The U.S. Government is absolved from any litigation which may ensue from the contractors infringing on the foreign patent rights which may be involved.
REEL - C
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3
The report is marked with the warning that "if the subject matter should be protected by British patents or patent applications, it should be protected by British patents or patent applications, thus publication cannot be held to give any protection against action or publication."
Reported by:

N. Shaw - British Civilian | Ministry
C. Whitston - British Civilian | of Supply.

BIOC Trip No: 1061
BIOC Target Number: 22/1 (e).

BIOC Black List Item 22
Miscellaneous Chemicals.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE
32, Bryanston Square, W. 1.
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Hydrazobenzene is produced at Leverkusen by the reduction of nitrobenzene. The first part of the reduction has been transferred to the amalgam process, permitting the zinc plant to concentrate in the 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. 6 x 20,000 square mercury cells with a 14 M x 60 cm. brine section and 4 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 II", and as they appear in the hydrazobenzene 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 the 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.N. sheet, welded. It is rather flimsy and is supported in an outer M.N. candle. The azobenzene 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, 150 m.m. above the amalgam outlet.

Published as BMFC Final Report No. 646.
A nickel gate stirrer, 60 r.p.m., reaches to within 5 m.m. of the bottom, so that both ammonia and acen- 
benezene are agitated. 5 turns of 40 m.m. bore Hi 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 

- 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 
carry's an open funnel rather higher than the outlet 
into which water is fed from a sprinkler.

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

- Nitrobenezene inlet.

- Connection to a 4" vent main leading to 
outside atmosphere.

Nickel is the only known material which will with-
stand the conditions in the reactor, and even this fails 
at the pots unless the pot is annealed after manufacture, 
Stainless Steel (W.N.), 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.

- 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 1/2" cast iron pots with conical bottom and sludge 
rin off.

- Treatment of Acenbenzenes. There is a battery of 12 
stirred, jacketed, cast iron pots adjacent to the cells, 
capacity guessed to be about 10 - 15 Ml, of which 
8 are normally used -

2 for Separation of Acenbenzenes and 1/3 HCl.
1 for Washing Acenbenzenes.
1 for treating the acid liquor from the 
reactor.

2.
Liquor transfer is by compressed air.

(5) tanks below the cells (about 15 l 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 60°C. The batch continues for 140,000 ampere hours, i.e., for 7 hours at 20,000 amps.

Analysis

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 0.2% 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  
<p>| | |</p>
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Azobenzene</td>
<td>75/90%</td>
</tr>
<tr>
<td>Hydrazobenzene</td>
<td>15/5%</td>
</tr>
<tr>
<td>Aniline</td>
<td>2/0.6%</td>
</tr>
<tr>
<td>Nitrobenzene</td>
<td>0/0.8%</td>
</tr>
</tbody>
</table>

50% 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 50% 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 phenol.

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

Hydrazobenzene has been made at 100°C, using a steam-jacketed pot, but muchiline was produced. It was not developed beyond the experimental stage.

Performance:

The mercury cell operates less efficiently about 55% because of high temperature and high amalgam concentration. The azobenzene is operated marginally on the cell process, the C16 and NaOH go to the cell process, which causes the following back to azobenzene:

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

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

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

\[
\text{Output: Actual: 1043.}  \\
\text{Actual: 500.398 tons as 100% azobenzene 472,305 = 96%}  \\
\text{Kc/to = 96% ineff.}
\]

Usage:

<table>
<thead>
<tr>
<th>Product</th>
<th>kg/kg</th>
<th>% Kc/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrobenzene</td>
<td>650.3</td>
<td>151</td>
</tr>
<tr>
<td>Chlorine Gas</td>
<td>714.4</td>
<td>246</td>
</tr>
<tr>
<td>NaOH liquor</td>
<td>118.7</td>
<td>295</td>
</tr>
<tr>
<td>Kc</td>
<td>227.2</td>
<td>4.8</td>
</tr>
<tr>
<td>Water</td>
<td>339.5</td>
<td>7.5</td>
</tr>
<tr>
<td>Steam</td>
<td>505.5</td>
<td>0.8</td>
</tr>
</tbody>
</table>

\[
\text{Kc} = \frac{\text{Kc}}{\text{Kc}}\]
Units:
Power A.C. kWh to 39161 74.1
" D.C. " 13878 286.3
Comp. Air m³ 28673 56.1
Process Labour 18300 hrs. 26.1

Drawings:
The following Drawings have been lodged with:
Board of Trade, German Division (Document Unit),
IOWN HOUSE, BERKELEY SQUARE, W.1. TEL: GROSVENOR

Drawing No.
00825. General layout.
117206. Reactor.
94191. Reactor Valve.
91704. Sludge Separator.

All applications for permission to inspect these
drawings should quote the following Reference Number:
KIOS/2003/1091/1316.
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.