

FILM STUDY GROUP  
SUBJECT INDEX AND REPORT  
T.O.M. REEL NO. 7

Prepared by  
HUMBLE OIL & REFINING COMPANY

L. L. Newman

Prepared and submitted by  
The Humble Oil and Refining Co. Film Study  
Group  
of API-TOM Study Committee.

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\* The Kleinuntersuchung gives all data required for the routine daily analysis of Abstreifer plant sample.

\*\* The Grossuntersuchung presents the scheme for the thorough analytical workup of the complete coal hydrogenation product (Abstreifer).

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# HUMBLE OIL & REFINING COMPANY

REFINING DEPARTMENT  
TECHNICAL AND RESEARCH DIVISION

LABORATORY

BAYTOWN, TEXAS

U. S. GOVERNMENT TECHNICAL OIL MISSION

Microfilm Reel-7

Translation by Martin S. Baer

Part I



R E S T R I C T E D

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U.S. GOVERNMENT TECHNICAL OIL MISSION  
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BAG 2746 TARGET NO.30/4.08 GELSENBERG  
(Orig.Iden.Reel 7A)

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04% Mo as (NH <sub>4</sub> ) <sub>2</sub> MoS <sub>4</sub>		

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U. S. Government Technical Oil Mission - Microfilm - Reel-7  
Bag 2746 Target No. 30/4.08 Gelsenberg  
(Orig. Idem. Reel 7A)

Item No. 5 Analytical Methods

Page 1

Note on Poor Laboratory Performance (Gelsenberg Benzin A.G. - Gelsenkirchen-Horst 5/29/40)

The plant laboratory has reported analyses varying as much as 100 per cent. The H<sub>2</sub>S concentration at the Claus-furnace inlet was reported from 20 to 60 per cent though it should be 35 to 40 per cent. Also, the Claus furnace exit gases were reported to contain 11 per cent H<sub>2</sub>S and three per cent SO<sub>2</sub>, causing the furnace to be run with insufficient air.

Page 2

Note on Recoveries from Coal Hydrogenation Residue (Gelsenberg Benzin A.G. - Gelsenkirchen Horst 5/28/40)

Coal hydrogenation residues from Scholven and Gelsenberg Benzin were analyzed by solvent fractionation by the Suida method. The analyses are as follows:

	Scholven	Gelsenberg Benzin		
		1	2	3
Neutral Oils, %	76.1	84.8	84.7	84.2
Oil Resins, %	14.1	4.5	3.6	4.5
Soft Asphalt, %	8.2	9.3	10.5	9.8
Hard Asphalt, %	1.7	1.4	1.2	1.5

It is noted that the sum of neutral oils and resins is approximately equal for all samples.

Pages 3 and 4

Method for Determination of HCN in Coal Distillation and Similar Gases  
(Bromocyanide Method)

Principle:

The gases are bubbled through potassium hydroxide and then measured through a gas meter. The hydroxide is made up to volume; acidified bromine water is then added. Both HCN and thiocyanogen yield bromocyanide (CNBr). Excess bromine is removed with phenol solution. Potassium iodide is added and the solution titrated with thiosulfate.

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Reagents:

Appr. 4N KOH = 224 g. KOH/l.  
8N H<sub>2</sub>SO<sub>4</sub> = 220 cc. Conc. H<sub>2</sub>SO<sub>4</sub>/l.  
Bromine Water = Several cc. Br<sub>2</sub> Shaken with Water  
Phenol Solution = 5%  
Thiosulfate Solution = 0.10 N  
Starch Solution  
Potassium Iodide, Cryst.

Procedure:

Run the gas sample through 60 cc. KOH and then through a wash bottle with 40 cc. KOH at the rate of 30 to 60 l./hour (one to two cubic feet per hour). Measure the flow through a gas meter. Use only relatively little gas (50 - 150 l. i.e., two to five cubic feet) so that no bicarbonate can form in the wash-bottle, since HCN is not quantitatively soluble in bicarbonate solution. For safety, check if phenolphthalein still turns red. Make up the solution plus wash to volume in a volumetric flask. Acidify bromine water with 30 cc. H<sub>2</sub>SO<sub>4</sub>, place in large glass stoppered flask, and add slowly half the solution from the volumetric flask through a pipette with tip immersed. If the bromine color disappears, add more bromine water until permanently deep yellow. Allow to stand five minutes after addition. In this manner, not only HCN but also HCNS is converted to bromocyanide. (HCNS + 4Br<sub>2</sub> + 4H<sub>2</sub>O → H<sub>2</sub>SO<sub>4</sub> + 7HBr + CNBr). Therefore any HCN previously converted to HCNS (in the process) is also reported in this test.

Excess bromine is removed by addition of several cc. phenol solution. After 15 minutes with repeated shaking add 0.5 g. KI. Allow to stand  $\frac{1}{2}$ -hour in the dark, then titrate with thiosulfate, using starch indicator. One cc. 0.1N thiosulfate is equivalent to 0.00135 g. HCN.

A check run was made to determine if HCN is lost with the CO<sub>2</sub> liberated when the bicarbonate solution is run into the acidified bromine water. No loss was observed.

Page 5

Determination of SO<sub>2</sub> in Presence of SO<sub>3</sub>

The gas is passed through 0.1 N iodine solution until the solution is just discolored. This determines SO<sub>2</sub>. The solution is titrated with 0.1 N NaOH using phenolphthalein indicator, to obtain total acidity.

Using a cc. of iodine solution and b cc. of NaOH:  $SO_2 = 0.003206a$   
 $SO_3 = 0.004003(b-2a)$

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(1) Determination of Bivalent Copper in Copper Solution

The analysis is made using the potassium iodide method, where one atom, Cu liberates one atom I which is titrated with  $\text{Na}_2\text{S}_2\text{O}_3$ . To save KI, a KI solution containing KCNS is used.

Method:

To the mixture of 10 cc. KI solution (166 g. KI plus 900 g. KCNS plus distilled  $\text{H}_2\text{O}$  to make 10 l.) and 10 cc. 0.1 N  $\text{H}_2\text{SO}_4$  add 9 drops starch solution and one cc. copper solution; titrate with 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$ .

Calculation:

$$\frac{\text{cc. 0.1 N Na}_2\text{S}_2\text{O}_3}{10} = \text{Mol Cu}^{++}/\text{l. Cu Solution}$$

(2) Determination of Monovalent Copper

$\text{Cu}^+$  is oxidized to  $\text{Cu}^{++}$  by  $\text{KMnO}_4$

Method:

50 cc.  $\text{H}_2\text{O}$  + 10 cc.  $\text{H}_2\text{SO}_4$  (1:2) are just colored with a few drops  $\text{KMnO}_4$  solution. 20 cc. of 0.1 N  $\text{KMnO}_4$  and then 1 cc. of copper solution are added; the solution is backtitrated with 0.1 N ferrous ammonium sulfate.

Calculation:

$$\frac{\text{cc. 0.1 N KMnO}_4 - \text{cc. 0.1 N Fe(NH}_4)_2(\text{SO}_4)_2}{10} = \text{Mol Cu}^+/\text{l. Cu Solution}$$

Page 7

A table for the conversion of volumes of  $\text{CO}_2$  into mols and gms. per 100 cc. copper solution.

Pages 8 and 9

Determination of HSCN in Water Gas

The following analysis was developed and checked in the "alkazid" laboratory of the "Leunawerke" to determine the damaging of "alkazid" solutions by HCN in the raw gas. Any HSCN in the gas is also determined. Since the originally small HCNS content of the generator - water gas is further diminished by the water wash, due to the higher acidity of HSCN relative to HCN, the amount of HSCN in these samples is insignificant.

-3-

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Principle:

The raw gas is passed through polysulfide solution. Ammonia is added, the solution is evaporated to low volume, sulfur filtered off, and iron ammonium alum is added to the filtrate.

The HCN content is determined colorimetrically by comparing with iron thiocyanate.

Equipment and Reagents:

Several small 10-bulb tubes (10-Kugelrohr)  
Ammonium polysulfide solution prepared by saturating 25%  $\text{NH}_4\text{OH}$  with  $\text{H}_2\text{S}$  and adding sublimed sulfur. Ready for use after three days with occasional shaking.

10%  $\text{HCl}$

Iron ammonium alum, 10%, slightly acidified with dil.  $\text{HNO}_3$  to reduce color.

Colorimeter with light blue filter and green plate.

Procedure:

Place two 10-bulb tubes, each with 10 cc. polysulfide solution, in series and pass through them an even stream of the water gas whose HCN content is to be determined. Measure the gas through a wet test meter after removing  $\text{NH}_3$  from it in an  $\text{H}_2\text{SO}_4$  bubbler.

The  $\text{CO}_2$  from the gas will precipitate the polysulfide sulfur, turning the solution from dark brown to yellow. When the sulfur begins to stick to the inside wall of the lowest bulb (after 15-25 l. of gas, i.e.,  $\frac{1}{2}$  to 1 cubic foot) turn off the gas stream and renew the polysulfide solution. Otherwise the sulfur on the glass wall will retain part of the HCNS, which is hard to wash out. After 50-100 l. (2 to 3 cubic feet) of gas have been run, collect the polysulfide in a 400 cc. beaker.

Add 10 cc.  $\text{NH}_4\text{OH}$  (to make certain that no HCNS will be lost in the evaporation), and evaporate on a hot plate at moderate heat. All the polysulfide sulfur precipitates until the solution is clear and nearly colorless. Filter off the sulfur, wash with hot water, and re-evaporate the filtrate (to about 30 cc.).

After cooling, acidify slightly with 10 per cent  $\text{HCl}$  (to turn litmus red) and add 30 cc. ferric ammonium sulfate.

Determine HCN colorimetrically by comparing with iron thiocyanate standard.

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Pages 10 and 11

Determination of Phenols in Gasolines, Gas Oils  
and Abstreifer\*Water (Buer-Scholven 7/23/40)

The product to be analyzed is shaken with sodium hydroxide to wash out the phenols. The phenol is then removed from the solution by heating and passing a stream of CO<sub>2</sub> through it. It is recovered in water which is titrated according to Koppeschar.

Procedure:

A measured amount of product is shaken three times with 10 per cent caustic in a separatory funnel. To remove neutral oils, the phenol solution is ether-washed. Phenol is removed from the ether with caustic and this added to the original caustic. Make up to 250 cc. or 1000 cc. in volumetric flask, depending on phenol content.

Place a 25 or 50 cc. aliquot in an Engler flask and add 12 per cent CuSO<sub>4</sub> solution for H<sub>2</sub>S removal until blue color remains. Start a rapid CO<sub>2</sub> stream through the flask. After absorption is essentially finished, reduce the CO<sub>2</sub> stream and heat the solution to boiling. Cool the product through a Liebig condenser and use an extended adapter dipping into a 250 cc. volumetric flask (cooled in an ice bath) as receiver. Distill in the CO<sub>2</sub> stream until nearly dry, add distilled water and continue distillation. Then wash condenser and adapter with water and fill the receiver to volume. If there are still phenol droplets in the distillate after filling, repeat experiment with smaller quantities, or titrate the whole distillate.

Normally for one titration 100 cc. of distillate are placed in an iodine flask and treated with an excess of 0.1 N bromide-bromate solution and 10 cc. conc. HCl. This precipitates the tribromophenol. After standing  $\frac{1}{2}$  hour, 15 cc. of 10 per cent KI is added and the amount of iodine equivalent to unreacted bromine back-titrated with 0.1 N thiosulfate.

From the cc. of bromide-bromate solution used, the phenol content is calculated by use of empirical factors.

The determination of phenol in phenol waters is similar. The samples are treated with caustic; if oily, they are ether washed and CuSO<sub>4</sub> solution is added. If strongly ammoniacal, the ammonia is first carefully removed from the open flask, using a small flame, before the distillation of phenol. It is important to have a sufficient excess of caustic.

~~The use of H<sub>2</sub>SO<sub>4</sub> instead of CO<sub>2</sub> for driving off phenol is not to be recommended since during the distillation SO<sub>2</sub> can be formed, which also consumes bromine.~~

\* This word is sometimes confusingly translated as "stripper." Actually the "Abstreifer" is the separator (tar pot or catch pot) in which the liquid hydro-generation product is separated from the gases, and consequently the liquid product withdrawn from it is also referred to as Abstreifer. A separate water phase is also withdrawn from the Abstreifer.

Determination of Mercaptan in Liquid Fuels (Hy. Werk Scholven 7/11/40)

Principle:

Mercaptans form mercaptides with copper solution. Therefore, if excess  $Cu^{++}$  is added to gasoline, a part of the copper is used to form mercaptides. The rest can be backtitrated with KCN solution. The deep blue ammoniacal copper salt solution is thereby discolored in the following reaction:  $2 Cu (NH_3)_4 SO_4 + 7 KCN + H_2O = [Cu_2 (CN)_6] K_3 (NH_4) + NH_4 CNO + 6NH_3 + 2K_2SO_4$ .

Method:

In a mixing cylinder shake 100 cc. of the gasoline with a fixed volume of copper tetramine solution of known copper content (1 mg./cc.). After vigorous shaking for  $\frac{1}{4}$ -hour titrate the excess copper with adjusted KCN solution in the cylinder in presence of the gasoline.

The KCN solution is so adjusted that 1 cc. Cu (1 mg. Cu) is equivalent to 1 cc. KCN solution.

Calculation:

Vol. Gasoline 100 cc.  
Vol. Cu Solution a cc.  
Vol. KCN Solution b cc.  
Cu Solution Consumed: a-b cc.

1 cc. Cu Solution = 1 mg. Cu  $\approx$  1.008 mg. Mercaptan Sulfur or 2.39 mg.  $C_3$ -Mercaptan.

Any  $H_2S$  in the gasoline must be washed out with saturated  $Na_2CO_3$  solution.

Preparation of Solutions:

(1) Copper tetramine solution

3.928 g.  $CuSO_4 \cdot 5H_2O$  are dissolved in 350 cc. dist. water. After cooling, about 25 cc.  $NaOH$  (5° Be.) are added dropwise until no more precipitate forms. Add cooled conc.  $NH_4OH$  until the ppt. is redissolved. Make up to 1000 cc. This solution is standardized electrolytically.

(2) KCN solution

3.5 g. KCN is dissolved in water. Solution made up to 1 l. Dilute so that 1 cc. KCN solution is equivalent to 1 cc. copper solution.



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Pages 13 and 14

Determination of Traces of Nitrogen in Hydrocarbons and Tars

Principle:

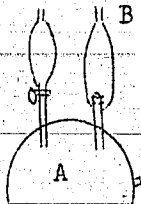
The nitrogen determination is made by the Kjeldahl method. If a compound with nitro groups is present, phenol is added before the decomposition (Method 1). In the presence of volatile sulfur compounds, oleum is added before the decomposition. If indicated, the two methods can be combined.

Method 1

About 10 to 20 grams of tar are placed in a 2 liter Kjeldahl flask with 250 cc. concentrated pure sulfuric acid, 10 grams phenol and 20 grams catalyst\* and heated one hour on a water bath (reduction). Continue heating carefully with open flame and raise temperature according to gas emission. The mixture clears in a short time and the sulfuric acid is then boiled vigorously for three more hours. After cooling dilute with water; distill off the ammonia as usual, using caustic, and titrate.

When using 10 grams of sample one cc. of .01 N acid is equivalent to a nitrogen content of about 0.001 per cent.

Method 2 (Especially for Low Boiling Hydrocarbons Such as Benzol)



In Vessel A as shown, place about 12 cc. of 10 per cent oleum. A scrubber B is provided to absorb escaping vapors in more oleum. Heat the oleum in A to 30 to 40° C. Then slowly add the sample through the dropping funnel. After 15 minutes ~~heat to about 100° F. for complete sulfonation.~~ Use 100 cc. of conc. H<sub>2</sub>SO<sub>4</sub> to wash all the oleum into a Kjeldahl flask. The Kjeldahl decomposition is started with mild heating and then carried on in the usual manner with addition of 8 gram "Selenium mixture" (900 grams anhydrous Na<sub>2</sub>SO<sub>4</sub>, 15 grams CuSO<sub>4</sub>, 70 grams HgO and 50 grams selenium dust). In the ammonia receiver use 0.1 or 0.02 N sulfuric acid. A blank is run to determine the sulfuric acid consumption of the reagent. (I.G. Analysis-20/25)

\* 720 g. anhydrous Na<sub>2</sub>SO<sub>4</sub>; 56 g. H<sub>2</sub>O; 10 g. Se; 12 g. CuSO<sub>4</sub>.

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Pages 15 and 16.

Determination of Ammonia in Recycle Gas (Gelsenberg-Benzin A.G. -  
Gelsenkirchen Horst 7/26/40)

This concerns the improvement of the sampling method for determining the ammonia from the recycle gas of the 6434 chamber at the Gelsenberg Plant. The main object was to avoid entrainment. The sampling tap was changed from the bottom to the top of the recycle line. It was shown that previous samples had always given excessively high  $\text{NH}_3$  values. The ammonia content of the recycle gas entering the 6434 chamber actually never exceeded the desired maximum value of 10 mg.

Pages 17 and 18

Changing the Method of Determination of Elemental Sulfur in Gaseous Fuel  
(Session of the "Treibgas Vereinigung" on 10/21/1940)

This concerns a change in the approved analysis for elemental sulfur in gaseous fuel. In the old method a sample of gas was withdrawn into a small cylinder and weighed. Then it was conducted into a small flask on a steam bath and evaporated. The residue, containing oil and elemental sulfur, was oxidized with nitric acid and magnesium oxide. The sulfuric acid so formed was precipitated with barium chloride and determined gravimetrically.

It was thought that this method gave high results since more elemental sulfur would be formed due to air in the sample bomb and also that oxidation of the residue would allow harmless sulfur compounds to form sulfuric acid. It was shown, however, that this method usually gave low results due to entrainment in the evaporation step and incomplete formation of sulfuric acid except on long standing.

A new method is proposed in which elemental sulfur is determined as mercuric sulfide. Analyses by this method are usually 50 per cent higher than those by the old method. The values have been proven to be more accurate.

It is suggested that the new method of analysis be adopted and that the allowable elemental sulfur content be raised from 0.10 to 0.15 mg./100 g. fuel gas.

Pages 19 through 22

Method for Determination of Elemental Sulfur in Gaseous Fuel

(1) Principle:

The fuel to be analyzed is transferred from the cylinder to a special Dewar flask, weighed, and evaporated. The residue containing the sulfur is dissolved in benzene and the sulfur converted to mercuric sulfide by shaking with mercury. The mercuric sulfide is decomposed with  $\text{HCl}$ . The  $\text{H}_2\text{S}$  so formed is bubbled into an iodine solution and determined by titration.

R E S T R I C T E D

(2) Apparatus:

(a) Evaporator

The evaporator is a glass cylinder of ca. 50 mm. i.d. and ca. 250 mm. length, with reduced diameter at the top, ending in a ground glass joint. It is jacketed like a Dewar flask, but with connection to a vacuum pump so that it can be used evacuated or air-filled. Before a run, the vessel (jacket air-filled) is immersed in liquid air until cold. By a ground glass jointed tube, the vessel is connected to a small scrubbing flask filled with benzene to make sure all sulfur is scrubbed from the gas.

(b) A shaking cylinder of about 50 cc. content, with wide neck and ground glass stopper.

(c) Decomposing flask

A 150 cc. round bottom flask with short neck and spring-held ground-glass stopper, and an exit tube directly above the bulb.

(d) A 500 cc. Bunsen retort

(3) Procedure:

With the evaporator (jacket airfilled) in liquid air, introduce sample through a liquid-air cooled coil. After the 100 to 150 cc. of gas have been introduced evacuate the jacket, remove the vessel from the liquid air bath, dip in strong alcohol, dry, and weigh.

Connect the wash flask (containing 20 cc. benzene) and begin evaporation with jacket evacuated. Continue evaporation admitting air to jacket slowly, and finish it with vessel in warm waterbath.

Dissolve the residue in the evaporator with the benzene from the scrubber. Pour into the shaking cylinder. Rinse and add rinsings. Total should be 40-50 cc.

Add 2-3 cc. mercury to the cylinder. Shake vigorously 10-15 minutes to convert all S to HgS. Filter through a 7 cm. diameter filter. If the benzene does not come out clear, refilter through the same filter. Take up any HgS in the cylinder with 1 per cent HCl, filter through a special filter, and wash. Hg is removed from the main filter by puncturing with a pin. It can be reused. After brief drying, place the filter papers in the decomposing flask, which is connected with a retort by means of a 25 cc. pipette.

The retort contains a measured volume of 0.01 N  $I_2$  solution, diluted with sufficient water to keep the retort water filled when inverted.

R E S T R I C T E D

Place 50 cc. conc. HCl in the flask, close quickly, and heat to boiling. Permit the first air to escape through the neck of the retort, then push the retort forward to force the gas up in it. Heat until the liquid in the bulb begins to turn brown. If distillation proceeds too long, I<sub>2</sub> may begin to escape from the retort neck. Withdraw pipette slowly after heating is discontinued. Wash the retort content into a 750 cc. Erlenmeyer flask and backtitrate unused iodine with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Calculate the sulfur from the iodine consumption as follows.

$$\frac{0.01 \text{ N Iodine Solution Consumed} \times 0.00016}{\text{Weight of Sample}} = \text{gS/100 g. Gas}$$

The water used must be completely free of nitrate and nitrite. This is checked by adding KI, dilute H<sub>2</sub>SO<sub>4</sub>, and starch solution. If the solution does not turn blue after 10-15 minutes, the water is nitrate free.

Note: Sketch of equipment on the microfilm (pp. 19 and 21).

Page 23

Determination of Moisture in Gases (Gelsenberg-Benzin A.G. -Gelsenkirchen-Horst 11/2/40)

To determine the moisture content of a gas, magnesium nitride seems particularly adapted. The equation is  $\text{Mg}_3\text{N}_2 + 6\text{H}_2\text{O} \longrightarrow 3\text{Mg}(\text{OH})_2 + 2\text{NH}_3$ . Warm, dry air is passed over the magnesium nitride to remove traces of H<sub>2</sub>O. The sample gas is then passed over the nitride and bubbled through 0.1 or 1/50 N H<sub>2</sub>SO<sub>4</sub> to absorb the ammonia. The excess acid is backtitrated. One gas sample yielded the following values:

2.23 mg. H <sub>2</sub> O/100 g. gas	2.72 mg. H <sub>2</sub> O/100 g. gas
2.25	2.17
2.43	3.90
2.73	3.48
2.95	2.34

Pages 24 through 27

Comments on Revised Sulfur Analysis 11-4-40 (Report on Visit by Dr. Sandler at Bochum)

It is reported that the sulfur analysis problem (see p. 17-22 of this microfilm) was investigated because analyses by Scholven and the "Benzolverband" disagreed. For some time, the Scholven fuel gas was high in sulfur and in mercaptans and disulfides. This has been remedied, presumably by doctor treating.

The new sulfur analysis was discussed. It would probably find application only as a referee method, since for plant purposes the colorimetric comparison of HgS precipitate on a mercury surface with the HgS ppt. from known amounts of sulfur is sufficient.

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For the colorimetric test, 50 cc. of benzene containing 0.05, 0.1, 0.15, and 0.5 mg. S are shaken vigorously with 2 cc. freshly added clean mercury. The sulfur content of an unknown can be obtained with fair accuracy by comparison of surfaces with these test samples.

The new test procedure was demonstrated and found much shorter than the old method. Dr. Sandlar has agreed that liquid nitrogen could be used instead of liquid air, and that an Erlenmeyer flask could be used instead of the Bunsen retort.

Page 28

Soda Determination in Slurry-Phase Product (Hy. Werk Scholven 7/6/40)

The method has been changed from steam extracting the product to boiling it in a beaker  $\frac{1}{2}$  hour and filtering. The method and calculations are given to determine per cent soda in solids and per cent soda in raw product.

Pages 29 and 30

Determination of Wet Gas in the "Abstreifer" (Hy.-Scholven 7/26/40)

A new method for thoroughly removing light gases in analyzing Abstreifer liquid yields more consistent results. In the old method (Kg-distillation) the light ends (gasbenzin) consisted of a coldtrap condensate and a smaller amount collected in the receiver, until the overhead temperature reached 35° C.

In the new method the Abstreifer liquid is heated to boiling and distilled through a column with a circulating icewater jacket held at +1° C. The stillpot reaches 100° C. in about an hour. The gases pass an icewater cooler, a small receiver for the higher boiling compounds, and a bubble-counter. They are then condensed in a cold trap at -80° C. This product is designated as FLUESSIGGAS (light ends). This material still contained 10 per cent boiling above +1° C. (off between 23° and 40°). The distillation is stopped when no more gas passes the bubble counter.

The stabilized Abstreifer is then fractionated into gasoline and gas oil in the usual column.

The new thorough stabilization exerts noticeable influence on the ASIM curve of the stabilized gasoline. The I.B.P. of the stabilized gasoline is considerably higher than that from a Kg distillation. Table I presents values from both methods.

R E S T R I C T E D

6434 Gasoline of March 7 and 8, 1940

<u>2 Kg Distillation (Old Method)</u>			<u>1 Kg Distillation (New Method)</u>				
	<u>°C.</u>	<u>°F.</u>	<u>%</u>		<u>°C.</u>	<u>°F.</u>	<u>%</u>
Light Ends	+35	95	14.5	Light Ends	+1	34	11.7
Gasoline	+155	311	49.7	Gasoline	+155	311	53.0
Gas Oil	166-278	331-532	35.0	Gas Oil	163-256	325-493	35.0
Loss			0.8	Loss			0.3
			100.0				100.0
Gasoline d <sub>15</sub>			0.721	Gasoline d <sub>15</sub>			0.719
A.P. (Aniline Point?)			+52° C.	Aniline Point			+53° C.

ASTM Distillation

<u>43° C.</u>	<u>109° F.</u>	<u>I.B.P.</u>		<u>48° C.</u>	<u>118° F.</u>
2.5%		50° C.	122° F.	1.0%	
9.5%		60°	140	8.5%	
21.0%		70°	158	26.0%	
34.0%		80°	176	43.0%	
48.5%		90°	194	57.5%	
62.0%		100°	212	70.0%	
84.0%		125°	257	89.0%	
90.0%		140°	284	94.0%	
93.0%		150°	302	96.0%	
95.0%		160°	320	97.0%	
		163°	325	98.0%	
97.0%		170°	338		
97.5%		172°	342		
1.0%		Bottoms		1.0%	
1.5%		Loss		1.0%	

Page 31

Organic Sulfur in Propane and Butane and Isobutane from Gaseous Fuel  
(Gelsenberg Benzin 11/1/40)

Sulfur was determined by burning, after rough distillation and Podbielniak redistillation of the fractions. A table is given with the results from two samples.

Page 32

Determination of Cyanide by Means of Polysulfide Solution

This is a short summation of the analysis previously presented on p. 8 and 9 of this microfilm.

Determination of Water in Propane-Butane Fraction (Gelsenberg Benzin A.G.)

Equipment for Purification of Oxygen

- 1 drying tower with  $\text{CaCl}_2$
- 1 U-tube with  $\text{P}_2\text{O}_5$
- 1 U-tube with magnesium perchlorate

Equipment for Analysis

- 1 "Andreas Hofer" bomb
- 1 Oil bath
- 1 U-tube with calcium carbide
- 6 Test tubes with Ilosway's reagent
- 1 Hot plate

Required Reagents

$\text{Fe}_2(\text{SO}_4)_3$  solution

100 g.  $\text{Fe}_2(\text{SO}_4)_3$ , 200 g.  $\text{H}_2\text{SO}_4$ , made to 1.1 with  $\text{H}_2\text{O}$

Ilosway's Reagent

5 g.  $\text{Cu}(\text{NO}_3)_2$ , 20 cc. 21%  $\text{NH}_4\text{OH}$ , 15 g. hydroxylamine hydrochloride; made to 250 cc.

0.10 N  $\text{KMnO}_4$  solution

Procedure:

Heat the oil bath containing the calcium carbide U-tube to  $160^\circ \text{C}$ . and sweep out with completely dry oxygen until no more copper acetylide precipitates. Weigh the gas sample in the bomb. Interrupt the oxygen stream and slowly pass the propane-butane stream over the calcium carbide. (Use 3-way stopcock.) The water in the fuel gas is converted to acetylene and precipitates as copper acetylide. After sufficient ppt. has formed, the stream is turned off and the gas remaining in the bomb weighed. The apparatus is flushed with dry oxygen until no more copper acetylide forms. Filter the ppt. through a fritted glass filter and wash with ammoniacal hydroxylamine hydrochloride. Rinse with distilled water until chloride free and dissolve in 40 cc. ferric sulfate solution. Titrate with 0.1 N  $\text{KMnO}_4$  until just red.

1 cc. 0.1 N  $\text{KMnO}_4$  is equivalent to 1.8 mg.  $\text{H}_2\text{O}$

R E S T R I C T E D

Pages 34 through 36

Notes on the Acetylene Content in Gases at the Linde Plant of Chemical Works Huels (Gelsenberg Benzin 5/22/42)

The explosion in No. II air separator (Lufttrenner) at the Gelsenberg Linde installation gave occasion for comparing acetylene concentration values at Gelsenberg and Huels.

It is shown that acetylene concentrations in the acetylene separator (Acetylenabschneider) and in the main condenser at Huels were often abnormally high, as much as 100 and 200 per cent above the given danger limit. One of the highest values was 3.127 C<sub>2</sub>H<sub>2</sub> p.m. which is over 200 per cent of the given danger limit and was found in the acetylene separator of App. III at Huels. At Gelsenberg, values are always considerably lower.

Maximum values found at Gelsenberg are 0.02 C<sub>2</sub>H<sub>2</sub> ppm. at the main condensers and 0.7 C<sub>2</sub>H<sub>2</sub> at the acetylene separator.

Huels does not analyze the oxygen fraction for hydrocarbons or the helium fraction for hydrogen. There seem to be no difficulties experienced, which is the more surprising since the odor of acetaldehyde was noticeable throughout the plant.

As for the explosion of the Gelsenberg air separator II, high acetylene concentration does not seem responsible, but it may be due to a dead end section in the reactor in which acetylene enrichment can take place, which would then be set off by liquid oxygen or other compounds.

Pages 136 and 137

Notes Concerning Discussion of Acetylene Contents of the LINDE Installation of "Hydrierwerk Scholven" (Discussion at Scholven on 5/22/42) (Written at Gelsenkirchen-Horst on 5/23/42)

It was found that Scholven does not make quantitative analyses of acetylene in liquid oxygen from the Linde installation. Acetylene is determined qualitatively with ammoniacal silver nitrate, and reported as trace, average, or high. According to this analysis, more or less liquid is taken out of the various air separators. This ranges up to 50 l./hr.

Air analyses for acetylene are not undertaken, and the liquid in the air separators has not been examined for hydrocarbon or the helium fraction for hydrogen, since 1936. The entering gases are also not examined for lube oil. Scholven leaves an air separator on stream 60 days after which it is thawed and blown down. If sufficient lube oil has collected after a year's operation, the equipment is rinsed thoroughly with trichloroethylene, after thawing.



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Pages 37 through 40

Colorimetric Determination of Nitrous Acid - Peter Gries Method

From F. P. Treadwell, Analytische Chemie, Vol. II, p. 285

This is also to be found in Treadwell and Hall, 9th Ed., Vol. II, p. 306

Pages 41 and 42

Determination of Paraffin in Asphalt

Weigh ca. 20 g. asphalt in an Erlenmeyer flask and dissolve in 30 cc. benzene with mild heating. Place in a 500 cc. or larger separatory funnel containing 300 cc. ordinary gasoline, and wash out the flask with an additional 100 cc. of gasoline. Refine by adding 30 cc. conc.  $H_2SO_4$  and shaking vigorously. Hard asphalts will settle out after 1 to 2 hours standing, often as a solid mass. Decant the solution and wash out the beaker with water. (Sulfonated products are relatively water soluble.) Repeat the acid treat two more times.

Neutralize by shaking with NaOH in 50 per cent alcohol; water wash. Proceed carefully to avoid forming emulsions. Place gasoline solution into distillation flask and distill to low bottoms. Place bottoms in a weighed dish and evaporate the rest of the solvent first at  $80^\circ$ , then at  $120^\circ C$ . Weigh the oily residuum.

Place an aliquot portion in a 20 cc. distillation flask and distill rapidly so that the total distillate comes over in 5 minutes. Dissolve the distillate in ether and slowly add an equal volume of alcohol. Chill at  $-20^\circ C$ . for  $\frac{1}{2}$  hour and filter off the paraffin.

Page 135

Acetylene Determination in Gas

Method:

The gas is passed through Ilosvay's reagent. A red-brown ppt. of  $Cu_2O$  forms. The ppt. is filtered and washed with dilute hydroxylamine solution until the Cu reaction is negative. Then wash with dist. water until chloride free. Decompose the ppt. with  $HNO_3$  and heat at red heat.

Ilosvay's Reagent:

1 g.  $Cu(NO_3)_2$  (or  $CuCl_2$  or  $CuSO_4$ ) is dissolved in a small amount of water in a 50 cc. volumetric flask. 4 cc. of conc.  $NH_4OH$  are added and then 3 g. hydroxylamine hydrochloride. Shake until deep blue color is lost, and dilute to volume.

R E S T R I C T E D

Calculation:

Weight CuO x 0.1634 = mg. C<sub>2</sub>H<sub>2</sub> in the measured amount of gas. This is converted to mg./m<sup>3</sup> or to mg./l. of liquid.

If result is to be reported as volume per cent C<sub>2</sub>H<sub>2</sub>:

$$1 \text{ g. CuO} \approx 140.8 \text{ cc. C}_2\text{H}_2$$

$$39.79 \text{ g. CuO} \approx 26 \text{ g. C}_2\text{H}_2$$

$$1 \text{ g. CuO} \approx 0.1634 \text{ g. C}_2\text{H}_2$$

$$26 \text{ g. C}_2\text{H}_2 \approx 22.400 \text{ cc.}$$

$$0.1634 \text{ g. C}_2\text{H}_2 \approx 1 \text{ g. CuO} \approx 140.8 \text{ cc. (at 760 mm. and 0°)}$$

(Note: I.G. uses not 22.4 but 24.4 l. per mol of gas for its calculations.)

:art

## Asphalt Analysis (Hydrierwerk Poelitz 4/10/42)

### Introduction

Bottoms from the coal liquefaction chambers, or other oils, may contain foreign matter in real or colloidal solution. The following paper considers which of these components may influence the oil yield during coking. The amount of benzene-insolubles determines the oil yield on coking, but there remain other materials in the oil after removal of these solids, which may lower the oil yield. Since the chemical structure of these materials is unknown, our efforts are limited to determining the total amount quantitatively and correlating it to the oil yield. The total of this material present in the oils from which the benzene insolubles have been removed is called total asphalt (Gesamtasphalt); this term has no significance, however, concerning the chemical properties of this material. A certain fraction of the total asphalt, characterized only by its analytical determination, is called partial asphalt (Teil asphalt).

### Determination of Total Asphalt

About half a gram of the sample is treated with 30 cc. benzene and the benzene insolubles filtered off. The filtrate is evaporated to 10-20 cc., and this amount is poured through a specially prepared alumina layer in a crucible. The alumina is rinsed with 200 cc. benzene. The weight of material which remains absorbed is termed the total asphalt.

### Determination of Partial Asphalt

The partial asphalt is determined exactly like the total asphalt, except that the wash solution consists of 200 cc. of ether.

### Rapid Determination of Total and Partial Asphalt

Rapid determinations are made by preparing standard samples of alumina with various amounts of "asphalt" absorbed on them, and using color comparison instead of the longer gravimetric method.

The above analytical methods appear in considerably more detail in the film.

A number of sample analytical results are given.

A mathematical derivation is made to permit calculating the expected oil yield on coking from the results of such an analysis, and the method is applied to a number of plant samples from several sources.

### Summary

Analytical methods were worked out for determining total and partial asphalt.

The partial asphalt is identical with asphalt values obtained by the benzene-precipitation method. (A hand-written footnote challenges this conclusion.)

The total asphalt values only are used in equation 1 to 3 and determine the oil yield during coking.

It is possible to determine the theoretical coking yield from equations given in the paper. (A big handwritten question mark in the margin opposite this statement.)

Approximation equations are as follows:

$$g \text{ oil from } 100g \text{ bottoms (Abschlaman)} = 100 - \left( \frac{\text{benzene insolubles}}{\text{total asphalt}} \right)$$

This equation is valid when the volatile components produce only gas during the coking of benzene insolubles plus total asphalt. Another equation is given for oil having a coking residuum.

Results obtained using these equations are applicable only in the absence of catalysts or other substances which can cause the conversion of asphalt to liquid products.

Finally it is mentioned that the above method of analysis takes five hours while the benzene precipitation method takes 20 hours. (This statement also is accompanied by a question mark in the margin.)

The equipment used for asphalt determination is sketched.

#### Appendix I:

Correlation of results from the above method with actual plant results.

#### "3d Communication"

Summary report on the work carried out at Scholven to produce a suitable method of asphalt analysis

Correlation of experimental results with operating data.

The following methods of asphalt analysis and their relative merits and correlation with plant data are discussed in considerable detail:

(1) S-Asphalt determination; (This seems to be the standard accepted method.)

(2) First method suggested by I. G. Ludwigshafen.

The pyridin-CS<sub>2</sub> soluble material is resolved into neutral oil, oil-resin resin-forming material, asphalt resins, asphaltenes, carbones, and carboids by suitable solvents, precipitants, and adsorbents. This method is not described in more detail. It is said to have produced poor results, probably due to lack of exact instructions.

(3) The Scholven modification of the method of Suida and Motz.  
(Petroleumzeitschrift 35, 527, (1939)).

This method is described in some detail. In this method the total product and not only the pyridin-CS<sub>2</sub> soluble is analyzed. The following fractions are determined:

- (a) CHCl<sub>3</sub> solubles at 20°
  - (a<sub>1</sub>) Neutral oil
  - (a<sub>2</sub>) Oil resins (pptd. with gasoline)
- (b) Pyridin solubles at 20°
  - (b<sub>1</sub>) Asphalt resins
- (c) Pyridin solubles at 50°
  - (c<sub>1</sub>) Hard asphalt
- (d) Pyridin-CS<sub>2</sub> solubles at 35°
  - (d<sub>1</sub>) Asphaltenes, carbenes, carboids
- (e) Pyridin-CS<sub>2</sub> insolubles

(4) New method for asphalt analysis, suggested by Scholven

The following are determined by this method:

- (a) Ash
- (b) Ash free pyridin insolubles
- (c) A-asphalt (hardest-to-dissolve asphalt) = chloroform insolubles - pyridin insoluble.
- (d) B-asphalt = benzene insoluble - chloroform insoluble
- (e) C-asphalt (easiest-to-dissolve asphalt) = CCl<sub>4</sub> insoluble - benzene insoluble
- (f) Oil resins = CCl<sub>4</sub> solubles - neutral oil
- (g) Neutral oil

Pages 74-85

Remarks on Fuel Analysis (Dr. Seibert Gelsenkirchen-Horst, 9/15/43)

This paper is in explanation of a standard method of calculation of heat values of solid fuels, as given in "Normblätter DIJ DVM 3716 and 3721 of the Deutsche Verband Für die Materialprüfung der Technik" (German ASTM) and in a treatise by Dr. Seibert, entitled "Bestimmung und Umrechnung von Heizwerten fester Brennstoffe," published in 1939.

Item No. 8 Analytical Methods

Page 187

Analytical methods for charge, intermediates, and end products of the high pressure hydrogenation in Ludwigshafen and Oppau. (The large majority of the analytical methods in this section are described in considerable detail, some with sketches of special apparatus included. Further literature references are also given in many cases.)

Pages 188-228

I Coal

A. Elemental Analysis

1. Carbon and Hydrogen

Determination according to Reihlen and Weinbrenner (Automatische Elementaranalyse, Chem. Fab. 7, 63 (1934)).

2. Nitrogen

Nitrogen determination may be carried out by several methods

Acc. to Dumas: Combustion over CuO

Acc. to Ter Meulen: Hydrogenation of the nitrogen over selenium catalyst, and titration of the NH<sub>3</sub>

Acc. to Kjeldahl: Wet decomposition with H<sub>2</sub>SO<sub>4</sub>

B. Proximate Analysis

1. Moisture Determination

a. "Coarse" Moisture

To prepare for analysis, the coal is reduced to 10 mm. grain size, spread out on tin pans and exposed to a mild air current for 48 hours. The weight loss is "Coarse" moisture.

b. Hygroscopic Moisture

Loss of weight of the fine-ground coal after two hours in a drying oven at 105° C.

2. Ash Determination

1 g Coal ashed in a quartz bowl in a muffle furnace. Ash analyzed for:

a. Silicates

b. Melting Point of Ash (Method described in film)

### 3. Coke Yield Determination

1 g Powdered coal in crucible heated in electric furnace to 850° C. for seven minutes. Residue weighed.

### 4. Volatile Matter

$$VM = 100 - (\text{moisture} + \text{ash} + (\text{crude coke} - \text{ash}))$$

### C. Sulfur

May be present as:

1. Pyrites or marcasite
2. Sulfate
3. Organic bound sulfur

A distinction is also made between combustible (or volatile) sulfur and residual sulfur in the ash.

#### Total Sulfur

By combustion with "Eschka" mixture (2 parts burned magnesia, 1 part anhydrous  $\text{Na}_2\text{CO}_3$  (pure)) in an oxygen stream in a quartz tube. (Equipment and method given in detail.)

#### Inorganic Sulfur

##### a. Pyrites

1 Kg Coal and 80 cc. dil.  $\text{HNO}_3$  (1:4) stored 2-3 days at room temperature, filtered, evaporated, and residue dissolved in HCl.

Fe: With  $\text{KMnO}_4$  or colorimetrically

$\text{SO}_4$ : With  $\text{BaCl}_2$

##### b. Sulfate

5 g Coal and 300 cc. 3% HCl digested 40 hours at 60° C., filtered and Fe and  $\text{SO}_4$  determined from this solution.

#### Volatile Sulfur

Determined by combustion in oxygen stream.

### D. Analytic Methods

All of the following methods are described in considerable detail. The page numbers are those appearing actually on the film.

1. Coal Sampling, page 7
2. Moisture Determination of the Coal

- a. With Xylene, page 8

From "Angew. Chemie" 27, 52 (1914) and DIN DVM 3721

- b. With Tetrachloroethane, page 9

For very low moisture content. From "Angew. Chemie" 39, 155 (1926)

- c. With Stream of Dry N<sub>2</sub>, page 9

3. Ash Determination, pages 11-12

4. Nitrogen Determination, acc. to Kjeldahl, pages 13-14

This method is not claimed as a precision method, Bibliography given for more precise methods.

5. Sulfur Determination

- a. Total Sulfur

- (1) Acc. to Eschka, pages 15-18

- (2) Combustion in a tube, pages 18-19

- b. Inorganic Sulfur

- (1) Pyrite sulfur, page 19

- (2) Sulfate sulfur, pages 19-20

6. Coke yield and Volatile Constituents of Coal, page 20

7. Coking Analysis Acc. to Fischer, pages 21-22

8. Specific Gravity of Coal, pages 23-24

9. Preparation for Microscopic Observation,

~~and Results of Microscopic Observation, pages 25-28.~~

10. Screen Analysis, pages 29-31

Note: Page 31 gives a correlation of German and American screen mesh numbers.

11. Deashing



For Hard Coal

- a. Lessing process
- b. Sophia-Jakoba process
- c. Chance process

For Soft Coal

- a. Base exchange with  $NH_4Cl$
- b. Chemical deashing with 10% HCl
12. Suspension Analysis (Settling Speed), page 34
13. Flotation, page 35
14. Swim-and-Sink Process, pages 36-37

In this process, liquids of various densities are used to resolve the finely ground coal (through 10,000 mesh/cm<sup>2</sup>) into components of various densities.

15. Sand Determination in Coal, page 38

By the swim-and-sink process.

16. Alkalinity of Coal, page 39
17. Iodine Number, page 40
18. Acid Number, page 41
19. Saponification Number, pages 41-42

Pages 229-237

II. Coal Paste and Grinding Oil

1. Water Determination, page 43

See Xylene method for coal.

2. Solids Determination (Benzene Insolubles), page 43
3. Ash in Solids, pages 43-44
4. Obtaining Oil Free of Solids and Specific Gravity, pages 44-45
5. Asphalt Determination, pages 45-46

6. Engler Distillation (By Weight), page 46
7. Settling Determination, page 47
8. Softening Point Acc. to Kramer-Sarnow, pages 47-48
9. Screen Analysis of Coal Paste, page 48-49
10. Wax Determination in the Oil, pages 49-50

Method described in Holde, 7th Ed., page 170, *ibid*, page 419 consists of distillation, using the 300° C. - coking temperature cut for a wax determination with 1:1 ether-alcohol at -21° C.

Pages 238-259

### III. Coal Abstreifer Products

#### 1. Water Determination

Analogous to xylene method for coal.

#### 2. Determining Specific Gravity

#### 3. Engler Distillation (By Weight)

#### 4. ASTM Distillation

#### 5. Aniline Point

#### 6. Unsaturation

Using absorption in 92%  $H_2SO_4$  for gasolines and in 90%  $H_2SO_4$  for gas oils.

#### 7. Phenol Analysis

By extraction of the oil with 10% NaOH, removal of oil from the NaOH by ether, acidifying the NaOH and separating the phenol layer, removing the last traces of phenol from the aqueous layer with ether, combining the phenol and phenol containing ether, drying with  $CaCl_2$ , filtering, driving off the ether, and weighing the residual phenol.

#### 8. Simplified Analysis of the Coal-Abstreifer

This is a combination of distillation and analysis of cuts, known as "Kleinuntersuchung" which is carried out on about 2.5 Kg of sample and is intended for the routine analysis of daily plant samples.

#### 9. Detailed Analysis of the Coal Abstreifer

- a. Fractionation  
Three fractions: Crude gasoline (170° C. cut point)  
Crude gas oil (170° - 325° C. " )  
Residuum above 325° C.
- b. Workup of the Gasoline Fraction
- c. Workup of the Gas Oil Fraction
- d. Workup of the Residuum

10. Abstreifer Water

- a. H<sub>2</sub>S Determination
- b. NH<sub>3</sub> Determination

Pages 260-266

IV. Hydrogenation Residue (Abschlamm)

- 1. Specific Gravity
- 2. Softening Point, Acc. to Kramer-Sarnow
- 3. Asphalt Content of the Residue
- 4. Viscosity Determination
- 5. Filtration Time
- 6. Solids Determination with recovery of the oil
- 7. Screen Analysis
- 8. Microscopic Examination

Note: Catalyst shows up in polarized light.

Pages 267-316

V. Gasoline Analysis

The following gasoline tests were already discussed under Section III.

Specific Gravity

Engler Distillation

ASTM Distillation

Aniline Point (I and II)

Phenol Determination

The following additional tests are used:

1. Loss of Gasoline on Refining

Volume loss upon treatment with 96%  $H_2SO_4$ .

2. Copper Dish Test

3. Glass Dish Test

4. Copper Strip Test

5. Qualitative Test for  $H_2S$  and Mercaptans - Doctor Test

6. Unsaturation by  $H_2SO_4$  Absorption

7. Calculating Gasoline Composition

Unsaturation (absorption in 92%  $H_2SO_4$ )

Aromatics (by aniline point method)

Naphthenes (from aniline points and unsat. analysis)

Paraffins (by difference)

8. Refractive Index ND

9. Small Amounts of Phenol

Colorimetric method of Folin-Denis.

10. Air Stream Method for Preformed Gum

11. Refining of Gasoline (Propane- $SO_2$  Extraction)

Includes considerable detail on workup of extract and raffinate.

12. Sulfur in Oils and Gasolines (Ludwigshafen Method)

By combustion to form  $H_2SO_4$  in a  $H_2O_2$  stream over platinum catalyst, and absorbing the  $H_2SO_4$  in 3%  $H_2O_2$ , neutralized with NaOH.

13. Chlorine in Oils and Gasolines, by combustion (Ludwigshafen method)

Same combustion equipment as for the above sulfur determination, but using 4% NaOH in the receiver.

14. Bomb Test (Storage Stability)

15. Reid Vapor Pressure

## 16. Octane Number

In the CFR or the IG motor.  
Both Motor and Research method.

Includes a number of graphs with correlations of octane number determinations by the various methods and other variables.

Pages 317-392

## VI. Gas Analysis

### Sampling

This includes a fairly detailed discussion of the various possible sealing liquids: mercury, water, aqueous solutions, and organic liquids.

Discussion of equipment:

Simple absorption vessels: Hempel pipettes or various modified pipettes are used.

Combination analytical apparatuses: Orsat apparatus is used with up to seven absorption pipettes. Hydrogen and hydrocarbon combustion is performed over  $\text{CuO}$  in quartz or NTC tubes. Gaseous hydrocarbons are analyzed by Podbielniak distillation after first being slowly bubbled through  $\text{KOH}$  for removal of  $\text{H}_2\text{S}$  and  $\text{CO}_2$ , then dried through  $\text{CaCl}_2$ , and having the condensables removed with liquid nitrogen. The condensables are then analyzed by Podbielniak distillation or in the Stack apparatus which utilizes the principle of fractional condensation.

### Analysis

#### 1. Absorptiometric and Combustion Method

##### a. Technical Gas Analysis, as follows:

$\text{CO}_2$  - in 50%  $\text{KOH}$   
Unsats - in bromine water (before reading volume loss, bromine vapors are absorbed in  $\text{KOH}$ )  
 $\text{O}_2$  - by phosphorus or alkaline pyrogallol  
 $\text{CO}$  - cuprous chloride (alkaline or in dil.  $\text{HCl}$ )  
 $\text{H}_2$  - by combustion over  $\text{CuO}$   
Saturated  $\text{HC}^{\text{S}}$  - combustion over  $\text{CuO}$  at red heat  
Average Carbon No. - from combustion data.

It is noted that the best  $\text{O}_2$  absorbent is chromous chloride solution.

##### b. Exact Gas Analysis

This was carried out with a special Orsat apparatus consisting of two measuring burettes, several absorption pipettes, and a 20 cm. combustion tube of NCT-3 material. The pipettes were filled as follows:

- 1st Pipette: 30% KOH for absorbing CO<sub>2</sub>
- 2nd Pipette: 75% H<sub>2</sub>SO<sub>4</sub> for absorbing butylone  
(Very temperature sensitive, since propylene is also absorbed)
- 3rd Pipette: 87% H<sub>2</sub>SO<sub>4</sub> for propylene absorption  
(An interpolation correction is necessary since ethylene is also absorbed)
- 4th Pipette: AgSO<sub>4</sub>-H<sub>2</sub>SO<sub>4</sub> for absorbing ethylene  
(This also absorbs CO from gases containing over 6% CO.)
- 5th Pipette: Fuming sulfuric acid (25% SO<sub>3</sub>) for heavier hydrocarbons.
- 6th Pipette: Alkaline pyrogallol for absorbing O<sub>2</sub>
- 7th Pipette: Ammoniacal CaCl<sub>2</sub> for absorbing CO  
(Two of these are used, since CO is absorbed so slowly.)
- 8th Pipette: Cuprous oxide-H<sub>2</sub>SO<sub>4</sub>-β naphthol for quick absorption of the remaining CO.
- 9th Pipette: Iodine pentoxide-fuming sulfuric acid for absorbing propane and butane.

2. Fractionation (Podbielniak distillation)
3. Fractional Condensation, Stock gas analysis method.
4. Missing
5. Titrimetric Methods

Absorption of H<sub>2</sub>S, CO<sub>2</sub>, and NH<sub>3</sub> in KOH and H<sub>2</sub>SO<sub>4</sub>.

The various process gases from the hydrogenation process, the methods of sampling and of analyzing them are discussed.

Determination of CO<sub>2</sub>, H<sub>2</sub>S, and NH<sub>3</sub>

H<sub>2</sub>S - treatment of the scrubber liquid with CdCl<sub>2</sub> and iodometric determination of the sulfide

CO<sub>2</sub> - treatment of the scrubber liquid with excess of hot 10% BaCl<sub>2</sub> to precipitate barium carbonate, filtering, redissolving the ppt. in HCl, boiling, and titrating to the methyl red end point.

NH<sub>3</sub> - driven out of alkaline solution and absorbed in H<sub>2</sub>SO<sub>4</sub> and titrated to methyl orange end point.

## Recovery of Light Hydrocarbons ("Gas-benzin")

Activated carbon is used for this purpose.

### Special Tests

#### 1. Carbonyls

##### a. Qualitative test for iron carbonyl:

Testing for metal precipitate from a test flame on a cold surface.

##### b. Qualitative test for iron carbonyl and nickel carbonyl:

Scrubbing through a solution of 90 cc. MeOH, 5 cc. H<sub>2</sub>O<sub>2</sub>, 5 cc. NH<sub>4</sub>OH · Fe(OH)<sub>3</sub> and CO<sub>2</sub> are formed. Fe and Ni are determined from the precipitate.

#### 2. Acetylene Acc. to Ilosway

The gas is passed through freshly prepared Ilosway's reagent (ammoniacal cuprous salt solution plus hydroxylamine hydrochloride). Acetylene causes a blood-red precipitate, allylene a yellow precipitate. O<sub>2</sub>, NO<sub>2</sub>, and H<sub>2</sub>S interfere. For oxygen-containing gases a 1% ammoniacal silver nitrate solution is recommended. The Ilosway method is covered in considerable detail.

#### 3. Determination of COS, CS<sub>2</sub>, and Mercaptans

The method was developed by the Oppau analytical laboratory (Report No. 1522, August 22, 1938 "Untersuchung von Saarferngas").

The gas is first passed through a tube of soda lime. The components are then determined in three parallel systems:

COS: The gas is passed through two bulbs with cobalt acetate to remove H<sub>2</sub>S; then through a CaCl<sub>2</sub> drier; a gas meter, a freezeout vessel with 10 cc. absolute alcohol cooled to -80° (to remove C<sub>6</sub>H<sub>6</sub>, thiophene, higher mercaptans, thioethers, CS<sub>2</sub>, etc.) and two scrubbers with MeOH-H<sub>2</sub>O-KOH. CO<sub>2</sub>S · (CH<sub>3</sub>)<sub>2</sub>K is formed, which is oxidized with "Perhydrol" (Trade name, probably for hydrogen peroxide). The solution is acidified and the sulfate determined as BaSO<sub>4</sub>.

CS<sub>2</sub>: The gas is passed through two bulbs with cobalt acetate to remove H<sub>2</sub>S; through a CaCl<sub>2</sub> drier; and then through two scrubbers with piperidine in monochlorobenzene, and a gas meter. CS<sub>2</sub> forms a piperidine compound which reacts with copper oleate to form an intensively brown copper salt which is used for colorimetric determination.

Mercaptans: After washing through cobalt acetate and a gas meter, the gas is passed through 0.1N mercuric chloride solution. The precipitate is determined gravimetrically.

#### 4. Organic Sulfur in Gases

Organic sulfur is determined by combustion with oxygen over a platinum catalyst in a quartz tube. The method is described in detail.

Pages 339-454

#### VII. Special Analytical Methods

##### A. Elemental Analysis

1. C-H Determination (Semimicro-and Micro combustion).
2. Determination of Volatile sulfur (combustion method)
3. Nitrogen Determination
  - a. Acc. to Dumas

Reference is made to Oppau Laboratory Report No. 1524 of June 3, 1938, by Dr. F. Grassner "Apparatur zur automatischen Mikro-Stickstoff-Bestimmung nach Dumas mit Hilfe von Synchronmotoren." The equipment is redescribed in the film.

##### b. Acc. to ter Meulen

This method is suitable for gasolines, oils, and other liquids. The method consists of catalytic hydrogenation over Nickel-magnesia catalysts. The catalyst preparation is given.

##### c. Acc. to Kjeldahl with Selenium Catalyst

This method is particularly suitable for tars and oils. This method is also described in pages 13 and 19 at the beginning of this microfilm.

##### B. Bromine Number

##### C. Iodine Number (Reference: Holde, page 764)

##### D. Acid and Saponification Number

The determination of micro acid and saponification numbers is also given.

The micro acid number determination consists merely of dissolving 5-20 mg. of sample in 5 cc. methyl alcohol (neutralized), heating if necessary, and titrating with 0.1N KOH, using phenolphthalein indicator.

The micro saponification number is determined by weighing out 5-20 mg. sample in a platinum dish, dissolving it in 5 cc. 0.1N alcoholic KOH in an Erlenmeyer with ground glass top, boiling  $\frac{1}{2}$ -1 hour with reflux, and backtitrating after cooling with 0.1N  $H_2SO_4$  or HCl. A blank run is to be made.



#### E. Hydroxyl Number (pyridine method)

All hydroxyl groups from alcohols and carboxylic acids are determined by this method. The pyridine method is generally to be preferred to the benzene method.

The sample is acetylated with a 1:3 mixture of acetic anhydride and pyridine, heated at 95° C. under reflux for 1 hour, and backtitrated with alcoholic KOH.

#### F. Hydroxylamine Number

The hydroxylamine number determines the carbonyl groups. The CO group in aldehydes and ketones is determined; COOH groups do not interfere.

The reagent consists of:

8 g Hydroxylamine hydrochloride  
16 cc. H<sub>2</sub>O  
160 cc. Methyl alcohol  
120 cc. 0.5N Alcoholic KOH  
2 cc. Bromphenol blue (0.1 g/100 cc. methyl alcohol)

0.5 g sample and 50 cc. reagent are boiled one hour, cooled, and titrated with 0.5N H<sub>2</sub>SO<sub>4</sub> after addition of a drop of bromphenol blue.

A blank is run with each determination.

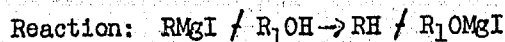
The hydroxylamine number is the mg. hydroxylamine hydrochloride consumed per gram of sample.

#### G. Alcohol Determination Acc. to Fischer

Addition of NO causes the formation of the alcohol-nitrite. The nitrite is volatilized out of the solution with a CO<sub>2</sub> current, washed with saturated NaHCO<sub>3</sub> solution, and bubbled into an acidified (HCl) potassium iodide solution. The iodine is titrated with 0.1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>; aldehydes and ketones do not interfere.

#### H. Determination of Active Hydrogen Acc. to Tschugaeff and Zerewitnioff

All hydrogen replaceable by metal is determined, including carbonyl hydrogen, alcoholic hydrogen, phenolic hydrogen, and amid and imid hydrogen.



~~The volume of gas liberated by the reaction is used to calculate the amount of OH present.~~

#### I. Determination of Methoxy and Ethoxy Groups

Methoxy and ethoxy compounds split off methyl or ethyl iodide when boiled with HI. The volatile iodides are removed with a CO<sub>2</sub> stream and collected in

$\text{AgNO}_3$ . The double salt  $\text{AgI} \cdot \text{AgNO}_3$  is formed. By addition of nitric acid the salt is split into  $\text{AgNO}_3$  and  $\text{AgI}$ , and the  $\text{AgI}$  determined gravimetrically.

J. Determination of Acetyl-(Benzene-) Groups Acc. to Kuhn and Roth

The acetyl number gives the mg KOH required to neutralize the acetic acid formed in the saponification of 1 g of acetylated substance.

The sample is saponified with  $\text{Ba}(\text{OH})_2$ , acidified with phosphoric acid, and the acetic acid distilled off with steam.

K. Halogen Determination

Nephelometric method, for halogen contents below 0.01%. This is a combustion method with the combustion similar to that for sulfur determination. The chloride is received in NaOH, the solution neutralized, filtered, and the filtrate added to  $\text{AgNO}_3$  and determined nephelometrically.

L. Determination of Traces of Water

Water is determined by the Karl Fischer method.

M. Improved Colorimetric Heating Value Determination of Liquid Fuels in Platinum Vessels

N. Analysis for Tetraethyl Lead

The Oppau method is used for this analysis. The lead is removed as lead bromide by the addition of the sample to a bromine-gasoline mixture. The lead bromide is extracted with water and determined, conveniently, as  $\text{PbCrO}_4$ .

O. Determination of Volatile Sulfur

1:1 HCl is added to the sample and the  $\text{H}_2\text{S}$  formed is driven out with nitrogen, absorbed in cadmium acetate and the sulfide determined.

Pages 455-474

VIII. Analysis of Raw, Drinking, Boiler, and Sower Water

A. Description of Water Treatment for Drinking Water

B. Analytical Methods

1. Alkalinity

2. "Eindickungszahl"

This is the ratio of the salt content of the boiler water to that of the drinking water.

### 3. Phosphates in Boiler and Drinking Water

A colorimetric method using molybdenum sulfonate (Sulfmolybdän) reagent to give a blue color after addition of tin foil.

### 4. Hardness Determination

The soap solution method of Bautron and Boudet is used.

### 5. Oxygen

The sample is caught air free, NaOH and manganous chloride are added, and shaken.  $\text{KHCO}_3$  is added and the sample is allowed to stand in the dark, and then filtered. Filtration is only necessary when  $\text{SO}_3$  is present in the solution. The precipitate is dissolved in 50 cc. 2:1 phosphoric acid containing a crystal of KI. Starch solution is added and the liberated iodine is titrated with 0.01N  $\text{Na}_2\text{S}_2\text{O}_3$  solution.

### 6. Total $\text{CO}_2$ in Raw, Drinking, and Boiler Water

The  $\text{CO}_2$  is removed from the water by a  $\text{CO}_2$  free air stream passed through the acidified water, and the  $\text{CO}_2$  is absorbed in  $\text{Ba}(\text{OH})_2$  solution, and determined by backtitration with oxalic acid.

### 7. Phosphate Determination, Quick Method with Strychnine

The time required for cloudiness or precipitation to appear in water after addition of the reagent indicates the amount of phosphates present. The literature is referred to for further details. The reference is given in the film at the end of the section.

### 8. Iron Determination

A photometric method using  $\text{NH}_4\text{Cl}$ , sulfosalicylic acid, and ammonium hydroxide.

### 9. Silicic Acid Determination

A photometric method using molybdenum sulfate reagent, hydroquinone solution, and carbonate-sulfite solution.

### 10. Carbonate Hardness

RESTRICTED

Technical Oil Missions  
Index Reel 7, Bag 2732, Target 30/4.11 Bottrop

Item No. 1 Catalyst Research

Pages 476-479

(A) Methods of Storing High Pressure Hydrogenation Catalyst for Air Raid Protection (5-17-41)

Directions are given for the air raid protection storage of catalysts in industrial use, since they all contain irreplaceable rare metals and since they are destroyed by radiant heat, water, and air. Storage in unused high pressure furnaces is recommended for the most valuable catalysts and in deep dry cellars for the others. No more than about 25 cubic meters (880 cubic feet) (usually 25 to 50 per cent of the total reserve) are to be stored in one place. Fire protection by CO<sub>2</sub> is preferred.

This is a list of catalysts according to their rare metal content: 5058, 6718, 7745, 6719, 6434, 7846, 7360, 5475, 7019.

Pages 480-483

(B) Experiments for the Hydrogenation of Soft Coal Gas Oil over Ruhroel Catalyst at 500 Atm. (7350 p.s.i.) (5-27-41)

Object: Preparation of aromatic aviation gasoline

In January, 1941, the Ammoniakwerk Merseburg began experiments for the one-step high pressure high temperature hydrogenation (Ruhroel process) of soft coal liquefaction oil to yield highly aromatic aviation gasoline. The catalysts for these experiments were furnished by Ruhroel and the experimental conditions determined in co-operation with Ruhroel.

The first experiment (1/7 - 1/17) was made with a strong hydrogenation catalyst (K-413). The feed was a mixture of equal parts of A-gas oil and B-gas oil from the 5058 chamber. This catalyst proved too active since a gasoline concentration of over 80 per cent was obtained in spite of a feed rate of 2 kg/l/hour.\* With a hydrogen partial pressure of 450 atm. (6600 p.s.i.), the final boiling point of the B-product was 225° (437° F.). Under these conditions, a recycle (Rueckfuehrung) of the B-product, was not possible. The phenol reduction was excellent in spite of the high feed rate (0.01 per cent in the Abstreifer).<sup>33</sup>

\* A rate of 1 kg/l/hr. is equal to 1 V/V/hr. for a feed of gravity 1.0. It means 1 kg. of feed per 1 of catalyst volume per hour. (The translator)

\*\*This word is sometimes confusingly translated as "stripper". Actually the Abstreifer is the separator (tar pot or catch pot) in which the liquid hydrogenation product is separated from the gases, and consequently the liquid product withdrawn from it is also referred to as Abstreifer, and sometimes, as Abstreifer product. A separate water phase is also withdrawn from the Abstreifer.

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The second experiment (2/8 - 5/17) was made with a milder catalyst (K-429). So as not to obtain misleading results because of the use of extraneous gas oil (such as 5058-B-product), the experiment was started with undiluted A-gas oil. In this procedure it had to be accepted that the high phenol content of the A-gas oil might damage the catalyst, particularly with respect to its ability to reduce phenols. When, therefore, phenol values of over 4 per cent were obtained in the Abstreifer later on in the experiment, this had to be blamed on the initial overload of the catalyst with phenols. The results of the first experiment indicate that with more careful operation a much better phenol reduction is possible.

The experiments with hard coal liquefaction oil (12 to 15 per cent phenol) in which a phenol reduction to 0.2-0.3 per cent was obtained indicate also that with careful operation a better phenol reduction (to below 0.5 per cent in the Abstreifer) would have been obtained.

The experiment was started at a pressure of 700 atm. (10,300 p.s.i.) and a feed rate of up to 1.8 kg/l/hour. Since the activity of the catalyst was too high at this pressure, the pressure was reduced to 500 atm. (7350 p.s.i.).

The experimental conditions during the main period (3/7 - 5/17) of the experiment were:

Total Pressure	500 Atm. (7350 p.s.i.)
Gas	Gasoline Recycle Gas (85% H <sub>2</sub> ) (Benzin-Kreislaufgas)
H <sub>2</sub> Partial Pressure	425 Atm. (6250 p.s.i.)
Temperature	26.5 mV (970° F.)
Feed Rate	1 Kg/l/hr.
Product/Gas Ratio	1:3000
Feed	60% Soft Coal A-Gas Oil, 40% Self-Produced B-Product

Under these experimental conditions, the Abstreifer had originally a gasoline content (cut pt. 165° C. (329° F.)) of about 55 per cent which fell slowly to about 45 per cent and remained constant at this value to the end of the run (over 1.5 months).

The following table shows the analytical data of the gasoline with an end point of 165° C. (329° F.) (=45 to 50 per cent of the Abstreifer) and an end point of 140° (284° F.) (40 to 45 per cent of the Abstreifer) in comparison to VT-702.

R E S T R I C T E D

	Gasoline from Experiment		
	-165° C. F.B.P. (329° F.)	-140° C. F.B.P. (284° F.)	VT-702
Density at 15°	0.769	0.756	0.719
ASTM Distillation:			
I.B.P.	40.0° C.	41.5° C.	45.0° C.
5 Vol. %	67.0° C.	65.0° C.	56.0° C.
15	83.0	77.5	64.5
25	92.0	84.5	69.5
35	99.5	91.5	76.5
45	107.0	97.5	84.0
55	115.5	103.0	92.0
65	124.0	108.0	98.5
75	133.0	114.0	108.0
85	142.0	123.0	120.0
95	163.0	137.0	137.5
F.B.P.	97% at 167° C. (333° F.)	97% at 141° C. (286° F.)	97.5% at 142° C. (288° F.)
Bottoms, %	0.9	0.7	0.8
Loss	2.1	2.3	1.7
10 Vol. %	74.0° C.	71.5° C.	62.0° C.
90 Vol. %	148.0° C.	128.0° C.	128.0° C.
100° Point (212° F)	36.0 Vol. %	51.0 Vol. %	66.5 Vol. %
Index (Kennziffer)	112.6	110.1	90.7
Aniline Point	22.8° C.	25.5° C.	49.0° C.
Iodine No.	4.14      4.16	2.63      2.70	1.07      1.08
Hydrocarbon Classes:			
Aromatics and			
Olefins, Vol. %	41*      (32)**	39	18
Naphthenes, Vol. %	24      (30)	26	34
Paraffins, Vol. %	35      (38)	35	48
Elements:			
C, %	87.07      86.96	87.27	84.70      84.86
H <sub>2</sub> , %	13.03      12.91	12.64	15.22      15.35
Octane No. (MM.):			
Clear	72.5      74.5	70.5	
0.05 Vol. % TEL (1.9 cc./Gal.)	83.5      86.0	85.0	
0.12 Vol. % TEL (4.55 cc./Gal.)	88.0      -	90.0	

\* Method Riesenfeld-Bandte (BVM 1940)

\*\* Aniline Point Method

Testing of the gasoline in a supercharge motor has been initiated. Samples were sent to the DWL for preparing of the supercharge curve.

Material balances have been set up to determine the extent of gas yield (Vergasung). The average gas yield based on gasoline plus gas produced is 26.5 per cent, according to Leuna. The yield of 165° E.P. (329° F.) gasoline was at the beginning of the run 0.36 kg/l/hour based on gasoline produced (Benzin Neubildung) (= 0.47 kg/l/hour yield, based on total gasoline) and remained after some time constant at 0.26 kg/l/hour based on gasoline produced (= 0.35 kg/l/hour based on total gasoline).

R E S T R I C T E D

At the end of the run the catalyst had retained its original form and hardness.

Pages 484-485

(C) Comparison of the Latest Ruhroel Catalysts with IG Catalyst 7019 (10-27-41)

Conditions: pressure, 550 atm. (8100 p.s.i.g.); gas type, fresh gas; product/gas ratio, 1:3000; temperature, 25.5 mV (940° F.); charge rate, 1.0 kg/l/hour.

	<u>7019</u>	<u>K-429</u>	<u>K-413</u>	<u>K-510</u>	<u>K-497</u>
Yield kg Gasoline/l/hour	0.20	0.42	0.48	0.53	0.55
Gas Yield Based on Gasoline / Gas Produced, %	20-24	20-24	20-24	20-24	20-24
<u>Abstreifer Analysis:</u>					
D <sub>15</sub> % at 165° (329° F.)	0.895	0.855	0.833	0.823	0.807
Phenol, %	0.025	0.25	0.080	0.020	0.005
Bases, %	0.35	0.09	0.007	0.005	0.003
<u>Gasoline Analysis - 165°</u>					
D <sub>15</sub> 4	0.812	0.792	0.788	0.780	0.778
<u>ASTM Distillation</u>					
I.B.P.	56° C.	44° C.	45° C.	-	39° C.
-100° (212° F.)	34%	49.5%	51%	56%	55%
F.B.P. 98%	179° C.	166° C.	160° C.	160° C.	155° C.
<u>Aniline Point</u>					
	-12.7° C.	-0.7° C.	-3.4° C.	-7.5° C.	-11.1° C.
<u>Aromatics and Olefins</u>					
Naphthenes	55%	50%	48%	45%	42%
Paraffins	40%	36%	36%	-	40%
	5%	14%	16%	-	18%
<u>Octane No. (MM.)</u>					
Unloaded	78.5	80.0	80.0	-	78.0
0.05 Vol. % TEL (1.9 cc. TEL/Gal.)	85.0	88.0	87.5	-	87.0
0.12 Vol. % TEL (4.55 cc. TEL/Gal.)	89.0	90.5	91.0	-	90.5
<hr/>					
C	88.8%	88.1%	87.7%	-	87.7%
H <sub>2</sub>	11.2%	11.9%	12.3%	-	12.3%

Pages 486-508

(D) Summary Report on Experiments with Vapor Phase Catalyst 7019 Ruhroel  
(from 1-19 to 3-7, 1940) (Ruhroel GMBH, Main Laboratory and "Technikum"  
April, 1940)

Contents:

- I. Analysis and Final Condition of Catalyst
- II. Investigation of the Catalyst in the 5.5 l. Furnace
  - 1. General Experimental Conditions
  - 2. Experiment at 300 atm. (4400 p.s.i.g.)
    - a. Pitch Gas Oil
    - b. Coal Gas Oil
  - 3. Experiments at 700 atm. (10,300 p.s.i.g.)
    - a. Coal Gas Oil
    - b. Pitch Gas Oil
  - 4. Analysis of the Liquid Reaction Products
  - 5. Analysis of the Reaction Gases

III. Summary

I. Analysis and Final Condition of the Catalyst

The catalyst was in the form of 8 mm. pills. It had the following composition:

Combustibles (coal)	81%
Ash	18.2%
Inorganic	
Sulfur	0.8%
Cr <sub>2</sub> O <sub>3</sub>	10.6% (= 7.3% Cr)
Al <sub>2</sub> O <sub>3</sub>	6.8% (= 3.5% Al)
Weight lost after three hours drying at 120° C.	
H <sub>2</sub> O	2.7%

The following weight and volume losses were found after a 49-day run:

Catalyst in	5440 cc.	= 4130 g.
" out	4100 cc.	= 3285 g.
Loss	1340 cc.	845 g.

The volume loss is about 25% and the weight loss about 20%.

The catalyst at the end of the run contained 9 volume per cent = 15 weight per cent dust or fines (Staub) and about 30 weight per cent granules (unter-korn).

The catalyst also carried considerable iron sulfide from the furnace wall. The fines contained 31.5 per cent ash (ash: 23.1% Fe). The granules contained 60.8 per cent ash (ash: 55.0% Fe). The actual weight loss is therefore about 35 per cent.

The catalyst removed was brittle and not very resistant to mechanical shock.



II. Investigation of the Catalyst in the 5.5 l. Furnace

1. General Experimental Conditions

The experimental furnace consisted of  $N_8$  pipe of 45 mm. (1.95 inch) inside diameter, about 5.5 m (18 feet) long. The pipe was heated by three electrical heating units. The foot and body of the furnace each had one further heating unit. The inside temperature of the furnace was measured at six places. The largest temperature differential at the six places was 0.3 mV (approximately 5.5° C. (9.7° F.)). The preheater was a coil of  $N_8$  pipe of 6 mm. (7/16 inch) inside diameter and 12 m (40 foot) length, heated in a lead bath.

The gas oil was fed by means of a Hofer pump with a Bosch filter directly ahead of the pump.

The gas was run straight through and was in all experiments so controlled that 14 cubic meter (400 cubic feet) an hour was released, as per Ludwigshafen instructions. All experiments were made with fresh gas (98 per cent  $H_2$ ). So as to simulate conditions when using recycle gas, the experimental pressure was reduced 50 atmospheres, that is, the 300 atm. experiment was run at 250 atm. fresh gas pressure and the 700 atm. experiment at 650 atm. The duration of the experiment was 49 days, divided as follows:

300 Atmospheres (4420 p.s.i.g.)

1 day: Increase temperature up to 26.5 mV. (970° F.)

1 day: 26.5 mV. without feed

Feed: Pitch Gas Oil

6 days: Increasing the feed rate from 0.2 to 1 kg/l/hour

12 days: Feed rate 1 kg/l/hour

Feed: Coal Gas Oil - Feed Rate 1 kg/l/hour

18 days at 26.5 mV. (970° F.)

1 day at 27.0 mV. (985° F.)

3 days at 27.5 mV. (1001° F.)

600 Atmospheres (8840 p.s.i.a.)

4 days: With coal gas oil at 26.5 mV. (972° F.)

3 days: With pitch gas oil at 26.5 mV. (972° F.)

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The temperatures were measured with iron constantan thermocouples. Terminal temperature was 40° C.\* (104° F.)

\* Since only two references were found on this film in which the terminal temperature was given, this value has been used throughout in calculating temperatures from the thermocouple readings in mV. (The Translator)

R E S T R I C T E D

2. Experiments at 300 Atmospheres (4420 p.s.i.g.)

a. Pitch Gas Oil

The feed, which remained constant throughout the series of experiments, consisted of A-gas oil from sump phase pitch hydrogenation at 700 atm. (10,300 p.s.i.g.) and B-gas oil from the vapor phase hydrogenation with K-157 at 700 atm. The composition was:

Density at 15°	0.970	
Engler Distillation:		
Initial Boiling Point	196° C.	385° F.
5 Vol. %	205	401
15 Vol. %	211	412
25 Vol. %	217	423
35 " "	220	428
45 " "	227	440
55 " "	232	449
65 " "	242	468
75 " "	255	491
85 " "	275	527
95 " "	311	592
F.B.P., % 331° (628° F.)	98	
Residue, %	1.5	
Loss, %	0.5	
-200°, % (392° F.)	2.0	
-300°, % (572° F.)	94.0	
Phenol, %	2.7	
Pyridine, %	2.4	
Viscosity at 20°	1.2° E.	
Conradson Test	0.05	
Elements:		
C, %	89.79	
H <sub>2</sub> , %	8.90	
S, %	0.13	
Cl, %	0.003	

The furnace was brought on temperature within 20 hours and then held on temperature 24 hours without feed. For protection of the catalyst the furnace was started at low feed rates and in the course of six days brought to a feed rate of 1 kg/l/hour. Table I indicates the results obtained. After the feed rate of 1 kg/l/hour had been attained it was held constant for ten days. Results are shown in Table II. It can be seen that during this period the catalytic effect of the catalyst dropped considerably (the low-boiling components decreased by 45 per cent, the higher boiling ones by 25 to 30 per cent). The phenol and pyridine reduction was consistently good. The Abstreifer contained less than 0.01 per cent phenol and less than 0.06 per cent pyridine. Gas yield was about 15 per cent based on gasoline (-195° (383° F.)) plus loss.

R E S T R I C T E D

After this, a two-day run was made with self-produced B-product (Table II).

Feed Analysis: 70 Per Cent B-Gas Oil, 30 Per Cent Pitch Gas Oil

Density at 15°	0.943
I.B.P.	190° C. (374° F.)
F.B.P.	301° C. (574° F.)
Phenol, %	0.79
Pyridine, %	1.00

No increase of yield on account of this oil was noticeable. It is interesting that the aniline point of the gasoline increased.

b. Coal Gas Oil

This gas oil was made by hard coal sump phase hydrogenation at 300° C. (572° F.) and was furnished by Hydrierwerk Scholven. The analysis was:

Density at 15°	0.974
Engler Distillation:	
I.B.P., °C.	177
F.B.P., °C.	350
200°, Vol. %	8.5
250°, Vol. %	38.5
300°, Vol. %	71.5
A.P. - °C.	-8.5
Phenol, %	20.0
Pyridine, %	2.4
Hard Asphalt	None
Conradson Test, %	0.024
Elements:	
C, %	86.9
H <sub>2</sub> , %	9.2
F, %	0.103
Cl, %	0.0013

Because of the high phenol content the coal gas oil was not fed straight but only 30 parts of this oil were added to 70 parts of B-product. Since B-product from coal gas oil was at first not available, B-product from pitch gas oil from the previous experiment was added. From the 21st to the 24th day this feed was used. The B-product from this period is already similar to the coal B-product. However, only after longer experimental duration is the feed product pure coal-A and -B gas oil.

The average analysis of this feed is:

Density at 15°	0.937
I.B.P.	195° (383° F.)
200° C., (392° F.)	1.5
300° C., (572° F.)	90.5
F.B.P.	335° (635° F.)
Phenol, %	5
Pyridine, %	1.2

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R E S T R I C T E D

R E S T R I C T E D

Table III shows the progress from the 25th to the 38th day. An increase in the aniline point of the gasoline as well as the B-gas oil is noticeable.

On the 38th day a sulfur containing feed (0.3 per cent ash) was run. On this one day, no increase in the catalyst performance was noted.

Since according to the Ludwigshafen indication, the decrease of catalyst performance should be countered by increasing the temperature, the run was continued at a higher temperature (see Table IV):

The phenol and pyridine reduction is also good for coal gas oil (phenol below 0.015 per cent, pyridine below 0.2 per cent). The gas yield at 26.5 mV. (955° F.) is about 12 to 15 per cent, and at 27.5 mV. (1001° F.) increases to about 18 per cent based on gasoline (-195°) plus loss.

3. Experiments at 700 Atmospheres (10,300 p.s.i.g.)

a. Coal Gas Oil

Experiments at higher pressures were also made with the coal A- and B-gas oil of the previous experiments. The results are shown in Table V. Aside from the increased yield, a strong increase in the aniline points is noticeable. Since the aniline point of the B-product continues to increase it is indicated that for a longer run duration a greater increase in the aniline point of the gasoline is also to be expected.

b. Pitch Gas Oil

Finally pitch gas oil was run for three days at 700 atm. The feed was the same as for the 300 atm. experiment. Results are shown in Table V.

4. Analysis of the Liquid Reaction Products

a. 300 atm. Pitch Gas Oil, Feed Rate 1 kg/l/hour, Temperature 26.5 mV. (970° F.)

The gasoline was washed with ten per cent NaOH, then with water, and finally filtered over CaCl<sub>2</sub>.

R E S T R I C T E D

Stream Day	9	12	16
Gasoline End Point	-195°	-180°	150°
Density at 15°	0.850	0.826	0.805
Engler Distillation:			
I.B.P.	51.0° C.	42.0° C.	38.5° C.
5 Vol. %	81.5° C.	69.0° C.	64.5° C.
15 " "	102.0° C.	87.0° C.	81.0° C.
25 " "	113.0° C.	95.0° C.	87.0° C.
35 " "	125.5° C.	101.0° C.	91.0° C.
45 " "	145.5° C.	109.5° C.	94.5° C.
55 " "	160.0° C.	121.0° C.	99.5° C.
65 " "	172.5° C.	135.0° C.	105.5° C.
75 " "	179.5° C.	152.0° C.	113.0° C.
85 " "	185.5° C.	173.0° C.	124.5° C.
95 " "	196.0° C.	189.0° C.	151.5° C.
F.B.P.	205.5° C.	197.5° C.	160.5° C.
100°	13.5 Vol. %	33.5 Vol. %	56.0 Vol. %
Refr. Index	1.4815	1.4708	1.4554
A.P.	-25°	-25°	-14°
Iodine No.	3.46	3.21	2.44
Arom. / Olef.	71%	66%	62%
Naphthenes	25%	30%	26%
Paraffines	4%	4%	12%
Elemental Analysis:			
C	89.30%	88.64%	-
H <sub>2</sub>	10.64%	11.34%	-

Stream Day	9	12	16
Gasoline End Point	-195°	-180°	-150°
Glass Bowl	1.1 mg	0.8 mg	0.2 mg
Bomb Test	68.6 mg	4.0 mg	3.0 mg
Octane No. (MM)			
Clear	78	79	79.5
0.5 TEL*	84	84.5	85.5
0.9 " *	-	85.5	-
1.2 " *	-	86.5	-
Benzene **	3.2 Vol. %	5 Vol. %	-
Toluene **	5.3 Vol. %	8 Vol. %	-
B-Product	> 195°	> 180°	> 150°
Density at 15°	0.942	0.932	0.930
I.B.P.	198° C.	186° C.	183° C.
F.B.P.	293° C.	295° C.	298° C.
A.P.	-24°	-14°	> -25°
Elemental Analysis:			
C	89.99%	89.83%	89.31%
H <sub>2</sub>	9.80%	9.78%	9.61%

\* The units here are doubtful, generally TEL is given as:-

Vol. % TEL (0.05 Vol. % = 1.9 cc./gal.)  
 (0.09 " " = 3.44 " )  
 (0.12 " " = 4.55 " )

\*\* By Podbielniak fractionation.

R E S T R I C T E D

b. 300 atm., Coal Gas Oil, Feed Rate 1 kg/l/hr.

A. Temperature 26.5 mV.

Stream Day	34 and 35	34 and 35	34 and 35
Gasoline	-180°	-150°	-110°
Density at 15°	0.822	0.808	0.780
Engler Distillation:			
I.B.P.	62.0° C.	55.0° C.	47.0° C.
5 Vol. %	84.5° C.	74.0° C.	61.0° C.
15 Vol. %	97.0° C.	85.5° C.	69.5° C.
25 Vol. %	100.5° C.	91.0° C.	74.5° C.
35 Vol. %	110.0° C.	95.0° C.	79.5° C.
45 Vol. %	121.0° C.	99.0° C.	80.5° C.
55 Vol. %	131.0° C.	104.0° C.	82.5° C.
65 Vol. %	141.0° C.	110.0° C.	84.5° C.
75 Vol. %	151.5° C.	118.0° C.	89.0° C.
85 Vol. %	162.5° C.	127.0° C.	94.0° C.
95 Vol. %	175.0° C.	144.5° C.	110.0° C.
F.B.P.	181.0° C.	159.0° C.	124.0° C.
100°	24.0 Vol. %	47.5 Vol. %	91.0 Vol. %
A.P.	-8.5°	-10.0°	+3.5°
Hydrocarbons			
Arom. / Olef.	61 Vol. %	58 Vol. %	45 Vol. %
Naphthenes	29 Vol. %	36 Vol. %	48 Vol. %
Paraffines	10 Vol. %	6 Vol. %	7 Vol. %
Octane No. (MM)			
Clear	75	76.5	77.5
0.5 TEL	83	84	86.0
0.9 TEL	86	86.5	90.0

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B. Temperature 27.5 mV.

Stream Day	40 and 41	40 and 41
Gasoline	-180°	-150°
Density at 15°	0.819	0.808
Angler Distillation:		
I.B.P.	56.0° C.	51.5° C.
5 Vol. %	81.5° C.	88.0° C.
15 Vol. %	94.0° C.	88.5° C.
25 Vol. %	102.0° C.	94.0° C.
35 Vol. %	108.5° C.	99.0° C.
45 Vol. %	117.5° C.	103.5° C.
55 Vol. %	126.5° C.	110.5° C.
65 Vol. %	136.5° C.	118.0° C.
75 Vol. %	147.0° C.	127.0° C.
85 Vol. %	159.0° C.	138.0° C.
95 Vol. %	175.0° C.	159.0° C.
F.B.P.	180.0° C.	170.0° C.
100°	23 Vol. %	38 Vol. %
A.P.	-12.0°	-11.5°
Hydrocarbons		
Arom / Olef.	62.0 Vol. %	60.0 Vol. %
Naphthenes	27.0 Vol. %	30.0 Vol. %
Paraffines	11.0 Vol. %	10.0 Vol. %
Octane No. (MM)		
Clear	76.0	76.5
0.5 TEL	83.5	84.5

R E S T R I C T E D

c. 600 atm., Coal Gas Oil, Feed 1/kg/1/Std., Temperature 26.5 mV.

	46	44	44
Stream Day			
Gasoline	-195°	-175°	-150°
Density at 15°	0.830	0.814	0.799
Engler Distillation:			
I.B.P.	46.5° C.	51.0° C.	46.0° C.
5 Vol. %	83.0° C.	79.5° C.	71.0° C.
15 Vol. %	101.5° C.	92.5° C.	83.0° C.
25 Vol. %	114.5° C.	99.5° C.	89.0° C.
35 Vol. %	127.0° C.	106.0° C.	93.0° C.
45 Vol. %	144.0° C.	116.0° C.	97.0° C.
55 Vol. %	158.5° C.	114.0° C.	101.0° C.
65 Vol. %	169.5° C.	124.0° C.	106.5° C.
75 Vol. %	178.5° C.	135.5° C.	114.0° C.
85 Vol. %	185.5° C.	147.0° C.	123.0° C.
95 Vol. %	200.0° C.	160.0° C.	141.5° C.
F.B.P.	212.5° C.	174.0° C.	154.5° C.
100°	13.5 Vol. %	25.5 Vol. %	53.0 Vol. %
A.P.	72.0°	-0.5°	-0.5°
Hydrocarbons			
Arom. / Olef.	55 Vol. %	54 Vol. %	52 Vol. %
Naphthenes	35 Vol. %	37 Vol. %	41 Vol. %
Paraffines	12 Vol. %	9 Vol. %	7 Vol. %
Elemental Analysis:			
C	87.88%	-	-
H <sub>2</sub>	12.04%	-	-
Octane No. (MM)			
Clear	70.5	75.5	76.5
0.5 TEL	79.0	83.0	84.5



R E S T R I C T E D

d. 600 atm., Pitch Gas Oil, Feed 1 kg/l/hr., Temperature 26.5 mV.

Stream Day	49	49
Gasoline	-180°	-150°
Density at 15°	0.821	0.803
Engler Distillation:		
I.B.P.	43.0° C.	35.0° C.
5 Vol. %	72.0° C.	62.0° C.
15 Vol. %	91.0° C.	78.0° C.
25 Vol. %	99.5° C.	85.5° C.
35 Vol. %	106.5° C.	90.5° C.
45 Vol. %	115.0° C.	94.0° C.
55 Vol. %	126.0° C.	98.0° C.
65 Vol. %	139.0° C.	103.0° C.
75 Vol. %	150.5° C.	110.0° C.
85 Vol. %	164.5° C.	123.0° C.
95 Vol. %	180.0° C.	151.5° C.
F.B.P.	185.0° C.	163.0° C.
100°	26 Vol. %	60 Vol. %
A.P.	-11.0°	-12.5°
Hydrocarbons		
Arom. / Olef.	60 vol. %	59 Vol. %
Naphthenes	35 Vol. %	39 Vol. %
Paraffines	5 Vol. %	2 Vol. %
Octane No. (MM)		
Clear	76.5	78.0
0.5 TEL	82.5	85.5
B-gas Oil	>180°	>150°
Density at 15°	0.888	0.882
I.B.P.	186° C.	175° C.
F.B.P.	272° C.	264° C.
A.P.	-1.0°	-3.5°
C	88.58%	88.41%
H <sub>2</sub>	11.35%	11.39%

R E S T R I C T E D

5. Analysis of Reaction Gases

a. 300 atm.

	<u>Pitch Gas Oil</u>		<u>Coal Gas Oil</u>			
	<u>26.5 mV.</u>		<u>26.5 mV.</u>		<u>27.5 mV.</u>	
	<u>17</u>		<u>30</u>		<u>41</u>	
Temperature	Chamber	Abstreifer	Chamber	Abstreifer	Chamber	Abstreifer
Stream Day	Outlet	Gas	Outlet	Gas	Outlet	Gas
Gas	Outlet	Gas	Outlet	Gas	Outlet	Gas
1/hr.	14000	-	14000	-	14000	-
1/1 <u>Abstreifer</u>	-	14.85	-	9.47	-	18.80
CO <sub>2</sub>	0.00	0.00	0.00	0.00	0.00	0.00
H <sub>2</sub> S	0.00	0.00	0.00	0.00	0.02	0.98
O <sub>2</sub>	0.00	0.00	0.00	0.00	0.00	0.00
H <sub>2</sub>	97.21	69.72	97.17	73.36	95.63	51.47
CO	0.15	0.12	0.11	0.09	0.21	0.10
N <sub>2</sub>	1.35	2.02	1.41	1.53	1.39	1.75
CH <sub>4</sub>	0.77	3.61	0.94	2.89	1.81	4.05
C <sub>2</sub> H <sub>6</sub>	0.35	6.65	0.16	5.58	0.39	12.66
C <sub>3</sub> H <sub>8</sub>	0.17	9.83	0.21	10.26	0.55	17.79
i-C <sub>4</sub> H <sub>10</sub>	0.00	0.55	0.00	0.77	0.00	1.82
n-C <sub>4</sub> H <sub>10</sub>	0.00	5.70	0.00	4.50	0.00	7.50
C <sub>5</sub> H <sub>12</sub> Plus	0.00	1.30	0.00	1.02	0.00	1.88

b. 600 atm.

	<u>Pitch Gas Oil</u>		<u>Coal Gas Oil</u>	
	<u>26.5 mV.</u>		<u>26.5 mV.</u>	
	<u>49</u>		<u>44</u>	
Temperature	Chamber	Abstreifer	Chamber	Abstreifer
Stream Day	Outlet	Gas	Outlet	Gas
Gas	Outlet	Gas	Outlet	Gas
1/hr.	14000	-	14000	-
1/1 <u>Abstreifer</u>	-	51.9	-	30.02
CO <sub>2</sub>	0.00	0.00	0.00	0.00
H <sub>2</sub> S	0.02	1.87	0.02	1.75
O <sub>2</sub>	0.00	0.00	0.00	0.00
H <sub>2</sub>	96.54	70.08	97.23	79.92
CO	0.03	0.04	0.04	0.13
N <sub>2</sub>	1.60	1.42	1.43	1.49
CH <sub>4</sub>	1.05	3.88	0.60	2.97
C <sub>2</sub> H <sub>6</sub>	0.36	5.60	0.43	5.24
C <sub>3</sub> H <sub>8</sub>	0.40	8.76	0.25	8.12
i-C <sub>4</sub> H <sub>10</sub>	0.00	0.84	0.00	1.27
n-C <sub>4</sub> H <sub>10</sub>	0.00	6.68	0.00	4.74
C <sub>5</sub> H <sub>12</sub>	0.00	0.83	0.00	1.37

R E S T R I C T E D

III. Summary

1. Catalyst 7019 after long use becomes brittle and is no longer abrasion proof. The coal which serves as carrier is slowly reduced by the hydrogen so that after some time the weight and volume loss become considerable.

2. During a 10-day run with pitch gas oil, a great loss in catalyst activity was noticed.

3. The yield with this catalyst at 300 atmospheres (4420 p.s.i.g.) is with both feeds 0.20 to 0.25 kg/l/hour of 180° (361° F.) E.P. gasoline (with about 25 volume per cent below 100° (212° F.)), 0.10 to 0.15 kg/l/hour of 150° (302° F.) gasoline (with about 55 per cent boiling below 100° (212° F.)). At 27.5 mV (1000° F.) the output is 0.35 kg/l/hour at a cut point of 180° (23 per cent below 100°) and 0.23 kg/l/hour at a cut point of 150° (40 per cent below 100°).

An increase in yield is also obtained by increasing the pressure from 300 to 700 atmospheres (4420 to 10,300 p.s.i.g.). For coal and gas oil this yield at 26.5 mV (970° F.) is: 0.3 kg/l/hour of 180° (361° F.) E.P. gasoline (25 per cent below 100°); 0.2 kg/l/hour of 150° (302° F.) E.P. gasoline (about 55 per cent below 100°).

4. The phenol and pyridine reduction is very good in all cases.

5. At 300 atmospheres when feeding pitch gas oil, the aniline point of gasoline and B-product remains below -25° (-13° F.). When adding B-product, the aniline point of the gasoline but not that of the C-product rises. With coal gas oil an increase of the aniline point of gasoline and B-product is noticeable. After a 14-day run the final equilibrium was probably not yet obtained. Increasing the temperature causes a small decrease of the aniline point. The increase of pressure to 700 atmospheres effects a rapid increase of the aniline point when feeding coal and pitch gas oil. Since that of the B-product increases very strongly the gasoline must also attain a very high aniline point after a longer run.

6. The gasoline quality is very much dependent on feed and reaction conditions. Generally the components below 100° (212° F.) are small so that the cut point for ordinary aviation gasoline must be between 150° (302° F.) and 180° (361° F.). It is noted in either case that the octane number decreases with increasing final boiling point. This is explained by the fact that the fraction between 150° and 180° is particularly rich in paraffin hydrocarbons. Treating experiments were made with the gasoline from the pitch gas oil. After only a simple caustic and water wash the gasoline showed satisfactory tests but it discolored on standing in the sunlight. ~~The gasoline from pitch gas oil at 300 atmospheres has the~~ octane numbers. They vary according to final boiling point between 78 and 79.5 (motor method; IG motor; cooling water temperature 150° (302° F.) The values in the CFR motor are about five units higher). The lead susceptibility of this gasoline is poor because of the high aromatics content.

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The gasoline from coal gas oil has octane numbers of 75 to 77.5. Since its lead susceptibility is better, however, the octane numbers with 0.5 per cent TEL (1.9 cc./gallon) equal those of equally leaded pitch gas oil gasoline (84 to 86). Temperature increase produces no significant change of the octane number. At a pressure of 600 atmospheres (8840 p.s.i.g.) the octane number of the gasoline from coal gas oil remains unchanged. That of the gasoline from pitch gas oil decreases to between 76.5 and 78.0. The lead susceptibility improves slightly.

7. Of the gas reaction products, the content of isobutane in the total butane is small (15 to 20 per cent).

R E S T R I C T E D

Table I

Variation of Abstrefiner from Pitch Gas Oil with Feed Rate

Temperature: 26.5 mV

Stream Day	Feed Rate kg/l/hr	D <sub>15</sub> 4	Aniline Point		Engler Distillation				
			Total Abstrefiner	(-356°F) (-180°C)	(>356°F) >180°C	(-212°F) (-100°C)	(-302°F) (-150°C)	(-356°F) (-180°C)	(-392°F) (-200°C)
3	0.20	0.802	48.0	45.0	419.2	44.5%	73.0%	84.0%	95.0%
4	0.26	0.803	45.8	43.7	415.5	46.0	73.0	85.0	96.0
5	0.33	0.813	40.0	-0.5	44.4	35.2	63.2	77.2	93.2
6	0.46	0.840	-15.0	-10.0	-18.5	20.0	47.5	61.5	85.5
7	0.62	0.860	-23.0	-19.5	< -25	10.5	36.0	49.0	71.5
8	0.82	0.877	< -25	-22.0	< -25	7.0	26.5	39.0	61.0

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Table II

Variation of Abstreifer from Pitch Gas Oil at calkg/l/hr Feed Rate with Run Length

Temperature: 26.5 mV

Stream Day	Feed Rate kg/l/Hr.	D <sub>4</sub> <sup>15</sup>	Aniline Point (-356° F.) (>356° F.)		(-212° F.) (-302° F.)		Engler Distillation (-356° F.) (-392° F.)		
			Total Abstreifer	>180°	-100°	-150°	-180°	-200°	
			Feed: Pitch - Gas Oil D <sub>4</sub> <sup>15</sup> = 0.970						
9	0.99	0.890	< -25	< -25	4.5%	19.5%	30.5%	50.5%	
10	1.00	0.892	"	"	4.5	17.5	30.0	47.5	
11	0.99	0.898	"	"	4.0	16.5	27.5	44.0	
12	0.96	0.900	"	"	5.5	15.5	26.5	45.5	
13	0.97	0.904	"	"	4.5	12.0	-	-	
14	0.96	0.904	"	"	4.5	13.5	24.5	43.5	
15	0.99	0.908	"	"	3.0	11.5	21.0	40.0	
16	0.96	0.908	"	"	2.2	10.7	21.2	38.2	
17	0.97	0.912	"	"	2.4	9.9	19.9	37.4	
18	0.96	0.912	"	"	2.8	11.3	20.3	37.3	
			Feed: 70% B-Product - 30% Pitch Gas Oil D <sub>4</sub> <sup>15</sup> = 0.943						
19	0.94	0.896	< -25	-18	1.8	9.8	22.8	51.3	
20	0.92	0.896	"	-21	2.2	10.7	21.7	49.2	

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R E S T R I C T E D

Table III

Variation of Abstreifer from Coal Gas Oil at calkg/l/hr Feed Rate with Run Length

Temperature 26.5 m.v.

Stream Day	Feed Rate kg/l/Std	D 15 D 4	Aniline Point		Engler Distillation			
			Total Abstreifer	(-356° F.) > 180°	(-212° F.) -100°	(-302° F.) -150°	(-356° F.) -180°	(-392° F.) -200°
25	0.90	0.886	-20.0	-11.5	1.9%	10.4%	24.9%	49.4%
26	0.91	0.886	-17.5	-12.0	2.0	11.5	26.0	48.0
27	0.95	0.890	-21.0	-12.0	1.5	9.0	22.0	47.5
28	0.94	0.885	-12.0	-9.5	2.2	11.2	24.7	47.7
29	0.97	0.886	-17.5	-10.0	2.1	10.1	23.6	46.1
30	0.98	0.886	-15.0	-9.0	1.5	9.5	23.0	45.9
31	0.95	0.886	-15.0	-9.0	1.5	10.0	22.5	44.9
32	1.01	0.894	-14.0	-7.0	1.0	6.0	16.0	35.5
33	0.95	0.887	-13.0	-6.0	1.5	10.0	23.0	42.0
34	0.99	0.889	-11.5	-6.0	2.5	12.0	24.0	39.5
35	0.94	0.890	-10.0	-5.0	2.0	10.0	20.5	38.5
36	0.96	0.890	-13.0	-5.0	1.5	8.5	18.0	36.0
37	0.94	0.890	-12.0	-5.0	2.0	9.5	19.0	37.5
38	0.94	0.889	-12.0	-5.0	2.5	9.0	19.0	37.5

Sulfured: 0.2%

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Table IV

Variation of Abstreifer from Coal Gas Oil with Reaction Temperature at Constant Feed Rate

Stream Day	Temperature mV	Temperature °F.	Feed Rate kE/l/Hr.	D	D	Aniline Point (-356° F.)		(-212° F.)		(-302° F.)		Engler Distillation	
						-180°	> 180°	-100°	-150°	-180°	-180°	-150°	-200°
38		26.5	0.94	0.889	15	Total	-5.0	-12.5	2.5%	9.0%	19.0%	37.5%	
39		27.0	0.95	0.879	4	Abstreifer	-4.0	-11.0	4.7	15.7	28.2	42.2	
40		27.5	0.96	0.874			-6.0	-15.0	6.0	23.0	35.0	54.0	
41		"	0.97	0.875			-5.5	-15.0	6.5	23.0	34.5	53.0	
42		"	0.95	0.876			-6.5	-16.0	5.5	21.5	33.5	52.5	

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(e) Experiments Concerning the Suitability of the Zinc Sulfide Catalyst for the Production of Gasoline in the Vapor Phase from Coal Diesel Oil  
(2/13/39)

Index:

- A. Purpose
- B. Materials
  - 1. Catalyst
  - 2. Feed
- C. Experimental Results
  - 1. Meggen zinc blend
  - 2. Zinc sulfide on Terrana
  - 3. Other catalysts
- D. Summary

A. Purpose

Ruhrroel GmbH has provided two catalysts which should produce low gas yields and very good antiknock gasoline from the sump-phase gas oils from hard-coal-pitch, and hard-coal-extract-hydrogenation.

B. Materials

1. Catalyst

Two batches of each of two catalysts were available.

(1) A Meggen zinc blend (K-36s) which according to Ruhrroel was composed as follows:

S	30.23%
Fe	4.74%
Al <sub>2</sub> O <sub>3</sub>	1.54%
Ca	0.013%
Zn	54.6%

The blend had been etched with hydrochloric acid at Welheim. The larger pieces had to be crushed somewhat to fit into our 50 cc. reactors so that the catalyst surface consisted partially of unetched surface.

(2) The same catalyst as 1, but delivered unetched. The etching was carried out in Leuna according to the Ruhrroel instruction:

The blend was crushed to 0.4-0.8 mm. and sifted. 20 Wt. % HCl was poured over it and it was warmed at 60° to 80° for one hour on a water bath. Thereupon the hydrochloric acid was decanted and the blend dried at 120° without previous washing.

(3) A synthetic zinc sulfide catalyst (K-42) which according to the information from Ruhrroel was prepared as follows:

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A mixture of 80% Terrana which had been etched with hydrofluoric acid and 20% zinc chloride was treated with H<sub>2</sub>S at 400°.

The catalyst was delivered in rather soft pills of 4 mm. diameter and 2-4 mm. height. The catalyst is supposed to harden by forming in a hydrogen stream at 450°.

(4) The same as 3, but a later delivery. It contained 0.60% Cl.

(5) Terrana etched with sulfuric acid.

(6) Schoenebeck Bentonite and aluminum hydroxide-1-1 mixture. Catalyst 5 and 6 were included in the experiment for comparison purposes.

II. Feed

1. Welheim Diesel Oil

(a) First Batch

d <sub>20</sub>	0.991					
Aniline Point	-20°					
Acid Oils*	3.2%					
Distillation	215°	220°	250°	280°	300°	307° C.
	0.6	42.0	78.4	95.4	98.8%	

(b) Second Batch

d <sub>20</sub>	0.995					
Aniline Point	<-20°					
Acid Oils	1.5%					
Distillation	226°	230°	250°	280°	300°	309° C.
	1.0	24.8	74.6	93.0	98.0%	

2. Since initially a sufficient amount of feed No. 1 was not available, a similar product from the Lutzkendorf sump phase experiments was used. This oil came from the hard coal tar which had been prepared for sump phase hydrogenation at Lutzkendorf. The oil is characterized by the following data:

d <sub>20</sub>	0.973					
Aniline Point	-20°					
Acid Oils	1.2%					
Distillation	212°	220°	250°	280°	300°	312° C.
	3.0	41.0	80.0	95.6	99.6%	

It differs from the Welheim oil mainly in the lower density.

\* Original Sauro Oelo

C. Experimental Results

Remarks: The experiments were carried out partially in small reactors of 40 cc. (Reactor 17) and 50 cc. (Reactor 18 and 19) catalyst volume and partially in large reactors of 4-5.4 l. catalyst volume. The tail gas (Ofengas) was vented. In the small reactors the tail gas and product gas were measured together. The total amount of gas given in the table includes therefore in these cases also the product gas.

Preliminary runs were carried out in the small reactors. In these the performance was determined by Engler analysis of the Abstreifer, and the light ends (Gasbenzin) were not considered. Since the gasoline so produced shows only a very small per cent boiling at 100° (212° F.), a considerable part of the light ends can be added to the gasoline without increasing its vapor pressure above the allowable. Therefore, the day and shift-balances for reactor 18 and KA-4 (kammer 4 = Chamber 4) in which the total light ends were added to the gasoline and this then stabilized sometimes show a considerably higher output.

1. Meggen zinc blend (36-s)

The results are summarized in Table I. Only preliminary experiments were made since it was found that at the various feed rates (0.25-1.0) only insufficient yield (max. 0.19) was obtained.

The efficiency of the catalyst seems largely dependent on the method of bringing on stream, since practically no effect was obtained in an experiment in KA-4 (Chamber 4) in the 4-liter reactor, in which, because of inability to adjust the feed pump, the unit was brought on stream with a feed rate of 0.75.

2. Zinc Sulfide on Teranna (K-47)

The preliminary experiments are summarized in Table II. Good yields were obtained at feed rates larger than 1. This increase in output with increasing feed rate is more noticeable at 26.5 (970° F.) than at 26 mV (955° F.). At a temperature of 26 mV. (955° F.) an increase in the feed rate of from 0.7-1.0 produced a yield increase from 0.32-0.37 (experiments 13 and 14). The increase of the feed rate to 1.5 produced only a yield increase to 0.4 (Experiment 25). This is also indicated by experiments in the 5.4 liter reactor (Table III, Experiment 1 and 2). When increasing the feed from 0.76 to 1.0 the output was not increased noticeably (from 0.33 to 0.34). Experiments 4 and 5 in Table III deviate from this rule. The situation is very different, however, at 26.5 mV. (970° F.). At this temperature the yield is 0.39, 0.45, and 0.52 at feed rates of 1.0, 1.2, and 1.5, respectively (Experiments 15 to 17). Naturally the produced gasoline becomes less "siedegerecht"\* with increasing feed rate. No conclusions can be drawn on this count from the preliminary experiments, since the light ends are not considered in them. From the experimental balances in Table III, however, it can be seen that with feed rates of above 0.4 about 10 to 15 per cent of the gasoline boiled below 100° (212° F.)

\* i.e. less is in the desirable boiling range

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The balance experiments (Table III) indicated a considerably higher yield than indicated in the preliminary remarks. The highest yield was obtained in Reactor 18 (Experiment 7); it was 0.57 at a feed rate of 1.33.

The corresponding experiments No. 8 (Reactor 18) and No. 3 (KA-4) indicate essentially good agreement. The small differences are partially due to differing experimental conditions. The oils used differ in phenol content (3.2 and 1.5 per cent, respectively); the gas/oil ratio is somewhat higher in KA-4 and consequently produces stronger hydrogenation. The two catalysts are from two different shipments. This may well be the cause of one significant difference between the two experiments, namely the considerably smaller gas yield in the experiment in KA-4 (only 70 per cent of Reactor 18). On the other hand, this could partially be caused by the difference in reactor type since in the smaller reactor the temperature cannot be as closely regulated as in the 5.4 liter reactor. Therefore, the experiments in KA-4 alone, are to be considered significant for judging the gas yield. It is to be noted that with an increase in temperature from 26 to 26.5 mV. (955-970° F.), the gas yield did not increase since the output increased in the same ratio. Phenol reduction is good only initially and soon decreases considerably. At equilibrium only about 50 per cent of the phenols are reduced.

The Engler final boiling point of the B-product is about 15 to 20° (27 to 36° F.) higher than the final boiling point of the feed. Since, however, it does not rise further after this value has been attained, there is no need for worry.

With a gas/oil ratio of 1.6 cubic meter gas per kg oil and lower, the octane numbers of the stabilized gasoline are at or above 100. At larger gas to oil ratios the octane numbers are from 95 to 98.

Catalyst life is very good; after a run length of 100 days there is no decrease in activity.

It can therefore be said in summary that in consideration of the quality of the gasoline a gas yield of 18 per cent is not excessive.

### 3. Other Catalysts

The fact that pure zinc sulfide proved inactive whereas the diluted catalyst (K-47) showed very good performance leads to the supposition that in the latter case the carrier rather than the zinc sulfide was active. To answer this question two experiments were carried out with high surface catalyst, one being H<sub>2</sub>SO<sub>4</sub> treated Terrana and the other a mixture of Bentonite and aluminum hydroxide. Two balance experiments are reported in Table III, from which it can be seen that these catalysts also yield high octane gasoline. However, the output is less than with the Welheim catalyst. Nevertheless we believe that in the case of the Welheim catalyst also the carrier is active and the influence of the zinc sulfide is small. Experiments should therefore be carried out in this direction.

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Summary

Two gasoline production catalysts from Ruhroel were tried. One was a natural zinc blende and the other zinc sulfide on Terrana.

The natural Zinc blende proved entirely inactive.

Zinc sulfide on Terrana at 26.5 mV. (970° F.) and 660 atmospheres (9700 p.s.i.g.) gave a yield of 0.5 gasoline of 100 octane number. The phenol reduction is insufficient so that the catalyst is only useful for phenol-poor gas oil. The gas yield is about 18 per cent. The durability of the catalyst was tried in a 100-day run. Other high surface catalysts give gasoline of equally good quality but in lesser yield.

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Table I

Experiments with Meggen Zinc Blends

No.	Oil	Catalyst	Furnace Temp. mV.	Furnace Temp. °F.	Pressure Atm.	Pressure p.s.i.	Feed Rate cc./cc./ Hr.	1 H <sub>2</sub> / cc. Oil	Yield g./ cc./ Hr.	Gasoline Conc. % <200 °C. (392 °F.)	% <100 °C. in Gasoline	O.N.	Method of Operating
1	Abstreifer KA2	K 36 s 1st shipment	26.0	955	640	9400	0.25	5.0	0.12	62-53*	4		Once Through
2	"	"	26.5	970	640	9400	0.5	2.5	0.16	52	3		"
3	Abstreifer KA2	K 36 s 2d shipment	26.0	955	660	9700	0.25	5.0	0.12	55	10-4		Once Through
4	"	"	26.0	955	660	9700	0.25	5.0	0.11	55	12		Recycle
5	"	"	26.0	955	660	9700	0.5	2.5	0.16	40	3		"
6	"	"	26.0	955	660	9700	0.75	1.7	0.19	37-33	3	>100	"
7	"	"	26.0	955	660	9700	1.0	1.9	0.15	20	2		"
8	"	"	26.0	955	660	9700	1.0	5.0	0.10	12	0		"

\* 62-53 means: Gradually Decreasing from 62 to 53

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Table II

Experiments with Zinc Sulfide on Terrana (K47)

No.	Oil	Catalyst	Furnace		Pressure		Feed Rate cc./Hr.	Yield G./cc./Hr.	Gasoline Conc. % <200° (392° F.)	% in Gasoline	O.N.	Method of Operating
			Temp. mK	°F.	Atm.	P.s.i.						
9	Abstreifer KA2	K 47 1st shipment	26	955	640	9400	0.2	0.1	90	49		Once Through
10	"	"	26	955	640	9400	0.3	0.12	90	58		"
11	"	"	26	955	640	9400	0.5	0.25	86	33		"
12	"	"	26	955	640	9400	0.7	0.38	78	21		"
13	"	"	26	955	640	9400	0.7	0.32	66	16		Recycle
14	"	"	26	955	660	9700	1.0	0.37	53	10	>100	"
15	"	"	26.5	970	660	9700	1.0	0.39	55	11	100	"
16	"	"	26.5	970	660	9700	1.2	0.45	52	8	>100	"
17	"	"	26.5	970	660	9700	1.5	0.52	49	6	>100	"
18	Welheim Diesel Oil	1st shipment	26.5	970	660	9700	1.0	0.37	51	7	>100	"
19	"	"	26.5	970	660	9700	1.5	0.47	45	5	>100	"
20	"	"	26.5	970	660	9700	1.5	0.44	40	5	>100	"
21	"	K 47 2d shipment	26	955	660	9700	0.2	0.1	75	22		Once Through
22	"	"	26	955	660	9700	0.5	0.25	60	10		Recycle
23	"	"	26	955	660	9700	1.0	0.32	33	5		"
24	"	"	26	955	660	9700	1.0	0.35	37	5		"
25	"	"	26	955	660	9700	1.5	0.4	32	5		"
26	2d shipment	"	26	955	660	9700	1.5	0.35	28	4	>100	"

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Table III

Balance - Experiments

	50 cc. Reactor (Furnace 19)		50 cc. Reactor (Furnace 18)		40 cc. Reactor (Furnace 17)		50 cc. Reactor (Furnace 16)	
	4	5	6	7	8	9	10	
5.4.1 Reactor (K47)	K47 2nd Batch		K47 1st Batch		Terrana attached with H <sub>2</sub> SO <sub>4</sub>		Bentonite + Al <sub>2</sub> O <sub>3</sub> (1:1)	
Catalyst	660	660	660	660	660	660	660	660
Pressure (atm)	26	26	26.5	26.5	26.5	26.5	26	26
Reactor Temp: mV/40°	Welheim Diesel Oil (2nd Batch)		Welheim Diesel Oil (1st Batch)		Welheim Diesel Oil (1st Batch)		Absstreifer KA2	
Oil	0.76	1.0	1.02	1.01	0.97	0.97	0.95	0.98
Feed Rate kg/l/hr.	2.5	1.85	1.85	1.6	1.5	1.5	1.5	4.0
m <sup>3</sup> H <sub>2</sub> /l oil	0.33	0.34	0.46	0.4	0.52	0.47	0.3	0.29
Yield kg/l/hr.	20.2	13.3	17.9	22.4	18.1	29	24.6	28.0
Gas Yield %	1.25	1.23	1.22	1.29	1.22	1.42	1.32	1.39
"Product Factor"	0.895	0.910	0.900	0.917	0.921	0.895	0.900	0.922
Products	50	40	52	46	40	58	55	37
Absstreifer	0.963	0.959	0.966	0.984	0.976	0.979	0.979	0.972
Gasoline > 200 Conc.	-18.3	-20	-18.2	-20	-20	-20	-20	-20
B-Product	326	346	337	330	325	320	347	305
Anilin Point, °C.	0.13	0.1	0.08	0.6	-	0.15	0.35	0.19
Acid Oils	0.830	0.830	0.830	0.859	0.854	0.840	8.844	0.853
Gasoline 200° d20	-10.5	-18.2	-19.6	-20	-20	-19.8	-20	-18.2
Anilin Point, °C.	0.52	0.58	0.58	0.45	0.53	0.52	0.51	0.52
Vapor pressure, atm.	95	96.5	97.5	100	100	100	100	100
Octane No.	0.23	0.1	0.1	1.2	1.6	0.15	0.35	0.35
IG Research	3.0	1.5	1.0	-	5	1.5	4	9
Acid Oils	20.4	13.4	15.0	8.5	8.0	18.5	14.0	12.0
Glass Dish mg/100 cc.								
>100° in gasoline Vol. %								

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