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B.I.O.S. FINAL REPORT No. 853
ITEM No. 22

0 MAY 1947

I.G. LEVERKUSEN.

AZOBENZENE.

(Miscellaneous Chemicals)

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE
Price 1s. 0d. net

I.G. LEVERKUSEN.

AZOBENZENE.

Reported by :

H. Shaw - British Civilian) Ministry
O. Whitston - British Civilian) of Supply.

BIOS Trip No: 1081
BIOS Target Number: 22/1 (e).

CIOS Black List Item 22
Miscellaneous Chemicals.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE
32, Bryanston Square, W. 1.

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I.G. LEVERKUSEN.

AZOBENZENE.

Process.

Reduction of nitrobenzene to Azobenzene with water and sodium amalgam obtained from Mercury Cells.

General.

Hydrazobenzene is produced at Leverkusen by zinc reduction of nitrobenzene. The first part of the reduction has been transferred to the amalgam process, permitting the zinc plant to concentrate on the azobenzene to Hydrazobenzene stage, and so increasing the output.

The process now worked in the amalgam cells will not produce hydrazobenzene in a practicable manner.

Plant.

(1). Cell. 8 x 20,000 ampere mercury cells with a 14M x 60 cm. brine section and 14 M x 20 cm. decomposer have been isolated for this process. The cells themselves are described in "German Chlorine Plants in the American, French and British Zones - Part I", and as they appear in the azobenzene process merely to provide the amalgam, they are not described here.

Each cell is provided with an azobenzene reactor, placed at the end of the brine cell and in line with it. The end box of the cell is modified to allow amalgam to flow either into the reactor or along the normal channel into the decomposer, depending on which of two outlets is closed. Spent amalgam from the reactor flows back into the normal decomposer and so back into the normal cell system.

(2) Azobenzene Reactor. A nickel pot, 1.1 M high x 1 M diameter made of 3 M.M. sheet, welded. It is rather flimsy and is supported in an outer M.S. cradle. The amalgam inlet is at the bottom, the outlet also at the bottom, but 85 m.m. above it. and the liquor outlet is in the side, 160 m.m. above the amalgam outlet.

M Published as BIOS Final Report No. 546.

A nickel gate stirrer, 80 r.p.m., reaches to within 5 m.m. of the bottom, so that both amalgam and azobenzene are agitated, 5 turns of 40 m.m. bore Ni pipe round the inside wall provide water cooling. The lid is nickel clad steel, flat, carrying a normal gland for the stirrer and 7 other flanges, through which pass

(i) Cooling water inlet and outlet. The reactor is at cell potential and is not earthed. Pipelines etc. to it are therefore broken with an insulating rubber sleeve and liquor flows are broken. The water inlet carries an open funnel rather higher than the outlet into which water is fed from a sprinkler.

(ii) Inlet for reaction water - provided with a sprinkler breaker enclosed in a sight glass.

(iii) Nitrobenzene inlet.

(iv) Connection to a 4" vent main leading to outside atmosphere.

Nickel is the only known material which will withstand the conditions in the reactor, and even this fails at the welds unless the pot is annealed after manufacture. Stainless Steel (V2A), and sprayed cement on iron have been found useless.

Difficulty was experienced with the run off valve, but a design was evolved which is satisfactory.

(3). Sludge Separator. After reaction, the contents of the pot are dropped to 2 sludge separators, along a steam jacketed 300 m.m. diameter collecting main, made for easy cleaning. The sludge separators are steam jacketed 5 M^3 cast iron pots with conical bottom and sludge run off.

(4). Treatment of Azobenzene. There is a battery of 12 stirred, jacketed, cast iron pots adjacent to the cells, capacity guessed to be about 10 - 15 M^3, of which 6 are normally used -

2 for Separation of Azobenzene and NaOH.
1 for Washing Azobenzene
1 for treating the NaOH liquor from the reactor.

2 for storing pure Azobenzene.

Liquor transfer is by compressed air.

(5) NaOH Clean Liquor Storage. 5 tanks below the cells (about 15 M³ each) with pumps.

Operation.

The reactors are worked batchwise. Charge is 120 Kgm. Nitrobenzene and 90 l of water, but the reactor has been designed so that the required amount of water is left behind. Only the Nitrobenzene is added. During the reaction, however, it is necessary to replace the water consumed and 200 l. of water per 120 kgm. of nitrobenzene is added gradually for this purpose, endeavouring to adjust the feed rate to the reaction rate, which is not constant. A 200 l water feed vessel gives better control than rotameter and is preferred.

The reaction is initially quite vigorous and cooling water has to be used to maintain the required temperature of 80°C. The batch continues for 140,000 ampere hours, i.e. for 7 hours at 20,000 amps.

Amalgam.

The cell and reactor contain 1300 kg. of mercury compared with 1000 in the normal cell. The brine cell is run hot deliberately, 70/80°C., to avoid excessive cooling of the reactor. It is also kept rather short of mercury in order to obtain fairly strong amalgam - normally 0.5% from the brine cell. In the early stages of the batch, all the sodium reacts, but towards the end only about half. On the average, about 90% of the sodium is used in the reactor.

Product.

The azobenzene produced melts at 70°C. and has to be kept in steam heated vessels.

Composition	Azobenzene	75/90%
	Hydrazobenzene	15/5%
	Aniline	2/3.5%
	Nitrobenzene	8/3%

55% NaOH liquor is produced simultaneously.

Sludge formation is the major problem with the process. Mercury reacts with some of the organic matter, forming copious sludge which has to be separated off and treated for mercury recovery. Treatment consists in stirring with amalgam, which reduces the azobenzene in the mixture to hydrazobenzene which is less potent as a sludge producer.

The 55% NaOH liquor, after separation from Azobenzene, is treated with 1% of its weight of chlorine, allowed to stand for 2 hours and is then used for making sodium fluoride. It is not suitable for sale or for organic preparations, as it contains phenols.

The NaOH made in the normal decomposer is of good quality and is mixed with the main production.

Hydrazobenzene has been made at 130°C, using a steam jacketed pot, but much aniline was produced. It was not developed beyond the experimental stage.

Performance.

The mercury cell operates less efficiently about (80%) because of high temperature and high amalgam concentration. The azobenzene is operated marginally on the cell process, the Cl$_2$ and NaOH go to the cell process, which charges the following back to azobenzene.

(1) Direct Services and labour incurred specially on its behalf.

(2) The loss of NaOH and Cl arising from the lower cell efficiency.

(3) Extra D.C. on the cells concerned.

		1943.		
Output	Actual	500.389 tons.	= 95%	
	as 100% Azobenzene	474.203	Kg/te = 94% Effy.	
Usages.				
Nitrobenzene	te	680.8		
Chlorine Gas	te	71.4	151	
NaOH Liquor	te	118.7	250	
Hg	kg	2277	4.8	
Water	M^3	3495	7.4	
Steam	te	305	0.6	

Usages

Power A.C.	KWh	to 35161	74.1
" D.C.	"	135782	286.0
Comp. Air	M^3	26573	56.1
Process Labour		12390 hrs.	26.1

Drawings.

The following Drawings have been lodged with:- Board of Trade, German Division (Documents Units), Lansdowne House, Berkeley Square, W.1. Tel: Grosvenor 4060, Ext: 2923.

Drawing No.	
94257.	General layout.
117583.	Reactor.
94191.	Reactor Valve.
91754.	Sludge Separator.

All applications for permission to inspect these drawings should quote the following Reference Number: BIOS/Docs/1081/1315.

TSDIN FORM 69 (13 MAR 47)

Shaw, H
Whitston, O

AUTHOR(S)

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ABSTRACT

Reduction of nitrobenzene to azobenzene with water and sodium amalgam obtained from mercury cells is described. The reaction is quite vigorous, and cooling water is used to maintain temperature at 80°C. The azobenzene produced melts at 70°C and has to be kept in steam-heated vessels. Nickel is only known material which will withstand the condition in the reactor. Production of amalgam is hindered because of inefficiency of mercury cells caused by high temperature and high amalgam concentration.