

RESTRICTED

APPENDIX B.

TRANSLATIONS OF
DOCUMENTS RELATING TO
RESEARCH ACTIVITIES

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RESTRICTED

The following is a rough translation of a manuscript by Dr. Grimme (said to be Director of the laboratory at Moers) dated January 15, 1942.

PROGRAM OF WORK FOR THE RESEARCH LABORATORIES

Research Laboratory No. 1

- 1) Dehydro-genation of hydrocarbons, in particular propane and butane to propylene and butylene, also the catalysts (chromoxide - aluminium oxide) developed therefor.
- 2) Conversion of alcohols to ketones.
- 3) Work with resins - preliminary.
 - (a) Manufacture of methylester of ethylacrylic acid
 - (b) Betamethyl gamma keto butanol
 - (c) Manufacture and purification of methylisopropenylketone and plexigum-like polymerisation products therefrom.
- 4) Development of the ketone resin, "Emekal", from methylethylketone and formaldehyde, also the working out of a process for a production of about 4 tons per month.
- 5) Manufacture of secondary butylstearate as plasticizer.
- 6) Manufacture of aluminium alcoholates. Experiments respecting different catalysts for the reaction of aluminium with alcohols. Application of butyl and propyl aluminium alcoholates for
 - (a) Pyrotechnical purposes
 - (b) Igniters for carbons
 - (c) The treatment of textile fibres
 - (d) For Lacquer technique and other purposes, especially the manufacture of emulsions.
 - (e) Medicinal purposes (Professor Engelhardt, Tubingen and Prof. Frey, Dusseldorf).
- 7) Development of the carbonic acid addition products of aluminium alcoholates, in particular of the butyl compounds.
- 8) Development of active aluminium hydroxide and oxide from aluminium alcoholates and their technical and medical application, also the development of an experimental unit for the production of above compounds.
- 9) Manufacture of a new gelation medium (Rh₆) out of butyl and our C₆ to C₉ fatty acids, also the Wittener-method-fatty acids. Application of the aluminium salts of these fatty acids for the purpose of "Wa Pruf 5 lc".

- 12) Developed contracts of OKH, "Wa Prüf 5 lc":
- (a) Development of smoke tubes (Parchlorit 7)
 - (b) Development of water stable friction devices (?) or grinding compounds- (?).
 - (c) Development of an igniter cartridge for oil (Parchlorit 7).
 - (d) Development of storage stable retarded devices (Parchlorit 7).
 - (e) Development of a membrane as a safety valve for flame throwing devices.
 - (f) Development of incendiary oil for filling of flame throwing devices.
 - (g) Development of burning tubes (Parchlorit 7).
 - (h) Development of a chemical time igniter (translator's concept is that this is a delayed fuse of some sort). Refer to our files W 109 820 XI/72 at "Chemischer Zeitzunder".

Research Laboratory No. 2

- 1) Manufacture of esters of acetic, propionic, and butyric acids with propyl, butyl and amyl alcohols.
- 2) Separation of olefines and paraffins through treatment with selective solvents, in particular with SO₂.
- 3) Manufacture of esters of phthallic acid, and other dicarbonic acids (plasticisers, thickeners and glyptals.
- 4) Reaction between dicarbonic acids, in particular phthalic acid and alcoholate.
- 5) Investigation of the oxidation products, Parestol and Pantoxyl, in a comprehensive sense. For example, resins of the type of alkya resins, soaps, washing media, salve basis for pharmacy and cosmetic purposes from fatty acids. Application of the oxy-acids as linseed oil substitutes, hydrogenation of the oxidation products to alcohols and esters.
- 6) Introduction of the sulphonic acid groups and nitro groups in the paraffin hydrocarbons.
- 7) Oxidation of the lower paraffins to dicarboxylic acids by means of nitric acid.
- 8) Dicarboxylic acids from Parestol or from paraffin or through the intermediary of the phenols from coke plants.

Research Laboratory No. 3

- 1) Conclusion of the theoretical work on secondary octyl acetate (trichloroacetic acid and benzene sulphonic acid as catalyst, H_2S formation).
- 2) Development of the production of secondary octyl acetate into semi-technical operation; corrosion problem, filtration.
- 3) Production of esters of secondary alcohols of high and low molecular weight.
- 4) Tests of the additivity of fatty acids to different olefine double bonds (iso-olefines, cyclic compounds).
- 5) Addition of H_2CO_3 and HCN on olefines.
- 6) Production in the wet way of pure olefines by means of the esters of secondary monovalent and polyvalent alcohols.
- 7) Experiments on the composition of kogasins. Fractionation, determination of branch chains and the position of the double bond.
- 8) Production of fatty acid esters of secondary alcohols from the pure substances for determination of their properties.
- 9) Explanation of the reaction mechanism of resin formation from ketones, in particular methyl ethyl ketone and formaldehyde; principal reaction, subsidiary reactions influence of single reactions on the quality of the resin.
- 10) Further experiments regarding methylol compounds, in particular mono- and di- methylol acid-amides.
- 11) Production of tasteless triglycerol esters from synthetic fatty acids of the Fischer Tropsch synthesis and explanation of the carrier of the scratchy after taste. Esters of "Wittener" fatty acids.

Research Laboratory No. 4

- 1) Purification of polymer benzin with phenolates.
- 2) Experiments in respect to the suitability of isopropyl alcohol as protection against freezing.

- 3) Production of ethers from alcohols and from olefines.
- 4) Production of the di-alkyl sulfates for alkylation.
- 5) Production of tertiary butyl alcohol.
- 6) Polymerisation of residue gas from the alcohol plant; production of products with high molecular weight.
- 7) Production of high molecular weight compounds from polymerisation of isobutylene.
- 8) Production of isobutylene by isomerisation of normal butylene.
- 9) Experiments on the reaction between olefines and aldehydes, especially formalin; production of glycols.
- 10) Chlorhydrins, olefine oxides and glycols from propylene and higher olefines -
 - (a) production of hydraulic liquids from glycols.
 - (b) polymerisation of olefine oxides.
 - (c) conversion of olefine oxides with paraffins to alcohols
- 11) Removal of CO₂ and H₂S from gases with aqueous ammonia.
- 12) Production of fuel for powdered coal motors from carbon monoxide.

(Signed) GRIMME

Treibstoffwerk, 15 January, 1942.

Treibstoffwerk
7 January 1942

Projects carried out from 1940 to the present date and the projects under way at present.

1. Dehydrogenation of hydrocarbons, particularly propane and butane to propylene and butylene over Chromia-Alumina Catalysts developed for this purpose. About 60 catalysts have been developed, of which the following in particular appear to be highly active and technically useful: -

K-33b: A mixed catalyst containing 10% Cr₂O₃ on synthetic Dawsonite.

K-57: A mixed catalyst containing 18% Cr₂O₃ and especially simple to prepare technically, since it is obtained directly by coprecipitation of aluminum chromate solution with ammonium bicarbonate. See (patent) applications St 59796 and St 61012 IVb/129g, "Process for Preparation of Active Aluminum Oxide-and Aluminum Oxide - Chromium Oxide Mixed Catalysts".

K-58/K-61: A mixed catalyst of high activity and exceptional mechanical strength obtained by decomposition of aluminum secondary butylate with water followed by direct adsorption of chromic acid - see application St. 59655 IVb/12m "Preparation and Use of Purest Hydroxides and Oxides of Aluminum and Magnesium", as well as addition application St 59894 "Process for Preparation of Activated Aluminum Oxide Catalysts".

2. Conversion of Ketones to Alcohols

A process has been developed for the thermal dehydrogenation of secondary alcohols, especially secondary butyl alcohol, to ketones, with the aid of a zinc catalyst (K-67) having a zinc content of 7-9%. The process has been put into use technically for the production of 30 tonnes per month of methylethylketone. In addition a process has been discovered for refining methylethylketone, which tends to turn yellow during storage, by distillation in the presence of calcium hydroxide.

3. Preliminary Work on Synthetic Resins.

Preparation of methyl ester of ethyl-acrylic acid (beta-methyl gamma-keto-butanol). Preparation and purification of methylisopropenyl ketone and its polymerization products resembling plexigum. For the decolorizing of methylisopropenylketone before its polymerization experiments have been carried out which proved barium hydroxide and other weak alkalis to be suitable. - see application St 60877 "Process for the purification of methylisopropenylketone". Furthermore, the preparation of cyclo-hexanone resin (AW-2-Resin), methylvinylketone, and others.

4. Development of the ketone resin "Emekal" from methylethylketone and formaldehyde, as well as the establishment of the process for a production of about 4 tonnes per month. Further improvement of the "Emekal" resin to an almost colorless and above all, hydrocarbon soluble resin type (for example Emekal 350) for the lacquer industry. See application St. 60911, "Process for preparation of light colored, easily soluble synthetic resins".
5. Preparation of secondary butyl stearate as a plasticizer.
6. Preparation of aluminum alcoholates. Investigation of different catalysts for the reaction of aluminum with alcohols. See application St 59625 IVc/120 and St 59626 IVc/120, "Process for the preparation of alcoholates of magnesium and aluminum".

Use of Aluminum Alcoholate (butyl and propyl) for: -

- (a) pyrotechnic purposes (application St 59322 IVd/78d, "Use of metal-alcohol compounds for pyrotechnic purposes")
- (b) carbon igniters (application St 59424 IVb/78d, "Process for improving the ignitability and combustion velocity of solid hydrocarbons").
- (c) treatment of textile fibres (application St 60815 IVc/8k, "Process for improving textile fibres").
- (d) lacquer technique and other purposes, especially the preparation of emulsions and suspensions with the aid of aluminum alcoholates")
- (e) medicinal purposes (Professor Engelhardt, Tübingen, as well as Professor Frey, Düsseldorf)

7. Development of the carbonic acid addition products of aluminum alcoholates, particularly of the butyl compounds (application St 59783 IVd/120, "Process for stabilization of metal alcoholates") Preparation of the material "Carbutal" or "Rh5" and its use as a gelation medium for lacquers and protective coatings (application St 60358 IV/10b, "Process of thickening and solidifying liquid organic compounds") Likewise a proposal for medicinal use of carbutals (Professor Engelhardt, Tübingen; Professor Frey, Düsseldorf).
8. Development of an aluminum alcoholate plant for a production of about 30 tons per month (application St 61255 IV/120, "Process for preparation of metal alcoholates; likewise St 59783 above).
9. Preparation of active aluminum hydroxide and oxide from aluminum alcoholates and their technical and medicinal application, as well as the development of an experimental plant for production of aluminum hydroxide and oxide.
10. Working out of a process for recovering pure aluminum from waste and remelted aluminum alloys (application St 61231 IVb/12m, "Process for separation of aluminum from aluminum alloys").
11. Preparation of a new gelatinizing material (Rh6) from butyl (Butal) and our C6 - C9 fatty acids, and also the fatty acids from the Wittener process. Use of the aluminum salts of these fattyacids for the purpose of "Wa Prüf 5 lc".
12. Development assignments of the "OKH., Wa Prüf 5".
 1. Development of smoke tubes (Parchlorit 7) X
 2. " " water resistant abrasive devices (Reibstätzen)
 3. " " an igniter cartridge for oil (Parchlorit 7) X
 4. " " storage stable delay devices

X See application St 59425 IVb/78i, "Process for preparation of finely dispersed materials, of importance in pyrotechnics, from paraffin wax from the Fischer-Tropsch synthese".

4. Development of storage stable delay devices (contd)
(Parchlorit 7) X
5. " " membranes as a safety valve for flame
throwing devices.
6. " " incendiary oil for filling flame
throwing devices, and in combination
with the above
7. " " an aluminum alcoholate plant (see above)
8. " " burning tubes (Parchlorit 7) X
9. " " a chemical time igniter (see special
application W 109820 XI/721, "Chemical
time igniter").

A few additional assignments are yet to be expected.

(signed)

Wiedmann

X See application St 59425 IVb/78i, "Process for preparation of finely dispersed materials, of importance in pyrotechnics, from paraffin wax from the Fischer-Tropsch synthese".

Treibstoffwerk
27 September 1943

To: General Manager Kost,
General Offices

Herewith I send you a review of the productivity of the scientists in the principal laboratories in the past report year.

Dr. Wiedmann

Work of Dr. W. in the past year:

- (A) Aluminum alcoholates and their decomposition in the most various ways. Technically useful products, especially thickeners, plastic masses and artificial materials were produced from the aluminum alcoholates and shown to be technically useful.
- (b) Aluminum hydroxide and oxide: intensive work was done on the production of the different modifications. Preparations were in part, taken up for their production on a semi-technical scale. Their usefulness as additives, as rubber filler, as catalysts, as a carrier for serums, as polishing means, and their usefulness for medical purposes was discussed and worked on in conjunction with numerous foreign firms.
- (c) Production of ketones, especially acetone was worked on further, and during the report year the technical production of acetone was built up and is being carried forward at present.
- (d) A series of developed projects of the Armed Forces (Army and Air Force) was worked upon and, in part, finished. In all of this work, Herr. Dr. W. proved himself to be extraordinarily valuable by reason of his fertility of ideas, his application to his work, and the pleasant way with which he did the work.

In the work mentioned above, he was supported with zeal and success by his assistant Dr. Josten.

Nine patents were applied for in respect to the work above mentioned from Dr. Wiedmann's division.

Dr. Schmitt

Aside from working upon any special current problems in the realm of organic chemistry, Dr. Schmitt was busy with the synthesis of polyvalent alcohols (glycerine substitutes). The work was carried

far enough in the course of the report year so that the technical planning in all particulars could be completed. In addition to this the conversion of acetone to diacetone alcohol and the conversion of the latter to glycol D was brought sufficiently to conclusion so that planning for a technical plant could be taken up.

Two patents were applied for in the above avenues of effort from Dr. Schmitt's division.

Aside from the above, Dr. Schmitt, together with Diploma Engineer Dr. Beier, oversaw the education of the apprentices.

Dr. Camper

The avenues in which Dr. Camper worked were:

- (a) Dicarboxylic acid from parastol fatty acids, etc., etc.
- (b) Nitrocarbonic acids from fatty acids and their conversion into amino acids.

Both of the above named problems, which are of importance for the complete synthetic production of artificial fibres, led to results of richest significance, which were described in different patent applications.

- (c) Obtaining higher alcohols from olefines of the benzine and kogasin fractions, and a new and what appears to be especially economic way to the solution of the above problem was worked out, whereby there was also obtained valuable information for the recovery of lower alcohols.
- (d) Isolation of olefines from synthesis product was further worked out in the laboratory and in the technical experimental plant. Dr. C. further supervised the extension of the laboratories which can be designated as complete in this report year.

Dr. C. was, in all of the above work, in which he showed great diligence and skill, assisted by Dr. Wollner, who could be promoted on the basis of his working out of the separation of olefines from kogasin.

Three patents were applied for in Dr. C.'s division.

Dr. Heusser

Dr. H. was primarily busy with the management of the alcohol plant which experienced a very considerable increase of production, as well as improvement in economies during the report year.

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Aside from this Dr. H. worked in the laboratory on the isolation of amyl alcohol from the pentane fractions, the concentration of sulphuric acid by means of submerged burners, and the separation of new kinds of subsidiary products from the alcohol plant (octanesulphones).

Dr. H. has shown himself as a diligent plant manager, exhibiting independence. One patent came from his division.

Diploma Engineer Beier

He dedicated himself with the greatest zeal and scientific attention to all problems of the control laboratories. It should be especially noted that in spite of hindrances from sickness he carried out his work regularly.

Dr. Dannefelser

Dr. D. has conscientiously carried through his work as the division head of the test stands, including the direction of the benzin laboratory, and the control tests of final products. Also he has given useful service in the negotiations with the service and government authorities.

(signed) GRIMME

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