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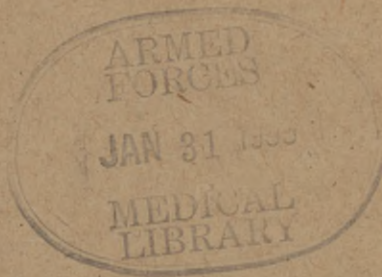
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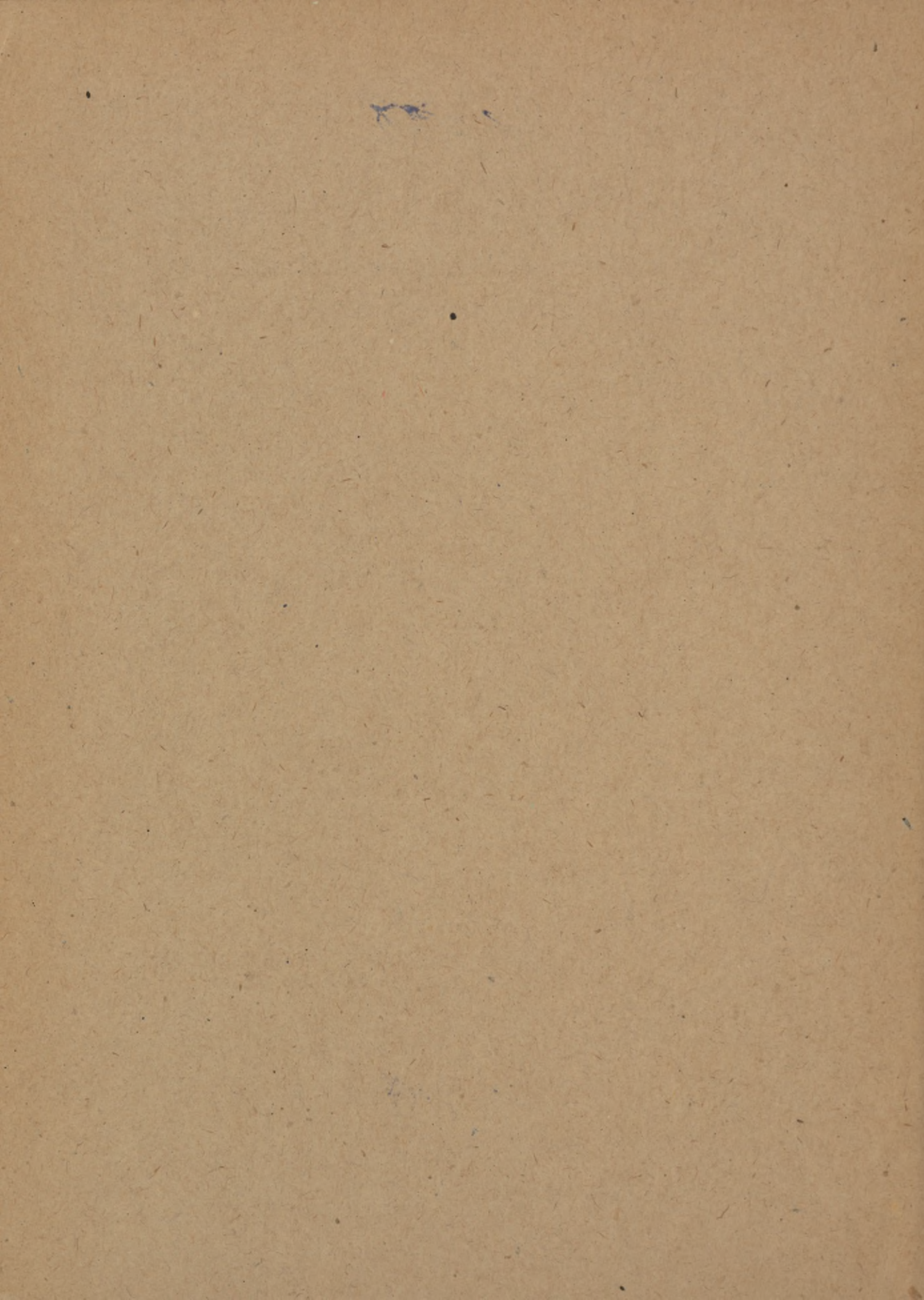
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PRODUCTION OF SYNTHETIC FATTY ACIDS
AND EDIBLE FATS
DEUTSCHE FETTSAUREWERKE, WITTEN



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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE



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REPORT ON
PRODUCTION OF SYNTHETIC FATTY ACIDS
AND EDIBLE FATS

DEUTSCHE FETTSÄUREWERKE AND MARKISCHE
SEIFENFABRIK
WITTEN (RUHR) GERMANY.

Reported By

E.L. BALDESCHWIELER, U.S.

on behalf of the

U.S. Technical Industrial Intelligence Committee

CIOS Target No. 22/459
Miscellaneous Chemicals

28 June 1945

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

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PRODUCTION OF SYNTHETIC
FATTY ACIDS AND EDIBLE FATS.

Location: The plants visited on 24 April 1945 are owned by the Deutsche Fettsäurewerke and its subsidiary the Markische Seifenfabrik. The two plants are located at Witten, Ruhr, and are on opposite sides of the road.

Personnel Interviewed: The plant is owned by Mr. Imhauser who was present with his son, Dr. A. Imhauser, chief chemist, also the chief analyst, Dr. Döpke. All parties seemed to be not only very willing, but rather anxious to furnish information. Dr. Imhauser explained that he was half Jewish and that both he and his father were in constant threat of death by the Nazis. Were it not for the fact that the plant was an important producer of edible fats, both men would have been sent to concentration camps early in the year. A Nazi director had been appointed over their heads and was in complete charge so that the Imhausers were only employees in their own plants.

Plant Personnel: The oxidation plant occupied 180 people, and the soap plant 400. These included 90 foreign laborers of whom 60 were Russian, 20 French and 10 Dutch. Much of the personnel was apparently still working and this included some of the Russians. The Nazi director had disappeared.

Production: The raw material is the wax or so-called "gatsch" from various Fischer-Tropsch plants, primarily from the Chemische Werke Essener Steinkohle A.G., at Kamen-Dortmund. The plant was designed for and produced 40,000 tons per year of fatty acids by oxidation of the gatsch. For the last year, the capacity was cut down to 20,000 tons, mostly due to lack of raw material caused by bombing of the Fischer-Tropsch units. A certain proportion of these fatty acids were used in the manufacture of edible fats, a total of 150 tons per month being produced. A sample of the fat has been obtained. Appearance and taste are very similar to oleomargarine.

Two other similar plants have been built by the I.G. Farbenindustrie in Germany, one at Heidebreck, Upper Silesia, and the other at Oppau. Each had a designed capacity of 20,000 tons fatty acids per year. A fourth is under construction at Magdeburg for the Hubbe and Fahrenholz Fabrik.

This plant is designed to operate at 25 atm. pressure but has never been operated. The elder Imhauser claims that this process is six times more expensive than that used in his plant.

Bomb Damage: Outside of broken windows in the office building and some damage to the shop, analytical laboratory and research pilot plant, the oxidation works appeared little damaged. The main building of the soap plant was, however, half destroyed.

Process: Two flow sheets are attached: No. 1 for the oxidation plant or manufacture of the fatty acids, and No. 2 for the manufacture of the edible fats. As indicated in flow sheet No. 1, the fatty acids to be used in the manufacture of edible fats are made by the same process as the other fatty acids, up to the pipe still step. At this stage, the distillation is carried out at 320°C. for ordinary fatty acids and at 380°C. for the edible acids. The properties of the acids are given in Table I.

TABLE I.

<u>Light Fraction.</u> (Vorlauf)	<u>Main Fraction.</u> (Hauptlauf)	<u>Heavy Fraction.</u> (Nachlauf)	<u>Residue.</u> (Rückstand)
Temp. "C" (up to: 120)	120-270	270-315	-
Yields % 15-20	55-60	7-12	7-13
Mol. Wt. C8-C10	C10-C20	C21-C28	-
Neut. No. 450-460	245	155-160	80-90
Sap. No. 450-460	255	175-180	110-120
Estn. No. 0-1	10	20	30
Ester No. for	1.5	10	-
Edible Fats -			
U.S.M. % 0	1.5	10.0	-
U.S.M. % for -			
Edible Fats -	Ca. 1	-	-

Total yields of fatty acids 80% of the charge to the still.

Neutralization, Saponification and Ester Numbers are expressed in milligram of Potassium hydroxide per gram of sample.

A carbon balance based on the analysis of the original sample is very difficult to obtain due to the fact that CO_2 escapes during the oxidation together with the volatile fatty acids, about 10-15% of the carbon being thus lost. There is also an additional loss of 1-2% in raw fatty acids during the distillation.

Additional explanations:

(1) The oxidation of the gatsch is carried out in aluminum vessels provided with an alloy steel head. There are 12 vessels, each of 20 tons capacity, and 24 vessels of 10 tons capacity. Eight of the latter are made entirely of alloy steel (chrome-nickel), but these are not as satisfactory as the aluminum vessels since this metal catalyzes the oxidation reaction. Alloy steel heads are necessary, however, since aluminum is corroded by the low molecular weight volatile fatty acids. The 20-ton vessels are 9 meters high and 2.7 meters interior diameter. Each vessel is provided with an air distributor, a bottom dished coil for heating and a middle coil for cooling, the oxidation reaction being exothermic. The air distributor and pipes are made of chrome-nickel steel. The air distributor has a diameter of 1.2 m., the top being provided with 1 mm. holes and the bottom with 3 or 4 large holes. Such a vessel is shown diagrammatically in the attached drawing. The oxidation is only carried on until about 30% of the gatsch has been oxidized, since experience has shown that further oxidation results in the formation of undesirable products such as ketones.

(2) The low molecular weight or volatile acids are condensed, absorbed in water and separated into two layers. The lower layer consists of an aqueous solution of formic, acetic and some propionic acids, the upper layer being a mixture of C_3 - C_{10} acids. The formic acid is used for the treatment of fodder silos and the acetic acid for the esterification of cellulose. The water-insoluble acids are sold for the manufacture of plasticizers, perfumes etc.

(3) The wash towers are 11 m. high and are provided with Jena glass lines to prevent corrosion by the water-soluble acids.

The saponification is carried out by adding the calculated amount of 38% NaOH . The reaction products separate into two layers. The upper layer shown in Flow Sheet No. 1 as USM(A) amounting to 30% consists of unreacted gatsch and is returned to the oxidation vessel.

The lower layer is an emulsion containing 30% soap and 30% gatsch which is fed into an autoclave and heated for one hour at 170-180°C. and 25 atmospheres pressure.

This treatment causes a further separation of gatsch, USM(3) which is returned to the oxidation vessel and a lower layer of soap solution containing 45% soap and 15% gatsch. The soap solution is then fed to a pipe still heated by gas at 320°C. for soap acids and 380°C. for edible acids when the water and remaining gatsch are driven off. The gatsch, USM(C) is recycled. The anhydrous soap is then run directly into water to form a 40% solution and the fatty acids liberated by addition of sulfuric acid. This reaction is carried out at 90-95°C.

(4) The vacuum distillation of the liberated fatty acids is carried out in four stages corresponding to the four cuts. The first stage (vorlauf or light fraction) is provided with a fractionating column. The next two stages are in parallel (hauptlauf or main fraction), and the last stage is for the heavy fraction (nachlauf), the residue (ruckstand) being collected from the kettle of the last stage.

The distillation is carried out in the presence of 5% of steam; the total steam consumption, including the vacuum steam injectors, is approximately equal to the weight of acids treated.

(5) The raw material for edible fat manufacture is a purified middle fraction from the vacuum still consisting of a mixture of C₈-C₂₀ acids. These acids are esterified with glycerine under the conditions indicated in the flow sheet to form a triglyceride. The remainder of the process consists of the purification of this glyceride which is finally sold as food after the addition of vitamins. This last operation is, however, carried out elsewhere.

This plant was large, airy and the walls covered with white tile for easy washing. With the exception of broken windows, it was virtually undamaged.

Soap Plant: As previously stated, this plant was badly damaged. The mixing kettles were of 100-ton capacity and it was evident that soap manufactures had been carried out on a large scale. The elder Imhauser admitted that even

with only 30% soap in the final product, they had not succeeded in removing the characteristic Fischer-Tropsch odor when washing hands. This odor is apparently due to a slight hydrolysis of the soap, and the odor is reminiscent of butyric acid. A sample of the pure soap has been obtained. (Toilet soap containing 30% of syn.acids).

Research Work: A fair amount of research work was carried out in a pilot plant provided with a 24 plate vacuum column operated at 1 mm. pressure. Pure alcohol and fatty acids from C₄ and C₁₁ have been separated and samples of octyl, nonyl, decyl, hexyl and undecyl alcohols, also caprylic, valerianic, nonylic and oenanthylic acids were exhibited. It was pointed out that the Fischer-Tropsch gatsch oxidation process provides the only industrial source of these products.

Documents: No documents were found, outside of a few analytical records which substantiated the statements outlined above. Consequently, no documents have been collected. The personnel interviewed claimed that all drawings and records had been burned.

Priority of Assessment: Since full statements were made by the persons interviewed and, moreover, since many of these statements check with those made by Dr. Keunecke (CIOS Report 12 on I.G. Production of Synthetic Fatty Acids), it is recommended that no further investigation of this target be carried out.

Schedule of Samples Obtained:

1. Synthetic fat as prepared for sale.
2. Toilet soap containing 30% synthetic acids.
3. Crude fatty acids before vac. dist.
4. Main products of distilled acids prepared
for soap manufacture.

E. L. BALDESCHWIELER.

Deutsche Fettsaure Werke
Flow Sheet No.1 Fatty Acid
Manufacture.

Fischer-Tropsch Gatsch (320°-450°C)

↓
 Centrifuge (to remove catalyst dust)

↓
 (1) Oxidation Vessel (Charge 8-20 tons plus
 0.2% $KMnO_4$ in 15% aqueous
 solution. 110°C. 50 m³ air/
 hr/ton wax. Time 20-24 hrs.
 1 part fresh gatsch to 1.8
 USM).

↓

(Volatile
 (H_2O , CO_2 plus)
 (2) (light oil plus)
 (low mol. wt.)
 (acids.)

↓
 (3) Count.Cur.Hot Water 2-3% water soluble
 Wash.

↓
 + Na_2CO_3
 (Neutralization)

(USM
 (Unsaponifiable material
 (1)(2)(3)(4). refer to
 additional explanations
 in the text.

Legend.

↓
 Saponification

Calc.Amt.38% $NaOH$.

↓
 USM (A)

Soap Solution to Autoclave
 1 hr.25 atm. 170-180°C.
 Continuous

↓
 USM (B)

Soap Solution to continuous
 Pipe Still.atm.press.(Gas heated)
 (320°C. soap acids)
 (380°C. edible acids)

↓
 Water plus
 USM (C)
 (Volatile)

Anhydrous Soap
 Run direct into water
 to give 40% solution.

↓
 H_2SO_4

Water Wash (For scraps, Neut.No.350-
 370; Ester No. 10; Sap.
 No.360-380)

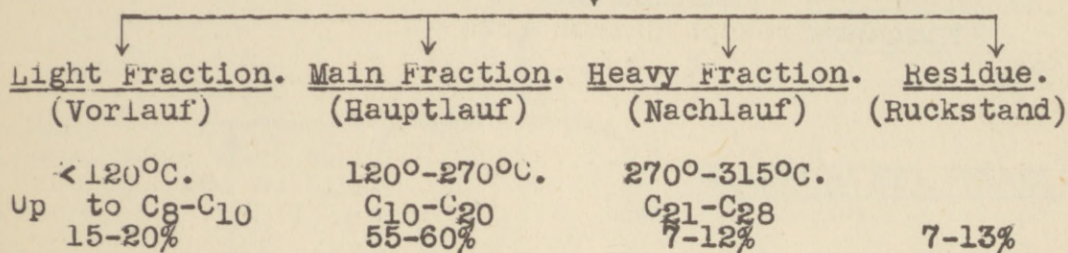
↓
 Crude acids

↓
 Vacuum Fractionation
 (4) 3-4 mm. 5% steam

(For edible fats, Neut.No.
 380; Sap.No.382; Ester
 No. 2).

(4) Vacuum Fractionation

3-4 mm. 5% steam.



Flow Sheet No. 2

Production of Edible Fats.

Middle Fraction C₈-C₂₀

Glycerine

(3-4% excess acid to form a triglyceride.
(2 mm. pressure, 0.2% metallic tin at 200°C.

Wash with acid to remove catalyst.

Wash with alkali to remove acid.

Treat with active charcoal & bleaching earth + filter pressing.

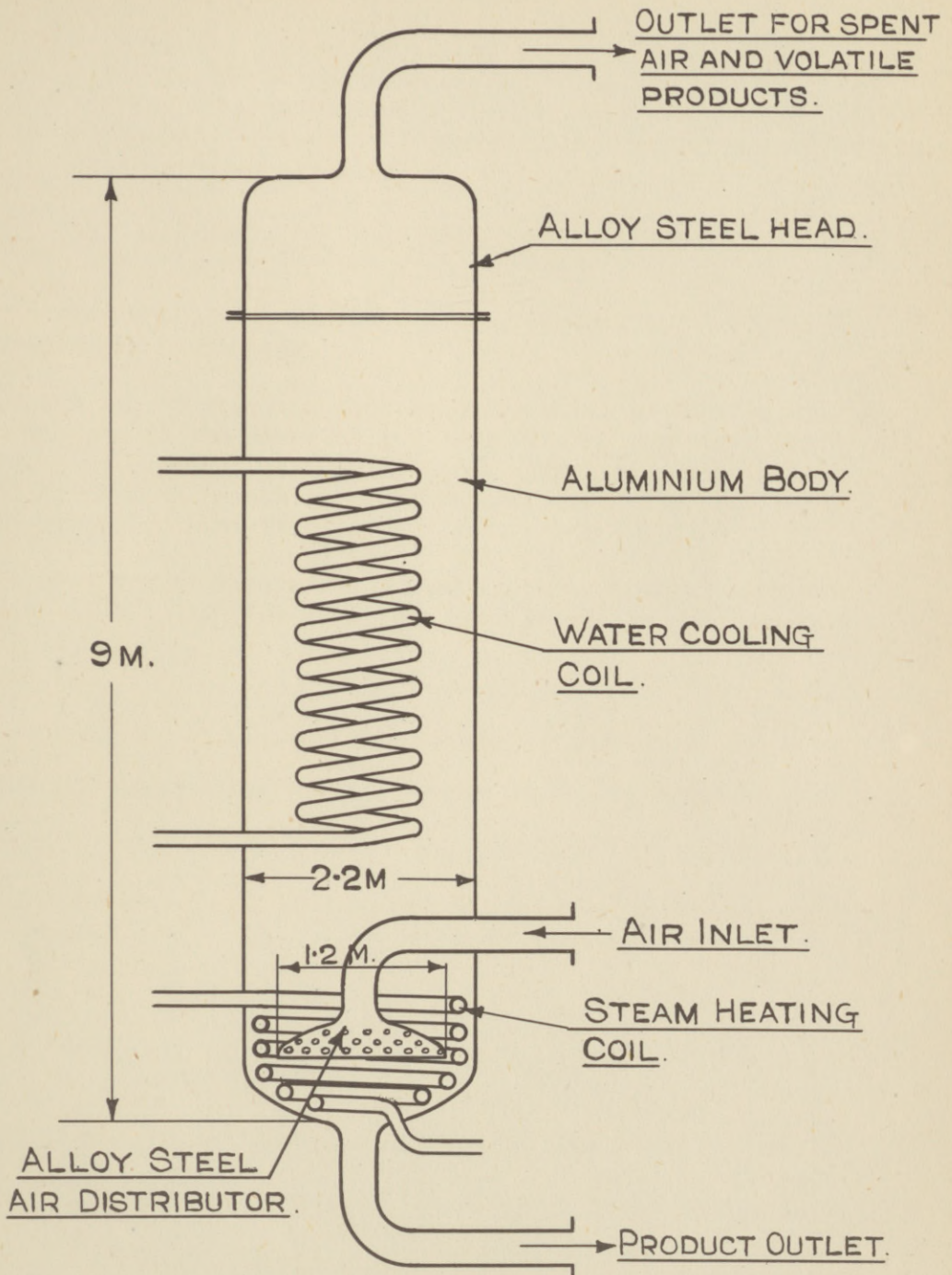
Steamed, 200°C. 2 mm. pressure.

Emulsification with 20% water.

Chilling.

Milling.

Sold for vitamin addition.



20 TON KETTLE FOR WAX OXIDATION.
DEUTSCHE FETTSÄURE WERKE, WITTEN.



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