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MANUFACTURE OF ACETALDEHYDE

I.G. FARBENINDUSTRIE

SCHKOPAU

Hasche & Brandy

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

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REPORT ON MANUFACTURE OF ACETALDEHYDE AT I.G. FARBENINDUSTRIE, SCHKOPAU

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On behalf of the

U.S. Technical Industrial Intelligence Committee

CIOS Target No. 22/82
Miscellaneous Chemicals

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COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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MANUFACTURE OF ACETALDEHYDE AT I.G. FARBENINDUSTRIE, SCHKOPAU

1. INTRODUCTION

Acetaldehyde was one of the most important chemicals in the wartime economy of Germany. The bulk of the production was from the I.G. Farbenindustrie plants at Schkopau, Huels and Knapsack which by the middle of 1942 had grown to 325,000 tons per year, distributed as follows:

	Ton/year
Schkopau	142,800
Huels	110,300
Knapsack	72,000

It is interesting to note, in this connection, that Germany built its industry for the manufacture of the major olefatic organic chemicals around calcium carbide as starting material. Butadiene for Buna, Solvents - such as butanol, acetone, ethanolacetic acid, anhydride and a large number of the more important vinyl compounds were made from Ca C_2 as basic raw material. Shortage in Germany of material petroleum resources and of molasses, and the obvious reasons for the mammoth calcium carbide industry. By the middle of May 1942 the production of calcium carbide amounted to a rate of 110,000 tons per month, and it was planned to expand it by the end of 1944 to 210,000 tons per month.

Of the three plants mentioned above as chief producers of acetaldehyde, Schkopau and Knapsack also had large calcium carbide plants with a yearly capacity of 352,000 and 294,000 tons respectively - or about half the German production. Approximately 70% of the combined calcium carbide production of Schkopau and Knapsack went to the three large I.G. acetaldehyde producers.

2. TYPE OF PROCESS EMPLOYED

The process for the hydration of acetylene to acetaldehyde was the conventional method using a liquid phase mecuric sulfate catalyst suspended in an aqueous solution of sulfuric acid and iron sulfate. The plants at Schkopau, Huels and Knapsack followed substantially the same procedure, with minor modifications on the regeneration of the catalyst and recovery of the acetaldehyde.

In the judgment of the members of our inspection team the operation at Schkopau was the most highly developed, and it will be described in detail.

3. DESCRIPTION OF THE OPERATION

A. Hydration of acetylene is carried out continuously with a liquid catalyst giving a 60% conversion per pass and a yield of acetaldehyde of 96.5%. About 0.5% of croton-aldehyde, 0.5-0.7% of acetone and a small amount of diacetyl, and about 2.0% of acetic acid are also formed. The latter is lost in the waste water to the sewer.

The operation of the process will be described by reference to the accompanying diagram. There are 12 hydration towers of rubber-lined steel construction 1.2 meters in diameter and 10 meters high to the expanded section. The latter is about 2.3 meters in diameter and 4 meters in height.

The catalyst solution contains 60 grams of H_2SO_4 per liter and 90 grams of Fe in the form of $FeSO_4$ and $Fe_2(SO_4)_3$. The ferric concentration of the solution is kept to about 20% of the total iron by nitric acid regeneration which will be described later. At the bottom of the tower a pool of mercury is maintained and the amount of mercury in the solution is about 0.5 grams per liter.

In operation the catalyst temperature is 97-98° C at the bottom and 94-95° C at the top. The pressure at the bottom is about 200 mm. above atmospheric pressure.

Acetylene gas having a purity of 97-98% is introduced in the bottom of the tower at a rate of 1000 cubic meters per hour plus recycle which averages about 75% acetylene. Along with the acetylene about 1.5 tons per hour of steam is introduced. The mixture of steam, acetaldehyde and unconverted acetylene passes from the tower to a stainless V4A cooler where the temperature is reduced to 60° C. Condensed steam and mercury is returned to the column. The mixture then passed to a stainless cooler where it drops to 25° C. The mercury returns to the tower and the condensate which contains 9% of acetaldehyde mixes with the absorbing water leaving the scrubbing tower. The acelytene gas mixture containing acetaldehyde vapor passes up through the scrubber counter-current to water for recovery. A solution with a 9% concentration of acetaldehyde passes to a stripping column where acetaldehyde is recovered using live steam for heating.

The scrubbing and recovery system consists of three sets of columns, each with an absorber and stripper. The columns are lead lined 1.8 meters in diameter

x 10 meters in length. They are packed with Raschig rings.

The stripping column operates under sufficient pressure to allow condensation of the acetaldehyde solution which is 80% concentration in water and also contains dissolved acelytene.

The primary distillation columns have a diameter of 1.6 meters and have 22 plates. Acelytene is removed from the top and passes back to the conversion system, while acetaldehyde is taken off at the bottom.

A final distillation column recovers acetaldehyde of 99.5% purity.

B. Regeneration of the Catalyst. A portion of the catalyst solution from the hydration tower is continuously withdrawn for regeneration. Approximately 2 cubic meters per hour from each reactor passes first to a separator which is lead-lined. Mercury sludge settles to the bottom and it overflows, mixing with a stream of 20% nitric acid and passes into the oxidizer of 1 cubic meter capacity and maintained at 98° C. From there it overflows into a rubber-lined steel column packed with Raschig rings. The nixtric oxide is stripped out with air and absorbed in a second tower.

C. Consumption of Chemicals. Dr. Walff, Director of the Schkopau plant, submitted the following figures on the consumption of chemicals in the process:

	Per Ton of Acetaldehyde
Mercury	0.05 kilograms
$Fe\ SO_4SH_2O$	5 "
Nitric Acid	2 "

4. COMMENTS

Although the mercury method for the hydration of acelytene was the only one used commercially, the Ludwigshafen I.G. plant operated a pilot plant based on Reppes method of producing the alkyl vinyl ethers. An investigation of this operation will be covered in a separate report.

R. L. Hasche

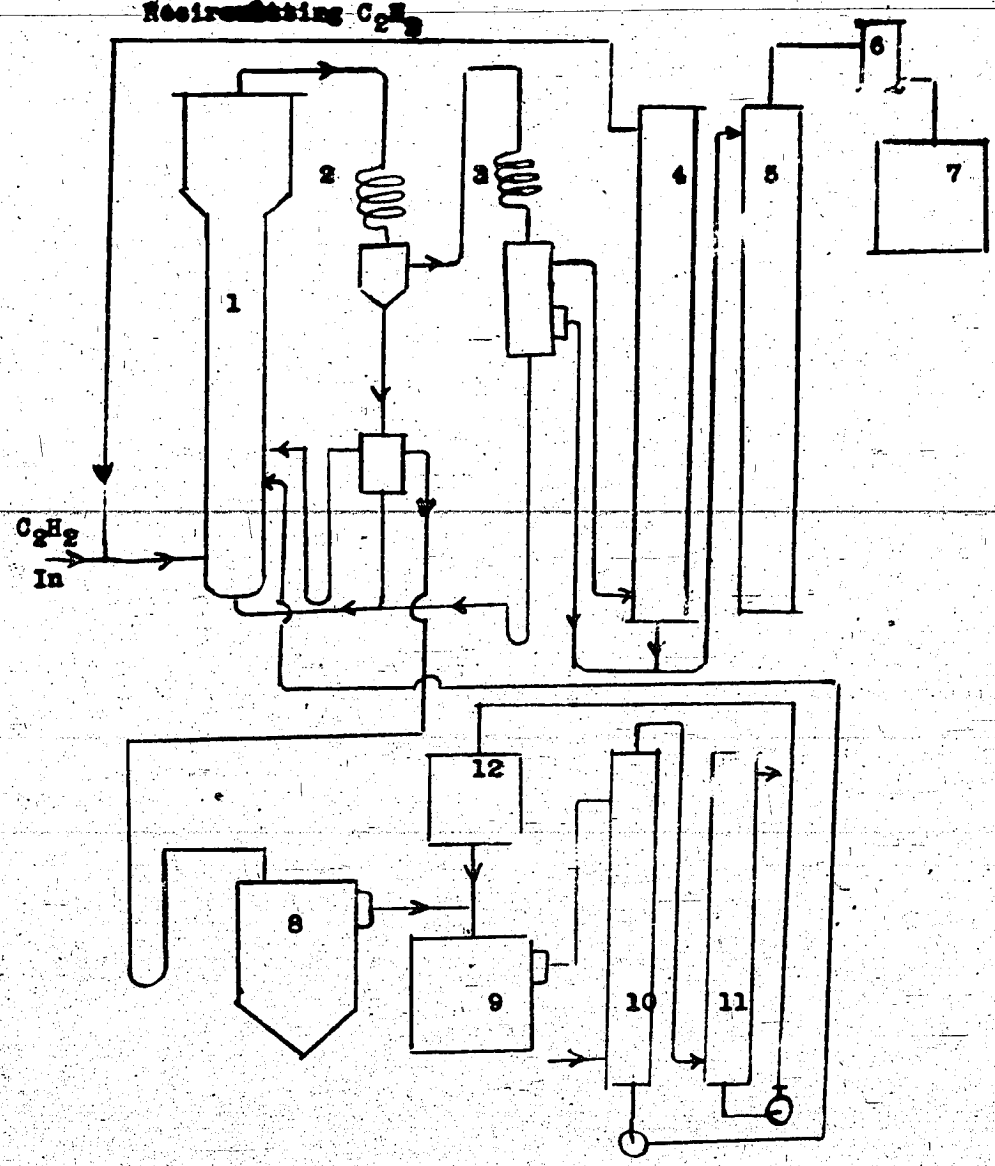

DIAGRAM OF PROCESS FOR CONVERSION OF ACETYLENE TO ACETALDEHYDE AT SCHKOPAU

Legend

1 Hydration Tower
2 Primary Cooler
3 Secondary Cooler
4 Aldehyde Scrubber
5 Aldehyde Scripper
6 Aldehyde Condenser
7 Crude Storage
8 Settling Tank
9 Oxidizer
10 Aerator
11 Nitric Acid Scrubber
12 Nitric Acid Storage

R.L.H.