked by the operators). Gland packing of fiber-reinforced rubber rings, with the gland nut simply tightened carefully with the hand. * Delivery about 170 USG/hr/unit at 100 psig.

A steady feed rate is important because fluctuations in the rate cause variations in the pot temperatures. (The flow of fuel gas to the preheater pots is set by hand, and consequently is not automatically readjusted to meet such variations. The convertor pot temperature is automatically controlled, via the fuel gas supply rate, but naturally the control lags, because of the sensible heat of the lead, the pot, and its setting).

* These rings are made for the stems of water valves, but they have been found to serve better than other packings [illegible]

Equipment List, contd

#4 Feed Pumps to the Conversion Units, contd

There is a Fisher strainer on the line from the feed tank, item #4, and the pumps deliver to twin filter vessels in the flow control room. See under "Process in Detail" above, page 30.

#5 Conversion Units

Drawings. See list under "Diphenyl Process Drawing List" subtitle "Conversion Unit", page 77, especially:

9C-5584-3	9C-8151	9C-9054
9C-7847-8	9C-8166-2	AA-5581
9C-7937-4	9C-9019-1	AA-7955
9C-8147-3	9C-9022	

In November 1950, Anniston had 12 units, of which two were of the two-pot type with special tubular heat exchangers, to transfer heat from the converted vapours to the incoming benzenevapour, and the remainder were of the 3-pot type. There were numerous minor differences from one unit to another, representing different stages in the design history of the process. The 3-pot units are preferred, and will be described in this report, because the design of the Newport plant is to be based on the 3-pot system.

A 3-pot unit consists of three essentially similar stainless steel vessels, about 6'-0" deep by 1'-10-3/4" I.D. (see drawings listed above), each set in its own gas-fired furnace. The benzene vapour passes through the three in series, called respectively 1st preheater, 2nd preheater, and convertor. The 1st preheater is in the middle of the row of three pots, with the 2nd preheater on one side of it and the convertor on the other side of it. The furnaces are of firebrick, with steel casing.

Each setting has eight gas burners, horizontally directed tangentially into it, four at one level and four at another, evenly spaced round the circumference, and there is a pressure gauge (about 22 p.s.i.g.) on each burner, between the control valve and the jet. The flue gases from pots 2 & 3 go downwards to cross flues leading to the bottom of the setting of pot #1, then up inside that casing, and out to a flue leading to the bottom of the setting of the vapourizer, item #6a.

There is a steel door, with stabbing hole, at the bottom of each setting, for running out lead in case of leakage from a pot, and there are access doors in the flues where needed.

Equipment List, contd

#5 Conversion Units, contd

By means of dampers, the flue gases may be by-passed away from the vapourizer by passing them into a vent stack. The temperatures of the three pots are discussed under "Process in Detail" on pages 33 and 51 above, and it will be seen that the #1 pot is at a much lower temperature than the other two. Actually the waste heat from pots 2 and 3 is almost enough for #1, but the burners in the #1 setting have a little work to do, and of course, would be needed if the pot had to be re-started from cold.

Benzene from the recycle feed tank, item #3, is forced by the pumps, item #4, against the back pressure of the whole conversion system, to the top of the vapourizer, item #6A. It is vapourized in passing down the coils, and the vapour bubbles through the lead in the three pots in series, then passes, via the lead trap, item #7, to the bottom of the fractionating column, item #8. Each lead pot has a dip pipe reaching nearly to the bottom of the pot, and the upper part of the pot has circular baffles to diminish the entrainment of the lead.

A great deal of experimentation has been devoted to such problems as the design of the dip pipe and of distributors, (see below), for use at its lower end, the design of the baffles, the choice of stainless steels for the different parts of the equipment, and the choice of gaskets for the pot covers. Something of this can be seen from the "Note on the Maintenance of the Lead Pots", on pages 41- above.

No attempt is made here to review the whole history, but only to describe the arrangement now used, with present-day ideas for improvement. Various old patterns of pots etc are still giving good service at Anniston.

The pots, and the fittings inside them, exposed to the hot lead, (but not the covers), are of stainless steel. See pages 88 & 89 below for the choice of the type of stainless steel, also page 22 above.

Anniston use steel covers for the pots. The #1 pots have flat covers, of steel plate welded on to the rim of the stainless pot. Some of these plate covers have heavy radial fins welded on for stiffness. The #2 pots have heavy cast steel domed covers, bolted on, with heavy cast steel (divided) backing flanges under the pot rims. The backing flange has four lugs, fitting into sockets on the furnace roof and supporting the pot so that the rim is several inches above the top plate of the furnace casing.

Equipment List, contd

#5 Conversion Units, contd

The gasket in this case is 5/16" round soft annealed copper, with a square section asbestos gasket inside it.

The 3rd pot is similar in construction, but has a gasket of 1" braided asbestos rope. Mr. Harden took a sample of this in August 1950. This thicker gasket is used because the edges of the flanges pull together in service at the high temperature, and they can remain in service longer with a thick gasket. To make the gasket, a piece of the asbestos rope is cut, long enough to lie just inside the bolt circle, plus a few inches over-lap. The ends are overlapped, and the groove between the overlapped parts is stuffed with strands of asbestos rope, and the overlap bound with a few thin wires. Liquid glue is used to help in making the overlap and in holding the ring in place between the flanges during assembly. In use, the joint ring becomes completely flattened and hard.

The use of restriction orifices after #3 pot, as discussed on page 34 above, has thrown much greater pressure on the joint of the pot and the asbestos rope gaskets are giving much more trouble. Vertical sheet iron fences, about 6" deep, in two halves bolted together, are being loosely fitted around the cover joints of some of the conversion pots to deflect any possible burst of flame due to a failure of a gasket.

London's proposal to use thicker covers and backing flanges, and to make them of stainless steel having a higher temperature yield point than the plain steel ones, is liked at Anniston, because it offers a chance of using copper gaskets on the conversion pots as well as on the preheaters. London's proposal to use slotted bolt holes is disliked as tending to favour the bending of the flanges.

London also suggested supporting the pot rim on a continuous ring instead of on the four lugs, but this arrangement would appear to make the cover bolts more difficult of access, to rob them of the cooling effect which exists under the Anniston arrangement, and to make the raising of the pot a little more difficult.

At Anniston the companion flange under the pot rim is in two halves, bolted together, as mentioned above, forming a circle, with four lugs at right angles to one another. Pairs of short pieces of angle iron are welded inside the box frame which composes the furnace roof, these pairs forming guides for the lugs on the companion flange. Steel packing pieces are placed first on the bottom of these guides to give the necessary elevation of the pot, then

Equipment List, contd

#5 Conversion Units, contd

the pot lowered in and secured by steel wedges driven in alongside the lugs. The pots should not be rigidly connected to the furnace casing because the continuous vibration of the pots would be communicated to the brickwork of the furnace, and tend to make it crumble. At Anniston they fit fairly closely into the round holes in the furnace roofs, but in some cases steel sheet sectors are used to bridge the gap. In any case, the joint is made good, either with clay, or more recently with a paste containing asbestos fiber (insulating compound 66). This helps to keep the pot rims a little cooler.

The bolts in the pot cover joint and in the adjoining pipe flanges, are of ordinary steel, at Anniston, and are never re-used, partly to save time and because they become stretched, but mainly because working conditions can be very uncomfortable because of the radiation of heat from other units. Newport, using stainless bolts, and having only one unit should be able to re-use the bolts. It is suggested to use nuts of dissimilar metal, to diminish galling. In cutting out the bolts on the pot cover, the Anniston welder uses an oxy-acetylene torch, and cuts through the base of the nut, flush with the upper surface of the cover, and the stub of the bolt is then knocked out downwards, the pot cover being high enough over the furnace roof to leave room for this and for the insertion of the new bolts. On the pipe joints, the bolts are cut between the flanges. The cover bolts have square heads, but hexagon nuts, and they go through the flanges, tail upwards. Small squares of steel (old nuts will serve), are welded under the companion flange, between the bolt holes, to prevent rotation of the bolt. The nuts are run on with an air-driven spanner, for speed, and tightened up after the pots are hot, with a 4' long ratchet spanner. Anniston advise strongly against attempting to use a tongue and groove joint on the pot covers because of the risk of distortion.

The attachment of the stainless pot rim (by welding) to the pot itself appears to be a line of weakness because the lead in the pot is very heavy and is gently bumping and vibrating whenever the vapour is passing. Cracks at this point are not unknown. Anniston have increased the thickness of the rim of the pot; formerly 5/8", now 1".

British makers of stainless steel agree that 309 stainless steel tends to be brittle at the pot temperature, but that 330 does not. MCL are experimenting with pots fabricated, by welding, from centrifugally cast sections of 330 stainless, welded either with 309 or with special electrodes.

Equipment List, contd

#5 Conversion Units, contd

(See the letter from Michigan Steel Casting Co. to Anniston, May 31/50). Anniston discussed this design, Aug.1/49. Heat treatment, after fabrication, is recommended for both 309 and 330 stainless.

Inconel, Eureka, nickel, and alloys rich in nickel are said to give poor service.

Anniston reported, Aug.1 & 5/49, that cast thermo wells of 309 had proved to be brittle. Centrifugally cast dip pipes of 309 were better than rolled, or mold cast, but were brittle or became brittle in service. Centrifugally cast dip pipes* have recently (1950) given excellent service. It is noted that in this method of casting the dross tends to form on the inside of the pipe, but no harm appears to have resulted. Thermo wells of cast 330 have recently given very good service.

(*of 310 stainless steel)

Carbon tends to build up in any crevices in the interior of the pots, and to exert a bursting pressure. Cast covers should be free from pinholes, blowholes and slag inclusions. Anniston warn against welding cover strips on the outside of welds on the pots. Outlet branches on the covers should not extend down into the pot, and the joint should be welded inside, to fill up any crevices. Sharp edges of metal also tend to attract deposits of carbon.

Any distributor at the bottom of the dip pipe must be such as will not increase impingement of the gas on the pot wall, because of erosion. Anniston are using a simple open-ended pipe with a renewable liner for the last few inches at the bottom and four 5/8" holes drilled radially through the wall about 4" from the bottom.

At a meeting at Anniston, Mar.16/49, a proposal was made to try a deflector which would discharge the vapours tangentially in a horizontal direction. This it was thought would give the lead a swirling motion, diminish vibration of the pots, increase the contact time of the vapours, diminish the size of the gas bubbles, and diminish the entrainment of lead. Narrow orifices must be avoided in the design of any deflector because of the building up of carbon.

This deflector had not been tried at Anniston, but London asked that the pots to be supplied for Newport should have a deflector. Standard dip pipes were, however, shipped in November/50 to avoid delay.

Equipment List, contd

#5 Conversion Units, contd

Texas City, Aug.10/49, sent MCL a drawing of the distributor they had developed for pots for propane cracking.

There is no heat insulation used anywhere in the conversion system, except that the vapour pipe from second preheater to the convertor is insulated on some of the units. It is desirable to avoid loss of heat anywhere along the line of flow, up to the top of the convertor pot, and at one time the pot lids were insulated. It was found, however, that greater distortion of the pot covers and bolts resulted.

The insulation of the vapour pipe from the second preheater to the conversion pot is not an easy task. In some cases, asbestos fiber, inside a sectional (lobster back) casing is used, and in other cases a special Johns Manville powdered product in a dust-proof steel casing. This insulation and casing adds to the weight of the pipe, and may cause sagging, and all these long pipes, from 2nd to 3rd pot, whether insulated or not, have support stirrups, which, however, appear to be out of use.

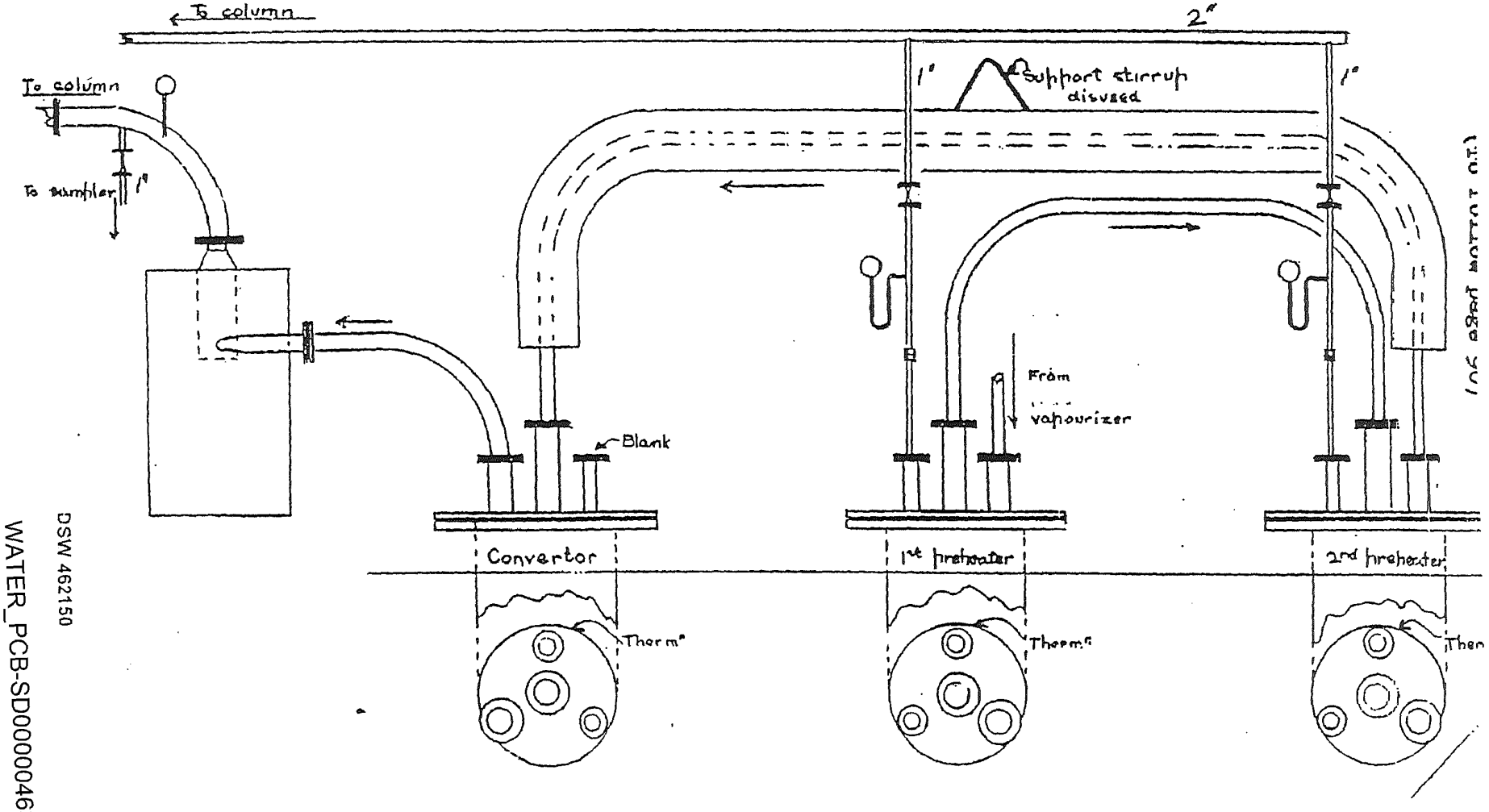
In addition to the vapour inlet and outlet and the thermo well, each pot cover has a fourth opening. On the convertor pots this is blanked, but on the 1st and 2nd preheaters this opening is used to connect the balancing line. These connections run in 1" pipe vertically for about 7'-0", and then join into a horizontal header running to the bottom of the column, item #8. There is a pipe coupling on this line about 2'-0" above the pot cover, then a branch for a pressure gauge, then a stop valve, which is normally closed. These gauges show the pressure in the space above the lead in pots 1 and 2, and the lines may be used to by-pass a pot in emergency, and to prevent lead from being forced backwards into the vapourizer. There is a pressure gauge on a branch in the vapour line from the convertor pot to the column, also a branch for the sampling condenser described under "Control Tests" on page 75 above.

There is a heavy I-beam about 18" deep, with 6" flanges, and with a chain block . . permanently installed, running along it , one for each row of 3 pots, with a hinged section of the floor to permit lowering a pot or a lead trap to the ground floor. When a pot is put out of action opportunity is taken to clear debris from the bottom of the furnace.

TO SHOW EQUALIZING LINE ON LEAD POTS

Scale ½" = 1'0"

BM Dec. 5.50



DSW 462150

Equipment List, contd

#6A Vapourisers

Drawings. 9C-7845-5 9C-9024
9C-8159-1 9C-9025

One per conversion unit. Each consists of a single coil or of two coils in parallel, in a brick lined steel stack through which the flue gases pass. Benzene passes downwards through the coils countercurrent to the flue gases.

The vapourizers first installed at Anniston had two coils in parallel, one inside the other with a common inlet header, and a control valve on each exit, leading to a Y pipe into the first preheater. This arrangement was found inconvenient, and has (except in the case of one unit) been abandoned in favour of a single coil. There was some doubt about the proper sharing of the flow between the two coils, and considerable trouble with carbon blockage. Anniston report 1642, Oct.15/41, states that corrosion of the vapourizer coils may occur by condensation of moisture from the flue gas, near the point of entry of the cool benzene.

A small leakage from a vapourizer coil into the flue may easily pass unnoticed, because the benzene will probably not ignite in the stream of flue gas, and the leakmay cause serious losses before it is discovered, As a safeguard against heavy losses a sample of the flue gas is aspirated every day from a point in the flue, immediately above the vapourizer coil on each unit, and is passed through a mixture, equal parts of sulphuric and nitric acid. For this purpose a $\frac{1}{4}$" steel pipe is permanently installed in the vapourizer flue and all these pipes are carried down to a test bench on the ground floor. The sulphuric-nitric mixture is put into small rubber stoppered bubbling bottles, which, for safety, are sheathed in simple steam-pipe sockets. The bench has a simple water aspirator, and it is the night operator's duty to test all units. He simply aspirates the gas through the acid for about 10 minutes, then smells the acid bottle.

Blockage of the vapourizer coil is rare (except by lead forced back from the first preheater). Traces of impurities left by evaporation of the benzene are simply carried forward in the vapour stream.

The flue gas must, of course, be by-passed round the vapourizer when the benzene flow is stopped. A coil should give over a year's service before needing repair.

Equipment List, contd

#68 By-Pass Flue

Drawing. 90-7845-5.

One per conversion unit, fitted with a damper. To allow flue gases to by-pass the vapouriser as necessary.

#7 Lead Trap

Drawing 90-8143-3.

One per conversion unit. This is a centrifugal type separator for removing any entrained lead. It is a closed, welded vessel which must be taken out at intervals to allow any lead to be melted out.

On some of the Anniston units, the lead trap is a relatively small steel vessel, so placed that lead can drain back from it to the convertor. In the newer units, the trap is larger and stands on a steel plate on the operating floor, which is nearly at the level of the flanges of the lead pots. These traps take the form of flat-bottomed cyclones 3'-6" high, 2'-0" diameter, with a central pipe 6" diameter, for the gas exit , and the usual tangential inlet for gas, high up the side. It has been found best not to carry the central pipe more than about 14" down inside the trap, otherwise its bottom end becomes choked too soon by lead and by solid debris (iron oxide, carbon, and shots of entrapped lead). In a trap, seen Nov/50, the layer of entrained lead was 14" deep, and there was considerable solid debris as well. After the lead had been melted out this vessel was cut open to permit the shortening of the inner tube, and opportunity has taken to dig out the solid debris.

Note on Emptying the Lead Trap

The unit is shut down and allowed to cool a little, and some diphenyl etc. may run back into the lead trap and may take fire when the trap is opened.

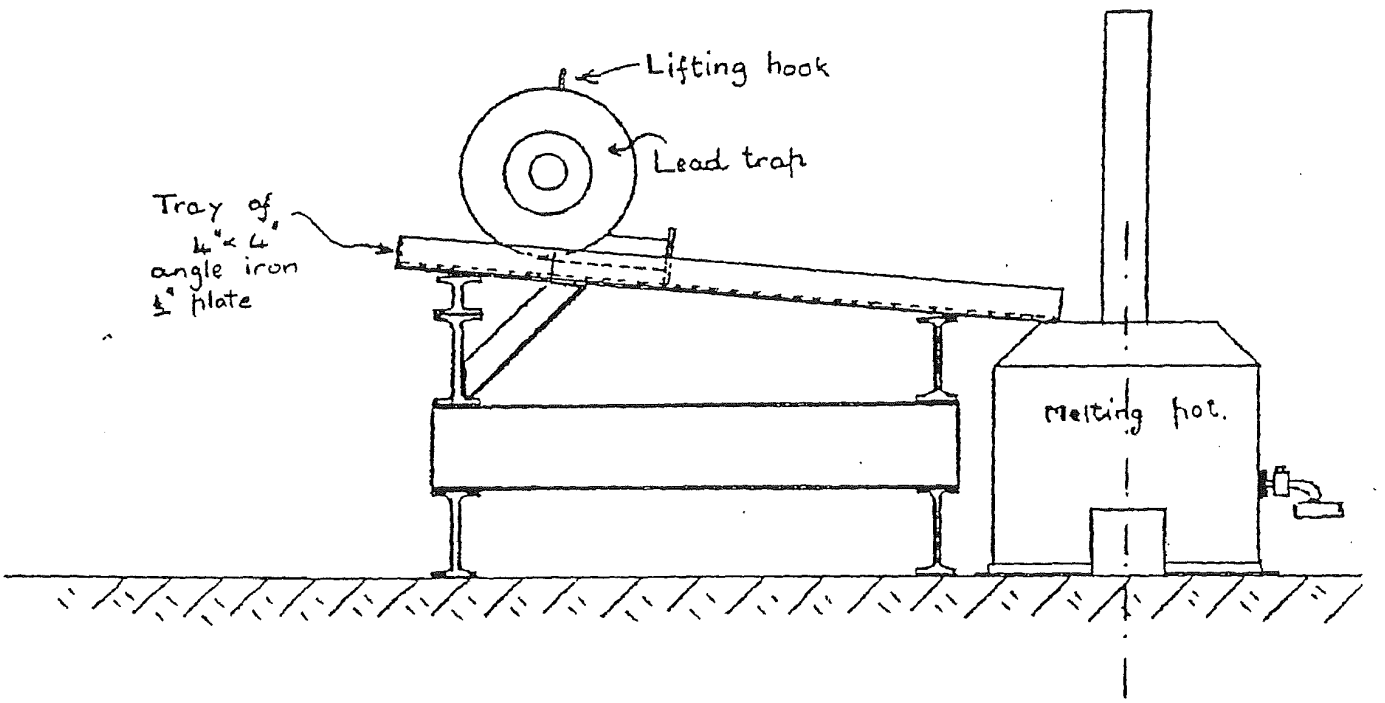
Each cyclone has a lifting hook welded on, opposite the gas inlet; this is for the yard crane; inside the shed the pot is lifted by a stirrup bolted to the central outlet flange. The cyclone is lowered to the ground floor, picked up by the yard crane, and taken to a melting table in the scrap yard, and put on the table with the vapour inlet downwards, (vessel on its side), and flames of natural gas played on the pot until the lead

(To follow page 92.)

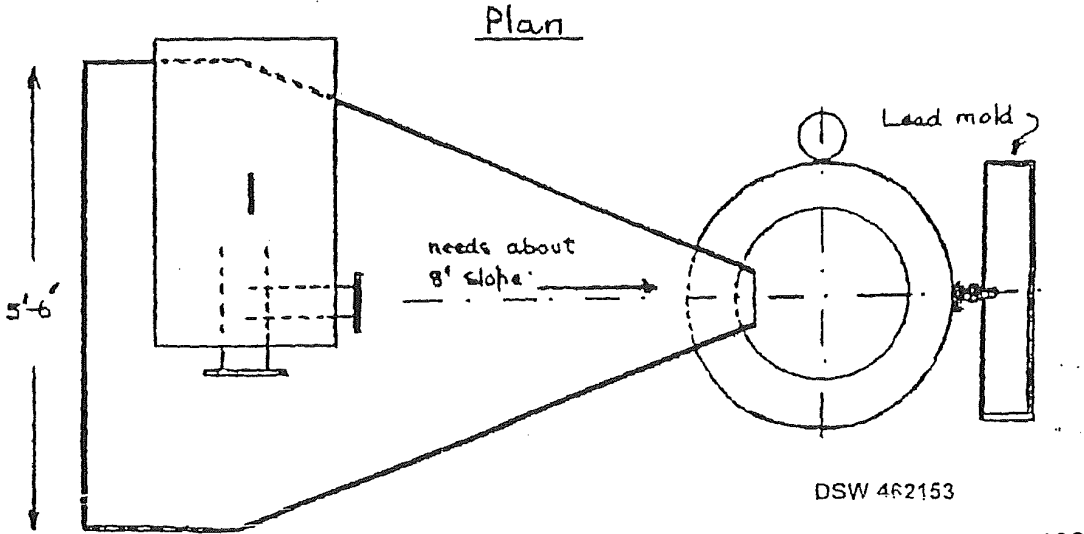
MELTING TABLE, ETC.
USED FOR CLEANING
THE LEAD TRAPS

Scale ½" = 1'-0" EA Dec. 2. 30.

Side view



Plan



DSW 462153

Equipment List, contd

#7 Lead Trap, contd

runs out on to the table and down into a lead melting pot, from which it can be run off at convenience into molds like those used to empty the pots. The melting pot has a brick setting, and a proper gas burner, but the cyclone is heated simply by open flames, confined somewhat by means of sheets of corrugated iron. The natural gas has a high calorific value, so the melting is quick.

The molds are the same as those used for emptying the lead pots, see page 39 above.

Note on Repairing Lead Trap & Column

These units are exposed to the blast of hot vapours opposite the gas inlets (especially the lead trap since the introduction of orifice plates in the vapour pipes from the convertor), and they wear thin. The unit being shut down, and purged with CO_2, external patches can be welded on. A useful trick first is to weld a pipe nipple on the center of the patch and to leave it open as a vent until the patch is welded to the vessel, then to put in a screw plug. Such repairs are done with the vessels in their working positions, but filled with CO_2.

The trap has to stand up to fairly heavy handling when being cleaned, apart from the high temperature during service, and it should be of heavy plating. Stainless is not necessary, and thin stainless would be unsuitable.

#8 Fractionating Columns on Conversion Units

Drawing 9C-8131-4.

One to each conversion unit. Vapour in at the bottom from lead trap #7. Benzene reflux in at the top from reflux box #12.

Vapour out at the top to condenser #11. Underflow out at bottom via carbon trap #9 to sump tanks #10. Thermometer well at point of entry of reflux line to column, and one in the exit from the carbon trap.

The column has 12 plates, but the caps have been removed from the lowest 2 plates because they became choked with entrained lead, etc. The lead would be above its melting point as it entered the column. The need for so many plates, for a comparatively rough separation of benzene from diphenyl, has been questioned, but plant experience indicates some difficulty in maintaining a reasonably

Equipment List, contd

#8 Fractionating Columns on Conversion Units, contd

low content of diphenyl in the distillate and of benzene in the under flow of the column.

Presumably the plate efficiency is low because of the dilution by hydrogen and possibly there is fouling of the plates by carbon carried forward in the vapor stream. The plates have the usual drain holes to prevent blockage by freezing. Design data were unavailable, May 14/48.

The crude diphenyl tanks are heat insulated, and any steam jacketed lines outdoors. The columns have no heat insulation.

Packed columns were used on the earlier units, but they became plugged with solid matter carried forward from the pots.

#9 Carbon Trap

Drawing 90-8143-3.

Underflow in from column. Side outlet at top to sump tank #10, with thermo well in the pipe bend. The outlet pipe goes down nearly to the ground level before rising again to the sump tanks, so forming a seal bend between the trap and the sump tank. This seal bend and all the piping are steam jacketed, inner tube 2" diameter.

These traps are removed from the unit for cleaning whenever necessary, about every third month. An opportunity may occur when the unit is shut down for some other purpose.

The temperature of the column trap is below the melting point of lead so that the lead that settles here is usually in finely divided solid particles. However, due to the nature of the infusible, highly carbonaceous organic materials that settle also, the lead is intermingled and agglomerated into a solid mass. Thus it is necessary to burn and melt out the trap to recover the lead. The proportion of carbon to lead is greater in the "carbon" trap than in the "lead" trap.

Anniston report 1642 of Oct.15/41 states that this carbon trap needs cleaning every 3-6 months, and that, although it has a bottom outlet plug, it has to be taken out and cleaned by burning. As seen at Anniston in November 1950, these pots were simple vertical steel cylinders about 3'-0" deep by about 1'-6" diameter with about a 9" flanged neck

Equipment List, contd

#9 Carbon Trap, contd

to connect to the bottom of the column. The cover also had an inspection plug and two lifting eyes to facilitate handling for maintenance.

#10 Sump Tanks for Crude Diphenyl

Horizontal, steel, insulated tanks. Underflow in, by gravity, from column #8 via carbon trap #9. Out by submerged pump #10A to Diphenyl Still #13. All liquor lines thoroughly steam jacketed. Vent to off-gas system. Steam coil connections. Varec liquid depth indicator. Vent with explosion disc.

Mr. Ellenburg, May 14/48, stated that these tanks are operated at about 125°C to prevent crystallization. When seen in November 1950 the vent pipes were quite hot to the touch.

#10A Submerged Pump

For transferring crude diphenyl from sump tank to diphenyl stills. One to each sump tank. Explosion proof motor.

#11 Condepsers on Conversion Units

Drawing 9C-9055.

One to each conversion unit. Tube and shell type, water through the tubes. Vapour in from column #8 and condensate out to Reflux Split Box #12, through a strainer with by-pass. Balancing line from top of Reflux Box #12 to condenser, with connection to hydrogen off-gas system. There is also an emergency vent to the atmosphere.

Anniston advise against the use of copper tubes, because of the sulphur impurities in the benzene. They advise making this condenser of ample capacity.

#12 Reflux Split Box.

DSW 462156

Drawing 9C-9055.

Standard Rotaweir, with sight glasses and levelling screws. One to each conversion unit. Forward flow returns via sight glass to recycle feed tank #3. Sight glass in reflux line. Both lines have deep seal bends, and there must be sufficient head between the reflux box and the return inlet

Equipment List, contd

#13 Diphenyl Still

Drawing 9C-8254.

Two are installed. Horizontal, insulated, steel tank. Submerged pump with water cooled bearings, and outlet to external gas-heated coil with branch to crude Santowax receiver #20.

Inlet from #10 sump tank and "Heads and Tails" receiver #18. On one dished end is return line from bottom of column #14. Return line from heating coil. Vapour outlet to bottom of column #14.

4" vent pipe, from a branch on the vapour pipe to the column, carried up through the roof. This vent pipe has a bursting disc between two flanges located about 6'-0" above the vapour pipe. In one case, the 6' length of pipe is jacketed (jacket empty), and in the other case it is bare. Immediately below the flange there is a thermo well, with a thermo couple giving a continuous record on the Multipoint temperature recorder in the instrument room. It is found that the discs slowly become perforated, probably by sulphur compounds, but even a small perforation is detected by the temperature rise. The pipe above the flange is supported on rods with a turn-buckle fitting by which the pipe can be lifted for easier replacement of the disc.

The bursting discs are B.S.&B. discs, type Al-Pb (lead on the concave surface) "Vinyl service". Bursting pressure 39 (in another case, 43) p.s.i.g., carried between two B.S.&B. type flanges.

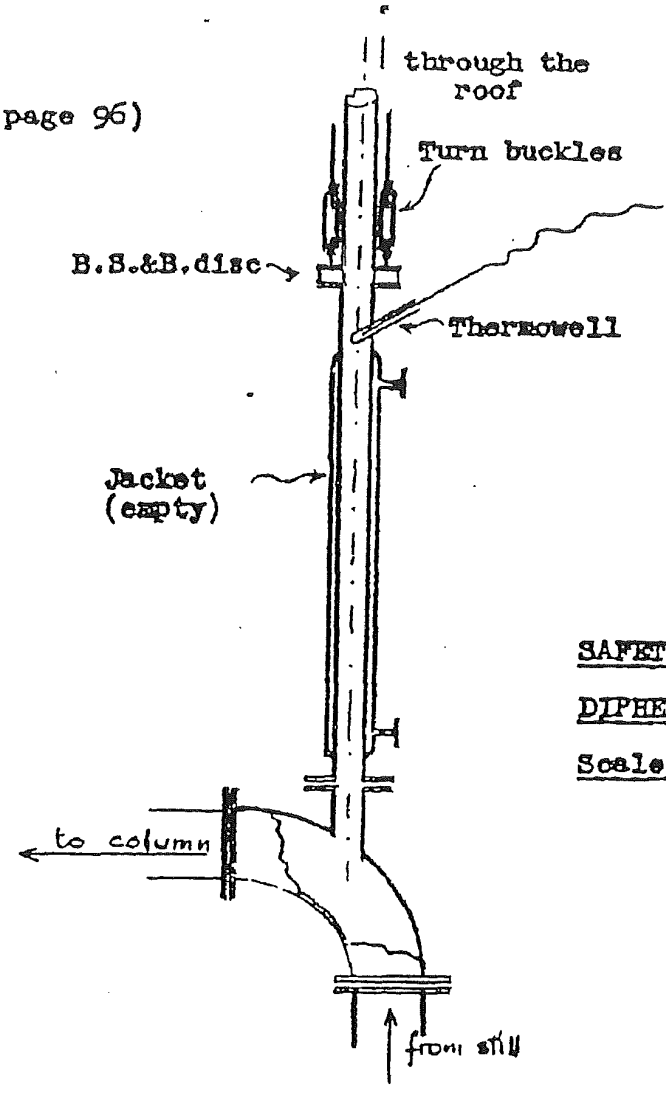
#13B Heating Coil

Drawings 9C-8211, 9C-8212.

One for each still. Supported in a steel casing with firebrick lining. Gas fired. Liquid circulated by pump #13A from and to still #13. Flow is countercurrent to flue gases.

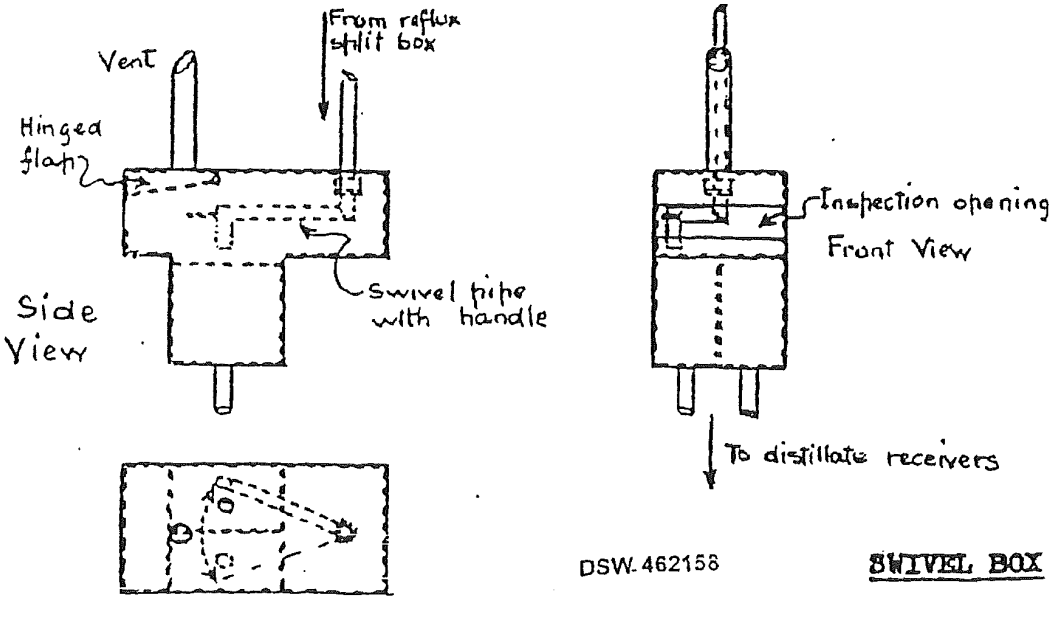
Half the casing hinges away on a steel track for access to the coil. The circulating lines are steam jacketed. Note, however, that if water is trapped in the jackets and the hot diphenyl circulated, a very high pressure would develop in the jackets.

(To follow page 96)



SAFETY VENT ON DIPHENYL STILL, ITEM 13

Scale ½" = 1'-0"



DSW. 462158

SWIVEL BOX

Equipment List, contd

#14 Column

Drawing 9C-7870.

One for each still. 20 plate column. Drain holes in plates. Vapour inlet at bottom from still #13. Reflux return from split box #16. Underflow from bottom back to still #13. Vapour out to condenser #15 via goose neck.

Anniston formerly used a packed column, with a direct-fired still, but the results were not so good as with the present bubble cap column. On May 12/49 Anniston expressed the opinion that the 20-plate column, proposed by London Central Engineering Dept. would be adequate. Special care is needed if the reflux line to the still is not to freeze.

#15 Condenser

Drawing 9C-9872.

One per still. Horizontal tube and shell. Vapour in from top of column #14. Condensate out to split box #16. Cooled by water through tubes, circulated in a closed system by pump #23 through heat exchanger #22 with make-up tank #24.

#16 Reflux Split Box

Drawings 9C-8223 and 9C-8224.

Standard Rotaweir, with sight glasses, and levelling screws. One for each still. Condensate in from condenser #15. Reflux out to top of column via sight glass. Forward flow to benzene receiver #21 or through box #17 to receivers #18 and #19. Vent pipe, steam jacketed for occasional melting out, (should have flame arrestor). The reflux box should have steam tracing for use while the diphenyl fraction is running.

#17 Swivel Boxes for Switching Diphenyl Still Distillates

Drawing 9C-7886

One for each still. Condensate in from Reflux Split Box #16, and out, by manual adjustment, to either "Heads and Tails" receiver #18, or Diphenyl receiver #19.

(To follow page 97).

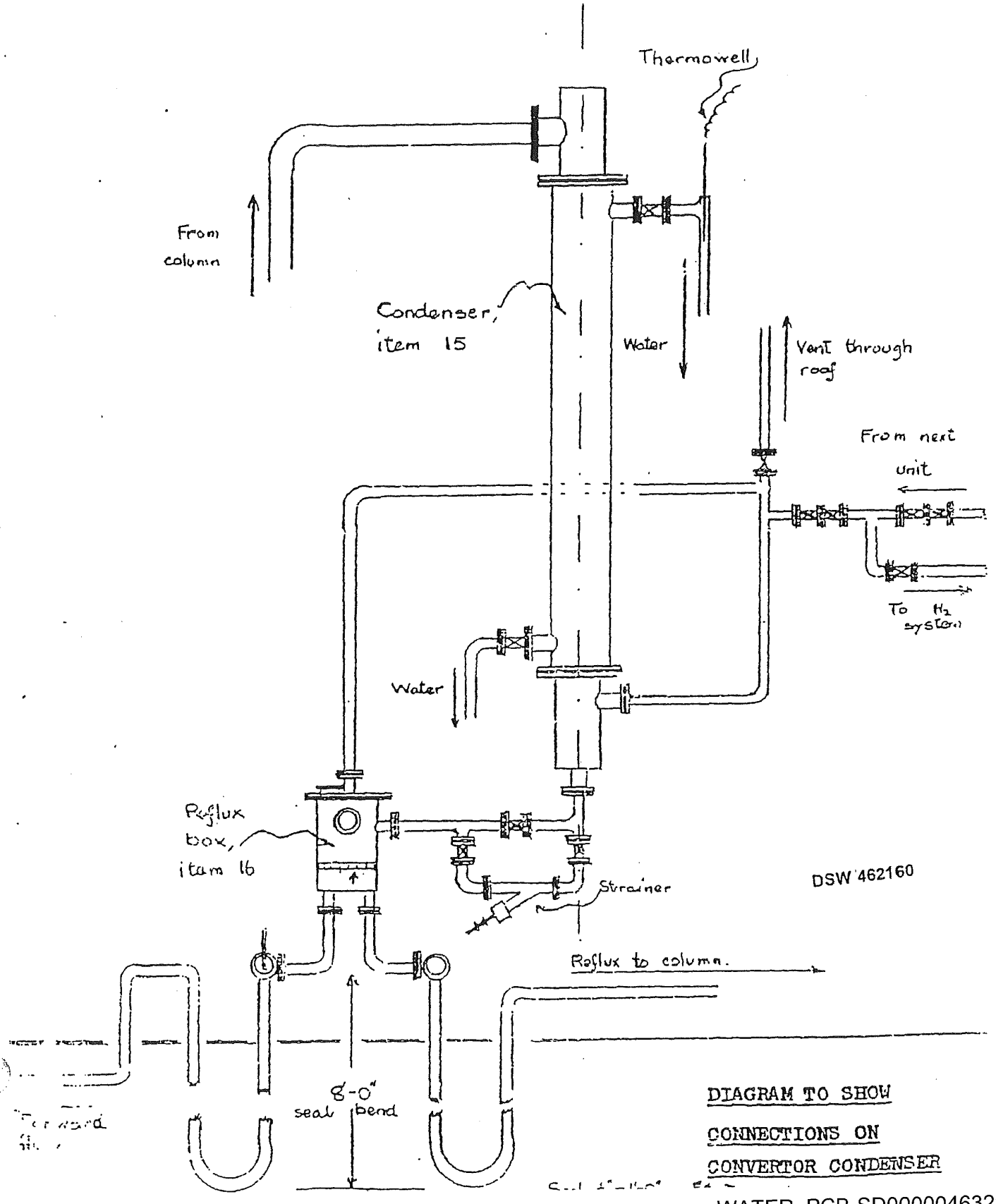


DIAGRAM TO SHOW CONNECTIONS ON CONVERTOR CONDENSER

Equipment List, cont'd

#18 "Heads and Tails" Receiver.

Drawing 9C-8124.

One for each still. Vertical tank with submerged pump, and manhole. "Heads" or "Tails" fraction of distillate in from Swivel Split Flow Box #17. Steam coil inlet and outlet. Submerged pump delivery line back to still. Vent line with lower portion steam jacketed and carrying bursting disc. Inspection hole with loose hinged cover. Bottom outlet.

#18A Submerged Pump

One per Heads and Tails receiver. For transferring Heads and Tails fraction to Stills #13.

#19 Diphenyl Receiver

Vertical tank fitted with submerged pump #19A.
Diphenyl fraction in from Swivel Split Flow Box #17.
Finished product out via pump delivery line to Aroclor Plant, Store tank or Flaker.
Vent with lower portion steam jacketed and with bursting disc. Manhole and cover. Handhole with loose hinged cover. Steam coil inlet and outlet.

#19A Submerged Pump

One for each Diphenyl receiver #19. For transferring diphenyl to Aroclor Plant, Store Tank or to Flaker. Explosion proof motor.

#20 Crude Santowax Receiver

Drawing 9C-7877.

Vertical tank with submerged pump #20A. Inlet from submerged pump #13A of still. Crude Santowax from outlet of submerged pump #20 to #34B of Santowax Distillation Unit.
Handhole and loose hinged cover. Manhole and cover.
Steam coil inlet and outlet.
Steam jacketed vent and bursting disc.
Crude Santowax to drums through bottom outlet.
Second steam jacketed vent with hand valve.

Equipment List, contd

#20A Submerged Pump

One for each crude Santowax receiver. For transferring crude Santowax to #34B Santowax still charging tank.

#21 Buffer Storage Tank

for recovered benzene. Vertical tank with manhole and vent. Recovered benzene in from reflux split box #16 and out to pump #21A. Inlet and outlet for steam coil. Gauge glass.

#21A Pump

Receiving recovered benzene from buffer storage tank, #21, and delivering to recycle feed tank #3. Explosion-proof motor.

#22 Heat Exchanger

One for each still. Receives hot circulating water from condenser #15 and delivers cooled water to pump #23. Cooling water through tubes.

The circulating water is maintained at 30-40°C during the benzene forerun, and about 80°C during the diphenyl fraction, (by hand control on the fresh cooling water through the tubes in each case). Compare page 47.

#23 Pump

One for each still. Receives circulating water from heat exchanger #22 and delivers to condensers #15. Explosion-proof motor.

#24 Make-up Water Tank

One for each still. Receives make-up pond water as necessary to replenish water lost by evaporation on circulating through condenser #15. Bottom outlet to circulating line. Overflow. Constant level device for controlling receipt of make-up water. Condensed water would, of course, be better for this make-up.

Equipment List, contd

#25 Cooler for Hydrogen

Drawings 9C-8040, Griscom Russel Co. NYB-14836.

Each of the four parallel units of the original cooler has been surrounded by a water jacket to increase the cooling surface.

Receives hydrogen, for cooling, from a main serving all the convertor units. There is a vent with hand controlled valve connected to the hydrogen inlet via a benzene trap. The latter is a small pot containing baffles and draining to the surge tank #26.

#26 Surge Tank for Hydrogen

Drawing 9C-8188

Receives cooled hydrogen and condensed benzene from cooler #25 and benzene from the vent to the hydrogen cooler. Hydrogen delivered to inlet of compressor #29. Condensed benzene flows by gravity through bottom outlet to either of the two tanks #27. Fitted with 55" water seal* to provide pressure release if necessary. Water seal has a vent with flame arrestor. Line to instrument controlling pressure within limits of 11-16 oz. Pressure gauge. Balancing line to benzene receivers #27.

When the pressure drops to 11 oz., the compressor low stage becomes inoperative and only the high stage operates. When the pressure reaches 16 oz both stages operate until the pressure falls again to 11 oz. (per sq.in.)

* through the roof,

#27 Benzene Receivers

Drawing 9C-8188.

One for each compressor. Receives condensed benzene from surge tank #26, from low pressure tank #31, and high pressure tank #33 through sight glasses. Benzene out via bottom outlet to pump #28. Balancing line to surge tank #26. Level gauges

#28 Pump

One for each compressor. Receiving condensed benzene from benzene receiver #27 and delivering to recycle feed tank #3.

Equipment List, contd

#29 Two Stage Hydrogen Compressors

Drawings 9C-8188,
9C-7366, wiring and controls, revised version sent to Mr. Buis, Jan.18/50.

Two are installed. The first stage compresses the hydrogen to 60#, and the second to 300# pressure. Provision for introducing CO_2 for purging purposes. The compressors themselves are water cooled and there is a small vertical tubular condenser after each stage, draining to the corresponding pressure tanks, items #31 & #33.

Dr. Hardy stated, November 17/48, that the compressors have: 316 stainless shafts and valve discs, Ni-resist cylinder liners and valve cages, Goetz gaskets, stainless steel or aluminum type, and that copper and its alloys are undesirable.

No copper or brass gaskets should be used, and generally copper and its alloys are avoided. Mr. Clegorn, Nov/50, mentioned gaskets of Babbitt metal for the compressors

Anniston were asked if un-lubricated cylinders with carbon rings would not be better, but they replied that finely divided carbon, which might come over in the hydrogen stream, would abrade the carbon rings. Drawing 9C-7366 showing the wiring and control system for a single compressor, was sent to London, May 24/48.

The compressors are fed rather liberally with lubricating oil to cope with the possible condensation of benzene in the cylinders, but Anniston have not noticed any trouble from lubricating oil getting into the process stream.

Mr. A. M. Ellenburg, June 30/48, gave a general discussion of the operation and control of the compressors, incidentally giving 150 and 300 p.s.i.g. as the two pressures, but stated that these values were not critical and that the choice turned on considerations of cylinder size and quantity of gas to be handled.

Progressive unloading would be satisfactory as an alternative to the "trigger, on & off" unloading control used at Anniston. By Nov./50 the solenoid unloading controls had been replaced by pneumatically operated controls, partly for reasons of safety. Electrical switches would in any case be outside the compressor room. The room is illuminated by flood lamps placed a few yards outside the windows.

Equipment List, contd

#30 Interstage Cooler

One for each compressor. Simply consists of a vertical, water-jacketed pipe between outlet from first stage of compressor and inlet to low pressure tank #31. Gas and vapor in at the top from the compressor. Condensate and gas out at the bottom to the low pressure tank, item #31.

#31 Low Pressure Tank

Drawing 9C-8188.

One for each compressor. Receives hydrogen at 60#, via interstage cooler #30 from first stage of compressor, and delivers to second stage of compressor. Condensed benzene blown to benzene receiver #27. Bottom drain cock. Pressure gauge.

The tank has a pop valve to open at 100 p.s.i.g. and a bursting disc (B.S.&B. aluminium, with lead facing on the pressure side), to open at 150 p.s.i.g. Both these have hooded vents well above the roof of the compressor room. The vents should be well away from the roof louvres of the conversion rooms, and the still furnace room. A connection, originally installed between item #31 and the gas inlet to the compressor, was removed.

#32 Cooler

Similar to #30, and cools the gas passing from outlet of second stage of the compressor to the high pressure tank #33.

#33 High Pressure Tank

Drawing 9C-8188.

One for each compressor. Receives hydrogen at 300# via cooler #33 from second stage of compressor and delivers to HB-40 department. Excess vented to atmosphere. Condensed benzene blown to condensed benzene receiver #27. Bottom drain cock. Pressure gauge. A connection, originally installed between item #33 and item #31, with a reducing valve, was removed.

There is a spring-operated release valve to atmosphere, opening at 300 lb gauge pressure, also a pop valve to open

Equipment List, contd

#33 High Pressure Tank, contd

at 380 lb and a rupture disc to open at 580 lb.

Anniston installed float type traps to discharge condensed benzene from the pressure tanks, items #31 & 33, but discarded them because of leakage past the valves. It is believed that traps made, for instance of 316 stainless steel, with hardened seatings, might serve. At present, the benzene is discharged by means of hand-operated valves, as described on page 44 above.

#34B Charging Tank for Santowax Still

Vertical tank with submerged pump #34D. #34B receives crude Santowax from crude Santowax receiver #20 via the submerged pump #20A. Return line from preheater #34C. Delivery of submerged pump is to preheater or, for the preheated material, to the still #35. There is an interconnection, low down on the side of the tanks, between #34A and #34B, also a 4" connection, about 8" from the top for use in case the bottom connection should be frozen at the start of the heating process. Inlet and outlet of heating coil. Manhole with hinged lid.

#34A Charging Tank

Similar to #34B, but only connections are the interconnecting line between the two. Vessel 34A simply adds to the capacity provided by 34B, and it has no pump of its own.

#34C Preheater

A gas fired coil within a brick-lined steel casing. On side of firewall remote from the still. Entry of crude Santowax from the pump #34D is at the top of the coil, and the flow is countercurrent to the flue gases. There are four gas burners.

#34D Submerged Pump for circulation

For circulation of crude Santowax through the Preheater #34C. Explosion proof motor.

Equipment List, contd

#35 Santowax Still

Drawings 9C-8550
9C-8551 General Layouts.
9C-8552

Horizontal tank with submerged pump to external gas heated coil #35A. Preheated charge in from submerged pump #34D of charge tank #34B. Return line from the still heater #35A. Underflow in from column #36.

Delivery line from submerged pump #35B to the still heater, #35A, or, for still bottoms, to the HB-40 department or to the pond. Vapour line to column #36, with bursting disc. Inlet and outlet for steam coil.

The temperature spread in and out of the Santowax still coil is normally 15-18°C on a Santowax R distillation. The pump has a capacity of 100 gpm and a 2" extra heavy pipe is used for the coil.

#35A Still Heater

Similar to Preheater #34C, but is longer and has eight gas burners. Gas supply controlled to give correct temperature spread between inlet and outlet of the coil.

#35B Submerged Pump

For circulating still charge through heater coils and discharging bottoms to 'pond'. Explosion proof motor.

#35 Column

12-plate column. 3 x 1/8" drain holes in each plate to prevent blockage by freezing. It is thought 1 x ¼" hole would be a better safeguard. Inlet for vapour at bottom from still #35. Inlet from reflux split box #38. Underflow from bottom back to still #35. Vapour out to condenser #37.

A much simpler column will serve if only Santowax R is to be made.

#37 Condenser DSW 462167

Single pipe in form of zig-zag and jacketed for water or steam. Receives vapour from top of column #36 and delivers condensate to reflux split box #38. It is important to ensure that water cannot be trapped in the jacket and raised to a very high temperature by the Santowax vapour.

Equipment List, contd

#37 Condenser, contd

Actually, only a dribble of water is used, in parallel flow with the vapour, and the water stream is so adjusted that the reflux split box temperature is around 200°C, (point #10 on the Multipoint recorder) for Santowax R. An earlier condenser, of shell and tube design, using Aroclor as a cooling medium, was discarded in favour of this simpler condenser, because of maintenance troubles.

There are no drawings, but Mr. Ellenburg, Aug.23/48, wrote the following description:

"The inner tube is a standard 2" black iron pipe, jacketed throughout its 45' of length by a 3" black iron pipe. ---- by extending a well-insulated 6" riser 8' above the top of the column, we were able to get 45' of condenser to connect to the original location of the split box as follows: Four 10' lengths of pipe were doubled back on each other to give a hair-pin shape condenser (a descending zig-zag. E.M.), with a pitch of about 1" per foot. Vertical sections about 9" in length were used to connect the long sections. A final vertical section of 2½' long connects the split flow box with the zig-zag part of the condenser. The inner pipe is jacketed continuously to prevent any cold spots on the inner tube. ----- performance under the --- heaviest load 2000 lb total condensate/hr------. ----- For the ortho and meta terphenyl fractions, where much lower condensate temperatures are permissible ---- the water rate can be increased.---- It would be worth while to provide a blanked off connection for sampling the condensate even if a complete (sampling) arrangement is not installed."

#38 Reflux Split Box

Standard type, but no reflux is used in the purification of crude Santowax to Santowax R.

#39 Santowax R Receiver

A vertical tank operating under a pressure of 50 mm of mercury absolute. Receives Santowax R from condenser #37 via #38. Inlet and outlet for steam coil. Connection to vacuum pump #40. Bottom outlet to the buffer vessel, item #39B. Valve for breaking vacuum.

Equipment List, contd

#39A Submerged Pump in item #39B

Delivers to the Aroclor plant, the Santowax R flaker, or the HB-40 plant as required.

#39B Santowax Distillate Receiver

Vertical cylindrical vessel 5' x 5'. Distillate in from the receiver, item #39, and out by submerged pump, item #39A. (Works under atmospheric pressure. Has steam coil.)

#40 Vacuum Pump

Manufactured by De Vine. Between item #39 and the vacuum pump, Anniston have a sublimate trap and a vacuum tank in the line, to allow settling of particles. No filter is employed because traces of terphenyl reaching the pump are either dissolved in the oil or discharged to atmosphere, and do no harm.

Equipment List, contd

Instrumentation

See Drawing 9C-9060.

A. Conversion Units

Each convertor unit has:

(a) A L.&N. 'Micromax' recording-controlling instrument for the temperature of the convertor pot, which operates by controlling the gas supply to the pot. This same instrument, still responding to the thermo-couple on the convertor, also controls the gas supply to #2 preheater. If automatic control should lead to towards a drop in temperature of the #2 preheater, then this is corrected by turning down burners on the convertor.

(b) A Foxboro "On-Off" type potentiometer temperature controller for the #2 preheater to prevent the temperature of this pot from getting too high. All pilot lines are taken from the gas line to the #1 preheater, which only has manual control of the supply.

The Foxboro controller is a safety device which will work if the convertor control does not operate. The convertor control should cut the gas off on both No. 2 pot and the convertor pot if the temperature gets too high in the convertor, but if the control fails the Foxboro cuts off the gas in No.2 pot. There is no definite limit on this control, but it operates at approximatdy 760°C.

(c) A L.&N. 'Micromax' 12-point recording instrument for temperatures at the following points:

#1 Vapour from outer vapouriser coil to #1 preheater.
#2 Vapour from inner vapouriser coil to #1 preheater.
#3 Vapour from #1 preheater.
#4 Vapour from #2 preheater.
#5 Vapour from convertor.
#6 #2 preheater lead.
#7 top of column
#8 condenser water outflow
#9 flue gas to vapouriser
#10 flue gas from vapouriser.
#11 #1 preheater lead.
#12 sump line from column.

DSW 462170

The points for units #4 and #5 have not been detailed here -- they are two-pot units of a different and less satisfactory design.

Equipment List, contd

A. Conversion Units, contd

(d) A Foxboro flow controller for the benzene supply to each unit, activated by an orifice in the feed line. Foxboro Pneumatic Pressure Transmitters have been installed. In November 1950 these were installed, with one spare, in a separate room which contained also the meters for the fuel gas, and which had a communicating door to the control room where most of the other instruments were located.

These indicate the flow and also transmit the orifice pressures to the control instrument. There are also located, within the Control Room, hand valves and pressure gauges for each benzol line. See also page 31 above.

B. Off-gas Treatment

(a) A temperature indicating instrument responding to thermocouples at the following points on the Hydrogen cooler

(1) Hydrogen in (2 points).
(2) Hydrogen out (4 points).
(3) Cooling water out (4 points).

(b) A controller responding to pressure changes at the hydrogen surge tank #26 and controlling solenoid (more recently pneumatic) operated valves on the compressors so as to maintain this pressure within the limits of 10-16 oz. per sq.in.

Rubber fittings must not be used inside instruments where they will be exposed to the process gas, because of the swelling which would be caused by the benzene in the gas.

C. Diphenyl Stills

Each still has:

(a) A L.& N. 'Micromax' controller similar to that used on the convertor pot, but controlling, via the gas supply, the spread of temperature between inlet and outlet to the heating coil.

(b) A L.& N. 'Micromax' multipoint recording instrument for temperatures of
#1 Still Body,
#2 Coil Inlet, this will be about the same as point #1.
#3 Outlet circulating line.
#4 Top of column.
#5 Reflux to column.
#6 Condenser Water.

DSW 462171

If the circulating pump should stop, the gas supply to

Equipment List, contd

D. Santowax Still

(a) A L.&N. 'Micromax' Controller similar to that used on the convertor pot, but controlling, via the gas supply the 'spread' of temperature between inlet and outlet to the heating coil.

(b) A L.& N. 'Micromax' multipoint recording instrument for temperatures of:

#1 Still body,
#2 Heater inlet,
#3 Heater outlet,
#4 Vapour leaving column,
#5
#6
#7 Top inside of column,
#8 Sight glass at receiver,
#9 Bottom inside of column,
#10 Condensate in flow box,
#11 Preheater inlet,
#12 Preheater outlet.

Specimen charts from most of these instruments were sent to Mr.W.M.Cooper, London, in October 1947.

Armiston suggest using a glass wool trap, about 1 pint size, to protect instrument lines from blockage by diphenyl etc., and using glycerin traps to protect instruments operating on benzene services.

COST OF EQUIPMENT

Armiston estimate for the installation of 3 diphenyl units (#11, 12, & 13), June 26/47, was given on page 58 of the report by Havercroft & Mather, September 1947, and is not repeated here, nor are the installed cost figures for other parts of the plant which were given on page 58a of that report.

COMMENTS ON THE PROCESS

See "Earlier Reports" listed on page 2 above, and "Note on Choice of Process" on page 6 above.

Effect of Variation in Benzene Quality

See also notes on benzene under "Specifications & Test Methods, page 54 above.

Anniston report 1642, Oct.15/41, states that, at that time, benzene with a distillation range of 79.4 to 81°C was used, but that the freezing point had to be 5.0°C minimum. Traces of toluene caused the formation of styrene etc. which accumulated in the "heads" fraction of the diphenyl distillation. The amount of this was only about 0.065% of the diphenyl made. Aliphatic impurities were limited in amount by the 5°C specification. The material contained 0.06 to 0.08% total sulphur, but this was not thought to cause any operating difficulty, and sulphur was found to delay the formation of carbon on the metal surfaces of the pots. Experimental work indicated that water in the benzene favoured the formation of carbon. (Excessive amounts, if pumped into the vapourizer, may cause a violent development of steam, endangering the joints of the equipment.)

The report refers to work by F.E.Hubbard & R.A.Ewing, Sept.17/41, showing that fresh benzene gives 1.5 to 1.8% more conversion per pass than does recycled benzene, and other reports mention even greater differences. See for examples pages 112 & 113 below. This effect might be explained in one of the following ways:

(1) The fresh benzene may contain some material which promotes the reaction.
(2) The recycled benzene may contain some material which inhibits the reaction.
(3) The recycled benzene may contain some material which destroys an inhibitor which is present in the fresh benzene.

Explanation (3) seems to be ruled out by the observation that in ordinary routine working, with about 80% recycled benzene, and about 20% fresh, the conversion goes quite well. As between (1) & (2) it is argued that some grades of fresh benzene show even higher conversion rates and it has been shown by experiment that the addition of certain chemicals does actually speed up the conversion.

Comments on the Process, contd

Effects of "Promotors" in Benzene

The report by Conover cited on page 3 above gives the following results in a laboratory unit.

Promotor Added 2% by weight	% Conversion at 800°C (Diphenyl in product)
None (recycled benzene)	10
None (fresh benzene)	14.5
Acetic Acid	23.0
Ethyl Alcohol	24.0
Ethyl Ether	24.5
n. Butyl Alcohol	26.0
sec. Butyl Alcohol	26.5
iso. Butyl Alcohol	27.5
Acetone	30.8

Effect of Size of Dose	% Conversion @ 750°C
2.0% iso. Propyl Alcohol	17.6
1.0 " " "	17.0
0.5 " " "	17.1
0.2 " " "	16.0
0.15 " " "	12.7
0.1 " " "	10.8
0.0 " " "	6.3

Comments on the Process, contd

Effect of "Promotors" in Benzene, contd

Promotors have not been used at Anniston in manufacturing work. Conover's report recommends that if the use of promotors in routine manufacture is planned, a trial batch ought first to be carried right through to Aroclors, to make sure the product does not contain any detrimental by-products.

If the plant is charged with fresh benzene the conversion figure for the standard conditions will be about 2½%, but this figure will fall as the unchanged benzene is recycled, and at the end of the day may be as low as 10%. The incoming benzene, though of very high purity, appears to contain enough of an unidentified promoter to have a marked effect on the rate of conversion.

The falling off in the conversion rate would naturally be very inconvenient in routine manufacture, so the feed of fresh benzene is made practically continuously into the recycled benzene tank, so that there is always about 20% of fresh benzene in the feed to the vapourizers. The reports just quoted discuss the conversion reaction in much greater detail.

Impurities likely to occur in the fresh benzene are toluene, paraffins, cycloparaffins, and similar products, boiling around 80°C. Pyrolysis of benzene which is contaminated with these will give diphenyl which is likely to be contamnated with a variety of products, and unless these contaminants are removed before the diphenyl is chlorinated to give Aroclors, the Aroclors made from the diphenyl are liable to be contaminated with chlor compounds in which the chlorine is more labile than in true Aroclors. Consequently the contaminated Aroclor may be rejected on the very stringent tests imposed by some electrical users of Aroclors.

The Diphenyl made by MCL is to be used to make Aroclors, and Mr. T. G. Crane, Oct.17/47, estimated that about half the Aroclor production would be as 1254 for electrical uses, so the question of stability of the Aroclors is of importance to MCL. Apparently, however, the trouble has been limited to "Pyranols", mixtures of Aroclors with trichlorbenzene, and it is the trichlorbenzene which has been mainly at fault. Mr. Ellenburg suggested that more intensive fractionation of the diphenyl might compensate for the use of inferior benzene.

Dr. Hardy, Dec.23/47, stated that benzene contaminated with paraffins, would yield styrene and apparently vinyl biphenyls, and that these products, on chlorination, would yield materials

Comments on the Process, contd

Effect of "Promotors" in Benzene, contd

liable to cause skin troubles. The chlorinated products might be stabilized by being held at high temperatures, but it was not certain that the stabilization would go far enough.

Toluene on pyrolysis is said to give some styrene, and it was believed at one time at Anniston that chlorine derivatives of styrene were causing skin troubles among the workers in the Aroclor plant. See page 16 above.

In a letter dated April 21/49, Mr. Ellenburg listed alkylated benzenes and alkylated biphenyls as possible by-products from the used impure benzene, and he stated that styrene and stilbene have been identified in the "heads" fraction of the diphenyl still.

Benzene homologues on pyrolysis give also dibenzyl or derivatives (M.Szwarc, "Nature", Sept.20/47).

Effect of Variation in Time-Temperature of Exposure

The process is normally run with the lead in the 3rd pot at not above 840°C and with a benzene flow rate of 170 U.S.Gal/hr., these conditions giving about 18% conversion of the benzene into diphenyl, terphenyls etc. The retention time corresponding to the given flow rate, naturally depends on the pressures at which the pots are operated. As the process is carried on day after day, the vapour passages become more and more restricted by the deposition of carbon in them, and this raises the pressure in the pots, and therefore the retention time increases. Also, orifice plate restrictions are being tried in the exits from the 3rd pots. When the pressures increase, from either cause, the practice is to reduce the temperatures enough to maintain the same conversion figure of about 18%. Increasing pressures also give less entrainment of lead because of the lower linear velocity of the vapours, and the lower temperatures should reduce the corrosion of the pots, but Anniston experience is showing that these advantages may be dearly bought, at the cost of increased maintenance of the pot covers, gaskets, etc.

See also under "Process in Detail", page 34 above.

Comments on the Process, contd

Effect of Variation in Time-Temperature of Exposure, contd

Amniston report 1642 of Oct.15/41 gives the following statement of the effect of variation in pot temperatures:

Temp. of lead in 1st pre-heater	Temp of lead in 2nd preheater	Temp of lead in convertor	% Conversion to crude diphenyl
580	725	840	18
700	725	840	20
580	720	840	18.0 to 19.0
580	775	825	17.7 to 17.9
580	800	825	21.1 to 21.9
#9 unit 580	800	800	17.9 to 19.3

Carbon formation becomes excessive above 840°C.

The effect of variation in pressure in the lead pots was studied by Conover & Huff of Plant "A", see report Oct.5/35. At 800°C under certain conditions they obtained 6.2% conversion at 5 psig, and 14.4% at 38 psig.

It is stated that ratio of terphenyls to benzene in the reaction product is about the same as the ratio of diphenyl to benzene. At 18% conversion, the ratio of diphenyl to terphenyls, etc., (high boilers) is about 4.5 to 1, and the amount of carbon formation is not too serious, but if the % conversion is pushed beyond 18%, the ratio of highboilers to diphenyl increases, also the ratio of carbon to diphenyl, so 18% appears to be about the economic balance for American conditions, even though Amniston cannot sell or use the whole output of high boilers.

See "Design Data for 4th Unit," Aug.9/39, for a curve of costs v. % conversion.

Raising the pot temperature gives an increase in output of diphenyl with relatively little increase in the consumption of fuel gas and at times small variations in conversion rate have been made at Amniston to get a greater output of diphenyl, at the cost of a higher proportion of wastage as highboilers.

Amniston report 1642 cites a report by Durgin, Heald & Perry, May 15/31, indicating that if highboilers are recycled in the benzene, the amount of HB in the crude diphenyl is not increased, but that it has a higher boiling range. Amniston experience up to November/50 indicates that recycling of diphenyl in the benzene increases the amount of carbon formation.

The benzene distillate from the diphenyl still is examined before being recycled to the conversion stage. It usually contains very little diphenyl, but if it contained more than about 3% it would be fed back gradually, or in an extreme case, returned to the heads & tails tank for redistillation. It was stated in September 1947 that conversion was reaching 13% in the second preheater and 18% in the convertor.

The extent to which the reaction proceeds in each of the 3 pots, naturally depends on the conditions of temperature and pressure in each pot. Anniston report 1642 gives the following figures for vapour leaving the second preheater:

2nd preheater lead temperature	% conversion
640°C	1.0
720-725°C	4.0 to 5.0
775°C	10.0

Distillation of the Crude Diphenyl

By-products, which boil between benzene and diphenyl, build up, only very slowly, in the continuous recycling of the "heads" fraction, and it is not difficult to run the fractionation so as to give diphenyl of the necessary quality. Mr.Ellenburg, May 12, 1949, stated that about 0.1% to 0.2% of the diphenyl yield was side-tracked in the form of the oil from the "heads" fraction. Anniston report 1642 gives 0.065% on the diphenyl as the amount of styrene, etc. formed.

Variation in the diphenyl distillation conditions might also go some way to offset the effects of impurities in the benzene used, see pages 55 & 113 above.

Benzene recovery from the Hydrogen

Anniston reported moderately good results from scrubbing the hydrogen with mineral oil. An activated alumina absorber was installed at Anniston, to take up benzene from the hydrogen, but it gave poor results and was discarded in favour of the present compression system. It is thought that the alumina was not properly re-activated. See Anniston reports 214,Sept.22/33, & 948, Aug.21/35.

Since there is a use for the compressed hydrogen, the compression system appears to be the best for Anniston conditions.

Originally the hydrogen at Anniston was piped to a gas main and burned, with natural gas, under the units, but difficulties in keeping the correct air-gas ratio in the burners soon caused a discontinuation of this use of hydrogen.

COST DATA

Labour Requirement

At Anniston in November 1950, one operator per shift could look after up to 5 conversion units and one diphenyl still. In addition, there was one chief operator per shift, (but his responsibilities included Aroclor & HB-40,) and there was a general day foreman, (Curry), covering a number of departments, including Aroclors.

Mr. Ellenburg wrote Aug.18/48, about the breakdown of the various service charges among the different products:

	Diphenyl Crude	Diphenyl Distilled
Steam, lbs	0.10	0.91
City water, cu.ft.	0.037	---
Recovered water, cu.ft.	0.49	4.40
Gas, cu.ft.	13.5	2.2
Electricity KWH	0.050	0.008

The units are given per pound of product -- for June/48, and he comments that there are seasonal variations in such figures.

Repair Work

The practice sheets, pages 120 & 121 below, give a record of the frequency of various repair jobs.

MONSANTO CHEMICAL COMPANY — COST REPORT — Anniston PLANT

PRODUCT Biphenyl — G-1300 — DEPT. NO. — MONTH August 1967

PRODUCTION—THIS MONTH 983,003 lb — YEAR TO DATE 7,641,387 — COST UNIT 100 lb

RATED CAPACITY — % OPERATED — DAYS OPERATED AT — % OF — BRACKET

PERCENT YIELD ON — STANDARD — ACTUAL — M. & E. INVESTMENT

PERCENT YIELD ON — STANDARD — ACTUAL — M. & E. EXP. RESERVE

MATERIAL AND UTILITY REQUIREMENTS

	Actual Quantity Used	Standard Quantity Used	Material Unit	Standard Material Unit Price	Quantity Standard	Actual This Mo.	Actual Yr. to Date	Last Year
Benzol	141,301	141,621	U.S. gall.	.19017 (year to date .19005)	18.04	17.97	17.93	17.07
STEAM								
ELECTRICITY								
COMPRESSED AIR								
WATER—PURCHASED								
WATER—PLANT								
FUEL—GAS, COAL, OIL								

ORGANIC DIVISION

Statement of Production Cost	Actual Cost	Efficiency Standard Cost	Variance	Current Month Actual	Current Month Standard	Year to Date Actual	Last Year Actual
Raw Materials							
Benzol	32 610 57	32 640 35	29 78	3.428	3.425	3.390	[illegible]
NET TOTAL MATLS.	32 610 57	32 640 35	29 78	3.422	3.425	3.399	3.644
STEAM	593 46	857 70	264 28	.062	.090	.066	.071
ELECTRICITY	668 75	714 75	46 00	.070	.075	.071	.075
COMPRESSED AIR							
WATER—PURCHASED	12 81	28 57	15 76	.001	.003	.001	.004
WATER—PLANT	1 466 87	1 572 46	105 59	.154	.165	.117	.147
FUEL—GAS, COAL, OIL	3 532 59	3 306 93	− 225 67	.371	.347	.347	.348
LABOR—OPS.	1 940 97	2 077 85	136 88	.204	.218	.203	.281
SUPERVISION—OPS.	67 50	181 07	113 57	.007	.019	.009	.046
S.S. TAX, COMP. INS., PENSIONS	129 74	143 95	13 21	.014	.015	.014	.020
REPAIRS—LABOR	733 10	1 591 52	858 42	.077	.167	.120	.105
REPAIRS—MATLS.	1 744 95	1 019 71	− 725 24	.183	.107	.218	.101
SUPPLIES	481 04	287 31	− 193 73	.047	.027	.024	.027
LABORATORY	61 12	70 24	9 12	.001	.008	.004	.003
CLOTHING AND LAUNDRY		9 53	9 53		.001		.001
DEPRECIATION & [illegible]						.001	
Stationery + printing / Development	38 43		− 38 43	.004	[illegible]	.003	
COST TO FINISH OPERATIONS							
TOTAL DIRECT COST	11 448 64	11 836 30	387 91	1.201	1.242	1.219	1.236
DEPRECIATION	2 970 49	2 858 01	− 121 77	.310	.300	.309	.467
CONTROLLABLE—F.I.E.	1 878 68	1 439 66	243 02	.164	.199	.173	.319
NON-CONTROLLABLE—F.I.E.	1 196 78	420 40	− 27 35	.125	.097	.108	.173
TOTAL INDIRECT COST							
TOTAL MFG. COST	49 936 00	50 051 72	115 61	5.239	5.283	5.104	5.809
TOTAL PACKING COST	808 88			.084		.057	.059
Total cost F.O.B. car				5.370		5.246	5.964

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MONSANTO CHEMICAL COMPANY COST REPORT *Anniston* PLANT

PRODUCT *Santoway R* *6-1301* DEPT. NO. ____ MONTH *August* 47

PRODUCTION—THIS MONTH *71,448 lb* YEAR TO DATE *714,102* COST UNIT *100 lb*

RATED CAPACITY ____ % OPERATED ____ DAYS OPERATED AT ____ % OF ____ CAPACITY

PERCENT YIELD ON ____ STANDARD ____ ACTUAL ____ M. & E. INVESTMENT ____

PERCENT YIELD ON ____ STANDARD ____ ACTUAL ____ M. & E. DEP. RESERVE ____

	ACTUAL QUANTITY USED	STANDARD QUANTITY USED	MATERIAL UNIT	STANDARD MATERIAL UNIT PRICE	QUANTITY STANDARD	QUANTITY USED PER UNIT PRODUCED: ACTUAL THIS MO.	YR. TO DATE	LAST YEAR
Crude santoway			lb	*N.V.*				

(distillation yield was stated to be about 85%.)

MATERIAL AND UTILITY USAGES

STEAM
ELECTRICITY
COMPRESSED AIR
WATER—PURCHASED
WATER—PLANT
FUEL—GAS, COAL, OIL

ORGANIC DIVISION

STATEMENT OF PRODUCTION COST	ACTUAL COST	INVENTORY STANDARD COST	VARIANCE	UNIT COSTS: CURRENT MONTH ACTUAL	CURRENT MONTH STANDARD	YEAR TO DATE ACTUAL	LAST YEAR ACTUAL
RAW MATERIALS							
Crude santoway	*N.V.*						
NET TOTAL MAT'LS							
DIRECT CONVERSION COST							
STEAM	59 94	44 01	14 07	.083	.103	.0[illegible]	.015
ELECTRICITY	25 00	26 57	1 57	.036	.037	.035	.0[illegible]
COMPRESSED AIR							
WATER—PURCHASED	1 47	27 31	25 80	.002	.038	.[illegible]	.019
WATER—PLANT	27 24	26 74	1 48	.038	.[illegible]	.[illegible]	.0[illegible]
FUEL—GAS, COAL, OIL							
LABOR—WAGES	139 47	244 28	104 81	.194	.3[illegible]	.2[illegible]	.3[illegible]
SUPERVISION—WAGES	22 57	9 [illegible]	– 13 61	.031	.013	.[illegible]	.018
S. S. TAX, COMP. INS. PENSIONS	10 34	15 81	5 46	.014	.022	.016	.02[illegible]
REPAIRS—MINOR	45 02	69 13	[illegible]	.363	.0[illegible]	.3[illegible]	.067
REPAIRS—MAJOR	[illegible]	37 34	32 82	.006	.052	.250	.067
SUPPLIES	5 52	7 19	1 67	.009	.01[illegible]	.018	.010
LABORATORY		27 30	27 30		.03[illegible]	.0[illegible]	.032
CLOTHING AND LAUNDRY		2 87	2 87		.004		
REFRIGERATION & ICE							
EXP. TO FINISH OPERATION							
TOTAL DIRECT COST	341 08	560 42	219 34	.473	.780	.926	.[illegible]
INDIRECT: DEPRECIATION	424 54	135 79	– 288 75	.591	.18[illegible]	.479	.[illegible]
CONTROLLABLE—F. I. E.	301 62	88 43	– 213 19	.4[illegible]	.137	.371	.[illegible]
NON-CONTROLLABLE—F. I. E.	157 15	38 08	– 120 07	.219	.052	.[illegible]	.[illegible]
TOTAL INDIRECT COST	904 [illegible]	272 30	– 632 61	1.258	.379	1.003	.476
TOTAL MFG. COST	1,245 39	832 72	– 412 67	1.733	1.159	1.927	1.200
TOTAL PRODUCTION COST							

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[redacted]

Month of Aug. 1947				Year To Date 19 47
¢	Pounds	Production	Pounds	¢
11.18	955,803	Diphenyl - Total	7,641,387	10.89
	3,468	" Lbs. Unit Day	3,788	
18.18	819,868	Santowax β	1,799,190	18.71
	16,879	Hydrogen	137,489	
Per Lb.	Gallons	Raw Materials Used	Gallons	Per Lb.
0.1797	171,381	Benzol - Actual	1,369,856	0.1795
0.1681	160,385	" Theoretical	1,293,956	0.1695
	11,996	" Loss	75,880	
0.9352		" Recovery		0.9446
Per Lb.	Quantity	Services	Quantity	Per Lb.
1.039	990,000	Steam - Lbs.	7,976,000	1.044
0.021	20,000	Water-City-Cu. Ft.	145,000	.019
5.247	5,000,000	Water-Recov'd-Cu. Ft.	38,405,000	5.026
19.446	18,532,000	Gas - Cu. Ft.	134,991,000	17.666
0.056	53,500	Electricity -KWH	413,000	.054
	8	Truck - Hours	11	
Lbs. M.H.	Man Hours	Labor	Man Hours	Lbs. M.H.
658	1,448	Operating	11,471	666
1,212	786	Maintenance	7,672	996
2,352	305	Packing	865	5,794
11,720	54	Shipping	189	24,140
¢	Unit-Hours	Operating Time	Unit Hours	¢
100.0	6,696	Hours - Calendar	52,488	100.00
0.63	42	Hours - Shut Down	3,464	6.60
99.37	6,654	Hours-Gross Operated	49,024	93.40
	52	Hours - Delay	508	
	6,602	Hours-Net Operated	48,516	92.43
0.78%		Delays % of Gross Oper		1.04
		Delays:		
1		Clean Gooseneaks (Large)	11	
2		Clean Gooseneaks (Small)	7	
		Clean Distributor	2	
1		Clean Carbon Trap	9	
1		Clean Lead Trap	8	
		Clean Column		
1		Clean Vaporizer Coil	2	
1		Change Distributor Conv.	12	
		Change Lead Pot	1	
		Change Lead Trap	4	
		Change Thermocouple		
1		Change Thermo-Well	3	
		Change Gooseneck Flange		
		Change Pressure Gauges		
		Repair Release Line		
		Miscellaneous	14	
		Clean #1 PH Dist.	2	
		Repair Reheater		
1		Repair Vaporizer Coil	1	
		Clean KHD Head	1	
		Clean Feed Lines	2	
		Change #2 PH Dist.	4	
1		Clean Condenser	1	
		Change Lead Pot #2 P. H.	1	
1		Repair Conv. head	3	

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DIPHENYL REPORT

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Mr. Kllenburg, January 20/48, gave the following breakdown of the fuel gas consumption:

	cu.ft./lb.
Aroclor Distillation	0.66
Biphenyl - Crude Units	16.7
Biphenyl - Distillation	2.2
Santowax R Distillation	1.5

Calorific value of the gas about 1040 B.th.U./cu.ft. at 60°F and atmospheric pressure.

C

Aug. 19 [illegible]				[illegible]
Pounds		Shipped and [illegible]		[illegible]
581,060		Shipped-Inter-Plant		[illegible]
		Shipped-Inter-Co.-Others		
59,165		Shipped-Outside-I		[illegible]
632,[illegible]		Shipped-Total		[illegible]
717,[illegible]		Produced		[illegible]
104		Rejected		[illegible]
Containers	Pounds	Containers Used	Pounds	Containers
8	581,060	Tank Cars-[illegible]	4,581,909	[illegible]
		Tank Cars-[illegible]		
		55 Gal. Blk. Steel [illegible]-Used		
		-Destroyed		
851	57,632	Black Barrels-Used	804,195	[illegible]
		-Destroyed		4
1	100	Black Bags-Used	450	5
		-Destroyed		
		55 Gal. Fibre Drum Used		
		-Destroyed		
		100# Fibre Drums-Used	85	1
		-Destroyed		[illegible]
		Paper Bags-Used		
		-Destroyed		
		Paper Liners-Used		886
		-Destroyed		

BIPHENYL
EXPORT

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WATER_PCB-SD0000046347

A C K N O W L E D G M E N T

This report is mainly a compilation of information drawn from Anniston reports and from discussions and correspondence with many members of the Anniston staff, whose willing and able help is gratefully remembered.

The reports made by Messrs Havercroft & Mather, September 1947, have been incorporated, also that of Mr. H. L. Harden, August 1950, and observations made by the author during visits to Anniston.

I N D E X

(Underlined figures denote the more important entry under any one subject)

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