CATALYTIC CRACKING WITH A FIXED-BED CATALYST

DESCRIPTION OF THE PILOT PLANT

The experiments were carried out in a shell-type reactor whose interior was brick-lined and which was insulated from the outside. The heat required for the reaction (360 - 540 B. t. u. per pound of oil) and the heat lost by radiation was obtained by superheating the charge stock above the required cracking temperature.

The catalyst was arranged in 2 beds, each of which had a capacity of 17.5 cu. ft. The height of each bed was 13.7 inches and the diameter 4.5 ft. The oil vapors passed from the top to the bottom of the reactor. The regenerating gas was introduced half-way between the 2 catalyst beds and distributed itself uniformly through both beds. During prolonged periods of cracking (exceeding 60 minutes) the temperature of the lower bed was, on the average, 18 - 36° lower than that of the upper bed. (See Table 1)

Table 1

Cracking Time min.	Space Velocity 1 vol/vol/hour		Space Velocity 0.6 vol/vol./hour		Space Velocity 0.5 vol/vol/hour		Space Velocity 0.4 vol/vol/hour		Av. Reactor Temperature
	Upper Bed	Lower Bed	Upper Bed	Lower Bed	Upper Bed	Lower Bed	Upper Bed	Lower Bed	
0	788°F	788°F	788°F	788°F	788°F	788°F	788°F	788°F	
20	792°F	784°F	792°F	784°F	792°F	786°F	797°F	786°F	
60			802°F	772°F	799°F	733°F	802°F	784°F	788°F
120					806°F	777°F	804°F	779°F	
180							806°F	777°F	

The distribution of carbon was not uniform for both catalyst beds. The amount of carbon deposit decreased uniformly from the top to the bottom in the direction of the flow of the oil vapors. 60% of the total carbon deposited on the catalyst was present in the upper bed, whereas 40% was deposited on the catalyst in the lower bed. Since both beds were regenerated by the same quantity of regenerating gas, uniform burning of the carbon deposits on each bed could not be obtained and this resulted in a certain difficulty when air alone was used as regenerating gas.

The oil preheater heated the gas oil charge (API Gravity about 34.2) from 68 - 878°F. The maximum throughput was 527 gallons per hour and the minimum 106 gallons per hour. The residence time in the tubes was at capacity throughput about 3 seconds and at a throughput of 155 gallons per hour about 5 seconds. The heat requirements were about 285,000 B. t. u. per barrel of gas oil.

The oil preheater was a tubular preheater with convection and radiation zones.

The cracking gas preheater which was originally designed as a steam heater raised the temperature of the cracking gas (density 1.0) from 68 to about 878°F. The heat requirements were about 281,000 B. t. u. per thousand cu. ft. of gas. The maximum throughput was 3,531 cu. ft. per hour.

The preheater for the regeneration gas was a tubular heater with convection and radiation zones. The regenerating gas, which is a mixture of air and nitrogen or air alone, was heated from 68 - 932°F. The maximum throughput was about 247,000 cu. ft. per hour and the minimum throughput was 35,314 cu. ft. per hour. The maximum pressure of the heated gases was 103 psig. and the minimum pressure 14.7 psig. The heat consumption at full throughput was about 4,760,000 B. t. u. per hour.

The blower for the regenerating gas had a capacity of about 254,000 cu. ft. at a pressure of 14.7 psig. and 68°F. It operated at 950 r. p. m. with a motor of 140 HP. The maximum pressure difference obtained was 250 mm. Hg. The turbo-compressor operated at 103 psig. and 68°F. at a capacity of about 240,000 cu. ft. per hour. It was operated by a motor of 14 HP at 675 r. p. m. The maximum pressure difference obtained was 125 mm. Hg.

The capacity of the tankage used in connection with the operations was 650 barrels. 5 measuring vessels of a capacity of 528 gallons each were used, together with a gas holder of a capacity of 885 cu. ft.

Mechanical Control of Valves.

The various operating periods were controlled by means of a contact drum which, on rotation, opens and closes the corresponding valves by electric operation of a hydraulic system. The control operation was arranged in such a way that after each operating period the drum had been turned once by 360° covering 20 contact positions. The mechanism operated as follows:

The oil level in the surge tank of the hydraulic system is kept at such a level during operations that the pressure is kept below 235 psig. A pump continuously circulates oil from the suction tank into the surge tank. The oil level is regulated by an overflow valve and the regulator "d" (see the attached diagram) maintains the pressure constant at 235 psig. When the contact drum is turned electrically or mechanically, a new position of the contact drum is reached and electric impulses are given to the corresponding electrically-operated valves which permit the oil to pass from the surge tank to the Sera-K oil pressure operated gate valves and allow the corresponding amount of oil to flow from the gate valve to the suction tank. In order to show the correct operation of the switching mechanism, the position of each oil pressure-operated gate valve is indicated on a lighted diagram on the panel board and irregularities in the operation are indicated by suitable signals. An arresting mechanism on the contact drum prevents further motion of the drum before the operating time of a valve (about 20 seconds) has elapsed. Ice-machine oil with a viscosity of about 85 SUS at 100 and a pour point of -90°F. is suitable for the hydraulic system.

The operation of the contact mechanism is described as follows:

Cracking.

The charge stock is pumped from storage through a measuring vessel into the preheater where it is vaporized. The oil vapors enter the reactor through valve 1. The reaction products leave the reactor through valve 4; after cooling they enter the product measuring vessel where separation into liquid and gaseous

components takes place. Part of the cracking gas is stored in the gas holder and the remainder is recycled through the cracking gas preheater and valve 6; the bulk, however, is returned into the plant.

Purging with Cracking Gas.

By switching from position "b" to "e", the feed stock is by-passed and the reactor can be purged with cracking gas (from the gas holder) which flows through valve 3 to 5. Valve 6 is closed. The oil which condenses in the cooler is separated in the separator.

Purging with Nitrogen.

The cracking gas is circulated using positions "f" to "i" and valve 6; reactor valves 3 and 5 are closed; valves 7, 8 and 9 are open. Nitrogen is introduced into the reactor by means of the blower after it has been preheated in the regenerating gas-preheater; the gas is passed into the reactor half-way between the catalyst beds. The purge gas leaves the reactor through 7 and 8, is cooled and purified by means of a centrifuge; it is partly vented and partly recycled through valve 11.

Catalyst Regeneration.

When regeneration is carried out with recycled gas at elevated pressure, the recycle system is brought up to the desired pressure using switch position "k". At position "l" the recycled gas is enriched with air which is introduced through the gate valve 14 proportional to the amount of carbon on the catalyst which has to be burned off. The pressure ranges from 44 - 90 psig. The pressure on the reactor is released at position "m" by opening the gate valve 12.

Regeneration with air alone is carried out with positions "n" through "p". The gate valve 11 is closed. Air is sucked in through the air filter of the blower and is passed into the reactor through the preheater and is finally vented through valves 12 and 13. At definite periods cold air is injected into the reactor through the regulating valves b and c. Positions "m" and "p" serve as protection for the blowers. The quantity of gas flowing through the reactor is regulated by regulating valve c.

The reactor is subsequently purged again with nitrogen analagous to the first nitrogen purge period. The reactor is closed in position "s", the gas is recycled through the reactor by-pass (valve 16). At position "u" a new cracking period starts.

The cracking catalyst is a synthetic aluminum silicate of the approximate composition $Al_2O_3 \cdot 3SiO_2$ which has been prepared by a special process. Two batches of 1 ton of the catalyst have been prepared for the catalytic cracking pilot plant and the first batch was of entirely unsatisfactory activity (it produced 20% of gasoline boiling below 345°F.). This catalyst was therefore only used for starting the unit and training the operating personnel. The second batch of catalyst was of somewhat better quality (gasoline yield - 29% of gasoline boiling below 345°F.). The particle size is of extreme importance for the activity of the catalyst. The cube-shaped catalyst should have a length of 0.24 - 0.4 inches. The conversion of middle oil amounted to 47% (28% of gasoline boiling below 345°F.). Experiments on a smaller scale indicated that the conversion increases when the particle size is kept at 0.15 - 0.25 inches; the conversion in this case is 60% with 32% of gasoline boiling below 345°F.

Because the catalyst did not have maximum activity and because a somewhat unsatisfactory charge stock was run, the unit was not operated for the purpose of obtaining detailed information on the quality of potential products. The purpose of the operation was to obtain mainly data on the regeneration of the catalyst and the control of thermal conditions in this operation. Samples for material balances were taken in such a way that aliquot parts of the liquid and gaseous reaction products were taken in the ratio in which they were produced and these samples were distilled and stabilized. An automatic gas sampler (Figure 3) was used for constant sampling of the cracked gas.

Regeneration of the Catalyst.

The problem in the regeneration of catalyst which has become inactive by carbon deposition consists in keeping the temperature below a maximum of $1,022°F.$ and, at the same time, carrying out the regeneration in the shortest possible time in order to reduce to a minimum the unproductive time of the unit. Combustion of the carbon is carried out by means of oxygen-containing gases which also serve for the dissipation of the heat liberated. The rate of combustion is proportional to the oxygen concentration, which means that a large amount of gas is required for the removal of heat. Since air alone is used for the regeneration, the quantity of gas required is larger than in the case where a gas enriched with oxygen is used and the quantity of gas to be used is limited economically by the pressure drop in the catalyst bed. In order to decrease the pressure drop, the catalyst can be arranged in several beds which are operated in series during the cracking period and in parallel during regeneration.

As long as the oxygen concentration in the gas has not reached that of air, much inert gas is needed. This leads to recycling of the regenerating gases in order to keep the amount of inert gas as small as possible.

In the Houdry process, combustion of the carbon deposit can be carried out with air alone without having to pass an extremely large amount of air over the catalyst because the heat of reaction is carried away by means of molten salt circulating through the catalyst. For the pilot plant, regeneration with direct removal of heat through the combustion gas was planned and this process was to be carried out at atmospheric pressure or at elevated pressure (up to about 90 psig.). It had, however, been previously found that regeneration of the catalyst will only be complete when the gases enter the reactor at about $750°F.$ Since, on the other hand, the maximum temperature in the catalyst bed must not exceed $1,022°F.,$ only a temperature difference of about $270°$ is available for the removal of heat. Laboratory tests had indicated that at a gas inlet temperature of $752°F.$ not all of the carbon deposited on the catalyst will be burned off but that a temperature of $860°F.$ was necessary to completely remove the carbon. This means that over the entire regeneration time the temperature difference cannot always be $270°F.$ If it is desired to completely remove the carbon. Experiments in a 20-gallon reactor indicated the magnitude of the average temperature difference over the entire regeneration time, i. e., not the time until the coke has been entirely burned off but the time at which the initial temperature of the catalyst (cracking temperature) has been reached again. The mean temperature difference, accordingly, lay at about $110°F.$ That meant, however, that only a third of the temperature difference of $270°F.$ could be utilized in practice. Consequently, one of the main objectives of the investigation was to find the factors which predominantly affect the mean temperature differential. Experiments in the 20-gallon reactor had shown that an increase in the mean temperature difference was to be expected when several catalyst layers were used in a reactor. By intensive cooling of the hot catalyst, the mean temperature difference should be further improved. It should also be tried whether the temperature difference of $270°$ cannot be increased by lowering the gas inlet temperature below $752°F.$

The Mechanism of Carbon Combustion.

Quantitative analysis of the catalyst containing a carbon deposit indicated that the carbon-hydrogen ratio of the deposit had the formula C_1H_1. It was furthermore found that, on the average, about a third of the carbon is burned to CO and two-thirds to CO_2. Under these conditions the heat of combustion for the carbon is calculated to be about 14,400 B. t. u. per pound. The amount of carbon deposited on the catalyst depends on the operating conditions and, especially, on the charge stock. Using a mixed base charge stock (end point 752°F.) which has been used as the charge stock for all pilot plant runs, the carbon to be removed amounts to about 2 - 4% by weight of the catalyst.

At the start of the regeneration period the catalyst has still the temperature of the cracking period, namely 752 - 788°F. Consequently, in the experiments to determine the ignition temperature of the carbon only the temperature of the regenerating air was varied. In these investigations it was found that the carbon consists of one portion which is easily ignited and a portion which is difficult to ignite. Whereas the first kind of carbon is ignited with air already at 482°F., the ignition temperature of the second portion lies around 815°F. If air is permitted to interact at 788°F. with catalyst containing carbon and if the removal of heat is sufficient to maintain the temperature of the catalyst at this level, the carbon ignites. The combustion, however, stops abruptly as soon as the easily-ignited deposit has been burned off. If the air temperature is raised to 815°F., the more difficultly-ignited carbon starts to burn. At 968°F. finally a small amount of residual carbon, approximately 5% of the total carbon, is burned off. Up to now, it could not be proved whether failure to burn the residual carbon affects the activity of the catalyst. The findings with respect to the different ignition temperatures of the carbon were confirmed in a special laboratory apparatus. In the first burning period the bulk of the combustion water is found in the regenerator waste gas. It can, therefore, be assumed that first the hydrogen-containing carbonaceous deposit and oil residues are burned off and that a carbon with low hydrogen content or free carbon remains on the catalyst.

Whereas the second burning period shows little relation to the concentration of oxygen in the regeneration gas, the combustion of the easily-ignited carbon is extremely dependent on the oxygen content of the regenerating gas. The amount of easily-ignited carbon is directly proportional to the extent of purging of the catalyst after cracking, but even after prolonged and intensive purging easily-ignited carbon is still present. For a normal purging period the ratio of the two carbon modifications amounts to about one-third easily-ignited and two-thirds difficultly-ignited carbon. The amount of difficultly-ignited carbon depends on the operating conditions (temperature reached by the catalyst during burning of the easily-ignited carbon, removal of heat by regeneration gases, temperature of regenerating gases).

Recycle Process.

Removal of the heat of combustion of the carbon requires gas quantities of 2,000 - 5,000 volumes per volume of catalyst. These large quantities of gas necessitate recycling of the inert gas-air mixture required for catalyst regeneration. According to the composition of the carbon deposit (C_1H_1), steam is produced during regeneration and the water vapor concentration in the recycle gas can reach 15%. Since the synthetic catalyst is susceptible to water, it was necessary to separate the water by condensation and this raises the cost for this type of operation

Consideration of the thermal conditions indicated that it would be advantageous to operate the regeneration step at elevated pressure (up to 90 psig.) and experiments in this direction were carried out.

Regeneration at Atmospheric Pressure.

For catalyst regeneration by a recycle process, it is advantageous to contact the catalyst with the regeneration gases at the ignition temperature of the more difficultly-ignited carbon because then stoppage of the combustion cannot occur. It could be clearly shown that otherwise an interruption in the combustion of the carbon occurs even when sufficient oxygen is present in the recycle gas. The regenerating time depends on the amount of carbon on the catalyst and the regeneration conditions.

Since no petroleum products were available, middle oil from the hydrogenation of brown coal was run in the unit and the operating times were increased in order to obtain a sufficiently large carbon deposit.

The amount of carbon burned off was calculated by assuming a heating value of 14,400 B. t. u. per pound. The amount of carbon deposit shown in Table 2 corresponds to definite operating practices using mixed-base natural oil products (boiling range 329 - 752°F.). The times required for purging with cracking gas and the first nitrogen purging period are not considered in the regenerating time since no exact times were obtained in the experimental unit and the connection to be added for the pumping periods is only very small in commercial operation. Regeneration was carried out using 106,000 - 175,000 cu. ft. of gas per hour which was introduced into the reactor at 815°F. As soon as the combustion of the carbon was finished, the inlet temperature of the gas was reduced to 788°F. in order to bring the temperature in the catalyst bed to the cracking temperature. Subsequently, oxygen in the circulated gas was replaced by nitrogen.

The following table shows the results of regeneration with circulated gas at atmospheric pressure:

Table 2

Cracking Conditions for Petroleum Gas Oil (329 - 752°F.)		Carbon on Catalyst % by Wt.	Regeneration Gas (cu.ft./hour)	Mean Temp. Difference °F.	Regeneration Time Minutes	Ratio of Cracking to Regeneration Period
vol. oil/ vol. catalyst/ hour	Cracking Time, Minutes					
1.0	20	2.2	106,000	115	90	1 : 4.5
			175,500	108	65	1 : 3.3
0.6	60	3.0	106,000	119	160	1 : 2.6
			175,500	111	110	1 : 1.7
0.5	120	3.8	106,000	122	200	1 : 1.7
			175,500	117	135	1 : 1.1
0.4	180	4.1	106,000	126	210	1 : 1.2
			175,500	120	140	1 : 0.8

When the carbon is removed completely in the recycle process the mean temperature difference decreases because of the additional heating required and the small heat of reaction of the residual carbon. For a space velocity of 1, a cracking time of 20 minutes (using the same stock as shown in the previous table) and a carbon deposit of 2.2% by weight, the ratio of reaction to regeneration time is 1 : 11, and the regeneration time is 220 minutes; the mean temperature difference in this case is 63 - 72°F. In a commercial unit it will, therefore, not be advisable to completely burn off the carbon, especially since there are no indications that the activity of the catalyst is appreciably affected by the residual carbon.

Regeneration at Elevated Pressure.

The recycle regeneration process at elevated pressure (106,000 cu. ft. of gas per hour at 47 psig. and 176,500 cu. ft. of gas per hour at 73.5 psig.) did not show any great advantage over the operation at atmospheric pressure with respect to the time required for combustion. The mean temperature difference was about 2 - 4° higher since the start of the combustion was somewhat earlier due to the increase in partial pressure of the oxygen in the regeneration gas; the temperatures of the catalyst bed rise more rapidly than during operation at atmospheric pressure. This, again, results in difficulties in the control of the air concentration of the regenerating gas since the maximum temperature is easily exceeded due to the greater speed of combustion. Sometimes, sudden peak temperatures are observed. Since the cracking process itself is operated at atmospheric pressure, the pressure in the reactor must be released prior to the cracking period and this, again, prolongs the regeneration time and requires complicated control mechanism and a pressure-resistant reactor.

Regeneration with Air Alone.

The result of the ignition experiments made it advisable to carry out regeneration with air alone without excessively increasing its quantity. Due to the difference in the ignition temperature of the two carbon modifications, the possibility presented itself to carry out combustion with air alone. At first, air of about 662°F. was used and the first third of the coke was burned off with an especially fast temperature rise and a mean temperature difference of about 360°F. The heat liberated heats the catalyst and the air to such an extent that the ignition temperature of the difficultly-ignited carbon is reached which is then burned off irrespective of the oxygen concentration. The difficulty in this case lies mainly in the change from the first to the second burning period because the "difficultly-ignited" carbon may ignite already while the first portion of the carbon is still burning if the temperature rise is too sudden. In this case it is impossible to avoid exceeding the maximum temperature. If the rise in the air temperature comes too late, combustion stops.

The following studies on the regeneration with air alone were carried out:

1. Removal of heat in the reactor by changing the amount of regenerating air.
2. Control of the temperature of the regenerating air by means of a preheater by-pass.
3. Control of the temperature of the regenerating air by the addition of cold air.
   1. In order to control the first temperature maximum (only possible with carbon deposits greater than 1% by weight of catalyst), and in order to avoid stoppage of the combustion during switch-over to the second burning period, the necessary burning temperature was maintained by

changing the amount of air, i. e., by increasing or decreasing the removal of heat in the reactor.

2. By means of a preheater by-pass, the temperature of the air was brought to the desired level, prior to introducing it into the reactor, by alternately admitting preheated air and air which had by-passed the preheater.

These two methods of catalyst regeneration with air alone are unsuited for commercial operation since the preheater lag is too large for continuous changes in throughput and for changes, over short periods of time. Consequently, this method of attack was abandoned soon.

3. The preheated regenerating air was mixed with a definite amount of cold air prior to its entrance into the reactor and in this way the inlet temperature was controlled. A constant amount of air could be maintained at the same temperature during the entire regenerating period. Only at the start of the regeneration and after termination of the burning time cold air was added in order to control ignition of the two modifications of carbon and in order to again reach the cracking temperature. Addition of cold air resulted in an increase in the amount of air passed over the catalyst which occurred exactly at the time when an increased amount of heat had to be removed from the reactor. The addition of cold air was controlled by air-operated valves.

It was attempted to conduct the entire regeneration in such a way that the air left the preheater with a temperature of 788°F. and was brought, by the addition of cold air, to the desired lower temperature at the beginning and at the end of the regeneration period. The choice of the temperature of 788°F. was made for the following 4 reasons:

1. After each regeneration the entire catalyst must be brought, as uniformly as possible, to 788°F.
2. The air in the reactor is heated rapidly to 815°F. by the hot catalyst so that ignition of the "difficultly-burned" carbon takes place.
3. The change from the first to the second burning period can be easily obtained with air of 788°F.
4. It must be attempted to carry out regeneration with an air inlet temperature as low as possible in order to obtain a large temperature difference.

In this type of regeneration with air alone, part of the "difficultly-ignitable" carbon was not completely removed since the catalyst which is contacted first by air will have a temperature only slightly higher than 788°F. The addition of cold air was controlled in such a way that the temperature in the last layers of the catalyst bed did not exceed 1,022°F.

In an extended test in which 98 batches of catalyst were regenerated, the operation was carried out with air of 788°F.; the catalyst contained 2.2% by weight of carbon. 106,000 cu. ft. of air were preheated and from the 1st to the 4th minute the addition of cold air was varied from 28,240 cu. ft. of cold air down to 0 cu. ft. The combustion period lasted 36 minutes. From the end of the combustion period (36 minutes) to the 50th minute, 28,250 cu. ft. of cold gas per hour were introduced into the reactor. The total regeneration time, including purging with nitrogen and bringing to temperature, was 70 minutes. The mean temperature difference in the run was 150°F. and the ratio of cracking to regeneration time was 1 : 3.5. The oil conversion decreased from batch to batch as indicated in the following table:

Table 3

Batch No.	Conversion % by Wt.	Gasoline % by Wt.	C₃ and C₄ % by Wt.	Residual Oil % by Wt.
1	46.0	28.5	10.5	54.0
11	45.5	28.0	10.2	54.5
30	45.0	27.5	10.0	55.0
50	43.0	27.0	9.8	57.0
75	42.0	26.0	9.7	58.0
95	40.5	25.5	9.5	59.5

In order to remove the carbon completely, it was necessary to bring all catalyst beds to a temperature of 968°F. This necessitated a continuous increase in the temperature of the regenerating air. Advantageously, this could be accomplished by operating the preheater at 968°F. and only lowering the air temperature at the start of the regeneration period and at the beginning of the cracking period by addition of cold air. This could not be done in the test runs because the capacity of the blower was insufficient.

Temperature control in the reactor was obtained by means of thermocouples which were arranged in iron thermocouple wells in the catalyst bed. The temperature curve obtained during regeneration with air alone showed a steeper rise as compared to the temperature for the recycle regeneration operation, but normally the maximum temperature of 1,022°F. was not exceeded. However, when the thermocouples were immersed directly into the catalyst bed without the use of thermocouple wells, a definite difference in the temperature curve of the 2 regeneration processes could be observed. When ignition of the 2 carbon modifications started, frequently temperature peaks were observed which were up to 200°F. higher than the temperatures indicated by the thermocouples in the well. In the case of regeneration by recycling of gas under pressure also, temperature maxima were observed when unprotected thermocouples were used, but the temperature peaks were not as high as in the other cases. Temperature peaks can be avoided by correct control of the added cold air; however, when the catalyst is regenerated with air alone, temperature peaks appear very suddenly. It is assumed that temperature peaks are detrimental to the catalyst and lead to a premature decrease in its activity.

If the carbon deposit on the catalyst exceeds 2%, regeneration with air alone becomes increasingly difficult because a too-rapid change to the second burning period will unavoidably lead to excessively high temperatures. A deposit of 3% of carbon on the catalyst requires 4 - 5,000 cu. ft. of air per cu. ft. of catalyst per hour.

Comparison of the 2 Regeneration Processes.

The only safe regeneration method with respect to apparatus design and conservation of the activity of the catalyst is regeneration with recycled gas at atmospheric pressure. The gas must be cooled after leaving the reactor in order to condense water vapors which are detrimental to the catalyst. Subsequently, the gases must again be preheated to the required temperature.

Only for carbon deposits up to 1% regeneration with air alone is advantageous. Since regeneration takes place in 1 pass, cooling of the combustion gases for the separation of water vapor is unnecessary.

Whereas for the regeneration in a cycle process, oxygen-free regeneration tail gases are available for purging with inert gases, a special inert gas manufacturing unit must be provided for a cracking unit in which the regeneration of the catalyst is carried out in 1-pass with air alone.

Purging of the Reactor.

Before the carbon-containing catalyst is regenerated by means of oxygen-containing gases, the reactor must be as free as possible from oil vapors since otherwise explosion of the oil vapor-regeneration gas mixture must be expected. Purging of the reactor after the cracking period is carried out by means of cracking gases. The reactor is subsequently purged with inert gas in order to replace the cracking gas by a non-combustible gas. After regeneration the reactor is freed from oxygen-containing regeneration gases by purging with inert gas. These purging times must be included in the regeneration time and should, therefore, be as short as possible.

Purging with Cracking Gas.

Purging of the reactor with cracking gas took place in a special cycle. A blower circulated the gas through preheater, reactor, cooler and centrifuge.

The purging effect proceeds asymptotically, i. e., for a given amount of purging gas a maximum of separated oil is obtained after a definite time. This is indicated in the following table:

Table 4

Purging Time Min.	Amount of Purging Gas cu.ft./hour	Amount of Oil Separated lbs.	Rate of Flow in Separator ft./sec.	Remarks
5		12.1		
10	3531	17.0	3.3	
15		19.1		
20		20.3		
5		22.4		
10	8830	25.8	7.9	
15		26.4		
5		21.2		
10	10,580	22.7	9.5	Fog formation
15		33.2		
5		21.2		
10	14,100	23.2	12.5	Strong fog formation
15		23.6		

Increasing the amount of purge gas decreased the time required to obtain maximum oil separated. However, the increase in the gas quantity was counteracted by the fact that the increased rate of flow of the purging gas atomized the oil

drops in the centrifugal separator and caused the formation of oil fog. When a quantity of purging gas exceeding 8,820 cu. ft. per hour was passed through the separator (diameter 7.9 inches) at a gas flow in the separator of less than 7.9 feet per second, fog was formed. These oil fogs could not be separated and, consequently, were recycled. Even when no fogs were observed, it was necessary to have at least 3 centrifugal separators in series in order to obtain satisfactory separation of the oil.

With the separators available, the optimum conditions consisted in a purge gas volume of 8,820 cu. ft. per hour and a purging time of 10 minutes. It must, however, be expected that shorter times can be reached for the cracking gas purging period when suitable separators with larger diameters are installed.

Purging with Inert Gas.

The purging experiments for the regeneration process using recycled gases indicate that a volume amounting to twice that of the recycled gas is sufficient for satisfactory purging. This would indicate that in a commercial unit a volume of inert gas corresponding to 2 - 3 times the reactor volume would be satisfactory.

The experiments on catalytic cracking in a fixed-bed reactor are considered completed, with the exception of catalyst development, since it is believed that sufficient design data have been obtained for the construction of a commercial unit.

The work on moving-bed catalytic cracking which has been started about a year ago seems to indicate that this process will be superior to catalytic cracking in a fixed-bed reactor.
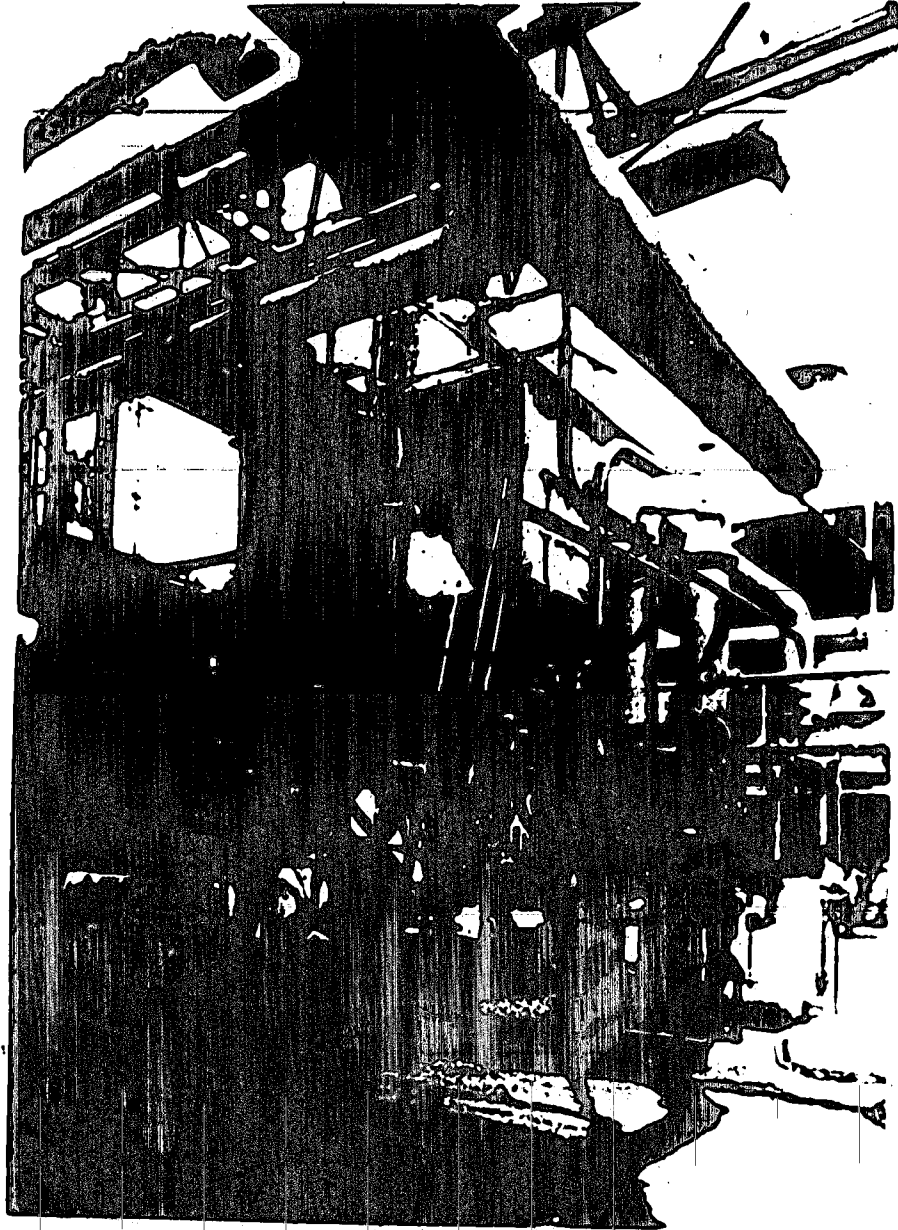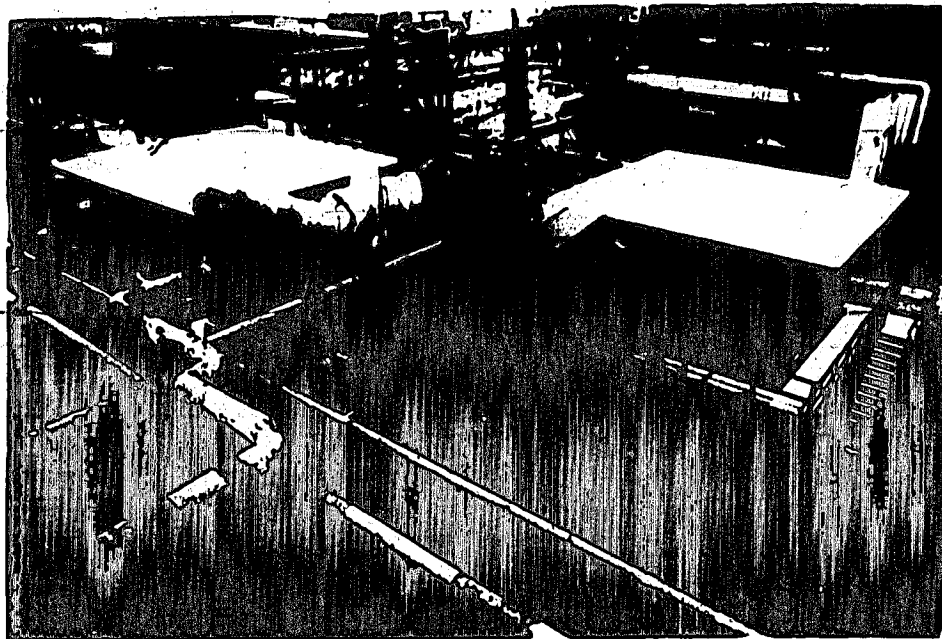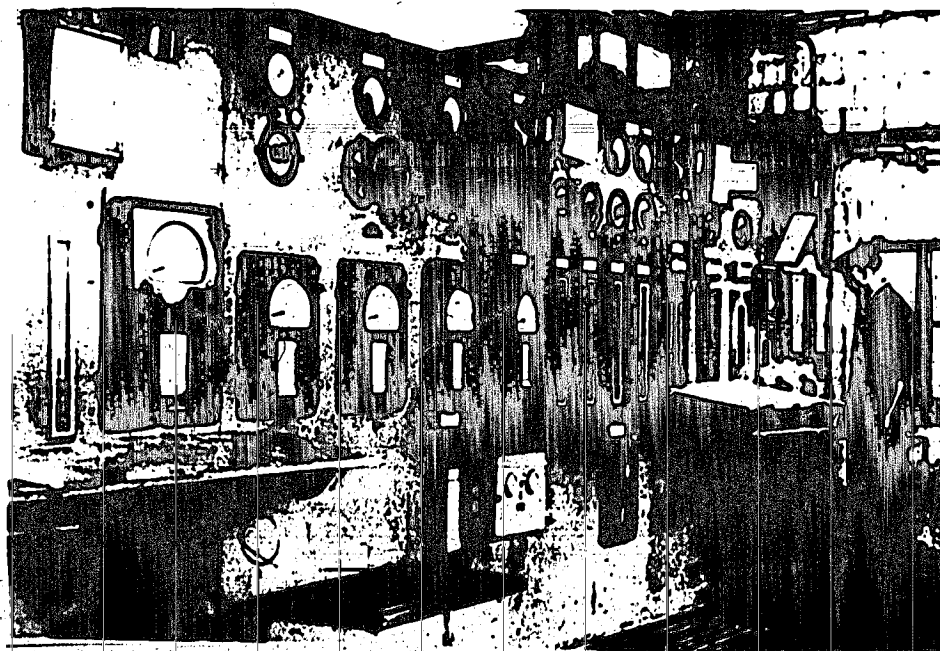
REACTOR

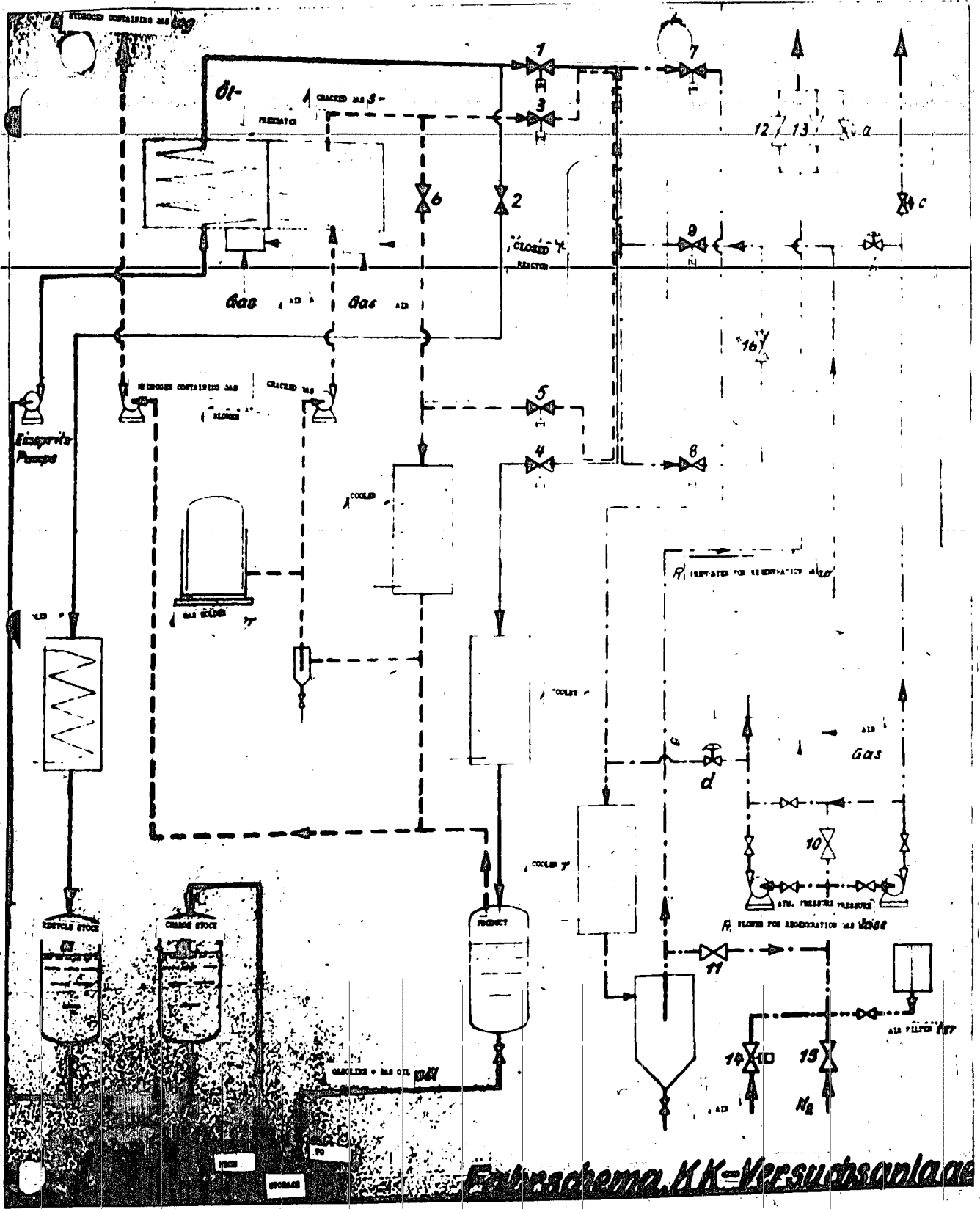LEFT, PREHEATER FOR REGENERATION GAS, RIGHT, PREHEATER FOR OIL
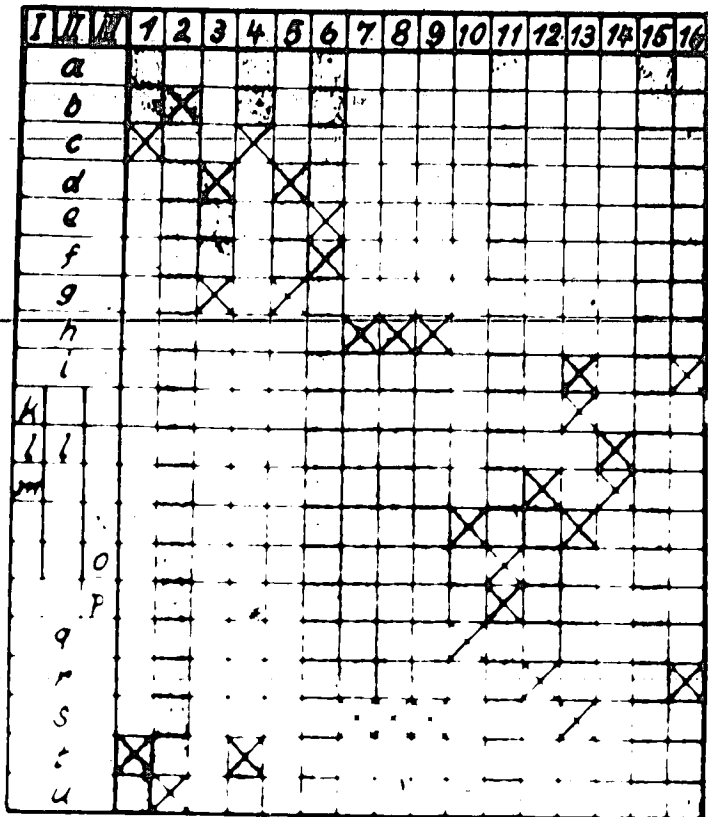
CENTER, REACTOR

B i l d   3

CONTROL PANEL

CONTACT DRUM, GROUP OF ELECTRIC SWITCH VALVES AND INDICATOR

Fließschema. K.K.-Versuchsanlage

Table header columns: I II III 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16

Rows: a b c d e f g h i k l m n o p q r s t u

GATE VALVES

CRACKING

PURGING WITH CRACKED GAS
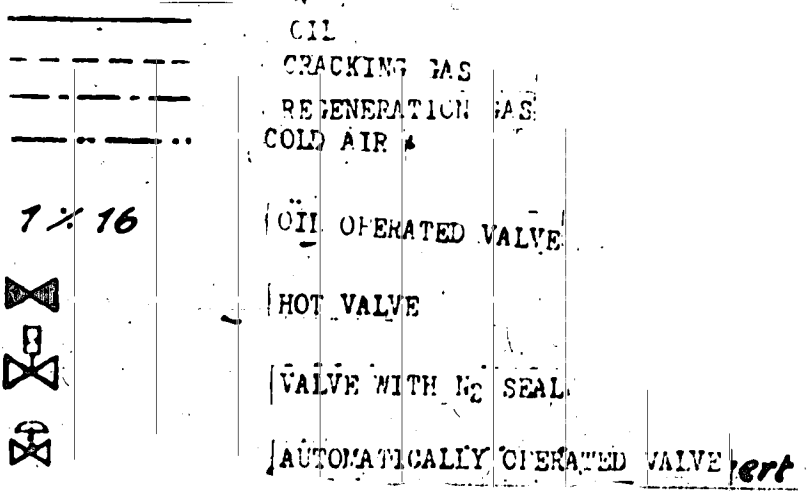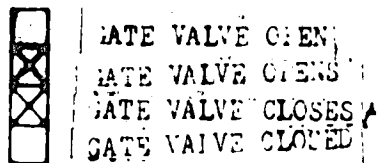
1. PURGE WITH N₂
OPERATED UNDER PRESSURE
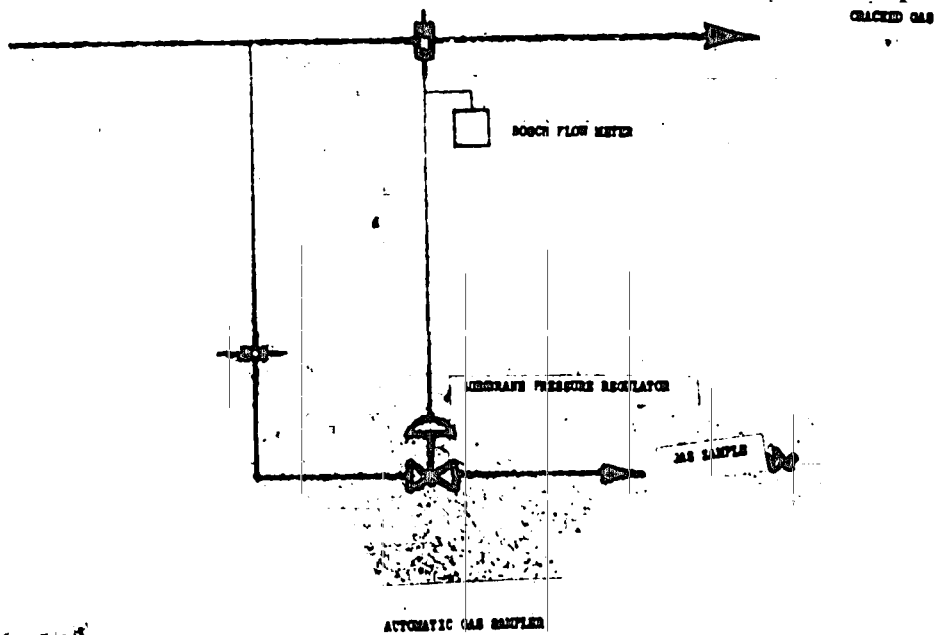RECYCLE REGENERATION
DEPRESSURING

Regeneration m. reiner Luft
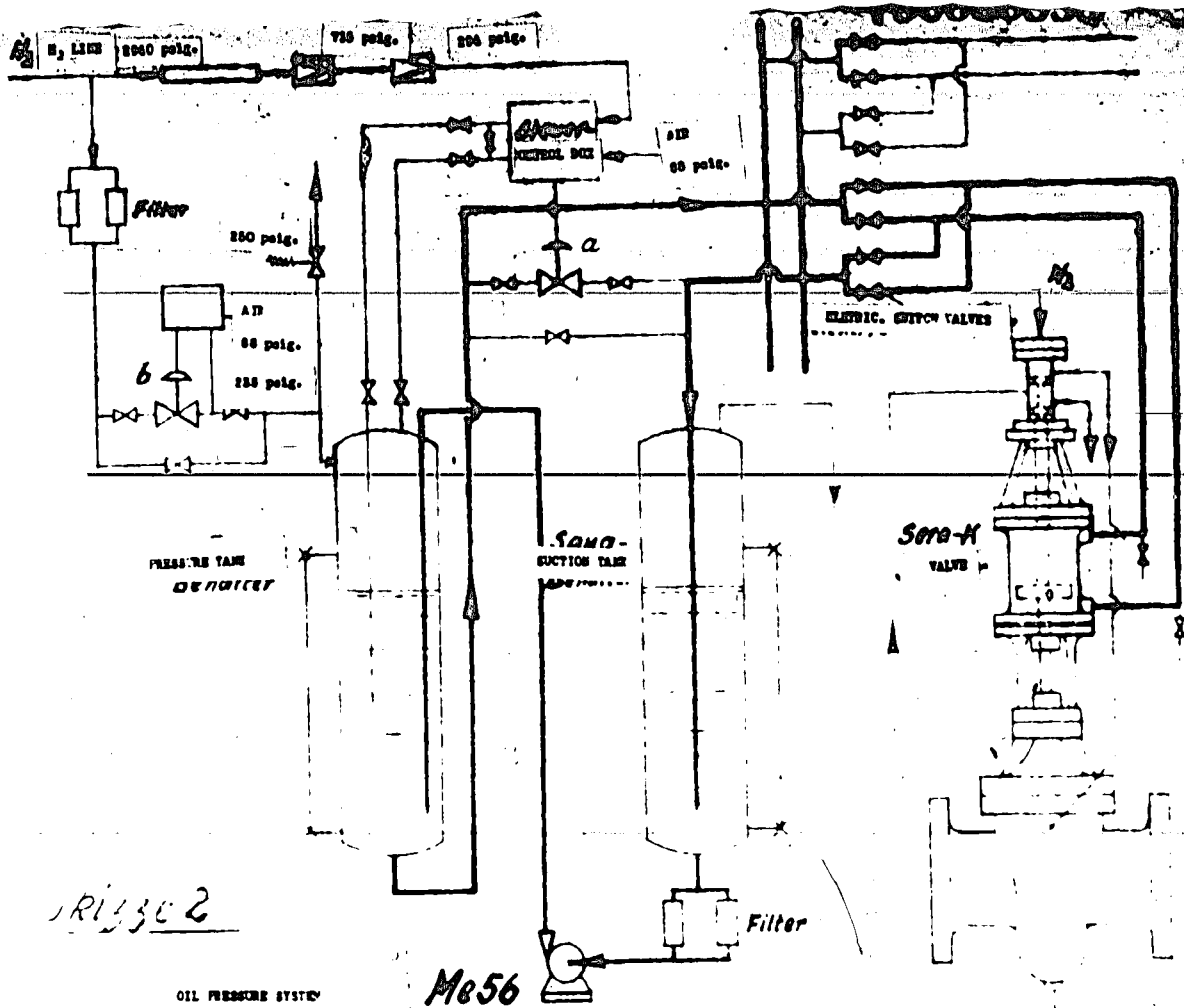
2. PURGE WITH N₂

CRACKING

I   *Regeneration* UNDER PRESSURE
II *Regeneration* AT ATMOSPHERIC PRESSURE
III *Regeneration* WITH AIR ALONE

GATE VALVE OPEN
GATE VALVE OPENS
GATE VALVE CLOSES
GATE VALVE CLOSED

——————— OIL
— — — — CRACKING GAS
— ·· — ·· REGENERATION GAS
— ·· — ·· COLD AIR

1 ÷ 16

OIL OPERATED VALVE

HOT VALVE

VALVE WITH N₂ SEAL

AUTOMATICALLY OPERATED VALVE

H₂ LINE 2940 psig. 735 psig. 206 psig.

Filter

250 psig.

AIR
88 psig.
235 psig.

CONTROL BOX
AIR
88 psig.

a

b

ELECTRIC. SWITCH VALVES

PRESSURE TANK
Druck

Saug-
SUCTION TANK

Servo-
VALVE

OIL PRESSURE SYSTEM

Filter

Mß56

Skizze 2

CRACKED GAS

BOSCH FLOW METER

MEMBRANE PRESSURE REGULATOR

GAS SAMPLE

AUTOMATIC GAS SAMPLER

Skizze 3