

FILM STUDY GROUP

REPORT ON MICROFILM REEL NO. 16

Prepared by

THE ATLANTIC REFINING COMPANY

SCANNING OF REEL #16

U. S. Government Technical Oil Mission

Bag 3043

19. COMPARISON OF MOTOR KNOCK TESTING.
LUDWIGSHAFEN, MAY 12, 1939

Frames 2608 - 2627.

Six stabilized fuels were rated in 14 engines by the C.F.R. method and in 12 engines (I.G. motors) for research and motor method octane numbers. No description of the I.G. motor is given. The average value obtained in different laboratories agreed within about three octane numbers, but a deviation of five octane numbers was possible. The deviation was somewhat greater in the C.F.R. motors than in the I.G. motors. The average value of C.F.R. and I.G. methods agree within about one octane number, although as a rule the I.G. motor gives somewhat lower values.

The fuels examined were benzol, motor gasoline, a mixture of the two, leaded gasoline, aviation fuel leaded and unleaded.

The location of the engines was in 14 places, and it is presumed that this investigation was to improve the agreement between cooperating organizations.

Reviewer's comment: A report upon German cooperative knock testing studies was issued by C.R.C. May 25, 1945, under the title "Report of the Association for Knock Rating, Ludwigshafen-Oppau, February 16-17, 1943."

MEASUREMENTS OF THE HEAT OF POLYMERIZATION,
LUDWIGSHAFEN, MAY 22, 1939.

Frame 2628+.

Fischer-Tropsch "gasol" (a mixture of C₃-C₆ paraffinic and olefinic hydrocarbons) is intended as charge stock to polymerization. The heat of polymerization was found to be 186-kcal/kg polymer. This is in good agreement with an earlier Leuna value of 173-kcal for isobutylene polymerization. The catalyst was known as V-11 and consisted of phosphoric acid and charcoal. The inlet temperature was approximately 150°C., the outlet about 226°C. and the pressure was about 31 atmospheres. Analysis of charge and spent gases is shown below.

<u>Component</u>	<u>Percent in Charge Gas</u>	<u>Percent in Spent Gas</u>
C ₂ H ₄	0.4	2.0
C ₃ H ₆	22.2	16.3
C ₄ H ₈	21.3	14.5
C ₅ H ₁₀	8.2	2.3

The liquid product was found to be 77% C₅-C₈. The description and data, with the exception of catalyst properties, is unusually complete (20 frames) and will permit ready checking by those who wish to make a critical survey of this subject.

TWO INTERMEDIATES FROM FUEL PRODUCTION
WITH IMPORTANT CHEMICAL USES

A report from the Ammoniakwerk Merseburg Laboratories August 7, 1939.

Frames 2648 - 2718.

This report had its origin in a desire to widen the application and uses of products derived from the hydrogenization of brown coal and is obviously a move to compete in the fields previously dominated by the bituminous coal by-products industry. These intermediate products of brown coal hydrogenation fall into two important groups: paraffinic and phenolic. The report which tends to be of a speculative nature, rather than the presentation of data is, nevertheless, believed to be of interest because it is the forerunner of applications developed at Merseburg. By-products in the preparation of these intermediates would be sulphonates (nepsin salts) and their esters. Detailed information should be sought upon: (a) recovery of phenol by extraction with tricresyl-phosphates, (b) the promotion of reaction in paraffinic hydrocarbons by means of light spectrum (3000 to 5000 Angstrom units), and (c) sulfo-chlorination involving the simultaneous use of SO₂ and chlorine.

RESEARCH ON SOAP FATTY ACIDS FROM TTH PARAFFIN,
OPPAU, AUGUST 8, 1940

Frame 2719+.

The fatty acids from TTH paraffin were converted to saturated esters, separated into single components and these latter were closely studied.

The acids consisted in greater part of straight-chain paraffinic acids, with a chain length of 9 to 25 carbon atoms; branch chain paraffin carbon acids were present from 1/2 to 1%; unsaturated acids were present in amount up to about 8%; material absorbed on silica gel (chiefly oxy-keto and dicarbon acids and their derivatives) amounted to about 15%.

The starting materials are derived by the oxidation of TTH paraffin, which itself appears to be a product of the Fischer-Tropsch process. The presently discussed investigation involved: (1) conversion of the acids into methyl esters, (2) separation of the oxy, keto- and dicarbon-acid esters and their derivatives, via absorption on silica gel, (3) hydrogenation of the olefinic acid ester to saturated esters, (4) fractionation, (5) study of the intermediate fractions C₁₁-C₁₂ and C₁₂-C₁₃, as esters of branched acids.

INFORMATION ON FATS AND FATTY ACIDS WITH AN
UNEVEN NUMBER OF CARBON ATOMS

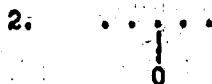
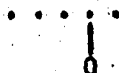
Report from the Oppau Laboratories of I.G., of uncertain date, but believed to be Spring of 1940.

Frame 2737+.

Typical page from "Literature Survey on the Constants of Fatty Acids with 6 to 20 Carbon Atoms"



Pentane carbon acid (2)

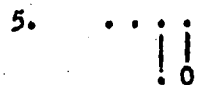


aus methyl propyl
 carbin iodide
 + KCN \rightarrow nitrile

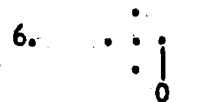
KP 193°/740 mm (1)
 D_0° : 0.9414 (1)
 D_0 18 : 09279 (1)



Ethyl ester
 KP 153°/751.5 mm (1)
 D_0° : 0.8816 (1)
 D_0 18 : 08670 (1)



Amide
 FP 95° (2)
 85° (3)



- (1) Saizen, A 193, 348
 (2) Kelbe, B 15, 311
 (3) Guerbet, C. r 153, 1709

- (1) The preparation of two saturated fats from cocoa fatty acid is described. A glyceride with uneven number of carbon atoms was made from a straight-chain fatty acid. The other was a glyceride made from straight-chain fatty acids consisting only of even number carbon atoms.
- (2) Both fats were compared with natural cocoa fat in rat feeding studies. In concentration of 5, 10 and 20%, there was no difference in the growth nor deposition of fatty materials during the research period, or in decomposition of the fat during deprivation of food.
- (3) The reabsorption of fats in humans shows no fundamental difference from that of the research on rats.
- (4) The respiratory quotient of rats as affected by saturated triglycerides of fatty acids with odd or even number of carbon atoms was studied.
- (5) The saturated glyceride having an uneven number of carbon atoms and derived from straight chain fatty acids, when tried on dogs compared to natural cocoa fat, showed no increased diacidurie.

LITERATURE SURVEY ON THE CONSTANTS OF FATTY
ACIDS WITH 6 TO 20 CARBON ATOMS,
OFFAU LABORATORIES, FEBRUARY 15, 1940

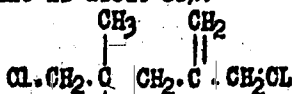
Frames 2764 - 3018.

Shown on the page facing is information typical of the entire presentation. The extensiveness of information on any one compound may vary from half to three times as much information as was indicated for the pentane carbon acid used as an illustration.

THE AMMONOLYSIS OF DI-ISOBUTENYLCHLORIDE,
LEUNA, SEPTEMBER 8, 1942

Frame 3019+.

A great excess of concentrated aqueous ammonia at a temperature of 160°C. is used. The yield of diamine is about 80%.



Di-isobutenylchloride, $\begin{array}{c} \text{CH}_3 \\ | \\ \text{Cl} \cdot \text{CH}_2 \cdot \text{C} \cdot \text{CH}_2 \text{Cl} \\ | \\ \text{CH}_3 \end{array}$, is made by dimerizing methyl allyl chloride. It was desired to replace its chlorine with the NH_2 groups, and so form a 1,5 diamine. The chlorine is replaced with great difficulty, up to the present study.

The description and detail in this report is considered by the reviewer to be ample for those interested in the subject to repeat the results shown. The writer of the report believed that the ammonolysis as described by him was novel and was not in conflict with other German patents.

STUDIES ON THE UTILITY OF ORGANO LEAD,
IRON CARBONYLS AS ANTI-KNOCK AGENTS,
LEUNA LABORATORIES, DECEMBER 28, 1943

Frame 3034+.

Diethyl lead-irontetracarbonyl and diphenyl lead-irontetracarbonyl were made and their usefulness as anti-knock materials was studied. Both compounds are inferior to tetraethyllead. They have less anti-knock effect, are easily decomposed and have limited solubility in gasoline.

This report describes the manufacture of possible substitutes for tetraethyllead. It is presumed that the difficulties in manufacture of lead tetraethyl and the corresponding simplicity in manufacture of carbonyls, prompted the research cited.

DISPERSION OF AIR IN LIQUIDS,
LUDWIGSHAFEN, JUNE 15, 1944

Frames 3042 - 3049.

This report was commented upon in the scanning of reel No. 25, and is not believed to be important.

WORK BY RESEARCH GROUPS AT MERSEBURG

Frames 3050 - 3127.

Summaries, usually less than one page, of individual problems. It is difficult to discuss these summaries since they appear apart from any supporting discussions and data. It is noted in some instances that they follow the formal report - as for example, Item ZuHy-28 recapitulates work on diethyllead-iron tetracarbonyl and diphenyllead-iron tetracarbonyl as anti-knock agents, which was reported in a previous section occupying several frames. Cited below is an enumeration of the reports with presumed date of issue.

Part I. Rv-Gruppe

- 1 - December, 1929 - Research on gasoline from middle oils.
- 2 - March, 1930 - Comparative product research and investigation of high boiling ends.
- 3 - March, 1931 - Behavior of pentane in benzene ovens.
- 4 - April, 1931 - Previous research on gasoline production with Ludwigshafen catalyst 5058.
- 5 - June, 1931 - Compilation of analysis on different middle oils.
- 6 - July, 1931 - Cracking of naphthalene over catalyst 5058 and hydrogen utilization in the refining of coal tars, etc.

- 7 - August, 1931 - Refining of coal tar oils.
- 8 - November, 1932 - Dehydrogenation process for improving anti-knock properties of gasolins.
- 9 - October, 1935 - Vaseline substitute from Leuna Hydrogenation Plant.
- 10 - July, 1936 - Gasoline refining research in Merseburg 333.
- 11 - October, 1936 - Refining of gasoline with metal chloride.
- 12 - October, 1936 - Hydrogenation of Steinkohle extract.
- 13 - December, 1936 - " " " "
- 14 - May, 1938 - Research report on the hydrogenation plant project at Lutzkendorf.
- 15 - December, 1938 - Production of diesel oil from coal middle oil by extraction with butane-SO₂.
- 16 - December, 1938 - Fractionation of Pott-Brosche coal extract.
- 17 - February, 1939 - Research on zinc sulphide catalyst for refining Steinkohle oils in the vapor phase.
- 18 - March, 1939 - Production of brown-coal cracked tar by extraction with selective solvents and separation of the paraffinic hydrocarbons.
- 19 - March, 1939 - Catalytic cracking of hydrocarbon.
- 20 - July, 1939 - Catalytic cracking with finely divided catalyst in the liquid phase.
- 21 - December, 1941 - HF process and plant at Moosbierbaum.
- 22 - July, 1942 - Aviation fuel by catalytic cracking.
- 23 - February, 1943 - The HF process.
- 24 - March, 1943 - Investigation of Estonian shale oil.
- 25 - August, 1943 - Research theory on catalyst regeneration in catalytic cracking.
- 26 - September, 1943 - Fixed bed catalytic cracking. Report on the KK Plant in Leuna.
- 27 - October, 1943 - Aromatic gasoline process.
- 28 - December, 1943 - Investigation on the utility of organo lead-iron-carbonyls as anti-knock agents.
- 29 - February, 1944 - Interim report on coal research.
- 30 - March, 1944 - Investigation of Russian petroleum.
- 31 - April, 1944 - 15 years of fuel research in Leuna Laboratories.
- 32 - June, 1944 - Catalytic cracking of pure hydrocarbon.
- 33 - July, 1944 - Investigation in comparison of redistillation residues from DHD and HF processes.
- 34 - August, 1944 - Rate of burning of coke laid down on the catalyst in the KK process.
- 35 - November, 1944 - Proposal for a Catalytic Cracking Plant with a capacity of 70,000 tons per year.
- 36 - (date uncertain) - Production of pure hydrocarbons boiling in the range of middle oil, by the pressure hydrogenation in the gas phase.

Part II. Kk-Gruppe

- 1 - November, 1929 - Research on desulphurization of hydrogenation spent gas.
- 2 - February, 1938 - Catalytic dehydrogenation of isobutane.
- 3 - December, 1941 - Investigation of gases containing hydrocarbon.
- 4 - February, 1943 - The isomerization of normal butane with AlCl₃.

- 5 - July, 1943 - Butane propane dehydrogenation in tubularovens with fixed catalyst.
- 6 - September, 1943 - Improvement in the separation of the lower paraffin-olefin mixtures with distillation aid (azeotrope).
- 7 - March, 1944 - Catalytic dehydrogenation of propane to propylene.
- 8 - August, 1944 - Separation of lower aliphatic olefins from olefin-paraffin mixtures with silver wash.
- 9 - October, 1944 - Production of Toluol by catalytic conversion of higher boiling aromatic hydrocarbons.

Part III: Other Departments

- 1 - October, 1926 - Compilation of work of the ammonia laboratories in wood and cellulose.
- 2 - October, 1926 - Research on the hydrogenation of furfural.
- 3 - January, 1927 - Research on the catalysis production of mono-methylamine from hydrocyanic acid, or from methylnitrite.
- 4 - January, 1927 - Exploratory research on the pressure heating of wood with lye and similar materials.
- 5 - March, 1927 - Separation of the oils from the tar residues obtained in coal hydrogenation by means of super-heated steam.
- 6 - March, 1931 - Investigation of the middle oils of different origin, and test methods.
- 7 - September, 1931 - Preliminary calculation for the production of aromatic hydrocarbons and olefins from Hy-gas.
- 8 - January, 1932 - Short summary on the technical production of fuel from gasoline, benzol, ethyl alcohol and methyl alcohol.
- 9 - October, 1932 - Isomerization of hydrocarbon (Hexane).
- 10 - August, 1935 - Heat of combustion of ethyl chloride.
- 11 - April, 1936 - Ethyl chloride production from vinyl chloride.
- 12 - April, 1936 - Conversion of dilute ethylene to ethylene oxide.
- 13 - April, 1936 - Hydrogenation of vinyl chloride to ethyl chloride.
- 14 - October -
December, 1936 - Research on hydrogenation of Steinkohle extract.
- 15 - Gas phase research -
- 16 - December, 1936 - Report on extract recovery and extract hydrogenation.
- 17 - July, 1937 - Research on hydrogenation of Steinkohle extract.
- 18 - March, 1938 - Catalytic dehydrogenation of isobutane.
- 19 - March, 1938 - Brown-coal extraction research at Leuna.
- 20 - April, 1938 - Catalyst VK6434 in the recycle.
- 21 - July, 1938 - Production of concentrated volatile olefins from paraffin hydrocarbons.
- 22 - October, 1938 - Testing of two diesel oils in motors.
- 23 - October, 1938 - Diesel oil from TTH residue and decomposition of this by cracking.
- 24 - March, 1940 - Report of analytical and separative work on hydro-carbon mixtures.
- 25 - April, 1940 to
August, 1942 - Investigation of knocking properties by (overload ?) method.

- 26 - May, 1942 - Laboratory column for close distillation.
- 27 - July, 1942 - Design of oven for catalytic exothermic and endothermic reactions.
- 28 - February, 1943 - Comparison of methods for determination of olefins.
- 29 - March, 1943 - Efficiency of the heat pump for fractionation.
- 30 - May, 1943 - Determination of the heating value of gasoline and oils from their densities.
- 31 - May, 1944 - Obscure; probably refers to a process group meeting.
- 32 - September, 1944 - Review of methods for determination of olefins.
- 33 - July, 1944 - Sapropel problems.
- 34 - September, 1944 - Research on the analysis of hydrocarbon mixtures by means of dyes.

The reviewer feels that the principal importance of this group of summaries is the insight which it gives to the research work carried on by the Merseburg group, leading up to processes and applications widely employed in the war years.

INDEX - MICROFILM - REEL #16
(Orig. Ident. Reel 16A)

Bag 3041 - Target 30/4:02

I.G. Farbenindustrie, A.G. Leuna

1 - Geissen Retort. Frames 2 - 10. A memorandum, August 25, 1936.

The new Geissen coking retorts in Edderitz and Bruckdorf were visited. Each oven has a daily capacity of 25 tons. Construction and operation of the retort were said to be simple. A pencil sketch of the retort shows it to be a vertical, cylindrical section concentric to a central combustion space. The retorting space has a number of equally spaced baffles (venetian blind) which probably are used to slow the passage of coal downward through the coking zone. Dried brown coal was used as charge stock. The approximate dimensions of the retort are 0.4 meter diameter and 10 meters high. The central heating tube is rotated at a speed of 3 rpm. A residence time of 4 minutes in the retort section is cited. The heater located at the top of the combustion space imparts the temperature of 1500°C. to this point, and the temperature of the coal about 1/3 way down is 460°C. A final temperature in the char of 520°C. is reached. The reviewer presumes that detailed prints and description of this process were obtained by the Technical Oil Mission, which would impart more authoritative information than the memorandum written by persons relatively unfamiliar with the process.

2 - Cost of Coke Plant Installation. Frames 11 - 17. A memorandum, February 29, 1940. Summaries of costs for a coking plant, charging about 1-1/3 million tons brown coal per year. The total cost of 31,000,000 RM included plant power facilities, coal drying, briquetting, retorting and gas purification.

3 - Improvement in the Lurgi Retort. Frames 18 - 22. A memorandum, November 15, 1943, discussing the Lurgi oven and features which the Leuna engineers believe capable of substantial improvement.

The material is of a speculative nature and without the prints of the Lurgi retort, the reviewer cannot take exception to the criticism offered by the Leuna engineers. This portion of the reel will probably be of interest when correlated with microfilm dealing specifically with the Lurgi process.

4 - Combined Hydrogenation and Carbonization Process. Frames 23 - 56. A Leuna report dated April 4, 1939.

The utilization of the hydrogenation residues when mixed with coal or in briquettes to be carbonized to give simultaneous recovery of hard coke and cracked tar, is primarily dependent on a certain asphalt content of the residue. About 11 to 14% asphalt is required to make strong coke.

From the mild hydrogenation of Steinkohle, decomposition amounting to 85% was attained, and the resulting residue was mixed with Steinkohle, and by retorting yields strong coke and at the same time permits recovery of cracked tar.

This method of processing was applied to the utilization of residue from hydrogenation at Scholven; this residue when used with different coals from the Ruhr, Saar, etc., gave strong coke. The proportions of slurry to coal are of the order 10 to 20% and 80 to 90%.

5 - Efficiency of Winkler Water Gas Generation. Frames 57 - 66, dated April 24, 1929. This memorandum consists of calculations presumably based upon test data secured at Leuna. The reel is illegible in many places; the reviewer did not find evidence that unusual operation or results are reported, and believes that more recent operating data will be secured by scrutiny of other reels.

6 - Report for 1935 on Winkler Generators, Leuna, January 21, 1936. Frames 67 - 95. Appears to be a methodical engineering report on the Winkler Generator Plant.

The installation consisted of five commercial generators and one pilot plant unit. Two machines were capable of producing 40,000 and 75,000 cubic meters producer gas per hour, one machine at a capacity of 20,000 cubic meters water gas per hour, a fourth generator was capable of 75,000 cubic meters of producer gas or 60,000 cubic meters of water gas per hour. The pilot unit had a capacity of approximately 3500 cubic meters per hour when used either for producer gas or water gas operations. A fifth generator was used as a retort and is placed in a stand-by capacity. This plant is interesting because oxygen was employed in water gas production. Waste heat boiler operating at about 220 and 750 lbs. pressure were in use. Oil and glycerine mixtures for lubricating the oxygen injection valves proved unsatisfactory, and since 1934 oil alone has been used.

The producers appeared to have been charged with a non-coking coal and temperatures in the combustion zone of 950-1000°C. are noted.

There is always danger of slagging where oxygen injection is employed. A temperature of 900°C. in the coal bed and 1030°C. on top was considered satisfactory.

The coal, presumably used as charge to both the producers and water gas generators is designated as Elise coal and during the year showed variable ash content between 15 and 25%.

The oxygen used averaged 98% purity. Its origin is not stated.

Analyses of the producer gas and water gas are shown.

<u>Component</u>	<u>Producer Gas</u>	<u>Water Gas</u>
CO ₂	10.6	23.3
H ₂	11.7	38.5
CO	21.7	35.3
N ₂	55.3	1.1

The efficiency of the producer was calculated at 60% and that of the water gas generator at 66%.

7 - Analysis of Water Gas from Various Processes.

A three-page memorandum, January, 1936 showing analyses of gas from various sources, such as the water gas from brown coal, briquetting and Hy-gas.

8 - Safety Precautions in Brown-Coal Plants. Frames 99 - 103.

A memorandum, May, 1939 discussing precautions taken against the repetition of an accident occurring in another plant. The coal drying installation was to be blanketed with CO₂ and an alarm device warned the operator when the oxygen content exceeded 4%. Dusty atmospheres were recognized as hazardous but were considered a secondary danger. The Winkler Generator Plant where air or oxygen is used in gasification, was provided with oxygen alarm and indicating devices when the temperatures in the generators became unusually high. Coal bunkers were to be blanketed with CO₂.

9 - Explosion Hazard in Pulverizing Plant. Frames 104 - 112. Memorandum from the I.G. Höchst Laboratory, September, 1941.

It was shown that certain dyestuffs and other materials which are being treated in a pulverizer-dryer, can be ignited by electrical sparks.

The experimental conditions are described and the conclusion reached that grinding and drying of such materials is to be done in an atmosphere deficient in oxygen.

10 - Firing of Salt Coals. Frames 113 - 135. A report from the Leuna Laboratories April 15, 1942.

This is an engineering survey on the utilization of some marginal quality fuels used for power generation purposes. It was shown that an Na_2O content of 20% or higher resulted in ash in which the alkali appears as carbonate or sulphate. The former melts at 800°C . and the latter at 884°C . The ash particles soften and adhere to the boiler in cooler spots. The deposition is very viscous and eventually closes off the vessel. On cooling, the incrustation becomes glassy hard and can be removed only with great difficulty.

Clay, added in amounts of 10 to 12%, hinders the formation of the carbonate and sulphate, tending rather to form sodium aluminum silicate whose melting point is about 1250 to 1300°C . Furthermore, the scale formed in lesser amounts is readily removed.

Reviewer's notes: The above discussion applies to specific German coals and the authors do not imply that universal relief was afforded by the addition of clay.

11 - Influence of Slag and Hydrogenation Residue on Fusion of Salt Coal Ash. Frames 136 - 150, Leuna, June 2, 1943.

The difficulties encountered in firing salt coal can be diminished by admixture of clay or kaolin. Investigation showed the significance of silicic acid and the benefits imparted by clay.

Clay and granulated slag, in amounts up to 12%, added to a coal whose ash had a softening point of 550° and a melting point of 635° , increased the resultant ash to a softening point of about 950° and a melting point of 1100° . The residue from hydrogenation steps, that is spent catalyst, whose analysis (on the water free basis) is about 13% SiO_2 , 16% Al_2O_3 , 18% Fe_2O_3 , 21% CaO , 4% MgO , gave somewhat similar increases in a softening and melting point of the ash being employed.

12 - Ashing of Salt Coals with Addition of Kaolin and CaCO_3 . Frames 151 - 160. A report from the Merseburg Laboratory, August 27, 1943.

Laboratory investigation showed the beneficial effects of the use of 10% or more of equal parts of kaolin and calcium carbonate or clay and limestone when added to salt brown coal. The ash softening point was raised and the SO_2 or SO_3 in the flue gas greatly diminished.

13 - A Use for Granulated Slag. Frames 161 - 183. A report September 3, 1940 from the Merseburg Laboratory.

The granulated clinker produced in a slagging type gas generator can be converted by a simple process into a cement-like material. The slag is ground with quicklime in proportions 1.6 to 1.0. The investigation showed that the material could be used for building purposes.

The details of this report are explicit, giving analyses of the raw materials, tests upon the product, indicating a careful investigation. (Note: the term "quicklime" here probably should be inferred as calcium hydroxide liquor).

14 - Cause of Slagging in Brasserie Generators. Frames 185 - 189.

A report from the Merseburg Laboratory May 24, 1941.

The Brasserie generator was equipped with revolving grate and has a coke capacity of about 24 cubic meters. In the stirring zone there was formed some compacting of clinker which could be only partially penetrated by the blast. This disrupts the operation of the generator and damages the clinker discharge mechanism. The fuel used has about 82% combustible matter and 18% ash (determined by combustion in oxygen at 700°C). Analyses of the ash from this combustion and of the clinker taken from the generator itself are given below:

<u>Component</u>	<u>Ash</u>	<u>Slag</u>
SiO ₂	4.1	7.6
Fe ₂ O ₃	10.4	23.8
Al ₂ O ₃	5.6	9.0
CaO	32.2	50.8
MgO	2.7	3.7
Alkalies	1.3	.9
SO ₃	42.2	1.3
Sulphide	.4	3.2
CO ₂	2.4	.9
Combustible	0	2.5

The decrease in SO₃ shown by analysis of the slag, was attributed to dissociation of gypsum in the somewhat higher and more oxidizing atmosphere of the generator operation.

15 - Preparation of Refractory from Pulverised Chrome Slag
Frame 190 - 209. Merseburg Laboratory, March 18, 1936.

This report describes the experimental work in developing a refractory from some specific material. The use of an electrolyte such as nitric acid and sodium hydroxide with kaolin and aluminum hydroxide, produced satisfactory binders when incorporated in the granulated chrome slag. A compromise was sought between materials which were good binders but influenced physical properties such as expansion, stability, resistance to slagging, etc.

16. Extraction of Brown Coal by the Pott Process. Frames 210 - 248.
An Oppau report dated January 8, 1938. A more correct title for this report would be "Analytical Investigation of Pott Extract from Brown Coal".

The so-called Pott extraction of brown coal with tetralin results in substances containing asphalt, which have complicated the chemical examination.

Hydrocarbons such as propane or butane were used to deasphalt the extract. The fraction extracted by propane was dissolved in ether and systematically subjected to various solvents. In view of the probable profound effect of a particular brown coal which might be investigated, the reviewer feels that statement of specific results is of little interest. Those persons undertaking the identification of components from the extraction of carbonaceous material may, however, be interested in the procedure and speculation pertaining to this Pott extract.

17 - Brown Coal Extraction. Frames 249 - 269. Herseburg March 15, 1938.

This report is in effect a review of the hydrogenation of brown coal, with particular reference to Pott-Broche process. The process as developed by the Stinnes interests, uses a mixture of tetralin and cresol as the solvent. The dried pulverized coal is treated at a pressure of 100 atmospheres and a temperature of 390 to 420°C. The tetralin is dehydrogenated to naphthalene. The extracted coal residue is easily separated by filtration. The naphthalene is readily rehydrogenated to tetralin. The process when applied to brown coals requires more tetralin, presumably because of their greater oxygen content. The report describes parallel or somewhat similar studies undertaken at the Stinnes plant in Karnap and at Leuna, and the present report may be called a review of these studies. The writer seems to be convinced of the advantages of the Pott-Broche extraction process preceding hydrogenation, as opposed to direct coal hydrogenation, citing the lower pressures, cheapness of material, simple equipment, etc. Hydrogen consumption was lower and no catalyst was necessary for the hydrogenation of the extract. Furthermore, there was no loss of catalyst in hydrogenation of the naphthalene to tetralin. Equipment and operation are described in general terms. The reviewer believes that the process had commercial application at a later date and that more specific information on operating details will be found in other reels.

18 - Inorganic Sulphur Content of the Gases Produced by the Water Gas Process with Respect to the Part of the Cycle. Oppau March 16, 1940. Frames 270 - 279.

The cycle on the water gas machine was a total of 4 minutes, of which 55 seconds was blast and 185 seconds run. Two types of experiment were made: (a) 95 seconds up run steam, 60 seconds down, 30 seconds up steam and (b) 35 seconds up steam, 60 seconds down steam, 90 seconds up steam. The make gas was analyzed for sulphur content, and in the first series showed a maximum at the end of the down steam period. Sulphur content of the gas fell to a minimum in the blast gas.

19 - Report for 1935 on Alkacid and Claus Kiln Plants. Frames 280 - 376.

Report dated Leuna, April 11, 1936.

The alkacid process was discovered by Dr. Baehr in 1931. By means of alkaline solution, H₂S and CO₂ can be removed from gases at low temperature. The solution is regenerated by heating to drive off the H₂S. More recent

development work showed that aqueous solutions of salts of amino acids were practical. It was said to resemble the Girdler (U.S.A.) process employing solutions of amino alcohol, as for example, triethanolamine. The early work was concerned with overcoming corrosion difficulties. Concentrated aluminum nitrate solution (specific gravity 1.15 to 1.18) did not attack aluminum under any condition; dilute solutions (less than 1.12 specific gravity) did. Once the corrosion had started, the reaction appeared to be auto-catalytic. By the introduction of water glass and phosphoric acid which combined with aluminum, corrosion was diminished.

This report is in effect an historical review of the development of the alkacid process, the recovery of concentrated hydrogen sulphide and conversion of this gas to elementary sulphur by means of the Claus kiln. The reaction involved is: $2 \text{H}_2\text{S} + \text{O}_2 \rightarrow 2 \text{S} + 2 \text{H}_2\text{O}$. The report contains numerous diagrams showing the development, and discusses the size and form apparently determined upon for a large size plant. There are no engineering drawings. This report will be found interesting from a patent viewpoint but lacks many essentials and details necessary for technical application.

20. Purification of Low Temperature Oven Gas by Alkacid Process.
Frames 377 - 402. Leuna, June 26, 1936.

The alkacid process was applied to several low temperature carbonization gases in varying types of equipment, as for example, packed columns and washers designed to create a body of foam to promote contact. There is a building up of thiosulphate which reduces the effectiveness of the process. This report again is of interest in tracing the development of the process, but lacks those details necessary for a proper understanding of the effectiveness of the process in full scale operation. It is apparent that approximately 90% of the H_2S is readily removed from an inert gas containing about 4% H_2S .

(This ends reel #16)