

of the liquid solution is still more substantial; painful wounds results from it. HF dissolves calcium in the human organism. HF meets the specification that the solvent must not react with the products to be treated.

In comparison to some other condensation agents (28), the use of HF offers the following technical advantages: The reaction mixture of HF and lignite Diesel fuel is at normal pressure and room temperature in the liquid phase. It is unnecessary to dilute the reaction mixture - as when sulfuric acid is used in refining - since HF will readily evaporate from the reaction mixture.

In discussing the price of HF we must refer to US conditions. HF is cheaper per molar weight than aluminum chloride and boron trifluoride. Sulfuric acid is cheaper, to be sure, yet with all that HF is superior, because a high percentage may be recovered by evaporation (16) and the formation of acid tar is avoided.

#### Conclusions and Basic Ideas of This Work

High grade Diesel fuel and lubricating oil have hitherto been manufactured from lignite-tar products, by means of catalytic condensation and a treatment with selective solvents. This operation takes place in two separate steps and involves a large expenditure in the production of lignite hydrocarbons, the approach of using concentrated HF as a catalyst and, at the same time, as a selective solvent.

The novelty of this method as compared with other solvent processes, lies in the feature that in one step a part of the charge stock undergoes a change in structure and also a selective refining.

We wanted to obtain high quality Diesel fuels and larger yields of lubricating oils, as well as a fuel oil of low solidification points by working up tar fractions with 95-100% HF.

Concentrated HF affects reaction boiling tar fractions which contain polymerizable and condensable hydrocarbons, in such a way that their physical and chemical structure is changed. The aliphatic and aromatic constituents of comparatively poor ignitibility are converted into high-molecular lubelike substances. In the extract are soluble, hydrogenous, sulfurous and nitrogenous compounds and high-molecular unsaturated hydrocarbons, which substances have the effect of reducing the burnability of Diesel fuel.

#### Selective Refinement of Diesel Fuel with 95-100% HF

By means of the solvent extraction method we aim at separating a mixture of liquids into a refined part and an extract. We operated by the simple extraction method. The oil mixture to be refined is brought into intimate contact with the extracting agent (HF). After the reaction has been derized and equilibrium established between the two liquid phases which evolve, the extract layer is separated from the refined oil layer and brought into a metal separating-funnel. The traveling of the interface is followed by measuring the differences in the conductivity of both layers.

For carrying out the tests an apparatus has been manufactured (fig. 1) consisting of a:

- (1) Cooling bath container,
- (2) Reactor with stirrer,
- (3) Electromotor,
- (4) Support.

The cooling bath container is equipped with an insulating jacket filled with glass wool (3). It serves as a container of the cooling liquid (K) and as a support to the reactor (R). At the bottom it is equipped with a socket (A) which is closed with a stopcock used for reading (sic) the cooling liquid. The inserted steel container is closed by means of a lid (D) with four hinged screwlocks; the lid is sealed with a lead ring. The stirrer (F) is passed through the lid and sealed by means of a lead seal. The thermometer socket (T) holds the thermometer. The reactants are fed through the inlet tube (B). The extract and the refined material are discharged through a steel tube which is attached at the bottom of the container; the tube is equipped with a chamber. Two platinum electrodes (Pt) are inserted in the chamber, fused in with sulfur; they serve for measuring the conductivity of the liquids passing through the chamber, for the purpose of observing the travelling of the interface. Above and below the chamber are the metal ground-in cocks (H<sub>1</sub> and H<sub>2</sub>).

When the polymerization is terminated, the paraffinic and condensed components are separated from the original hydrocarbon mixture by selective refining with HF, and gather in the reformer layer. The extract contains all the asphalt constituents, oxygenates, sulfurates and nitrogenous compounds and high-molecular unsaturated hydrocarbons. The reformer layer contains about 5% by weight of the HF charge and the extract 95% by weight. The hydrofluoric acid is evaporated at 100°C. The refined layer contains the diesel and lubricating oil fractions. The extract consists of a suitable fuel oil of low melting point.

All experiments have been carried out with the same amount of HF Diesel fuel. Feed stock used in each of the tests was 500 g of Diesel fuel. Table 2 summarizes the analytical data of the starting-material Diesel fuel.

In order to determine the order of reformation obtained with HF, the original Diesel fuel and all the conversion products obtained from it, have been subjected to distillation at subatmospheric pressure, being divided into two fractions: one Diesel fraction the boiling range of which at 12 Torr goes up to 220°C (350°C at 760 mm), and another lubricating-oil fraction the boiling range of which goes from 220°C up to the cracking limit.

Table 2 registers the values obtained for yields, and the analytical data concerning the Diesel and lubricating oil fractions.

We have run a series of tests with regard to the alkylation and polymerization power of HF, using a commercial Diesel fuel of the Auhaltische Kohlenwerke. We tested the effects which the reaction time, the ratio of HF to lignite Diesel fuel, the reaction temperature and the discharge temperature, have upon the conversion rate, the yields and properties of the reaction products.

Reactions, when the mixing ratio and temperature are constant while the reaction periods vary.

The original Diesel fuel has been reacted with 96-100% HF for different lengths of time of the components the ratio and the temperature of the mixture being constant. The reaction took place in the equipment described above.

800 g of Diesel fuel are charged to the steel containers and cooled to  $-2^{\circ}\text{C}$ . 800 g of hydrofluoric acid are discharged from a steel bomb weighed in a 1 liter steel flask and then cooled to a temperature lower than the test temperature. They are added to the stock slowly and under constant stirring. The reaction temperature has been observed at the thermometer inserted in the socket. During the initial 30 minutes, the temperature fluctuated  $\pm 2^{\circ}\text{C}$ . Thereafter the reaction temperature has been held constant at  $\pm 1^{\circ}\text{C}$ . The respective conversion periods were: 30, 60, 90, and 270 minutes. The automatic stirrer was turned off when the reaction was terminated. After a few minutes the two phases separated. In order to obtain a uniform equilibrium, in all of the tests, extract and refined material have been separated at a uniform temperature of  $+10^{\circ}\text{C}$  and after a settling period of 15 min. HF was separated from extract and refined material in an evaporator. After evaporating the HF, we washed the refined oil and the extract with warm lye and sodium chloride solution, for the purpose of removing any acid constituents. Both the refined material and the extract have been dried over calcined sodium sulfate, filtered and weighed.

The refined material has been separated into a Diesel oil fraction of a boiling range of up to  $220^{\circ}\text{C}$  at 12 Torr, and into a lubricating oil fraction of a boiling range of up to the cracking temperature. The lubricating oil fraction has been distilled at 4 Torr. In order to remove any traces of HF the stock has been distilled over calcined calcium-oxide powder. None of the fractions contained any HF compounds.

Tables 3, 4 and 5 give the yields and analytical data.

In fig. 2-5 the yields of extract and refined material, or fuel oil, Diesel oil and lubricating oil and the analytical data have been plotted as a function of the reaction period. With reaction periods of 30 to 90 minutes the yields of refined material (fig. 2) went up from 64 to 59% of the feed stock, and the yields of Diesel oil (fig. 3) went up from 31.5 to 24% by weight, of lubricating oil from 17.5 to 20% by weight, calculated on the basis of the original hydrocarbon mixture, fuel oil going down from 45 to 42% by weight. At a reaction time of 75 minutes, the main process with regard to the conversion of olefins and aromatics in the presence of 96-100% HF is practically terminated. Consequently, a longer reaction period cannot substantially change the yields. The condensation of olefins and aromatics does not cause a substantial reduction in the specific gravity (fig. 4) of the Diesel fuel and lubricant fraction; the olefins are converted to higher molecular olefins and, with aromatics, to alkylated compounds. With increasingly long reaction periods the characteristic viscosity of the lubricating oils (fig. 4) goes up and with it the "Polheche" (\*). These findings are conformant to A. V. Schmidt's, stated in his studies on uniform alkylate aromatic

(\*) The "Polheche" value is similar to but not identical with the viscosity index in that it is an evaluation of the rate of change of viscosity with temperature (M.B.).

compounds, saying that the specific gravities and refractive indexes of alkyl-benzols (29) decrease with an increase in the number of C-atoms in the side chain, while the characteristic viscosity slowly increases. If the molecules contain more than 10 C-atoms, the properties of the paraffinic side chain exercise an increasingly greater effect upon the nature of the molecules.

The mean iodine number of the Diesel fuel is 26, that of the lubricating oil 37 (fig. 5). The maximum values of the octane numbers — determined by the Bardon aerometer method — are reached after a 75 minutes reaction time (fig. 5) and remain about constant. The average solidification point of the Diesel fuel is the same as that of the feed stock; that of the lubricating oil is +8°C. The sulfur content — it is 0.0% by weight in the Diesel fuel and 0.5% by weight in the lubricating oil — is substantially improved by selective reformation. The Conradson test, which represents an approximation value of the coking-tendency of Diesel and lubricating oils, gives a value of 0.0% by weight for Diesel fuel and an average value of 0.25% by weight for lubricating oil (Tables 4 and 5).

(Rest of text — there was obviously at least one more section in the original — and bibliography are not included in the reel. M.B.)

M. Both

MB/ed

TABLE 2

Analytical Data of ASL Diesel Oil

Specific Gravity at 20°C	0.9087
Boiling Number	293
Cetane Number (Aerometer Method by Bardon)	45
Iodine Number	83.0
Solidification Point (°C)	-7.5
Sulfur Content (% by Weight)	1.22
Ash Content (% by Weight)	0.14
Conradson Number (% by Weight)	0.15

Yields and analytical data of the feed stock Diesel oil, separated into a Diesel oil fraction and a lubricant fraction.

Yields	640 g Diesel Oil	80%
	145 g Lubricating Oil	18%
	15 g dist. loss and residue	2%

0590

Properties of Diesel Fuel

Specific Gravity at 20°C	0.900
Boiling Number	280
Cetane Number (Aerometer Method by Harder)	45
Iodine Number	92.8
Solidification Point (°C)	-14
Sulfur Content (% by Weight)	0.59
Ashes Content (% by Weight)	0.0
Conradson Number (% by Weight)	0.16

Properties of Lubricating Oil

Specific Gravity at 20°C	0.9449
Iodine Number	73.0
Solidification Point (°C)	+13
Sulfur Content (% by Weight)	0.93
Ashes (% by Weight)	0.01
Conradson Number (% by Weight)	0.20
Viscosity in "E at 20°C	6.6
"    50°C	2.3
in c St at 20°C	50.7
"    50°C	14.4
Viscosity - polhoehc	1.79

TABLE 3

	Yields in % by Weight			
Reaction Time in Minutes	30	60	90	270
Extract	46.0	43.7	41.2	45.0
Refined Product	54.0	56.3	58.8	55.0

	Yields in % by Weight			
Reaction Time in Minutes	30	60	90	270
Fuel Oil	45.0	43.0	42.0	44.0
Diesel Oil	31.5	33.0	34.0	30.0
Lubricating Oil	17.5	19.5	20.0	23.0
Washing and Dist. Losses	6.0	4.5	4.0	3.0

H. Beth  
July 8, 1948

Distribution: All Divisions  
AEM (25)

0591

TABLE 1

## Physico-Chemical Properties of Selective Agents

Translation Book #176  
Reel 113 Dec. 7

Name	Molecular Weight	Specific Gravity	Fp in °C	Kp in °C	Kr in °C	F Kr in Atm.	At D in °C	Mol. Heat. Cp in Cal. Mol <sup>-1</sup> degree <sup>-1</sup>	Evaporation Enthalpy		Vapor Pressure			
									At ΔT <sub>K</sub> in °C	in K Cal Mol <sup>-1</sup>	10 Torr	100 Torr	760 Torr	5 Atm.
Methanol	32.04	0.7923 <sup>20</sup>	-97.1	64.7	24.0	99	20	18.44	20	9.19	-15.7	20.9	64.7	112
Ethanol	46.07	0.7892 <sup>20</sup>	-114	78.3	24.3	63	200	25.66	20	10.33	-2.7	34.9	78.3	126
Phenol	94.11	1.0545 <sup>45</sup>	41	181.4	419	50	22.6	31.8	183	11.50	-12.5	26.4	80.3	144
-o		1.0482 <sup>20</sup>	31	191	422	-	0.30	53.9			77.6	127.4	190.1	-
-m Cresol	108.13	1.034 <sup>20</sup>	10.9	202	432	45	0.20	51.8	201	10.9	87.8	138.0	200.5	-
-p		1.0347 <sup>20</sup>	33.8	202	426	-	0.20	52.6			88.5	138.4	201.6	-
Aniline	93.12	1.0217 <sup>20</sup>	-6.2	184.4	425	52	25	45.6	184	9.71	69.2	119.4	183.9	-
Furfurol	96.08	1.1594 <sup>20</sup>	-36	161.6	-	-	-	-	-	-	-	-	-	-
Acetone	58.08	0.7960 <sup>15</sup>	-95	56.3	235	47	17.20	29.9	27.5	7.713	-32	7.3	56.13	109
Benzol	78.11	0.8786 <sup>20</sup>	5.49	80.12	288	48	18	31.8	20	8.073	-12.5	26.4	80.3	144
Nitrobenzol	123.11	1.2229 <sup>0</sup>	5.7	210.9	-	-	30	44.1	209	11.67	85.4	139	208.3	-
Propane	44.09	2.0037(*)	-189	-42.6	97	42	0	16.12	-30	4.32	-115	-83	-44.5	0
Liquid Sulfur Dioxide	64.06	1.46-10	-75.5	-10.0	197	88	25	9.52	-10	5.96	-75	-47.7	-10	-
Hydrofluoric Acid	20.01	0.987	-85	19.5	230	-	18	6.67	19.5	6.15	-66	-29	19.5	-

(\*) kg/m<sup>3</sup>

MB/ed  
8-3-48

0592 TABLE 1  
Diesel Fuel - Properties

Translation Book #176

	Specific Gravity D <sub>20</sub> <sup>20</sup>	Boiling Characteristic Number	Cetane No. (*)	Solidification Point °C	Iodine Number	Sulfur Content % by Weight	Ashes % by Weight	Conradson Number % by Weight
Diesel Fuel, Reaction Time - 30 Minutes	0.859	294	66	-4	23.2	0.0	0.0	0.0
Diesel Fuel, Reaction Time - 60 Minutes	0.856	294	67	-4	25.9	0.0	0.0	0.0
Diesel Fuel, Reaction Time - 90 Minutes	0.854	293	69	-5	25.6	0.0	0.0	0.0
Diesel Fuel, Reaction Time - 270 Minutes	0.850	287	69	-7	24.1	0.0	0.0	0.0

(\*) Determined by the aerometer method by Marder.

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8-3-48

0593

TABLE 5

Lubricating Oil - Properties

Translation Book #176

	Specific Gravity D <sub>4</sub> <sup>20</sup>	Iodine Number	Solidification Point °C	Ashes % by Weight	Sulfur Content % by Weight	Conradson Number % by Weight	Viscosity				Polhoehn
							°E		c St.		
							20°C	50°C	20°C	50°C	
Lube, Reaction Time - 30 Minutes	0.9204	40.1	+8.5	0.0	0.51	0.42	21.6	4.3	176	32.1	27
Lube, Reaction Time - 60 Minutes	0.9185	37.8	+7	0.0	0.55	0.28	24.0	4.6	181	34.5	2.4
Lube, Reaction Time - 90 Minutes	0.9162	37.3	+8.5	0.0	0.51	0.29	21.8	4.4	156	30.8	2.3
Lube, Reaction Time - 240 Minutes	0.9126	37.0	+8	0.0	0.51	0.25	16.0	3.7	123	26.7	2.1

MB/ed  
8-3-48



Translation Book # 176

Condenser and  
Cooling Bath Vessel

0594

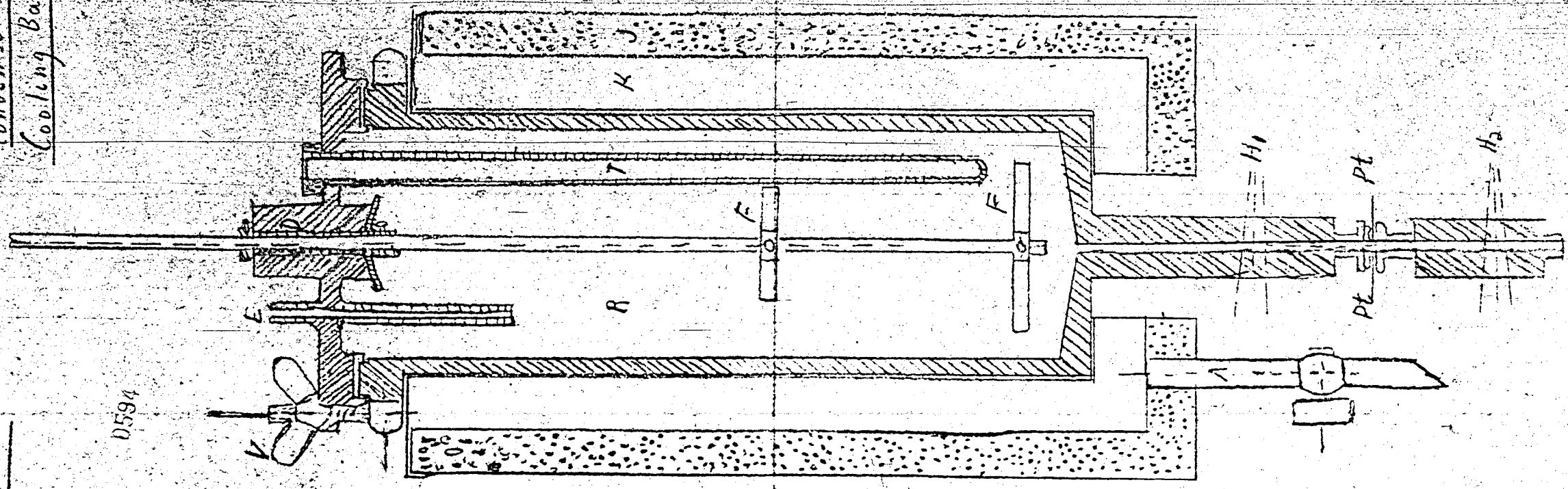


FIGURE 1

Translation Book #176

Condensing agent: 96-100% HF  
Reaction Temperature: +0°C  
Ratio of Diesel Oil to HF = 1:1

Fig. 2

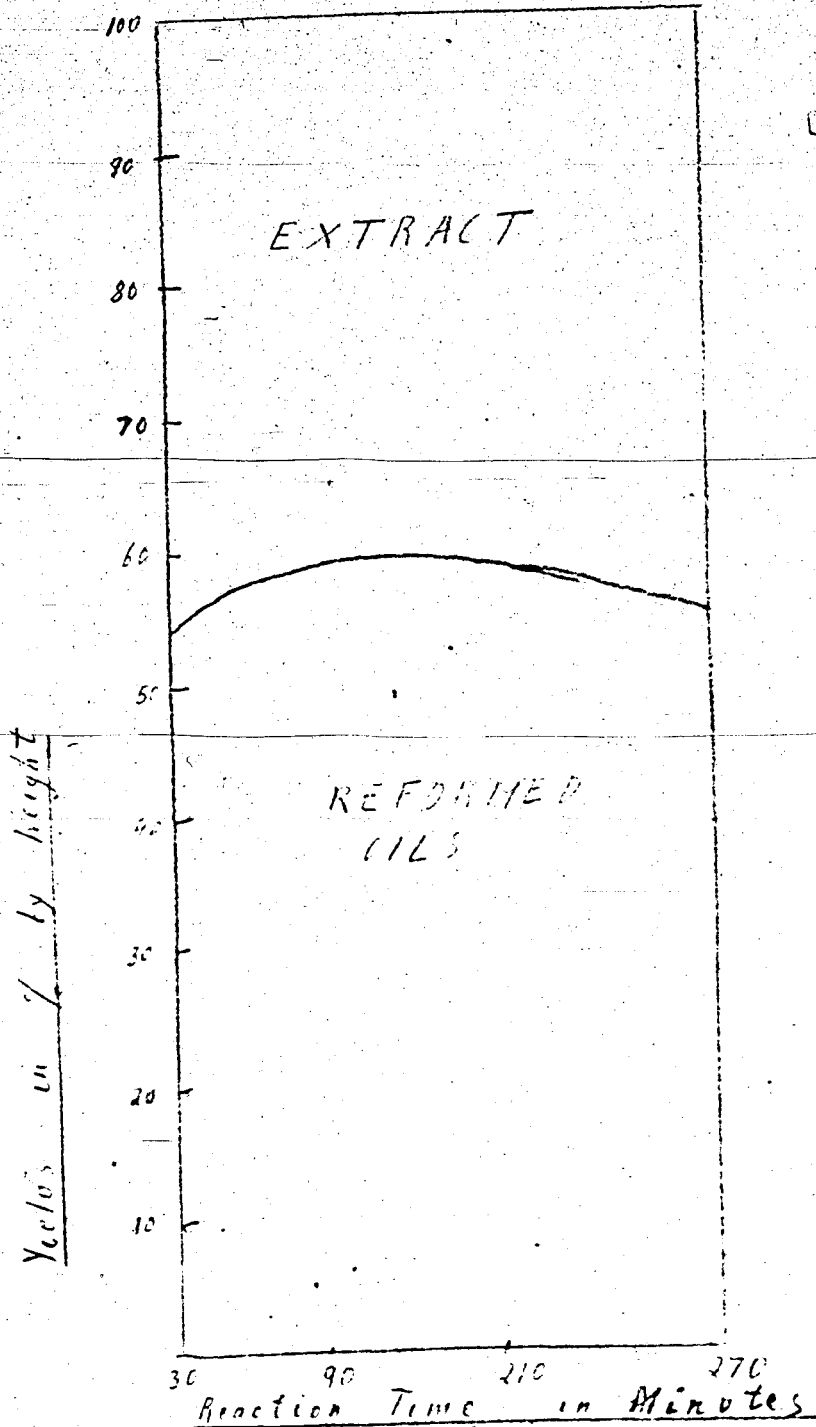


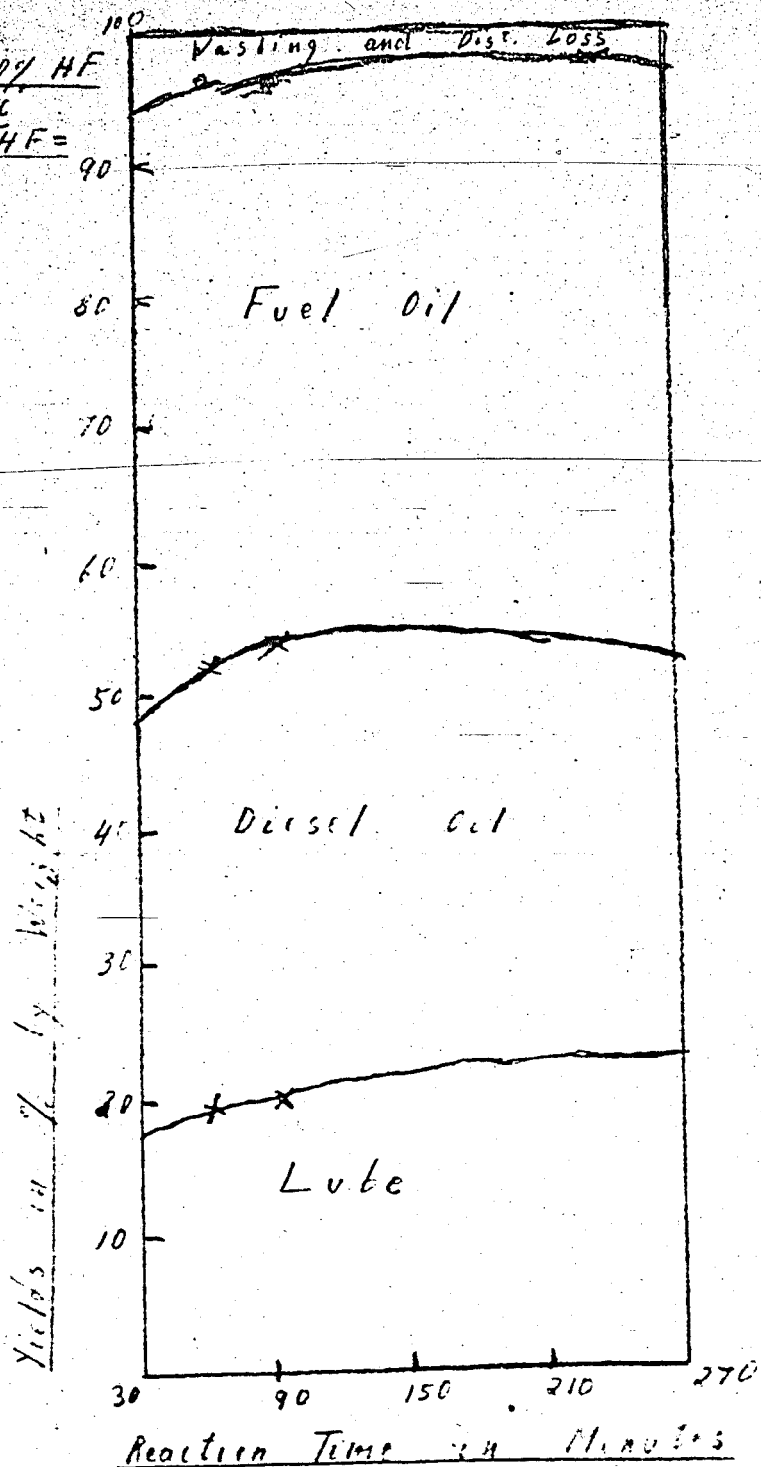
Fig. 3

0596

Condens. Agent: 96-100% HF

Reaction Temp.: +0°C

Ratio Diesel Oil : HF =  
= 1:1

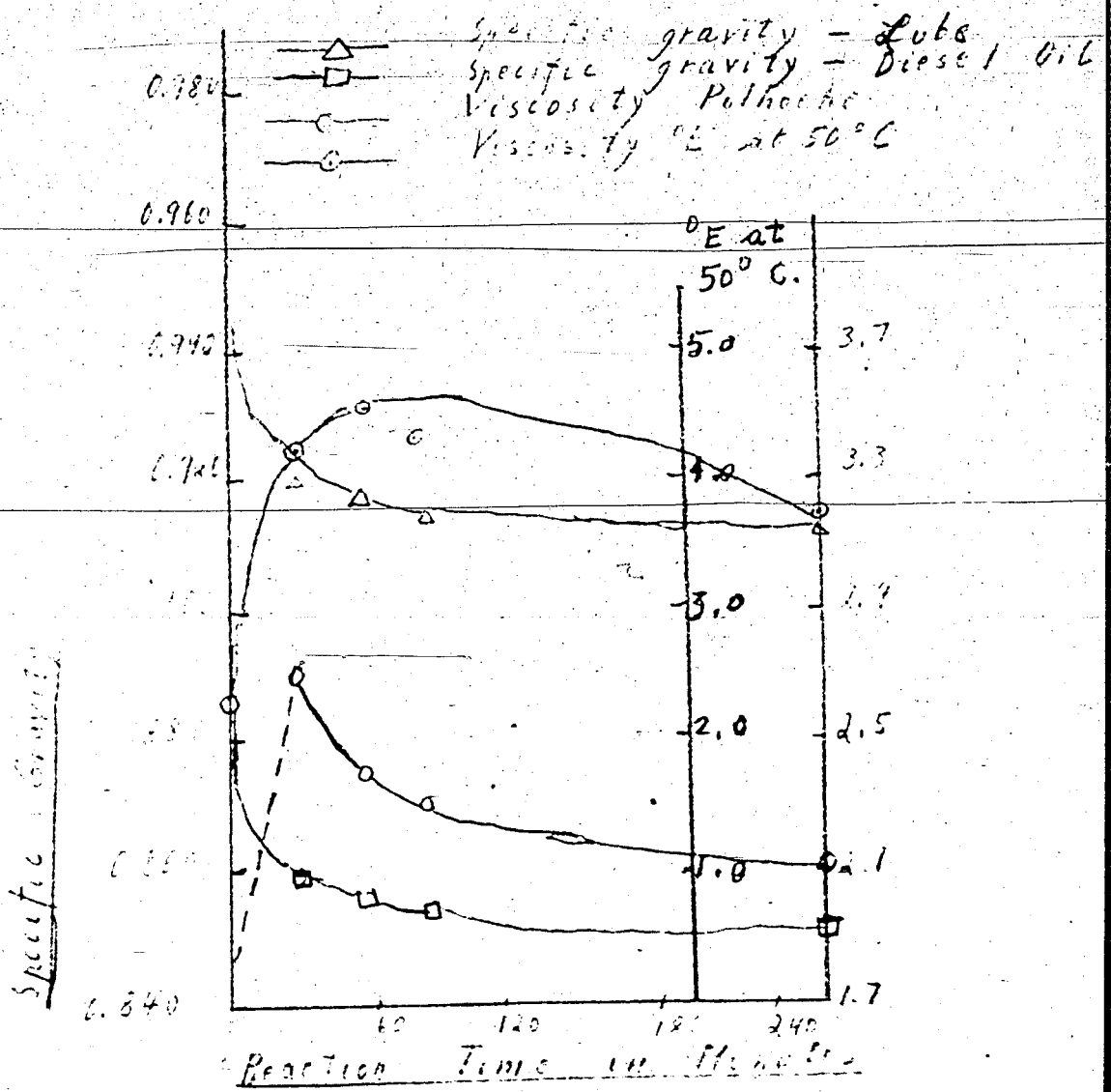


Translation Book #176

Fig. 4

0597

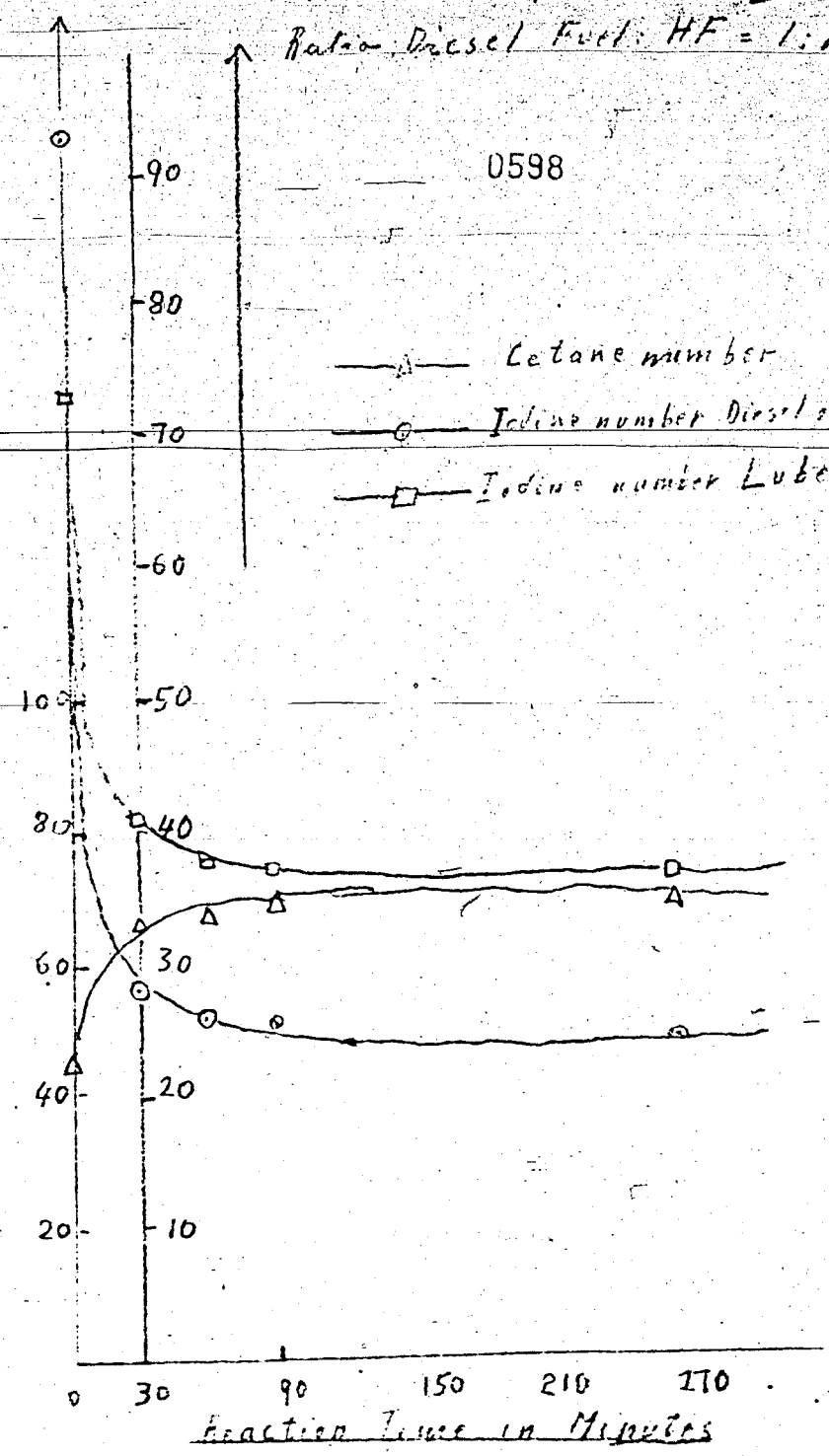
Condens. Agent 98-100% HF  
Reaction Temp.  $+ 0^{\circ}C$   
Ratio: Diesel Oil : HF = 1:1



Translation Book # 176

Fig. 5

Condens. Agent: 96-100% HF  
Reaction Temperature:  $\pm 0^{\circ}\text{C}$   
Ratio Diesel Fuel: HF = 1:1

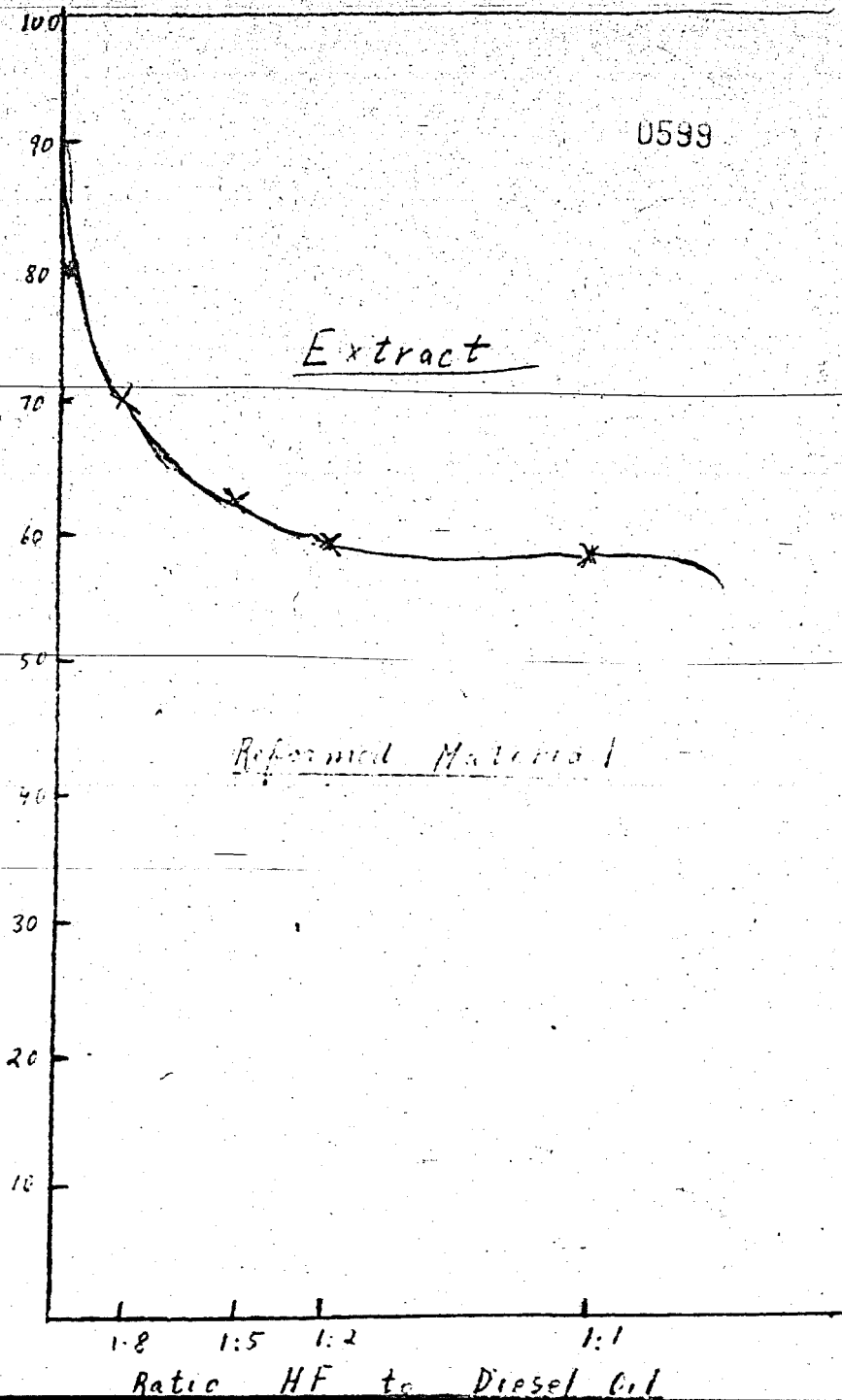


Translation Book #176

Fig. 6

Condens. Agent: 96-100% HF  
Reaction Temperature: + 0°C  
Reaction Time: 75 minutes

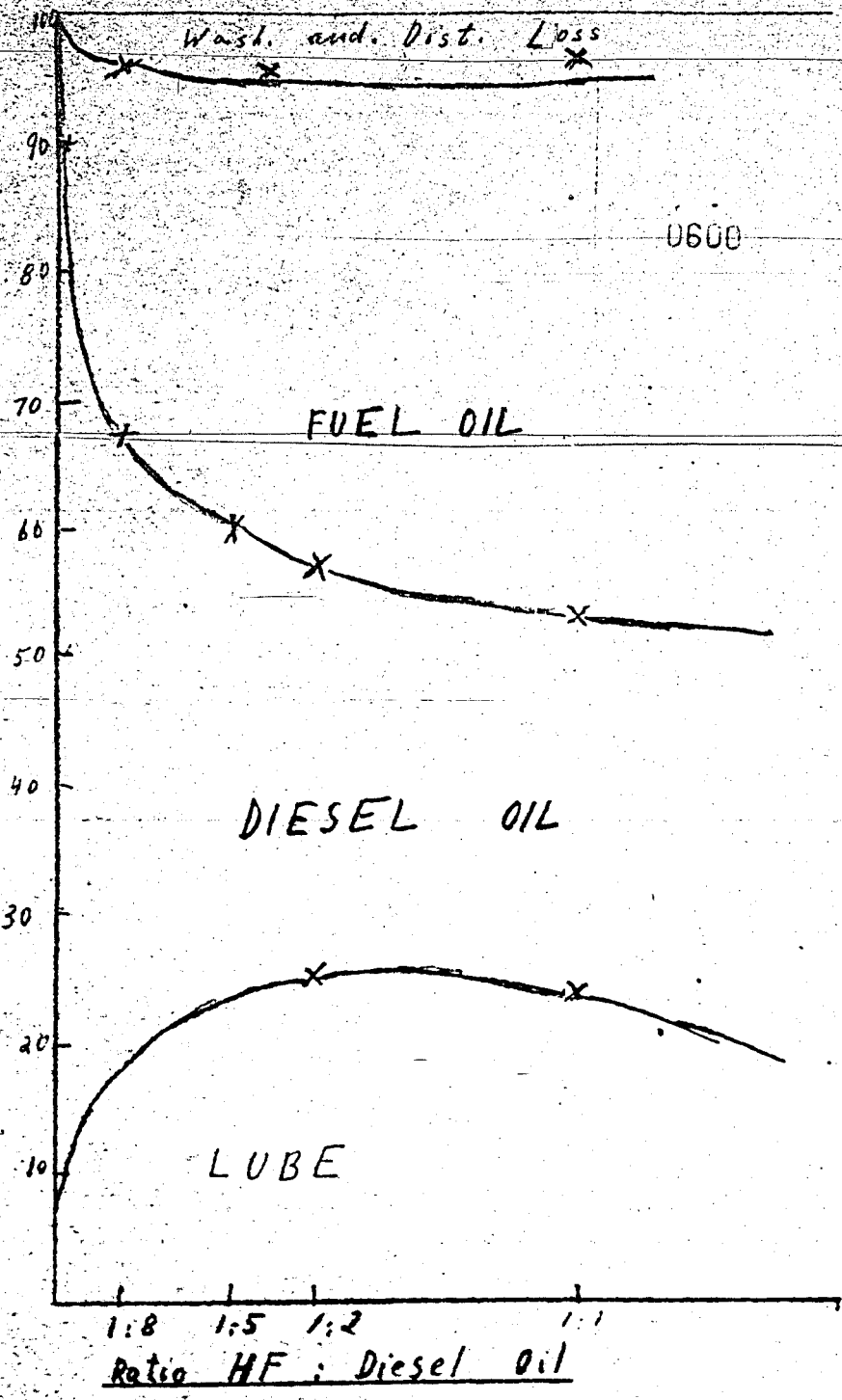
Yield in % by Weight



Translation Book # 176

Fig. 7

Condens. Agent: 96-100% HF  
Reaction Temperature:  $\pm 0^{\circ}\text{C}$   
Reaction Time: 75 minutes

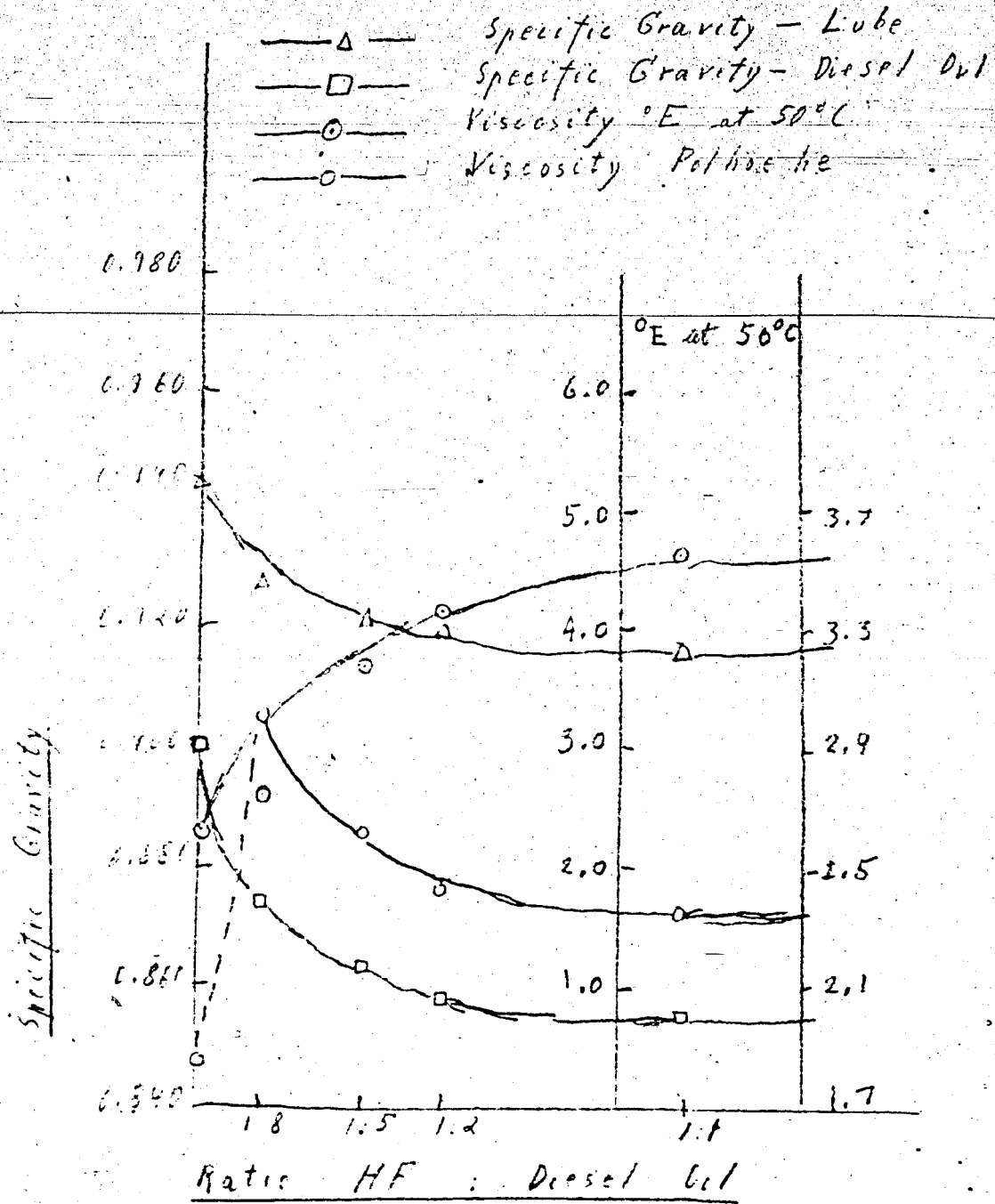


Translation Book #176

Condens. Agent: 96-100% HF  
Reaction Temperature: + 0°C  
Reaction Time: 75 minutes

Fig. 8

0601





Translation Book No. 196  
 Reel 195  
 Part 12, Dec. '9  
 Frame 31504

0602

August 31, 1948

Flow SheetPreparation of HF - Catalyst

## Explanation

- No. 11: Storage Tank for ortho - p acid - 20 %  
 Capacity : 20,000 liter  
 Covered with lead
- No. 12: Container --- 80 %  
 Capacity : 200 liter ; 600 lb ; 300 kg.  
 Iron, rubber lined
- No. 13: Condenser & Cooler
- No. 14: Measuring apparatus (2 pieces)  
 Capacity 40 liters  
 Iron, rubber lined
- No. 15: Reactor  
 at 1000  
 Temperature 250° C
- Charging: 2 hours  
 Boiling: 4 hours  
 Number of kettles: 3 pieces and 1 in reserve  
 Charge in each kettle: 35 liter  
 Height of liquid: 105 cm (?)
- No. 16: Cooler
- No. 17: Kneading apparatus  
 Capacity: 35 liter each (charge 35 liter; loose)  
 Kneading period: 4 hours  
 Charging period: 2 hours  
 Number of machines: 2 pieces and 1 in reserve
- No. 18: Scales for the kneading machines. (Charge: 10 + 11 kg.  
 i.e. 35 liters)

0603

No. 49: Vacuum drier

Drying period: 12 hours at 140° C  
14 mm Hg abs. ....(illegible)  
Drying area: 30 m<sup>2</sup> each drier  
Number of driers: 2 pieces and 1 in reserve.

No. 50: Measuring apparatus  
Capacity 30 liters  
Measuring capacity 22 liters

No. 51: No text (H.B.)

Performance: 190 tons per year; 25 kg/m finished catalyst  
Bulk weight: 0.8

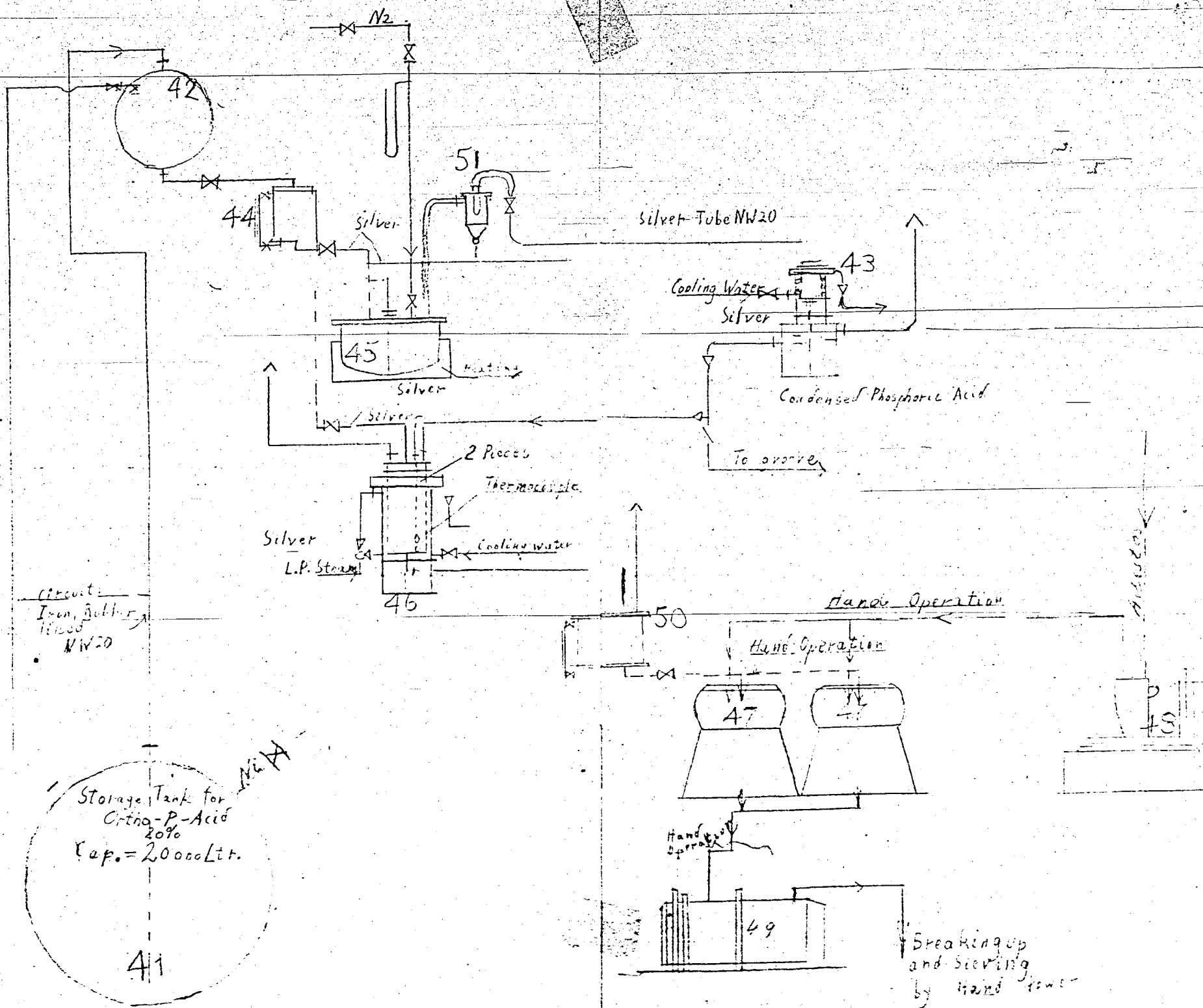
Each finished charge of the kneading machines yields  
32 liters of paste -- bulk weight: 1.05

*M. Beth*

M. Beth

MB/mb

SPILLWAY



circuit  
Iron, Rubber  
11/1000  
NW20

Storage Tank for  
Citric-P-Acid  
20%  
Cap. = 20,000 Lit.  
41

Silver  
L.P. Storage  
Cooling water

Silver-Tube NW20

Cooling Water  
Silver

Condensed Phosphoric Acid

Hand Operation

Hand Operation

Breaking up  
and Sieving  
by hand power

DETECTION OF ISO-AND ALICYCLIC COMPOUNDS IN SATURATED  
HYDROCARBON MIXTURES

By Dr. Doznow-Petroleum Institute of Hanover Technical School-July 7, 1944  
T.O.M. Recl. 73, Item 5 Pages 587-607

As previously reported, I have discovered a simple color reaction sensitive to 0.01 percent for the detection of hydrocarbons with tertiary carbon atoms.

In this determination, 1 ccm. of hydrocarbon mixture, 0.5 ccm. of a one-half molar solution of  $AlEt_3$  in  $CS_2$ , and 0.075 ccm. of a 10% emulsion of chlorosulfonic acid ester in  $CS_2$  are allowed to react for five minutes at room temperature. The oil layer which has formed is then decanted, and a small quantity of methanol is added dropwise. The appearance of an orange to blue color indicates the presence of tertiary hydrocarbons.

The purest normal hydrocarbons, as for example pentane obtained by repeated distillations of petroleum ether, produce no color. (Isomer-free heptane can also be produced from oenanthic alcohol by means of the hydrazone.) The purest cyclopentane, likewise, does not show this color reaction. All of the aliphatic and alicyclic hydrocarbons with tertiary carbon atoms which we have investigated, however, do give a decided color.

The general applicability of this method of analysis and its limitations have been further established.

Literature covering research on petroleum and synthetic hydrocarbons indicates great interest in the composition and structure of the components of the various fractions. Recent discussions concern compounds which contain very reactive chemical groups. These reactive linkages are the aromatic and olefinic double bonds and the oxygen, sulfur, and nitrogen containing groups.

It is more difficult, however, to determine the chemical structure of saturated hydrocarbons (the paraffins and the cycloparaffins). These are slow to react, and consequently most chemical reactions are not applicable to them. When these compounds react, moreover, no absolute conclusion about the starting materials could be formed because of the great number of their isomers.

The recent tendency to make fuller use of oil and synthetic hydrocarbons has brought forward many questions concerning their structure. The effort to construct more efficient gasoline and diesel motors brings up new problems about the relation between the process of combustion and the composition and structure of the fuel. Structural questions are also raised in work with lubricating oil in the manufacture of fatty acids and other important products from paraffins. A great deal of progress has already been made toward an understanding of hydrocarbon construction; nevertheless it becomes more and more obvious that chemical methods required for obtaining a closer insight are still lacking. In modern chemistry the wider application of catalysis to isomerization, aromatization, cyclization, and hydrocarbon synthesis reactions, increases the need for

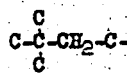
better analytical methods for detecting changes in the structure of the products.

The known methods for the determination of structure and isomerization have been investigated and none of them were found to be satisfactory.

All saturated aliphatic and alicyclic hydrocarbons can be classified into the following groups:



I



II



III



IV

Experience has shown that the tertiary bound carbon atom possesses the movable hydrogen, and is, accordingly, quite reactive. Many attempts have been made to find chemical reagents which would attack this tertiary carbon atom specifically, and thereby distinguish this group of compounds. Nitric acid, fuming sulfuric acid, mixed acid, chlorosulfonic acid, and oxidizing agents such as  $\text{H}_2\text{O}_2$ , have been tried, but none of these are sufficiently specific.

Chlorosulfonic acid and antimony pentachloride show a great differentiation between tertiary and other hydrocarbons, therefore these two reagents were further investigated. Young (1) and Marcussen (2) used chlorosulfonic acid to separate tertiary iso compounds from paraffins. Later, in order to determine the content of normal paraffins in ceresins, use was made of the similar reaction of Holze and Schmeemann (3), in which the isoparaffins heated with chlorosulfonic acid were partly decomposed to carbon. Sheppard (4) and co-worker also used this reagent for the isolation of normal paraffins from  $\text{C}_5$  to  $\text{C}_{12}$  and they gave the constants for the compounds purified by this method. According to Von Schearschmidt (5), all reagents used for purification which are insoluble in hydrocarbons are not very satisfactory and show little differentiation in chemical effect. He defined a satisfactory reagent as one which is completely soluble in hydrocarbons and reactive specifically with the tertiary carbon atom. He believed antimony pentachloride to be such a reagent because it preferentially attacks the tertiary carbon atom. A closer inspection of the report made by Von Schearschmidt and co-worker, however, questions the practicability of their reaction; Koch and Hiltnerath previously mentioned the same doubts (6).

In the search for other reagents, the idea of using chlorosulfonic acid again occurred to us. We used, however, the ethyl ester (which is completely soluble in hydrocarbons) in the hope of converting the hydrocarbons to their sulfonic acid ester. We then investigated methyl cyclohexane (a hydrocarbon with a tertiary carbon atom). Upon prolonged heating of equimolar amounts, the condensate from the reflux cooler separated into layers. The upper layer had the boiling point of the converted hydrocarbon, while the heavier layer contained diethyl sulfate.

In order to shorten the reaction time and to thereby avoid the formation of diethyl sulfate, the reaction was carried out in the presence of one mole of aluminum bromide.  $\text{AlBr}_3$  proved to be very suitable because it dissolves readily in hydrocarbons. The reaction proceeded so violently

with the production of hydrocarbon halide, that cooling was essential. Ice water served to halt the reaction and it produced a strong resinification. The product was dissolved in benzene or ether, whereupon a strong green coloring could be observed. After steaming off the solvents, a small quantity of a higher boiling material (as well as the diethyl sulfate and the starting material) was found. We performed the same experiment with triptane in order to rule out the possibility of ring cleavage or of rearrangement. The product was found to be of essentially the same nature as that from methyl cyclohexane. The observation that at the end of the reaction unchanged hydrocarbon was always present led to the assumption that the reaction was incomplete or perhaps reversible.

In the search for water-like substances to produce milder reaction, alcohols were next used for the decomposition. After the addition of methanol (or other alcohols) to the conversion products, a red-violet color appeared. Generally the pure tertiary hydrocarbons (particularly those free of olefins) exhibited typical color reactions.

This reaction was then carried out by adding  $AlPr_3$  to a mixture of hydrocarbons and chlorosulfonic acid ester. The reaction mixture became warm and isomerization (attributable to the  $AlPr_3$ ) occurred. Cyclohexane yielded a blue-green color though it has no tertiary carbon atom (provided that methyl cyclopentane, a compound of the same molecular weight, has been removed). We were therefore of the opinion that isomerization had taken place, but later investigations revealed that the cyclohexane was not sufficiently pure.

In order to decrease the possibility of isomerization, the reaction was carried out under mild conditions, i.e., a solution of  $AlPr_3$  was used and a few drops of chlorosulfonic acid ester were added.  $CS_2$  proved to be a suitable solvent for  $AlPr_3$  in this reaction. Upon varying the concentration of  $AlPr_3$ , a point was reached where even cyclohexane produced no color reaction. This concentration was found to be half molar. To approximately 0.1 cc. of hydrocarbon were added about 5 cc. of half molar  $AlPr_3$  solution in  $CS_2$  and about 3 drops of chlorosulfonic acid ester. After three minutes reaction, methanol was added to the separated oil.

When the above procedure was applied to cyclohexane, no color appeared. Methyl cyclohexane, however, yielded a clear green color.

In the case of n-octane (pure, synthetic, from Schering) there was observed a clear red color which revealed the presence of tertiary hydrocarbon impurities. After the octane was purified again the same way, it showed no color.

In order to establish the sensitivity of the reaction to isocompounds, it was necessary to check mixtures of the purest normal paraffins against isocompounds. Therefore we investigated pure synthetic Schering n-octane in mixture with pure synthetic 2,5-dimethylhexane. It proved that if 1 ml. of this mixture is reacted for three minutes with the same amount of a one-half molar  $AlPr_3$  solution in  $CS_2$  and 0.25 ml. of a 10% emulsion of chlorosulfonic acid ester in  $CS_2$ , and is decomposed with methanol by the method mentioned above, not only a 1% solution but also a 0.1% solution of iso-octane in n-octane would give a clear color reaction. Under similar conditions a 0.01% solution caused the appearance of a weak rose color which, however, appeared also with n-octane alone. It was therefore

apparent that even the supposedly pure n-octane contained a small quantity of isocompounds whose presence was revealed in this more sensitive reaction process or that during the three minute reaction the  $AlPr_3$  encouraged isomerization.

The results obtained from an experiment on the purest available n-heptane from Hayl & Co. were very similar. A mixture of methyl cyclohexane in n-heptane which was analyzed showed that even a 0.1% solution of methyl cyclohexane in n-heptane gives a clear green color, provided that to 2.5 ml. of this solution the same amount of 1/2 molar  $AlPr_3$  and a drop of chlorosulfonic acid are added and the mixture allowed to react for three minutes. Under the same conditions, the n-heptane alone gave a weak green color which appeared after approximately a one-half hour contact of the conversion products with methanol. The n-heptane, therefore, could also have contained a small quantity of isocompounds.

Methyl cyclohexane mixed with cyclohexane could readily be detected by a clear green color, while the cyclohexane, which had been more thoroughly purified, showed a golden yellow color.

In order to determine the sensitivity limits more accurately and to decide whether isomerization occurred during the reaction it was necessary to produce a very pure hydrocarbon. For this purpose oeanthol was distilled several times over a rectifying column one meter in length with glass rings. The fraction boiling at constant temperature (71.8-72.0), 49 mm., was converted according to standard procedure into the hydrazone, washed with ether, and dried at 35° for 25 hours. It was then heated with a mole of sodium ethylate at 160° and the n-heptane was separated as it was produced and removed by means of a Vigreux column. The distillate was washed with water, sulfuric acid (1.84), caustic soda and again with water, dried with  $CaCl_2$  and distilled from the sodium through the column mentioned above. The n-heptane so obtained showed an R.I. of  $n_D^{20} = 1.3878 \pm 0.0001$ . This n-heptane from oeanthol gave no color reaction in the test. A 0.01% solution of 2,2,4-trimethyl pentane (I.G.  $n_D^{20} = 1.3920$ ) in n-heptane showed red coloring when 2 ml. of this mixture had been allowed to react with 0.5 ml. of one-half molar  $AlPr_3$  and  $E_2SO_4$  and 0.075 ml. of a 10% solution of ester in  $CS_2$  for five minutes. 2,5-dimethyl hexane could be detected in 0.01% solution, and 2,2,3-trimethyl butane (triptans) in not less than a 0.1% solution, apparently because the methyl groups restrain the tertiary H-atom. The testing of 2 ml. of a 0.01% solution of methylcyclohexane resulted, after five minutes, in a clear reaction - in this case a green color.

Isocompounds with quaternary carbon atoms cannot be detected by this color reaction. The tetramethyl butane and the 2,2-dimethyl butane which were obtained by means of the pinacole through splitting with sodium alcoholate and were distilled through the column of Koch-Kilgerath were tested to confirm this statement.

Pentane obtained from petroleum ether through distillation in the same column yielded practically no color reaction. We have, furthermore, submitted paraffin oil D.A.E.6 and solid commercial paraffins to our test and have observed similar color reactions.

In another set of experiments, purest cyclohexane of  $n_D^{20}=1.4267$  was investigated by the same treatment. This yielded droplets of reddish tinge, but the red subsequently changed into a golden yellow. This result causes one to suspect that methyl cyclohexane is present in detectable amounts in the narrowest fractions of cyclohexane obtained by the usual means. Thermodynamic computations show that at equilibrium at room temperature a few percent of methyl cyclopentane is contained and Benitzescu (7) has demonstrated experimentally that at the boiling point of cyclohexane about 22.8% of methyl cyclopentane is present. Mizushima and co-worker proved by means of the Raman spectrograph that methylcyclopentane is the only isomer.

We therefore prepared methyl cyclopentane according to the process of Benitzescu (I.O.) mentioned above and carried out a careful distillation. In concentrations ranging from 1% to 100% there appeared a red orange color which soon changed to a dirty red brown. With greater dilution (0.1%) the color remained golden yellow, showing the presence of cyclohexane. We have, nevertheless, investigated a mixture of methyl cyclohexane in cyclohexane. The green color of the methyl cyclohexane covered up the yellow gold color of the methyl cyclopentane which was present. In a 0.01% solution a green color with brown tint (caused by the presence of the methyl cyclopentane) appeared.

Cyclopentane ( $n_D^{20}=1.4064$ ) fractionated in the column of Koch-Hilberath yielded no reaction. On the other hand, cycloheptane ( $n_D^{20}=1.4450$ ) showed a deep green color which may perhaps be attributed to the instability of the seven ring system and the fact that cycloheptane is at equilibrium with methyl cyclohexane at constant temperature.

A bordeaux red color, however, was observed in the case of dimethyl cyclohexane and cyclooctane although these two preparations are not well defined compounds.

We have not yet determined the sensitivity limits of higher paraffins and cyclic hydrocarbons because the materials needed for their manufacture have not been available.

We have sought to purify higher commercial compounds by means of this reaction. Such experiments have, up to this time, given satisfactory results and a report on this subject will subsequently be made in collaboration with Dr. Schmeissermann.

This problem has also been worked on by Scharschmidt and co-worker who used the  $SbCl_5$  method to some extent for the approximate quantitative determination of hydrocarbons. We have established the fact, from our reaction, that the Scharschmidt reaction cannot be used for complete purification nor for quantitative determinations.

For this purpose, Scharschmidt had obtained a mixture of 50% n-heptane and 5% methyl cyclohexane and had treated the same with so much  $SbCl_5$  that the methyl cyclohexane became quantitatively converted. There resulted a mixture which, according to the aniline point method, showed a content of approximately two percent isocompounds, whereupon Scharschmidt concluded that since the aniline point method shows an accuracy of the same order, purification was possible.



We have, therefore, produced a mixture of n-heptane with two percent methyl cyclohexane and have treated it with two moles of  $SbCl_5$  according to the method of Schaarschmidt. Upon completion of reaction, the hydrocarbons obtained yielded a deep green color with violet sheen, which signified the fact that the methyl cyclohexane was not removed by this procedure and also that, just as Schaarschmidt had suspected, normal hydrocarbons also were attacked. This purification was repeated twice with the products obtained and yielded ever stronger reactions. It is therefore known that this reaction can be used as an aid to purification but not for complete purification.

Even n-heptane, after two hours of treatment with  $SbCl_5$  at  $0^\circ$  has not been attacked. The reaction occurs only in the presence of an isocompound, as Schaarschmidt had surmised.

Progress in the qualitative determination of isocompounds can now be reported. Our reaction can easily and rapidly be carried out, and it permits the detection of a quantity of about 0.01% of isocompounds and requires, in most cases, the use of only 1 ccm. of hydrocarbon mixture. Schaarschmidt ascribed to the reaction carried out by this method a sensitivity limit of not lower than 0.1% and based this opinion on the fact that a normal hydrocarbon evidenced the beginning of reaction after twenty-four hours and the same hydrocarbon with about 0.1% isocompound showed first signs of reaction after twenty hours.

It is now possible to prove the presence of tertiary hydrocarbons in mixtures, and this reaction can be used as well as the known physical methods.

It is planned to put this reaction on a still broader basis and to determine whether difference in place isomers (as for ex. dimethyl cyclohexanes) can be discovered, and to establish the limitations of the application of this reaction to the quantitative determination of hydrocarbons.

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8/20/48

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*G. S. O'Connell Wash. 7-26*

Refining of Light Oil Originating from  
Low Temperature Carbonization of Coal

US11

*G. S. O'Connell, W. H.*

Light oils originating from low temperature carbonization of coal or from cracking the low temperature coal tar differ from those which are obtained from the normal high temperature carbonization of coal insofar as they contain more aliphatic, phenolic and unsaturated compounds and less aromatic ones than a high temperature light oil. The refining of such light oils requires somewhat modified processes in order to cut down refining losses.

Refining such light oils requires numerous laboratory experiments which have to be carried out in order to determine the most suitable refining procedure. For these reasons no kitchen ready recipe can be given, but the following general ideas on the subject may be of interest. Without doubt, it is possible to obtain a water white finished product with a low gum content if the crude light oil is treated with large amounts of concentrated sulfuric acid. However, the losses are very high and besides the diolefines, monoolefines and aromatics are removed too. The content of saturated paraffinic hydrocarbons accumulates in the refined product, thus decreasing its antiknock value. Since the diolefines polymerize very readily, they are mostly responsible for the formation of "gums" during storing. A suitable refining method should remove as many diolefines as possible without attacking the monoolefines, which should be protected against the influence of oxygen by an admixture of suitable inhibitors. The best refining method - hydrogenation excepted - would be such a selective one which removes only the diolefines without affecting other hydrocarbons. Since during commercial operation it is almost impossible to obtain such a high selectivity, the refining process should be carried out in such a manner as to extract as few monoolefines as possible.

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The reaction of the di- and monoolefines with sulfuric acid is accompanied by an evolution of heat. The higher the olefine content the larger the heat evolution. The degree of heat evolution can be easily shown by a simple laboratory experiment. A small Dewar vacuum flask is equipped with a thermometer which is inserted into the flask by means of a tight fitting cork stopper. We fill now equal volumes of light oil and concentrated sulfuric acid into the flask, shake vigorously and read the highest temperature shown by the thermometer. Testing various light oils under equal conditions allows to set up an order of their reactivity. We observe easily that the temperatures rise higher, when we examine a light oil originating from low temperature carbonization of coal or lignite or from cracking the low temperature tar obtained by low temperature carbonization. It is evident that at higher temperatures the reaction of the sulfuric acid with the olefinic hydrocarbons will be stronger than at lower ones, which means that the temperature should be controlled in order to prevent excessive reaction with the acid. When, some 15 years ago, we tried to crack lignite tar to gasoline in cooperation with the Universal Oil Products Company of Chicago, we had to refine the cracked gasoline in order to obtain a suitable motor fuel. Applying the sulfuric acid treatment, we found out that the best results were obtained when we removed the heat of reaction as well as possible and kept the temperature of the content of the laboratory agitator between  $5$  and  $10^{\circ}\text{C}$  ( $41-50^{\circ}\text{F}$ ). Furthermore, it was better to apply small volumes of concentrated 66 Bé sulfuric acid than larger volumes of a diluted one. The acid treated product was neutralized with caustic soda solution and the redistillation had to be carried out as quickly as possible. Transferring the laboratory experiments to commercial operation requires the application of an agitator which is equipped with a jacket, through which cold water or an artificially refrigerated brine solution can be circulated. Such an agitator

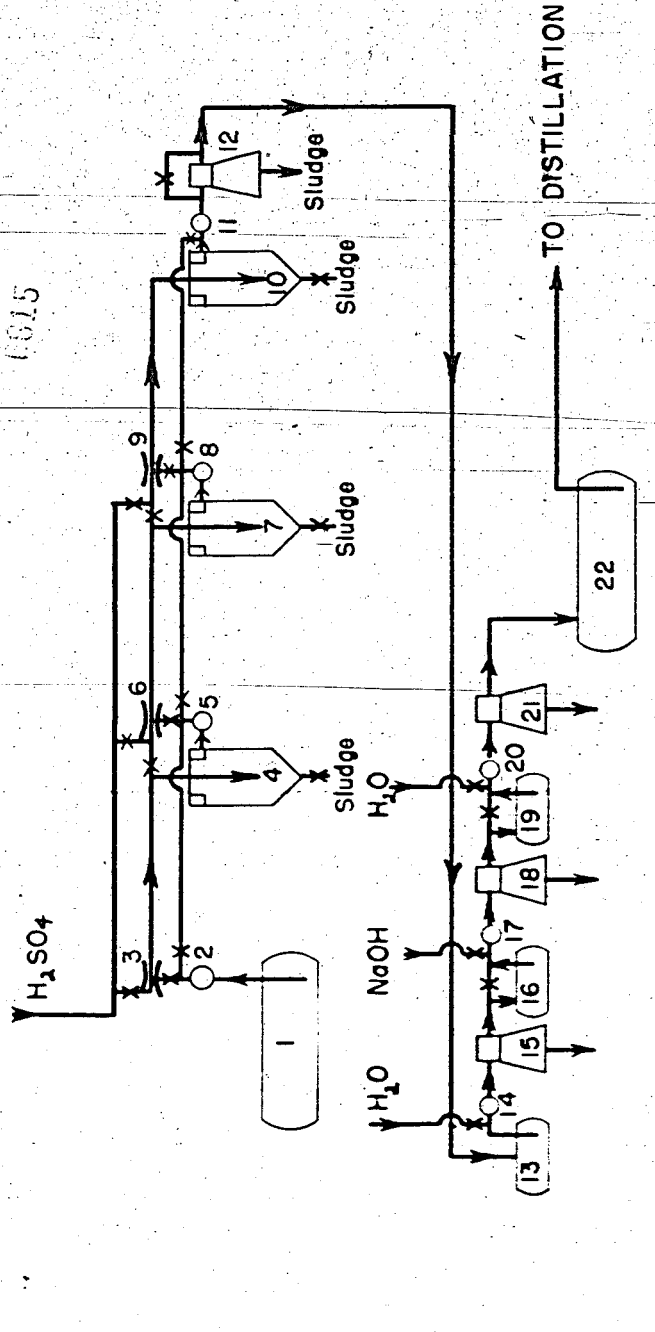
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cannot be lined with acid proof clay which would result in a decreased heat transmission. The most suitable temperature must be found out by trial. The acid may be added in several portions, whereby each addition is followed by agitation, settling and withdrawal of the sludge. In order to prevent polymerization of olefinic hydrocarbons, a continuous distillation of the refined product should be employed, in order to keep the hydrocarbons at elevated temperature for only a short period of time. Some kind of inhibitor should be admixed to the finished product.

Another process which may be used if light oils have to be refined which contain high amounts of unsaturated hydrocarbons may be considered more a modification of the distilling procedure. Normally, when the refined product is redistilled the final temperatures become so high that direct steam must be applied in order to vaporize the heavy fractions. It was found out that some of the polymerized products were slightly decomposed under the influence of the elevated temperature, thus increasing the gum content of the distillate, or in order to keep the gum content low the distillation must be interrupted at a somewhat lower temperature than would be necessary in order to obtain the highest yields of distillate. A decomposition of the distillate can be prevented if the distillation is carried out at a temperature of 80-90°C (176-194°F). This is possible by applying an increasing vacuum pressure during the course of the distillation, so that the distilling temperature is kept within the above mentioned limits. By doing this, the yields can be slightly raised, a little weaker acid than usual can be employed or the amount of acid can be lessened. Small scale experiments showed that the process is of value when light oils which contain high amounts of unsaturated hydrocarbons have to be refined. The combination of the vacuum pressure distillation with the temperature controlled acid treatment is a very mild kind of acid refining which should enable the diolefinic hydrocarbons to be almost selectively removed. The addition of small amounts of inhibitor to the finished product furnishes a stable gasoline.

The reaction of the sulfuric acid with the unsaturated hydrocarbons can be controlled by a limited contact time between the acid and the light oil. The acid refining equipment consists of a number of injector mixers each followed by a reaction tube and a separating vessel. The reaction tube should be designed in such a manner that no separation of the hydrocarbon-acid-mixture takes place. The time of reaction can be controlled by the length of the reaction tube and the number of the employed injector mixers. The separation of the acid from the light oil should be as complete as possible during the passage through the separating vessels. The final separation behind the last separating vessel can be carried out by means of a centrifuge. The neutralization with water and caustic soda solution should be carried out by means of mixing pumps and centrifuges. The refined product is distilled and blended with inhibitor in order to obtain a stable product. The following sketch represents a flow sheet of the proposed process.

CONTINUOUS LIGHT-OIL REFINING PLANT  
(Controlled Contact Time)



- |                                     |                                     |                 |
|-------------------------------------|-------------------------------------|-----------------|
| 1 STORAGE TANK                      | 8 FEED PUMP                         | 16 FEED TANK    |
| 2 FEED PUMP                         | 9 INJECTOR MIXER WITH REACTION TUBE | 17 MIXING PUMP  |
| 3 INJECTOR MIXER WITH REACTION TUBE | 10 SEPARATING VESSEL                | 18 CENTRIFUGE   |
| 4 SEPARATING VESSEL                 | 11 FEED PUMP                        | 19 FEED TANK    |
| 5 FEED PUMP                         | 12 CENTRIFUGE                       | 20 MIXING PUMP  |
| 6 INJECTOR MIXER WITH REACTION TUBE | 13 FEED TANK                        | 21 CENTRIFUGE   |
| 7 SEPARATING VESSEL                 | 14 MIXING PUMP                      | 22 STORAGE TANK |
|                                     | 15 CENTRIFUGE                       |                 |

0616

It should be investigated, too, whether it is advisable to treat the total recovered product or to separate the raw product in various fractions prior to the refining procedure. Since it is possible that the diolefines are not equally distributed over the entire boiling range, fractions may be recovered which do not need exhaustive refining. Such a procedure could preserve monoolefines, thus cutting down the losses.

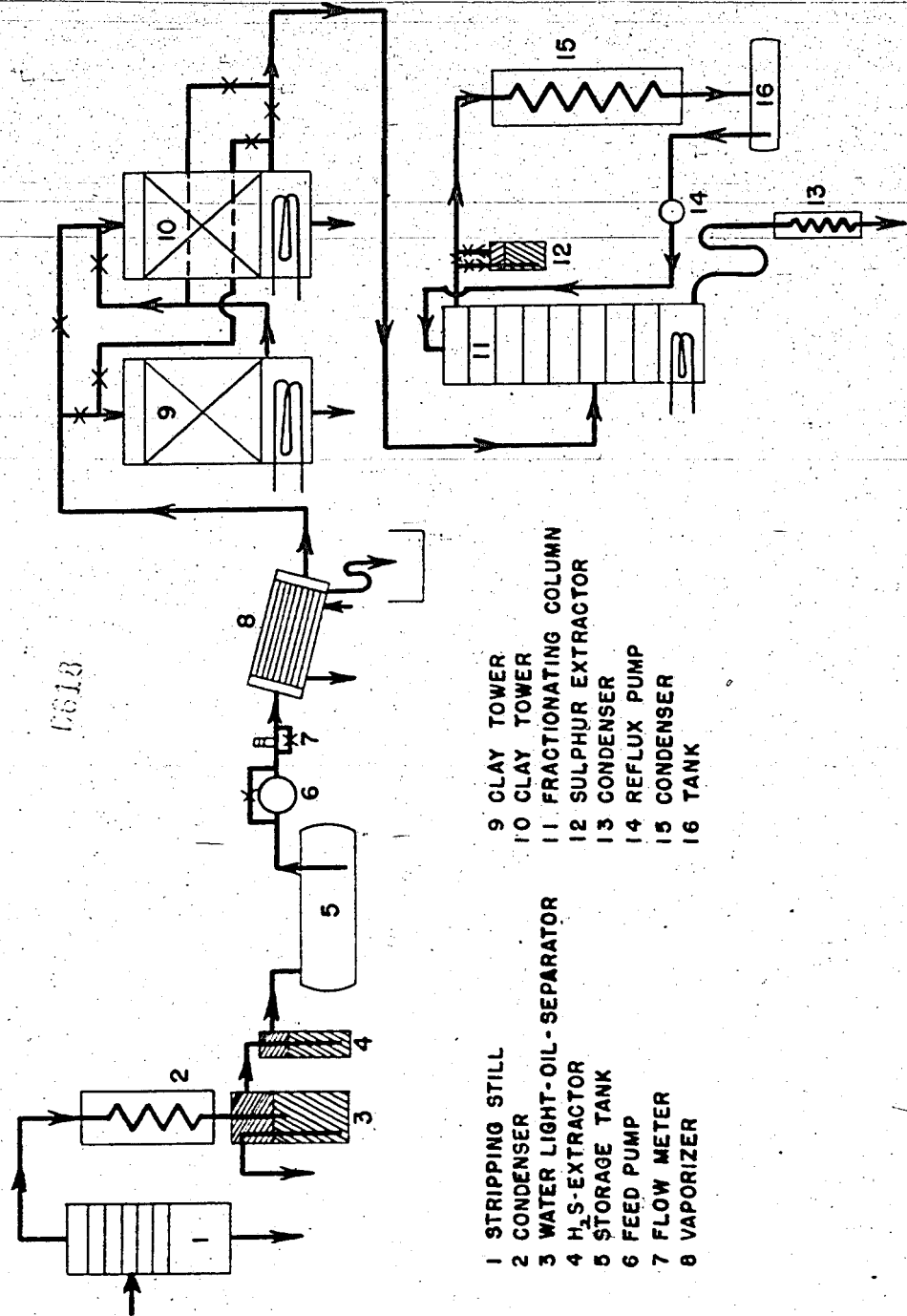
The vapor phase treatment with activated clays should be tried, too. The following sketch represents a flow sheet of such a vapor phase plant which we employed for refining light oil originating from high temperature carbonization of coal. With slight modifications the plant can be employed for the treatment of cracked gasoline, too. In case of light oil refining the wash-oil is stripped from the light oil by passing through the stripping still (1). The stripped oil is withdrawn from the bottom of the still, cooled and returned to the light oil scrubbers. The light oil vapors leaving at the top are condensed (2) and separated from water (3). The water free product is forced through a bath of caustic soda (4) in order to extract the hydrogen sulfide from the crude light oil which is stored in (5). From now on the flow sheet applies to both the treatment of light oil or cracked gasoline as well. A feed pump (6) sucks the product from tank (5) and pumps it over a flow meter (7) into the vaporizer (8) which is heated by steam. The heavy residue which has not been vaporized is withdrawn, cooled and disposed of. The superheated light oil vapors are admitted in the top of the clay tower (9) and are flowing downwards to the bottom. The gummy polymers, which due to the elevated temperature are not too viscous and which have not been adsorbed by the clay, are recovered in the lower part of the tower which is equipped with a steam coil in order to vaporize light products, should they have been condensed. The liquid polymers are continuously or periodically withdrawn. The light oil vapors are then

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admitted in the top of the clay tower (10) which takes care of the final refining. Gummy polymers are withdrawn and disposed of. The two clay towers in series are connected with a system of valve and gas piping connections to permit changing the order of the towers so that the vapors before leaving the plant come in contact with almost fresh clay. The refined vapors are admitted in the fractionating column (11) and separated into motor benzol and heavy residue. The fractionating is aided by a stream of reflux withdrawn from tank (16) by reflux pump (14). The heavy residue is discharged at the bottom of the still, cooled and can be used as heavy solvent. The gasoline vapors, leaving at the top of the column, before being condensed (15), are led through a bath of caustic soda solution (12) (spec. grav. 1.45) which removes sulfur and hydrogen sulfide. The refined product is stored in tank (16). Since some of the resins are adsorbed by the activated clay, a high boiling solvent should be percolated through the bed, which, by extracting the clay, opens its pores and extends its useful life. Provisions should be made in order to refresh the clay by burning out the carbon residue; however, the operation of such a furnace is economical only if a large plant is operated. The finished product should be blended with inhibitor.



VAPOR-PHASE REFINING OF LIGHT OIL WITH ACTIVATED CLAY



0619

Besides  $H_2S$  the light oil or cracked gasoline contains sometimes mercaptan, which is not entirely removed if the product is treated with sulfuric acid at low temperatures or with activated clay. In order to produce a gasoline which meets the specifications with respect to the sulfur content, a treatment with doctor solution is necessary.

In order to present a more complete picture concerning the methods which can be employed, hydrogenation processes must be mentioned. High pressure hydrogenation (vapor phase treatment with fixed bed catalysts) gives high yields and a finished product of good quality; however, it is complicated and expensive.

A somewhat modified hydrogenation process is the "Rostin process" which uses a finely divided iron ore (minette) over which the gasoline vapors are led together with hydrogen containing gases at  $350^{\circ}C$  ( $662^{\circ}F$ ).

Washington, D. C.  
August 13, 1947

Dr. Walter H. Oppelt

DR. WALTER H. OPPELT

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Auth. J.9.02.4/30/47

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TRANSLATION

made in office

AZIENDA CARBONI ITALIANI

194.412

Stabilimento  
Distillazione

DESCRIPTION OF THE PLANT

General description

1829

The plant carries out low temperature distillation of fines from the Sulcis coal mines nearby and it is proposed to produce industrial coke from the fines, at the same time recovering tar and light fuel oils.

The plant is located on an unfinished navigable canal, which connects the Gulf of Palmas and S. Antioco Bay. When the work is completed, ships transporting the solid and liquid fuels can tie up to the quay of the works which is also connected with the Railway line (F.M.S.) which passes nearby.

The fuel to be processed arrives in wagons by the above mentioned route and is unloaded either directly into feed hoppers or into nearby bins, which are divided into compartments for the various grades of coal which are used.

The plant is essentially comprised of the following units (See Fig. 1):

1. A boiler house to produce steam for the various services.
2. A battery of gas generators to supply gas, mixed with distillation gases, to the distillation ovens.
3. Two batteries of ovens and their equipment (only one battery is in operation at the present time);
4. A plant for separation of tar.
5. A plant for separation of light oils.

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6. A tar distillation plant.

7. "Benzine" and oil refinery.

Consumption of raw material and the recovery of the various products are shown in Appendix No. 2.

#### Boiler House.

The boiler plant, (Fig. 1) produces steam for the various services connected with the process.

Steam is used:

- For the production of crude gas
- For removal of Tar from crude gas
- For separation of light oils ("Benzine")
- For Tar distillation
- For rectification of the light fraction
- For liquifying "C" Tar before filling drums.

The plant consists of two mechanically fired Babcock and Wilcox boilers one of which is normally in reserve.

Feed water is treated with sodium phosphate.

No recuperators nor condensers are employed.

Tar burners with which the unit is supplied are used intermittently.

Certain data on the operation are to be found in Appendix No. 2.

#### Gas Plant.

This plant produces gas necessary for heating the distillation ovens. It consists of a battery of four Stein Gas Producers.

with revolving grate for automatic discharge of clinker and with a water jacket for the production <sup>of</sup> steam for blowing mixed with the air, under the grate.

One fan is sufficient to blow air for the gas producers, two of which suffice for the battery of ovens now in use.

A mechanical conveyor feeds coal to the producer feed hoppers which have a double seal.

The gas from each producer passes through a hydraulic lock into a sea water spray washer. It then passes to the Theisen Centrifugal washer where it undergoes final tar separation and its pressure is brought up to that point necessary for the operation of the oven burners.

The gas, after passing through the collector which retains the drops of water and tar in suspension, passes to a second washing tower with Easchig Rings and sea water sprays where it deposits the medium oils. From here, through meters for the measurement of flow and through automatic regulating valves, the gas is fed to the distillation ovens.

Residue gas from the distillation plant is mixed with the producers gas ahead of the Thiesen plant.

All apparatus <sup>for</sup> the measurement and control of gas conditions at various points in the circuit are centralised in one cabin together with apparatus for the automatic regulation of the pressure of the mixture of air and steam blown into the producers, which pressure is a function of the pressure in the main collector (Askania System).

In Appendix No. 3 will be found the more important data relating to this unit.

Distillation Ovens and Accessories

The coal used and its preparation.

A mixture of so-called Fat coking coal and so-called Lean coal is fed to the ovens. The latter (non-coking coal) is added because the coking coal when used alone produces an excessively porous and fragile coke. The proportions of the mixture are established daily on the result of laboratory analyses.

From the hoppers the two types of coal are fed by adjusted disc feeders and a belt conveyor to a hopper above two Cars disintegrators which have the double function of crushing to the correct size (Minimum dimension 1 mm) and of thoroughly mixing the coal.

From the disintegrators the coal passed on two belt conveyors to the hoppers of the distillation ovens.

Distillation ovens.

The unit consists of two batteries of two ovens each. Only the second battery (ovens 3 and 4) is in operation at present.

Each oven (Fig. No. 2) consists of four horizontal tunnels, one above the other, these are the distillation chambers. Their dimensions are: Length 50 meters, inside width 1,60 meters, and

height 0,25 meters. Inside these tunnels, on a refractory bed heated from below, metal pans containing the coal to be distilled run in opposite directions in each oven.

The fuel gas is piped to each section of the oven (27 sections) and then distributed to two burners (main and secondary). Air for the burners is supplied by fans.

The combustion chamber is of rectangular section built of firebrick and partitioned into five communicating compartments, the burners are placed in the first and the third compartments.

The burnt gas after passing the five compartments, passes to a vertical tower which is common to the flues of the four combustion chambers of each section of the oven.

From the towers, fumes pass through a collecting chamber to the chimney. The pans containing coal travel slowly along the tunnels and are heated by the combustion chambers underneath.

The gases produced in the distillation chambers are drawn out through nine rising columns and pass to a container for preliminary tar separation (locally known as a "Bariletto"), from there they pass to the section for the final extraction of tar and to that for the extraction of light oils.

The coke produced is conveyed to the Extinguishers in small buckets and then conveyed to the warehouse or shipped directly to consumers.

Material of which the ovens are constructed.

The body of the battery is built of silica-alumina bricks at 30% alumina; near the burners the alumina content is 40 to 42.

The bed of the distillation chamber is of interlocking slabs, these also are of silica-alumina refractory material at 30 to 32% alumina with a fusion point of 1500° C.

Material of which the pans are constructed.

The number of pans in the circuit is 1168. They circulate in pairs. The dimensions are 0,75 x 0,75 x 0,20 and they are divided into eight compartments by partitions parallel with the direction of travel.

At the present time most of the pans are of cast iron with 3% Chromium, other pans of ordinary and cr-Ti cast iron are also in use. The former have a greater resistance to corrosion and to distortion but are slightly more fragile than the latter. Experiments have been carried out and the best material so far found is a steel with special heat treatment. These will be adopted as soon as possible. With the new material there will be an improvement as regards fragility, distortion and resistance to the corrosive agents liberated in distillation, the pans will also be considerably lighter so that double sized pans, 0,75 x 1,50 x 0,20 m. can be adopted.

Automatic Control of the Oven.

At intervals of about eight minutes an electric clock operates a selector which automatically engages and disengages the motors of the various components and completes the cycle (lasting



—about three minutes) of operations from feeding coal to discharging coke. Movement of the pans is effected by two rams, normal to each other, one being the principal and the other secondary. Figure No. 3 illustrates their operations.

The pressures of the distillation gas, fuel gas and air are regulated by the "Askania" System, the essential feature of which is butterfly valves operated by "Servo" Oil Pump in turn operated by the pressures themselves.

The control of distillation, through the inspection of temperature and thence regulation of combustion, is by means of thermo couples placed in nine sections (eight couples to a section). Some of these are connected to a central recording station where a visual alarm signal shows the zones in which the temperature is too high, others are connected to direct reading pyrometers. (See figure No. 4).

Some data relating to the operation of the ovens is given in Appendix 4.

#### Tar separation

The first condensation of tar takes place in the water jacketed columns and in the preliminary tar separators (called "Barietti") and further tar is deposited in surface condenser.

The tar is collected in a small tank from which it is pumped to two steam jacketed tanks for decantation, overflow pipes take the tar to an lung tank and finally to a main tank of 1000 cubic meters.

From the batteries of surface condensers the gas passes two Beale Badoni hollow cylinder tar extractors (2 are also in reserve) and then a Rotary Pelouze "Labyrinth" Tar Separator to remove the last traces of tar. The tar after decantation is sent to the main tar tank.

Separation of light oils.

After leaving the tar Extraction Plant the gas passes a sea water spray washer and then two oil washing towers. After this treatment it is collected as already mentioned, in the producer plant tanks to be mixed with gas from the producer plant for heating the ovens.

The cold "Benzine" carrying oil is pumped to a heat exchanger and is heated to 40° C., it then goes to two steam preheaters, where the temperature reaches 120° C., and then to a plate distillation tower into which steam is injected in counter current.

While the oil freed from the "Benzine" passes to a cooler of sea water spray before returning to the circuit, the mixed vapours of water and "Gasolin" enter the aforementioned heat exchanger producing a light spirit vapour which passes directly to the rectifier and to heavy fractions which follow the same path after passing through an auxiliary heater.

From the rectifier through a condenser the water-gasoline mixture passes to a separator (locally known as a "Fiorentina") from which the gasoline, which is separated goes into an intermediate recovery tank. See Appendix No. 5 for information regarding this section.

Tar Distillation Plant

This consists of two "proab" continuous distillation retorts which until now have only been used very little and are rather of an experimental nature.

The tar from a decantation tank passes through measuring boxes and is pumped to a heat exchanger where it is preheated at the expense of "Benzine vapours" from the fractional distillation plant before being fed to retorts. These retorts are cylindrical and upright. They are heated by distillation gas the products of combustion of which circulate through external coils.

The tar, preheated as mentioned above, is brought up to 250° C. by superheated steam and distilled.

The pitch obtained is continuously syphoned from the bottom and taken through steam jacketed pipes first to a cooler and then to collecting tanks.

Vapour from the dome of the retort passes to the bottom of a plated fractional separating column.

"Benzine" vapour from the outlet at the top is cooled, and condense in containers where they are separated from the water by decantation. From the upper, middle and lower parts of the plated column, are derived paraffin, heavy and medium oils respectively. All products are sent to their appropriate tanks.

Appendix No. 6 gives the details of the oils.

Refinery for light oils.

The "Gasoline" and the Tar "Benzine", either separately or mixed in various proportions undergo a series of chemical washings to remove acid, before refining.

After washing, the "Benzine" is placed in a rectifier heated at first by internal steam coils and subsequently by direct injection of steam.

While "Benzine" vapours pass to condensers, in which decantation also takes place, and then to tanks the residues remain at the bottom of the vessel.

Appendix No. 7 gives some data on the system of washing plant and on the characteristics on the products.

The plant has its own laboratory for the control of operations and for research. It has also all necessary accessory services (Workshops, sea-water pumps, air compressors etc.).

APPENDIX No. 1

0630

RAW MATERIALS CONSUMED UNDER PRESENT PROGRAM

RAW MATERIALS	AVERAGE MONTHLY CONSUMPTION
Coking coal for ovens (0+10)	1680 tons
Non-coking coal for ovens (0+10)	720 tons
Lump-coal for gas producer plants (1 <sup>o</sup> +30)	680 tons
Coal for boilers (0+10)	120 tons
Electric power	35000 KWH.

PRODUCTION UNDER PRESENT PROGRAM

PRODUCTS	Quantity monthly (tons)	Percentage distilled coal
Coke	1900	78
Tar D.	226	9
Light Fuel	16	0,65
Oil G.	5	0,39
Tar G.	48	2

APPENDIX No. 2

COKE

0631

Lump 0 + 10 m/m

Immediate analysis:	Moisture	7.50 %
	Ash	22.12 %
	Volatile Substances	34.08 %
	Fixed Carbon	36.30 %
Elementary analysis:	Carbon	54.35 %
(dry sample)	Hydrogen	4.38 %
	Total Sulphur	7.58 %

Calorific value 5310 Cal/Kg.

Hourly consumption 165 Kg.

Hourly steam production 990 Kg.

Characteristic of steam produced: p = 7.5 Kg/cm<sup>2</sup>  
t = 200° C.

APPENDIX No. 3

a) - RAW MATERIALS

1°) - Quality: Coal =

Lump 10 + 30 m/m

Immediate analysis:	Moisture	5.52 %
	Ash	18.46 %
	Volatile Substances	34.52 %
	Fixed Carbon	41.50 %

0632

Elementary analysis:	Carbon	56.16 %
(dry sample)	Hydrogen	4.79 %
	Total Sulphur	8.79 %

Calorific value	5550 Cal/Kg.
Hourly consumption	950 Kg.

b) - PRODUCTS

1°) - Quality + Gas misto (Gas G) =

Density at 0° 760 m/m Hg:  $d = 1.096 \text{ Kg/Mc N}$

Chemical analysis/	CO <sub>2</sub> .... =	6.9 %
	H <sub>2</sub> S .... =	2.2 %
	O <sub>2</sub> .... =	0.3 %
	C <sub>n</sub> H <sub>m</sub> .... =	0.2 %
	CO .... =	23.9 %
	H <sub>2</sub> .... =	16.9 %
	C <sub>2</sub> H <sub>6</sub> .... =	0.2 %
	C H <sub>4</sub> .... =	2.6 %
	N <sub>2</sub> .... =	46.8 %

Calorific value 1668 Cal/Nm<sup>3</sup>

Hourly production 2886 mc

Specific production 3.00 mc/Kg carb. gassified

Percentage gassified coal 33.3

Conditions of gas upon leaving reucer plant:

Temperature 227° C.

Pressure 10 m/m H<sub>2</sub>O

0633

200 - Quality - Tar =

Density 15° C / d = 1.058 Kg/dmc  
Moisture 0.58 %

Elementary analysis:	Hydrogen	9.37 %
(on anhydride)	Carbon	79.33 %
	Sulphur	4.52 %
	Ash	0.08 %
Calorific value		9136 Cal/Kg.

Distillation Cycle =

Starts at 104.5° C

"	130° C	distills	0.1 %
"	150° C	"	0.3 %
"	200° C	"	0.8 %
"	250° C	"	1.2 %
"	300° C	"	11.8 %
"	350° C	"	34.5 %
"	385° C	"	66.0 %

Hourly production	75 Kg.
Percentage of coal gassified	7.9 %



0634

3°) - Quality - medium oil =

Density at 15°C : d = 0.9077 Kg/dmc  
Moisture traces

DISTILLATION CYCLE =

Starts at 138°C

" 170°C	distills	1.5 %
" 200°C	"	6.5 %
" 250°C	"	55 %
" 298°C	"	94 %

Hourly production 7 Kg.

Percentage of coal gassified 0.75 %

RESIDUE EXTRACTED

Moisture 21.79 %  
Elementary analysis / Incombustible carbon 9.92 %  
(dry sample) Total sulphur 2.37 %

Hourly production 186 Kg.

Percentage of coal gassified 19.6 %

0835

a) - RAW MATERIALS -1°) - Quality/Coking Coal - greasy agglomerating =

Lump : 0 + 10 m/m

Immediate analysis : Moisture	8.1 %
Ash	14.1 %
Volatile Substances	37.2 %
Fixed Carbon	40.6 %
Elementary Analysis: Total Sulphur (on dry sample)	9.1 %
Calorific value	6190 Cal/Kg

2°) - Quality : Non-coking coal - non-agglomerating

Lump : 0 + 10 m/m

Immediate analysis : Moisture	7.0 %
Ash	12.9 %
Volatile Substances	37.2 %
Fixed Carbon	38.0 %
Elementary analysis: Total Sulphur (on dry sample)	7.9 %
Calorific value	5365 Cal/Kg

3°) - Quality - Mixed coal - agglomerated

Lump : 0 + 8 m/m

Percentage used	coking	79.9 %
	non-coking	20.1 %
Immediate analysis: Moisture		7.30 %
Ash		15.72 %

0633

	Volatile Substances	36.28 %
	Fixed Carbon	40.70 %
Elementary analysis:	Carbon	63.97 %
(dry sample)	Hydrogen	4.89 %
	Total sulphur	8.39 %
Calorific value		6075 Cal/Kg.
Coal distilled per hour		3500 Kg.
Loadings per hour		7
Amount of coal per load		500 Kg.

4°) - Quality - Gas for heating the ovens

Mixture of gas from gas producer plants  
and distillation gases (Gas N)

Density at 0° a 760 mm Hg :  $d = 1.104 \text{ Kg/Nmc.}$

Chemical analysis :	CO <sub>2</sub> .....	= .....	7.9 %
	H <sub>2</sub> S .....	= .....	3.84 %
	O <sub>2</sub> .....	= .....	0.7 %
	CnHm .....	= .....	0.7% $\left\{ \begin{array}{l} C_2H_4 = 0.25 \\ C_4H_8 = 0.5 \end{array} \right.$
	CO .....	= .....	19.9 %
	H <sub>2</sub> .....	= .....	15.7 %
	C <sub>2</sub> H <sub>6</sub> .....	= .....	0.9 %
	CH <sub>4</sub> .....	= .....	6.4 %
	N <sub>2</sub> .....	= .....	44.4 %
(calculated) calorific value			2360 Cal/ Nmc.
Quantity of gas burned per hour			3501.5 mc.N

PRODUCTS -

1°) - Quality - semi-coke =

0837

1) Lump : > 30 m/m

Immediate analysis :	Moisture	7.40 %
	Ash	20.31 %
	Volatile substances	10.19 %
	Fixed carbon	62.10 %
Elementary analysis:	Carbon	63.76 %
	Hydrogen	3.09 %
	Total sulphur	6.83 %
Calorific value		5365 Cal/Kg.

Hourly production 2240 Kg.  
Percentage of coal loaded 64 %

2) Lump : > 30 m/m

Immediate analysis :	Moisture	14.10 %
	Ash	20.23 %
	Volatile substances	8.31 %
	Fixed carbon	57.36 %
Elementary analysis :	Carbon	59.74 %
(on dry sample)	Hydrogen	2.74 %
	Total Sulphur	6.97 %
Calorific value		5007 CAL/KG.

Hourly production 490 Kg.  
Percentage of coal loaded 14 %  
Total hourly production 2730  
Percentage of coal loaded 78 %

0638

2°) - Quality - Distillation gas (Gas D) =

Density at 0°C. at 760° Hg. : d = 1.172 Kg/Nmc.

Chemical analysis :	CO <sub>2</sub> .....	=	.....	11.3 %
	H <sub>2</sub> S .....	=	.....	11.3 %
	O <sub>2</sub> .....	=	.....	2.7 %
	C <sub>4</sub> H <sub>8</sub> .....	=	.....	2.9 %
	CO .....	=	.....	3.1 %
	C <sub>2</sub> H <sub>6</sub> .....	=	.....	4.5 %
	H <sub>2</sub> .....	=	.....	9.2 %
	CH <sub>4</sub> .....	=	.....	23.8 %
	N <sub>2</sub> .....	=	.....	31.2 %

Calorific value 4942 Cal/Nmc

Supposing that all the nitrogen is derived from infiltration air (that of the coal passes through ammonia water) the following data is held to be true:

Density at 0°C. and 760 /m Hg. : d = 1.06

Chemical analysis :	CO <sub>2</sub> .....	=	.....	9.17 %
	H <sub>2</sub> S .....	=	.....	17.84 %
	O <sub>2</sub> .....	=	.....	4.26 %
	C <sub>4</sub> H <sub>8</sub> .....	=	.....	4.56 %
	CO .....	=	.....	4.90 %
	H <sub>2</sub> .....	=	.....	14.54 %
	C <sub>2</sub> H <sub>6</sub> .....	=	.....	7.12 %
	CH <sub>4</sub> .....	=	.....	34.60 %

Calorific value (calculated) 7795 Cal/Nmc.

Hourly production 615.5 Nmc

Hourly production of gas D purified by infiltration air 250 Kg.

Percentage of coal distilled 7.2 %

3°9 - Quality: Distillation Tar (Tar D) = 0839

Density at 15°C. : d = 1.002

Moisture 3.35 %

Elementary analysis	:	Hydrogen	9.35 %
(on anidride)		Carbon	80.87 %
		Sulphur	5.20 %
		Ash	0.04 %

Calorific value 9359 Cal/Kg.

Distillation Cycle =

starts at 96.5° C.

" 110° C. . . . .	distills . . . . .	4.8 %
" 150° C. . . . .	" . . . . .	6.1 %
" 200° C. . . . .	" . . . . .	15.3 %
" 250° C. . . . .	" . . . . .	33.5 %
" 300° C. . . . .	" . . . . .	44.2 %
" 350° C. . . . .	" . . . . .	64.5 %
" 375° C. . . . .	" . . . . .	82.0 %

Hourly production (anidride) 302 Kg.

Percentage of distilled coal 9.28 %

4°) - Quality - Gasoline =

Color . . . . . Citrus yellow

Aspect . . . . . Clear

Deposit . . . . . None

Density at 15° C. 0.7664 Kg/cm<sup>3</sup>.

Reaction to metilarancio - Alkaline

Phenol content. . 3.3%

0840

Elementary analysis :	Hydrogen	13.15 %
(on anidride)	Carbon	83.28 %
	Suplhur	3.57 %

Distillation Cycle (Kraemer & Spilker)

Starts at 40°C.

10 %	"	66°C. . . . .	60%	at	107.5°C
20 %	"	74.5°C. . . . .	70%	at	120
30 %	"	82°C. . . . .	80%	at	140.5°C
40 %	"	89.3°C. . . . .	90%	at	174
50 %	"	97.5°C. . . . .	93%	at	180

Hourly production	29 Kg.
Percentage of coal loaded	0.84 %

DATA RELATIVE TO THE OPERATION OF THE OVENS

Average temperature in the ovens:

- In combustion chamber : OVEN No. 3:T = 537.7°C  
" " 4/T = 554.9°C
- In distillation chamber: OVEN No. 3:T = 450.4°C  
" " 4:T = 442.4°C

Average depression of distillation gas in the "Bariletto"

OVEN No. 3	:	1.5	m/m	H <sub>2</sub> O
" "	:	2.3	m/m	H <sub>2</sub> O

0641

CALCULATED BALANCE OF PRODUCTS OBTAINED IN REFERENCE  
TO CIST CO. I  
Loaded during one hour of operation

DIVISIONS	COAL		PRODUCTS	
	Kg/hr	%	Kg/hr	%
Moist coal loaded	3500	100.0		
Coke: Lump			2240.=	64.=
"    Fines			490.=	14.=
Anidride Tar			320.=	9.28
Water in the Tar			10.=	0.28
Ammonia water			8.=	0.22
Gasoline			29.=	0.84
Gasoline remaining in the gas			3.48	0.01
Gas (purified by infiltration air)			252.=	7.20
Loss thru gas burned by infiltration air, escaping gas, combustion of coal fines, dispersal of fines and of gas.			147.52	4.17
			3500.=	100.=



0642

THERMO-CHEMICAL BALANCE OF PRODUCTS DERIVED  
FROM THE DISTILLATION OF ONE KG OF MOIST MIXTURE  
 (Refer to calorific value)

DIVISIONS	Quantity of coal	
	calorific/value	%
Calorific value of 1 Kg. of coal	6075	100
Latent heat of the coke		
- Lump 0.64 x 5365	3433.60	56.5
- Fines 0.14 x 5007	700.98	11.53
Latent heat in Gas D.		
0.072 x $\frac{7795}{1.06}$	526.03	8.65
Latent heat in Tar D.		
0.0928 x 9359	868.51	14.29
Latent heat in the Gasoline		
0.0084 x 11800	99.12	1.63
Latent heat in the Gasoline left in the gas		
0.0001 x 11.800	1.18	0.01
Evaporation heat of the water		
(0.0028 + 0.0022) x 100	0.50	0.005
Loss	445.08	7.375
Thermo-chemical rendition : 92.625 %		

0643

PUREWASHING OIL

Density at 15°C : d = 0.979

Viscosity Engler at 20°C:E = 3°

Distillation Cycle -

Starts at 157°C

" 200°C . . . . . distills . . . . . 5.5%  
 " 250°C . . . . . " . . . . . 36.5%  
 " 300°C . . . . . " . . . . . 77.0%

Up to " 336°C . . . . . " . . . . .

Quantity in-circulation . . . . . 8 mc.

Capacity of oil . . . . . 1763 Kg/hr

WASHING OIL SEPARATED FROM BENZINE (after one months use)

Density at 15°C : d = 0.980

Viscosity Engler at 20°C:E = 2.49°

Distillation Cycle

Starts at 156°C

" 200°C . . . . . distills . . . . . 4.6%  
 " 250°C . . . . . " . . . . . 30.0%  
 " 300°C . . . . . " . . . . . 76.0%

Up to at 326°C.

Gasoline content of gas before removal of benzine : 128.86 g/cN

" " " after the " " " : 13.86 g/cN

" extracted : 115.00 g/cN

Percentage of the quantity contained in the gas 88%

CHARACTERISTICS OF CLAUDE TAR OIL

1) - Quality: Light oil =

Density at + 15°C : d = 759 Kg/m<sup>3</sup>  
 Sulphur = 3.261 %

Distillation Cycle -

Starts at 55°C

"	98°C . . . . .	distills . . . . .	5%
"	105°C . . . . .	" . . . . .	10%
"	152°C . . . . .	" . . . . .	50%
"	205°C . . . . .	" . . . . .	95%

2) - Quality: medium oil

Density 15°C : d = 0.9483  
 Sulphur = 3.595 %  
 Phenol soluble  
 in soda = 32%  
 Base = 2%  
 Point of inflammability P.M. = 95°C.  
 Point of combustion P.M. = 97°C

Distillation Cycle

starts at 180°C.

"	196°C . . . . .	distills . . . . .	5%
"	200°C . . . . .	" . . . . .	10%
"	216°C . . . . .	" . . . . .	50%
"	289°C . . . . .	" . . . . .	95%

PITCHY RESIDUE

3) - Quality: Heavy oil =

Density at 15°C = 0.9674  
 Sulphur = 4.165 %

Point of inflammability P.M. = 103°C  
Point of combustion P.M. = 118°C

0540

Distillation Cycle

starts at 205°C

" 224°C . . . . .	distills . . . . .	5%
" 229°C . . . . .	" . . . . .	10%
" 262°C . . . . .	" . . . . .	50%
" 339°C . . . . .	" . . . . .	95%

4) - Quality - Paraffin oil

Density at 15°C : d = 1.0171  
Sulphur = 4.952 %  
Point of inflammability P.M. = 134°C  
Point of combustion P.M. = 169°C

Distillation cycle

starts at 222°C

" 266°C = . . . . .	distills . . . . .	5%
" 260°C . . . . .	" . . . . .	10%
" 338°C . . . . .	" . . . . .	50%
" 394°C . . . . .	" . . . . .	95%

5) - Quality - Pitch =

Softening point: 70°C.

0646

PRODUCTION OF CRUDE OIL IN REFERENCE TO DISTILLED TAR

DIVISIONS	Percentages
Tar	100%
Light Oil	20
Medium Oil	15.5
Heavy Oil	14.5
Paraffin Oil	36.4
Pitch	13.6 100.0

APPENDIX No. 7

TYPE OF WASHING FOR GASOLINE -

1°) - Washing 8% Na <sub>2</sub> CO <sub>3</sub> at 20%	agitation	20'
	decantation	30'
2°) - Water washing	shower	10'
	decantation	10'
3°) - washing with 0.6% H <sub>2</sub> SO <sub>4</sub> 61 Bé	agitation	20'
	decantation	20'
4°) - Washing with 2% H <sub>2</sub> SO <sub>4</sub> 61 Bé	agitation	20'
	decantation	20'
5°) - Water washing	shower	10'
	decantation	10'
6°) - washing with 2% NaOHa 14 Bé	agitation	20'
	decantation	20'
7°) - water washing	shower	20'
	decantation	10'

0647

8°) - Washing with 2% Piombito sodico at 18 Bé	agitation 20' decantation 20'
9°) - Water washing	shower 10' decantation 10'
Loss thru washing	13.7 %
Loss thru refining	14.1 %
Total loss	27.8 %

Refined gasoline - average sample

Color Clear opalescence yellow  
Aspect clear  
Deposit None  
Density at 15°C: 0.746  
Corrosion teste on copper plate : negative  
Sulphur tenor : 1.04%

Distillation Cycle

Starts at 66°C	
" 88°C . . . . . distills . . . . .	5%
" 92°C . . . . . "	10%
" 96°C . . . . . "	20%
" 100°C . . . . . "	30%
" 105°C . . . . . "	40%
" 111°C . . . . . "	50%
" 117°C . . . . . "	60%
" 123°C . . . . . "	70%
" 130°C . . . . . "	80%
" 140°C . . . . . "	90%
" 158°C . . . . . "	93%

Dry point at 160°C.

CC48

TYPE OF WASHING FOR 50% TAR BENZINE AND 50% GASOLINE

1°) - Washing 10% NaOH at 15 Bé	agitation	30'
	decantation	30'
2°) - washing 5% NaOH at 15 Bé	agitation	30'
	decantation	30'
3°) - washing 5% water	agitation	20'
	decantation	20'
4°) - Washing 5% H <sub>2</sub> SO <sub>4</sub> a 61 Bé	agitation	60'
	decantation	60'
5°) - Washing 5% water	agitation	20'
	decantation	20'
6°) - Washing 2% Na <sub>2</sub> CO <sub>3</sub> 15 Bé	agitation	30'
	decantation	30'
7°) - water washing	agitation	20'
	decantation	20'
8°) - Washing with Piombito sodico	agitation	20'
	decantation	30'
9°) - water washing	agitation	10'
	decantation	20'

Refined benzine Mixture - average sample

Color clear opalescence orange  
Aspect clear  
Deposit None

Density at 15°C : 0.778

Sulphur : 1.29 %

Corrosion tests made on copper plate:

after 18 hours slight tarnishing

Distillation Cycle

Starts at 68°C

" 105°C . . . . . distills . . . . . 5%

" 112°C . . . . . " . . . . . 10%

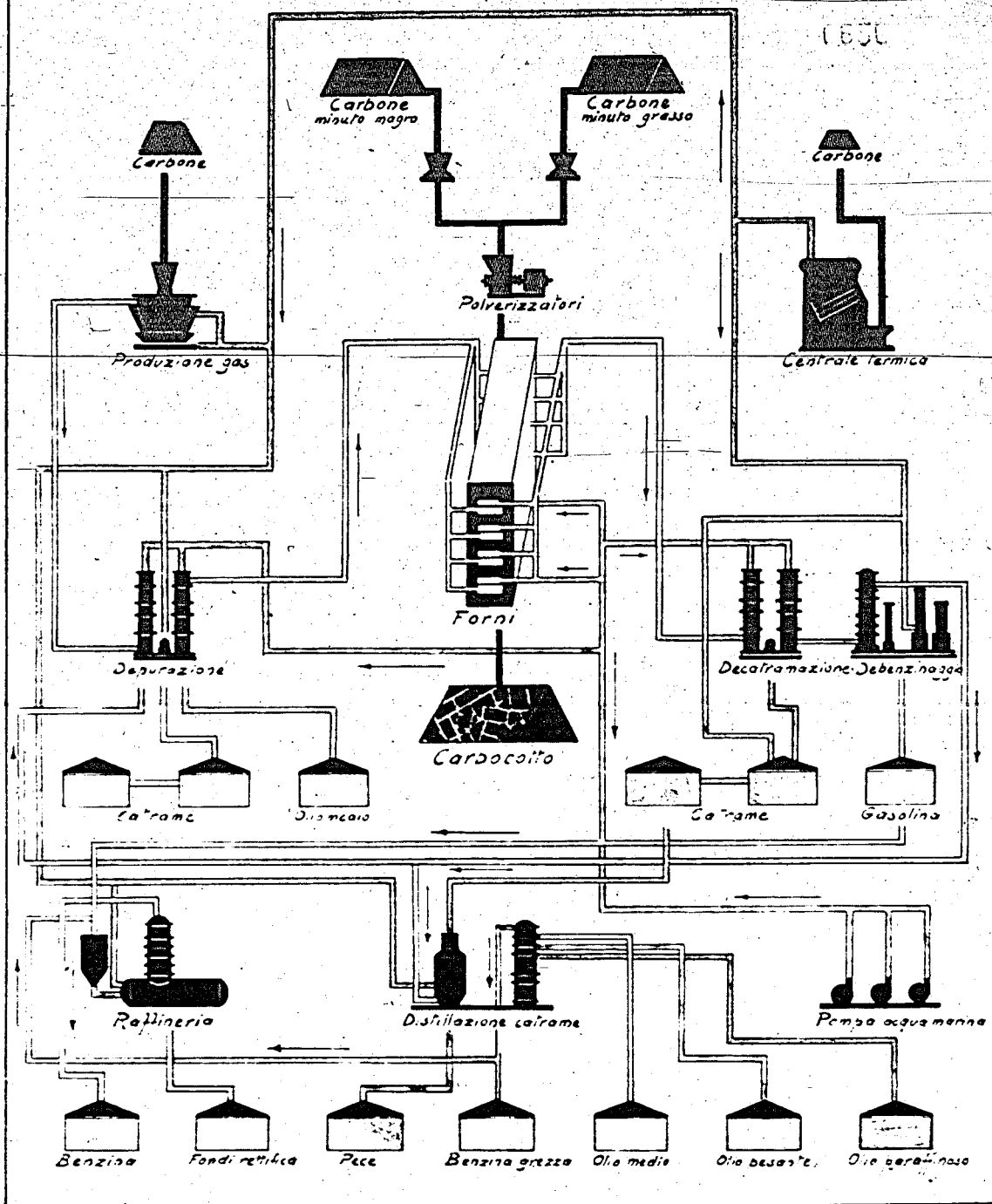
0643

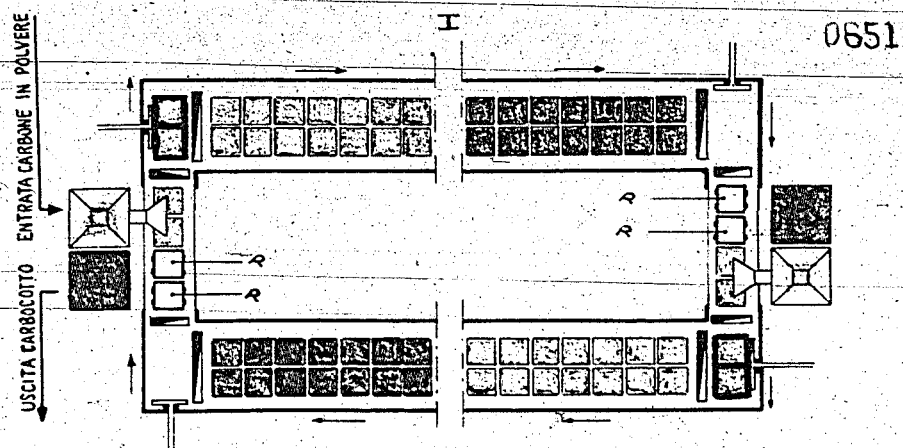
AT 126° C . . . . .	distills . . . . .	20%
" 134° C . . . . .	" . . . . .	30%
" 136° C . . . . .	" . . . . .	40%
" 139° C . . . . .	" . . . . .	50%
" 148° C . . . . .	" . . . . .	60%
" 156° C . . . . .	" . . . . .	70%
" 163° C . . . . .	" . . . . .	80%
" 170° C . . . . .	" . . . . .	90%
" 177° C . . . . .	" . . . . .	98%

Dry point at 181°C.



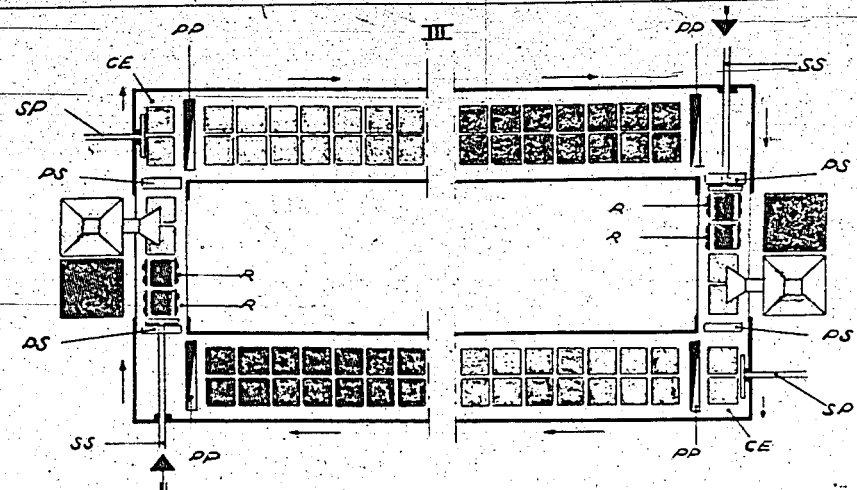
# SCHEMA DI LAVORAZIONE



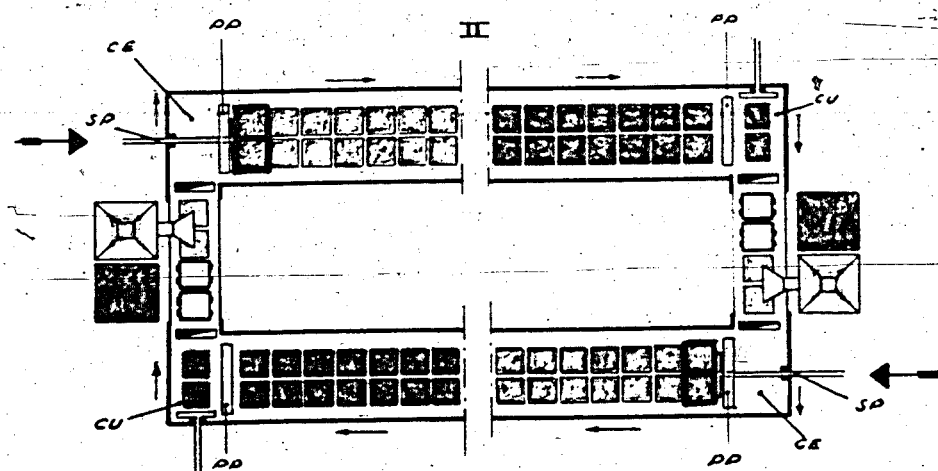


I)<sup>o</sup> =  
 I ROVESCIATORI (R) CHE ALLA FINE DEL CICLO PRECEDENTE AVEVANO COMPIUTO UNA ROTAZIONE DI 180° VUOTANDO LE CASSETTE DEL LORO CONTENUTO (CARBOCOTTO) SI RIALZANO RIPRENENDO LA LORO POSIZIONE NORMALE

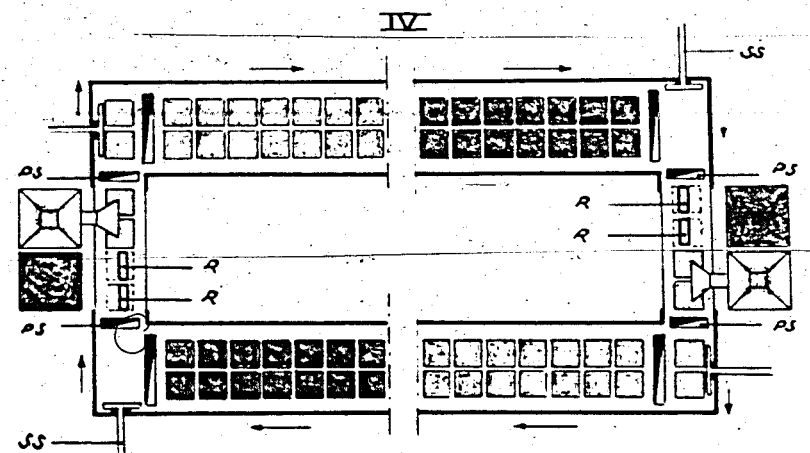
- CASSETTE VUOTE
- ▒ CASSETTE PIENE DI CARBONE IN POLVERE
- CASSETTE PIENE DI CARBOCOTTO



III)<sup>o</sup>  
 LE MACCHINE DI SPINTA PRINCIPALI (SP) RIPRENDO LA POSIZIONE PRIMITIVA ;  
 SI CHIUDONO LE PORTE PRINCIPALI (PP) INTERCETTANDO LA COMUNICAZIONE FRA FORNI E AVANCAMERE ;  
 SI APRONO LE PORTE SECONDARIE (PS) STABILENDO UNA COMUNICAZIONE FRA LE AVANCAMERE E L'ESTERNO ;  
 LE MACCHINE DI SPINTA SECONDARIE (SS) AGENDO NEL SENSO DELLE FRECCE PORTANO LE CASSETTE CARICHE DI CARBOCOTTO NEI ROVESCIATORI ;  
 CONTEMPORANEAMENTE LE CASSETTE VUOTE CHE SI TROVANO SUI ROVESCIATORI VANNO A RIEMPIRSI DI POLVERE DI CARBONE SOTTO LE TRAMOGGE DI CARICO SPINGENDO NELLE AVANCAMERE (CE) QUELLE GIÀ PRECEDENTEMENTE RIEMPIE DI CARBONE .

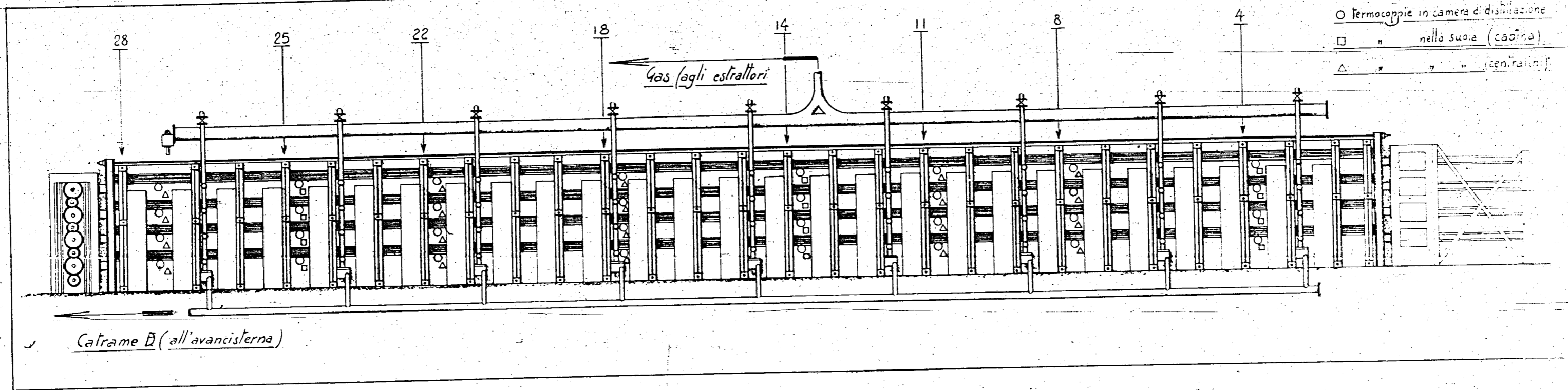


II)<sup>o</sup> =  
 SI APRONO LE PORTE PRINCIPALI (PP) STABILENDO UNA COMUNICAZIONE DIRETTA FRA L'AMBIENTE DEI FORNI E LE AVANCAMERE ;  
 LE MACCHINE DI SPINTA PRINCIPALI (SP) AGENDO NEL SENSO DELLE FRECCE INTRODUCONO NEI FORNI LE CASSETTE CARICHE DI COMBUSTIBILE DA TRATTARE CHE SI TROVANO NELLE AVANCAMERE DI ENTRATA (CE) ;  
 CONTEMPORANEAMENTE LE CASSETTE CARICHE DI CARBOCOTTO ENTRANO NELLE AVANCAMERE DI USCITA (CU).



IV)<sup>o</sup> =  
 LE MACCHINE DI SPINTA SECONDARIE (SS) RIPRENDO LA POSIZIONE NORMALE ;  
 SI CHIUDONO LE PORTE SECONDARIE (PS) INTERCETTANDO LA COMUNICAZIONE FRA AVANCAMERE E AMBIENTE ESTERNO ;  
 I ROVESCIATORI SI CAPOVOLGONO SCARICANDO IL CARBOCOTTO E RESTANO IN TALE POSIZIONE FINO ALL'INIZIO DEL CICLO

1852



1853

A. Ca. I.  
DISTILLAZIONE  
STABILIMENTO DI S. ANTONIO  
PLANIMETRIA  
GENERALE  
A 1:1000

PER SANTIAGO

PER SANTIAGO

N

CAYALE

