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THE OXO PLANE RULERCHEMIE OBERTAINENTHOLDEN

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REPORT ON INSPECTION OF THE OXO PLANT AT RUHRCHEMIE OBERHAUSEN-HOLDEN

Reported by:

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On behalf of the U.S. Technical Industrial Intelligence Committee

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1. INTRODUCTION

The existence of the Oxo unit, which is adjacent to the Ruhrchemie plant at Oberhausen-Holden, was discovered after considerable questioning of Dr. Spanier and Dr. Schreiber of Ruhrchemie. In previous conversations at the Ludwigshafen and Wolfen I.G. plants, we had learned that experimental work had been done by Ruhrchemie on the process for the production of aldehydes and acids by the interaction of olefines and water gas.

2. DEVELOPMENT OF THE PROCESS

The experimental work on the process was done by Ruhrchemie previous to 1938 by Dr. Landgraf, who is now in
charge of the commercial plant. A separate company was
formed called the Oxo Gesellschaft, and the stock is jointly
held by Ruhrchemie, the I.G. and Henkel. The combination
indicates the importance which was apparently attached to
the process by the participating companies.

The process consists of producing olefines of the C11-C17 fraction, obtained by the Fischer-Tropsch method, with water gas in the liquid phase at approximately 150 atmospheres pressure. The reaction results in a mixture of straight chain aliphatic aldehydes and ketones, the former predominating. The aldehydes are then reduced in a second step to form the corresponding alcohols. The products form a very valuable class of detergents for synthetic fibers, being non-acid and non-alkaline. The process is equally well adapted to the production of lower aldehydes, particularly propionaldehyde, by the interaction of ethylene and water gas.

Construction of the plant was begun in 1938 and was scheduled to be in operation in 1942. However, it was given a lower priority due to war conditions and was not completed. It has been damaged but slightly and could be put in operation in an estimated period of three months to produce 10,000 metric tons per year of products with an extension to 25,000 tons per year with the addition of a small amount of auxiliary equipment.

3. DESCRIPTION OF PROCESS

It was originally intended that the Ruhrchemie would supply the olefines to the Oxo unit. However, the Ruhrchemie plant has been badly damaged and it is now proposed to produce the olefines on the site of the Oxo plant. Olefines are produced by a Dubbs type of cracking furnace from a C20-C40 Fischer-Tropsch fraction.

Yields of the olefines of approximately 50% have been obtained in the cracking unit operating at a temperature of 400° to 450° C. The reaction is carried out batch-wise in a converter 12 meters in length with an internal diameter of 400 mm. The converters are vertical and provided at the bottom with a shaft and bearings so that they can be tipped to a horizontal position at the ground level for repair and cleaning. There are now four of these units, and provision for an additional six. The attached sketch shows the general arrangement of a single 0x0 unit.

The catalyst unit is charged 3/4 full with a suspension of the catalyst in the liquid olefines of the C11 C17 fraction. In case the process is operated on gaseous olefines, the catalyst is suspended in Diesel oil. The catalyst, which is a standard Fischer-Tropsch material, is composed of 90% cobalt, 7% thoria and 5% magnesia, and is deposited as carbonates on hieselguhr. The content is about 50%. A suspension of catalyst and Diesel oil is made up in agitated vessels and then pumped to a pressure vessel, one being provided for each converted and thence charged into the converter. Each converter is provided with means for heating and cooling, which consists of 30 cooling tubes extending 3/4 of the distance from the bottom into the chamber. The tubes are 30 mm. I.D. x 38 mm. 0.D.

Mater gas is compressed to 300 atmospheres in fourstage compressors and reduced to 150 atmospheres, at which
pressure it is introduced into the bottom of the converter.
The operating temperatures vary from 150° to 180° C. The
relative amounts of aldehyde and ketone can be varied by
controlling the temperature - the higher temperature favoring the aldehyde but reducing the yield due to polymerization. Yields of alcohols obtained after the second stage
of reduction amount to approximately 1 lb. of olefine
charged.

The capacity of the converter in total liquid products is 700 to 720 liters per batch, which corresponds to 500 kilograms of product. The actual time for the reaction is 20 to 30 minutes, and the total time, including charging and discharging the converter is approximately 1 hour.

The water gas, is recirculated through the converter and fresh material added. The compositions of the inlet and residue gases are as follows:

Inlet Gas	1
Carbon dioxide	6.0%
Carbon monoxide	38-39%
Hydrogen	48-49%
Inerts	Balance

Residue	
CO2 and Inerts	20-30%
Carbon monoxide	15-20%
Hydrogen	40-50%

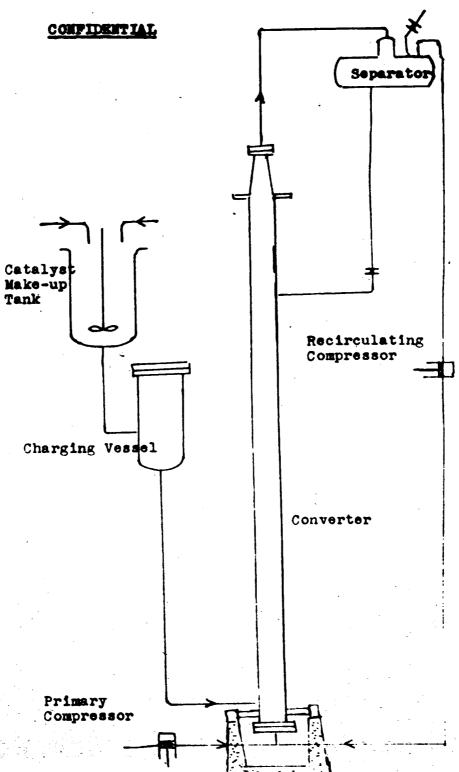
The quantity of recirculation gas is 200 cubic meters per hour and of fresh makeup gas 40-50 cubic meters per hour.

The catalyst can be reused 50 to 100 times depending on the type of olefines employed. When the operation is carried out with ethylene, temperatures as low as 85°C can be used and under optimum conditions the product is composed of 70% of propunaldehyde and 30% of diethyl ketones by weight.

Separation of the oxygenated compounds from the unreacted olefines is accomplished by fractional distillation. Extraction methods were investigated in the early development stages but abandoned as impractical.

Reduction of the higher aldehydes to alcohols for use as detergent was a very simple process in which a Raney type of nickel catalyst was used.

R. L. Hasche



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