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CATALYTIC DEHYDROGENATION OF ISOBUTANE

Laboratory experiments were carried out on the conditions for catalytic dehydrogenation of isobutane at temperatures not exceeding 1,112°F. The work includes the effect of pressure and vacuum, as well as the presence of oxygen. No pilot plant data were obtained in the program whose results are given in the subject report.

The experiments were carried out in tubes of high-melting glass or quartz of a diameter of 0.64 - 0.79 inches. The gases were preheated before reaching the reaction space proper and the upper part of the reactor tube was used as a preheater. This part of the tube was filled with pieces of quartz. The reaction space proper which contained the catalyst had a volume of approximately 1.83 cubic inches (30 cc.). The remaining part of the tube was tapered in order to increase the gas velocity and thereby avoid undesirable side reactions.

The tubes were heated electrically by a jacket and the temperatures inside the tubes were measured as usual by iron-constantan or chromium-nickel thermocouples.

The gases were collected as usual over concentrated sodium chloride solution. In the experiments in which oxygen was used, the exit gas was passed through drying towers filled with caustic and the water, CO₂ and oxygen containing by-products formed were trapped here. After prolonged use the by-products mentioned could be detected in the material with which the towers were filled.

In the experiments under vacuum the gases were collected in the receiver shown in Figure 1. This apparatus permits to obtain samples for analysis and make gas measurements at any time without disturbing the vacuum in the apparatus. The operation of the apparatus is described as follows:

"A" is a flask whose capacity is chosen according to the capacity of the apparatus; in the experiments described here the volume of the flask was 5 liters.

"B" is a cylindrical vessel which will resist vacuum and pressure; it is standardized and has the same volume as the flask "A". The entire arrangement should be set up in such a way that the ends of tubes 2 and 3 are at the same level as shown in the figure. Before use, "A" and tubes 3 and 4 are completely filled with the absorption liquid, e.g. saturated sodium chloride solution. The connection of this measuring device with the apparatus is made by the three-way stop-cock "A".

During the experiment the gas leaving the reaction space escapes through 1. By means of 5 the same pressure as in the experimental apparatus is obtained in "B" and, therefore, also in 1. From 1, the gas is permitted to enter "A" through a and 2, whereby the corresponding volume of confining liquid flows over 3 into "B". By exact measurement of the liquid volume and the time required to obtain this volume, the amount of gas flowing in the unit of time is determined. By changing the position of "c" and "d", atmospheric pressure is produced in the container "B" and the volume of gas in "A" assumes the volume corresponding to atmospheric pressure. The gas samples for analysis are now taken through f. The confining liquid in "B" is returned to "A" by means of compressed air through 6 and e and the line 4. The apparatus is now ready for further sampling.

In order to avoid erroneous results due to the decrease of the activity of the catalyst, the catalyst tube was filled with fresh catalyst for each temperature investigated. Each temperature was maintained for 1 day and as many samples as possible were taken during this period. The analytical data given in the tables are mostly average values and maximum values which are obtained immediately after starting with a new catalyst were not considered.

Isobutylene was determined by standing over sulphuric acid (65%), whereas the n-butylene and propylene was determined by slight shaking with 87% sulphuric acid.

Ethylene was determined, as usual, with bromine water.

The total amount of olefins was determined by treating a sample of gas as received with bromine water.

In the experiments in which oxygen was used, oxygen was determined by means of chromous chloride solution and carbon monoxide was determined with cuprous chloride solution.

Catalysts

1. Activated charcoal, sulphidic and oxidic catalysts, except chromium oxide.

The catalysts given below were not found suitable for the dehydrogenation of isobutane and had an exceedingly short life: Sulphides of the 6th and 8th group of the Periodic System, ZnS, oxides of 5th and 8th group of the Periodic System, V_2O_5 , Mn_2O_5 , activated charcoal with various additives, poisoned catalysts like activated charcoal treated with platinum and with PCl_3 , graphitic acid. The methanol catalyst also was unsatisfactory.

2. Catalysts on the basis of chromium oxide.

Chromium oxide with additions of earth alkali chloride and alkali chloride was found most suitable. A good catalyst was found soon in $Cr_2O_3 \cdot 5\% BaCl_2$. With this catalyst the systematic experiments described below have been made.

The following catalysts were also tested: $Cr_2O_3 \cdot 20\% BaCl_2$, $Cr_2O_3 \cdot 5\% Ba(OH)_2$, $Cr_2O_3 \cdot 5\% MgCl_2$, $Cr_2O_3 \cdot 5\% LiCl$, $Cr_2O_3 \cdot 5\% NaCl$, $Cr_2O_3 \cdot 5\% KCl$. The manufacture and method of precipitation of the chromium oxide is of highest importance for the activity of these catalysts. The catalysts which were at first made from molten chromium oxide (chromic acid is molten and reduced at 662 - 842°F. in a current of hydrogen) showed low resistance at the experimental conditions chosen. After a few hours already decomposition was observed and the catalyst was carried away with the gases in the form of a powder. The catalyst volume decreased to 1/3 and more from its original volume. These disadvantages could be avoided by the use of precipitated chromium oxide.

According to known methods, chromium is precipitated as $Cr(OH)_3$, decanted, washed and filtered. The moist $Cr(OH)_3$ is kneaded with a concentrated solution of the desired additive, dried and formed into pills. Before use the catalyst is dried in vacuum at 752°F. (12 cubic inches of catalyst dried in 12 hours.)

The catalyst prepared in this way showed great strength at the temperatures used for the dehydrogenation of isobutane. In experiments extending over

500 - 1,000 hours, in most cases no loss of catalyst was found or the loss amounted to only a few percent of the original charge. When the catalyst was removed from the reactor, no changes were found except darkening in color. The original mechanical strength of the catalyst remained the same. No carbon deposition was observed in the laboratory tests.

Since the chromium oxide catalyst used in these experiments showed a decreased activity in extended use (up to 1,000 hours) further variations in the manufacture of the catalyst were attempted. Precipitation of the chromium hydroxide with ammonia in methyl alcohol solution was without success.

The addition of earth alkali and alkali chloride resulted in a considerable increase in the activity of the chromium oxide. For instance $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$ not only was more active as compared to Cr_2O_3 , but showed also an increase in the selectivity, i.e. the ratio of dehydration to cracking became more favorable.

Not only the amount of additive material but also the kind of halogen ion seems to be of importance. $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$ was found to be more effective than $\text{Cr}_2\text{O}_3 \cdot 20\% \text{BaCl}_2$. The combination $\text{Cr}_2\text{O}_3 \cdot 5\% \text{Ba}(\text{OH})_2$ showed even smaller activity.

The results of the study of many catalysts brought out the noticeable superiority of chromium oxide containing earth alkali and alkali chloride. It would appear justified to make large-scale experiments with the $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$ catalyst.

Experiments with Chrome Oxide Catalyst.

1. Comparison with other catalysts.

Table 2 and Figures 2 and 3 show the results obtained with two sulphidic catalysts at atmospheric pressure and reduced pressure as compared to the catalyst $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$.

2. Experiments with chromium oxide catalyst containing alkali and earth alkali additives.

Table 2 and Figures 4 and 5 show the results obtained with catalyst $\text{Cr}_2\text{O}_3 \cdot 5\% \text{LiCl}$, $\text{Cr}_2\text{O}_3 \cdot 5\% \text{NaCl}$, $\text{Cr}_2\text{O}_3 \cdot 5\% \text{KCl}$, $\text{Cr}_2\text{O}_3 \cdot 5\% \text{MgCl}_2$ and $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$. The addition of NaCl , KCl and BaCl_2 gave the best results, barium chloride being the most satisfactory additive (see Figure 5).

3. Experiments with chromium oxide catalyst with barium chloride and barium hydroxide additives.

The good results obtained with catalyst $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$ led to further variations of the combination chromium oxide - barium compounds.

Table 3 and Figure 5 show results obtained in these tests. Figure 6 shows the difference of Cr_2O_3 without additives and the combination $\text{Cr}_2\text{O}_3 \cdot \text{BaCl}_2$.

All these experiments make it advisable to investigate this latter combination in greater detail.

Systematic Experiments with $\text{Cr}_2\text{O}_3 \cdot 5\% \text{BaCl}_2$.

Theoretical considerations indicate that the best results should be obtained under vacuum in the presence of oxygen. It was to be anticipated that at

lower temperatures under vacuum the same results should be obtained as at atmospheric pressure. Since the dehydrogenation of isobutane is an endothermic reaction and requires considerable supply of energy, it was intended to provide half of the heat required by the addition of limited amounts of oxygen to combine with the hydrogen liberated in the reaction; better distribution of the heat over the reaction space also was expected from this measure. By the removal of hydrogen in the form of water, furthermore, the equilibrium should be shifted to one side.

1. Experiments at reduced pressure without the addition of oxygen.

The results shown in Table 4 are plotted in Figure 2 - 9, 14 and 15, which show the relation of conversion and yields to temperature, whereas Figures 10 - 13 show the isobutylene yield in relation to the space velocity.

The data permit the following conclusions:

- a. Higher conversions are obtained with increasing temperature and simultaneously the isobutane cracking increases. (Figures 9 and 10)
- b. With increasing space velocity which is equivalent to a shortening of the residence time, higher isobutylene yields are obtained but the conversion is decreased. (Figures 10 - 14)
- c. Reduction in pressure corresponds to shortening of the residence time and, consequently, conversion is decreased and also the yields of cracking products.

2. Experiments under reduced pressure and in the presence of oxygen.

Details are given in Table 5 and the corresponding diagrams. The comparison shown in Figures 16 - 21 indicate that no improved results are obtained by addition of oxygen.

In the experiments with oxygen, the formation of very small amounts of CO₂ were found. No CO was formed in the presence of Cr₂O₃ · 5% BaCl₂. However, the reaction gas contained oxygen which means that the oxygen added has not been completely used up.

3. Experiments at atmospheric pressure with and without the addition of oxygen.

The results of these experiments are given in Table 6 and in Figures 22 - 24.

Experiments carried out under these conditions confirm the findings made in the experiments carried out under vacuum. With increasing temperatures the conversion is increased but the formation of cracking products also increases. By increasing the space velocity, less isobutylene is cracked and conversion is smaller.

Analogous to the vacuum experiments, it was found that the addition of oxygen is without advantage but has certain disadvantages because the greater part of the oxygen used (often up to 100%) was found in the reaction gas. Carbon monoxide was not found in the experiments with chromium oxide catalyst. CO₂ in amounts corresponding to a maximum of 1 - 2% of the isobutane charged were found. Oxygen containing organic compounds were not determined in the analysis.

Conditions under which less than 20% of the isobutane charged was consumed per pass were not investigated because of the lack of economic importance (increased cost of isobutane separation, increased cost when the amount of recycle gases is increased). Best yields of isobutylene were obtained with Cr₂O₃ · 5% BaCl₂ at a temperature of 1,112°F., pressure of 14.7 psig., and a space velocity of 266 volumes per volume of catalyst per hour. The conversion of the butylene charged per pass was 35.04% of which 78.8% were dehydrogenated to isobutylene. This corresponds to 10.9 pounds of isobutylene per cubic foot of catalyst per hour.

Table 1

Catalyst	Temperature °F	Pressure psia.	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀ Charged	Conversion of isoC ₄ H ₁₀ Reacted	
				%	Dehydro- genation %	Cracking %
2NiS·WS ₃ (#118)	842	7.4	66	12.1	71.4	28.6
	932	7.4	66	13.5	79.0	21.0
	1022	7.4	66	17.8	83.7	16.3
	1112	7.4	66	30.4	80.5	19.5
Cr ₂ O ₃ ·5%BaCl ₂	842	7.4	66	20.85	82.6	17.4
	932	7.4	66	23.20	82.7	17.3
	1022	7.4	66	27.31	81.7	18.3
	1112	7.4	66	42.02	78.2	21.8
3NiS·MoS ₃	842	14.7	133	6.30	71.8	28.2
	932	14.7	133	9.30	88.4	11.6
	1022	14.7	133	18.26	84.6	15.4
	1112	14.7	133	28.81	81.5	18.5
Cr ₂ O ₃ ·5%BaCl ₂	842	14.7	133	19.56	87.8	12.2
	932	14.7	133	23.69	90.0	10.0
	1022	14.7	133	28.58	84.8	15.2
	1112	14.7	133	47.63	78.4	21.6

Table 2

Catalyst	Temperature °F	Pressure psia.	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀	Conversion of isoC ₄ H ₁₀ Reacted	
				Charged %	Dehydro- genation %	Cracking %
Cr ₂ O ₃ ·5%LiCl	842	14.7	133	9.18	75.6	24.4
	932	14.7	133	10.13	90.2	9.8
	1022	14.7	133	19.94	86.4	13.6
	1112	14.7	133	31.65	70.8	29.2
Cr ₂ O ₃ ·5%NaCl	842	14.7	133	9.75	90.6	9.4
	932	14.7	133	9.65	86.0	14.0
	1022	14.7	133	28.16	88.5	11.5
	1112	14.7	133	37.98	82.6	17.4
Cr ₂ O ₃ ·5%KCl	842	14.7	133	10.09	91.2	8.8
	932	14.7	133	19.34	93.8	6.2
	1022	14.7	133	26.01	77.7	22.3
	1112	14.7	133	47.15	78.8	21.2
Cr ₂ O ₃ ·5%MgCl ₂	842	14.7	133	9.10	80.0	20.0
	932	14.7	133	8.77	85.2	14.8
	1022	14.7	133	12.21	82.1	17.9
	1112	14.7	133	23.81	80.1	19.9
Cr ₂ O ₃ ·5%BaCl ₂	842	14.7	133	19.58	87.8	12.2
	932	14.7	133	23.69	90.0	10.0
	1022	14.7	133	28.55	84.8	15.2
	1112	14.7	133	47.63	78.4	21.6

Table 3

Catalyst	Temperature of	Pressure psia.	O ₂ %	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀ Charged %	Conversion of isoC ₄ H ₁₀ Reacted	
						Dehydro- genation %	Cracking %
Cr ₂ O ₃	842	3.7	15	66	13.0	91.5	8.5
	1022	3.7	15	66	14.0	87.7	12.3
	1112	3.7	15	66	24.4	84.1	15.9
Cr ₂ O ₃ ·5%BaCl ₂	842	3.7	15	66	10.86	91.2	8.8
	932	3.7	15	66	23.55	92.6	7.4
	1022	3.7	15	66	25.96	90.4	9.6
	1112	3.7	15	66	27.50	89.7	10.3
Cr ₂ O ₃ ·5%BaCl ₂	842	14.7	-	133	19.56	87.8	12.2
	932	14.7	-	133	23.69	90.0	10.0
	1022	14.7	-	133	28.58	84.8	15.2
	1112	14.7	-	133	47.63	78.4	21.6
Cr ₂ O ₃ ·20%BaCl ₂	842	14.7	-	133	9.95	83.4	16.6
	1022	14.7	-	133	14.97	81.5	18.5
	1112	14.7	-	133	22.71	74.7	25.3
Cr ₂ O ₃ ·5%Ba(OH) ₂	842	14.7	-	133	14.67	92.5	7.5
	932	14.7	-	133	18.37	83.6	16.4
	1022	14.7	-	133	17.25	86.5	13.5
	1112	14.7	-	133	25.86	83.1	16.9

Table 4

Temperature °F	Pressure psia.	Residence Time (seconds) (cold gas)	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀ Charged	Conversion of isoC ₄ H ₁₀ Reacted	
				%	Dehydro- genation %	Cracking %
842	11.0	9	66	15.20	91.0	9.0
932	11.0	9	66	28.32	90.6	9.8
1022	11.0	9	66	29.70	86.6	14.4
1112	11.0	9	66	33.20	72.0	28.0
842	11.0	4.5	133	15.10	93.7	6.3
932	11.0	4.5	133	23.40	93.6	6.6
1022	11.0	4.5	133	31.80	85.6	14.4
1112	11.0	4.5	133	34.80	76.4	23.6
842	11.0	2.25	266	14.22	89.4	10.6
932	11.0	2.25	266	25.30	87.8	12.2
1022	11.0	2.25	266	22.90	87.9	12.1
1112	11.0	2.25	266	27.80	89.4	10.6
842	11.0	1.12	532	10.81	82.0	18.0
932	11.0	1.12	532	17.64	89.0	11.0
1022	11.0	1.12	532	18.32	91.6	8.4
1112	11.0	1.12	532	19.67	83.6	16.6
842	11.0	0.73	833	6.38	89.9	10.1
932	11.0	0.73	833	6.04	93.7	6.3
1022	11.0	0.73	833	7.76	82.1	17.9
1112	11.0	1.08	550	16.80	76.2	23.8
842	7.4	5.95	66	20.85	82.6	17.4
932	7.4	5.95	66	23.20	82.7	17.3
1022	7.4	5.95	66	27.31	81.7	18.3
1112	7.4	5.95	66	42.02	78.2	21.8
842	3.7	3.0	66	17.05	91.2	8.8
932	3.7	3.0	66	17.70	93.8	6.2
1022	3.7	3.0	66	15.50	92.4	7.6
1112	3.7	3.0	66	24.35	86.4	13.6

Table 6

Temperature °F	Pressure psia.	O ₂ %	Residence Time (seconds) (cold gas)	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀ Charged	Conversion of isoC ₄ H ₁₀ Reacted	
					%	Dehydro- genation %	Crackin %
842	11.0	5.0	8.2	66	16.72	96.5	3.5
932	11.0	5.0	8.2	66	20.96	89.4	10.6
1022	11.0	5.0	8.2	66	22.29	87.2	12.8
1112	11.0	5.0	8.2	66	30.75	80.0	20.0
842	11.0	15.0	6.5	66	5.35	76.4	24.6
932	11.0	15.0	6.5	66	23.7	82.7	17.3
1022	11.0	15.0	6.5	66	31.95	79.5	20.6
1112	11.0	15.0	6.5	66	33.40	76.0	24.0
842	11.0	15.0	3.2	133	13.54	94.5	5.5
932	11.0	15.0	3.2	133	21.52	90.2	9.8
1022	11.0	15.0	3.2	133	23.11	84.6	15.4
1112	11.0	15.0	3.2	133	31.46	87.1	12.9
842	11.0	15.0	1.6	266	9.07	82.8	17.2
932	11.0	15.0	1.6	266	14.90	88.0	12.0
1022	11.0	15.0	1.6	266	18.16	82.5	17.5
1112	11.0	15.0	1.6	266	27.13	67.8	32.2
842	7.4	15.0	4.3	66	18.9	87.9	12.1
932	7.4	15.0	4.3	66	19.6	86.8	13.2
977	7.4	15.0	4.3	66	20.2	81.9	18.1
1022	7.4	15.0	4.3	66	22.65	81.6	18.5
1112	7.4	15.0	4.3	66	39.80	80.1	19.9
842	3.7	15.0	2.15	66	10.86	91.2	8.8
932	3.7	15.0	2.15	66	23.55	92.6	7.4
1022	3.7	15.0	2.15	66	25.96	90.4	9.6
1112	3.7	15.0	2.15	66	27.50	89.7	10.3

Table 6

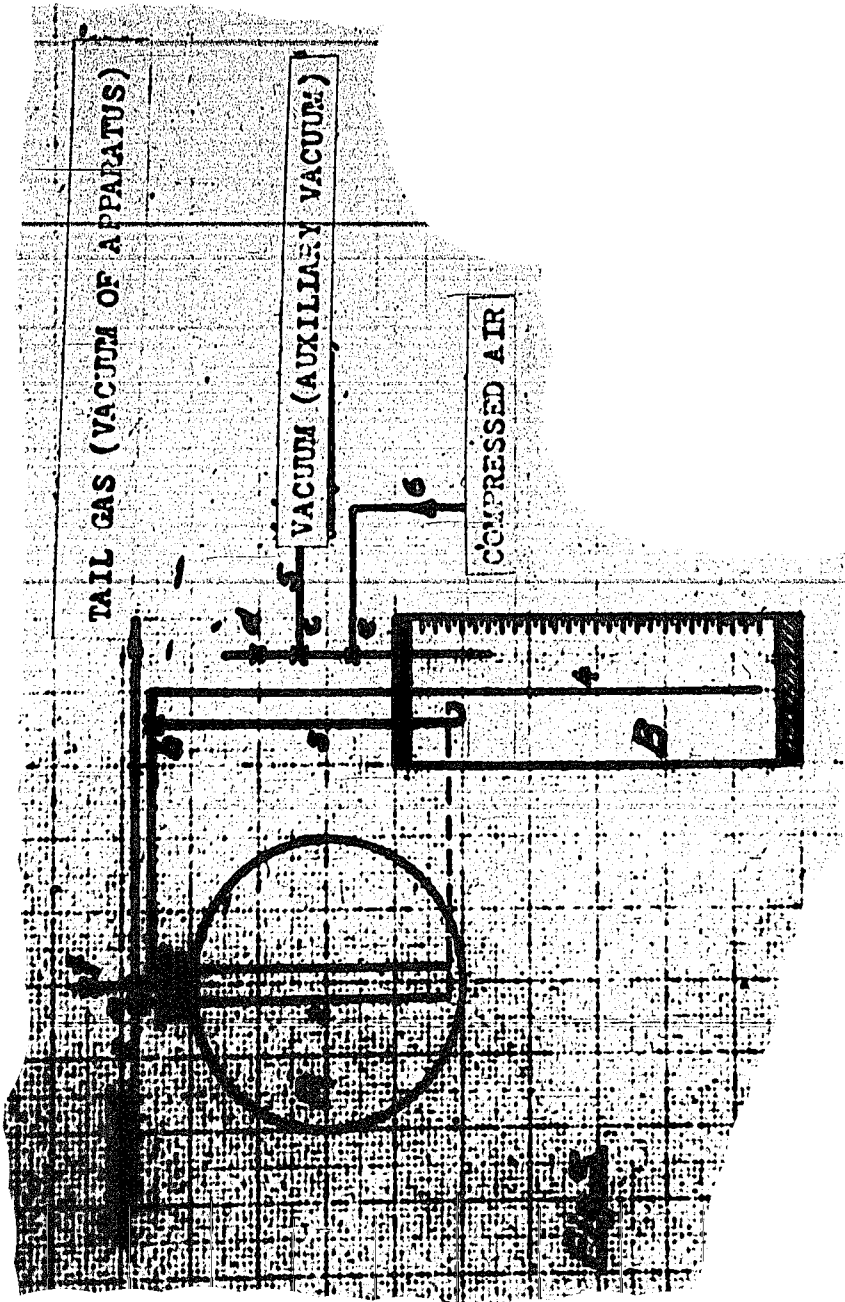
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					%	Dehydro- genation %	Cracking %
842	14.7	-	5.95	133	19.56	87.8	12.2
932	14.7	-	5.95	133	23.69	90.0	10.0
1022	14.7	-	5.95	133	28.58	84.8	15.2
1112	14.7	-	5.95	133	47.63	78.4	21.6
842	14.7	-	3.0	266	11.75	90.0	10.0
932	14.7	-	3.0	266	22.24	89.4	10.6
1022	14.7	-	3.0	266	20.28	87.5	12.5
1112	14.7	-	3.0	266	35.44	78.8	21.2
842	14.7	10	5.35	133	17.48	88.3	11.7
932	14.7	10	5.35	133	18.83	90.9	9.1
1022	14.7	10	5.35	133	24.53	80.6	19.4
1112	14.7	10	5.35	133	43.88	75.9	24.1
842	14.7	15	5.15	133	12.71	90.5	9.5
932	14.7	15	5.15	133	14.09	90.8	9.2
1022	14.7	15	5.15	133	24.60	84.0	16.0
1112	14.7	15	5.15	133	42.90	77.5	22.4

Table 7

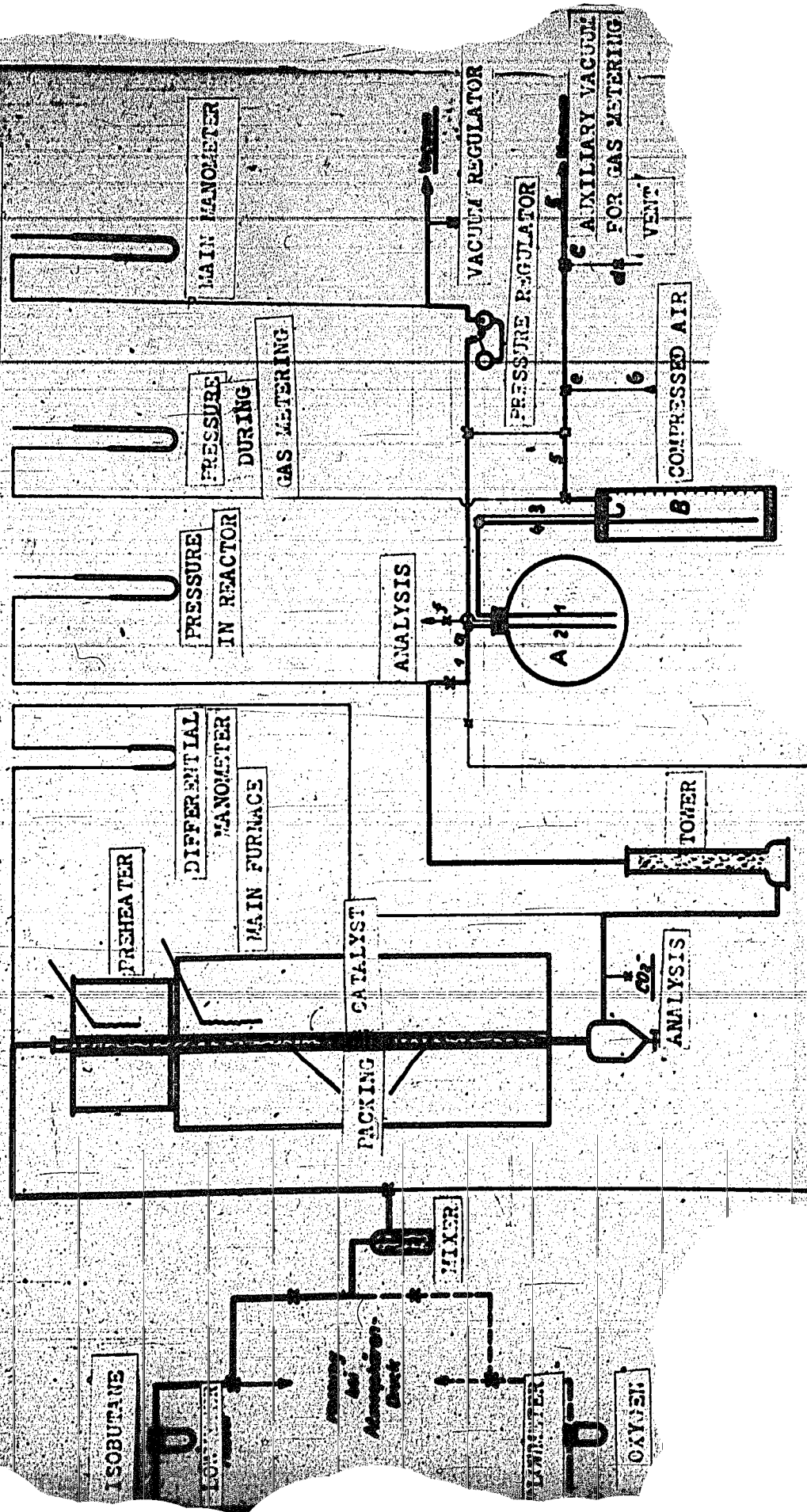
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					%	Dehydro- genation %	Cracking %
842	14.7	-	5.95	133	19.56	87.8	12.2
932	14.7	-	5.95	133	23.69	90.0	10.0
1022	14.7	-	5.95	133	28.58	84.8	15.2
1112	14.7	-	5.95	133	47.63	78.4	21.6
842	14.7	10	5.35	133	17.48	88.8	11.7
932	14.7	10	5.35	133	18.83	90.9	9.1
1022	14.7	10	5.35	133	24.53	80.6	19.4
1112	14.7	10	5.35	133	43.88	75.9	24.1
842	7.4	-	5.95	66	20.85	82.6	17.4
932	7.4	-	5.95	66	23.20	82.7	17.3
1022	7.4	-	5.95	66	27.31	81.7	18.3
1112	7.4	-	5.95	66	42.02	78.2	21.8
842	11.0	15	6.5	66	5.35	75.4	24.6
932	11.0	15	6.5	66	23.70	82.7	17.3
1022	11.0	15	6.5	66	31.95	79.5	20.5
1112	11.0	15	6.5	66	33.40	76.0	24.0

Table 8

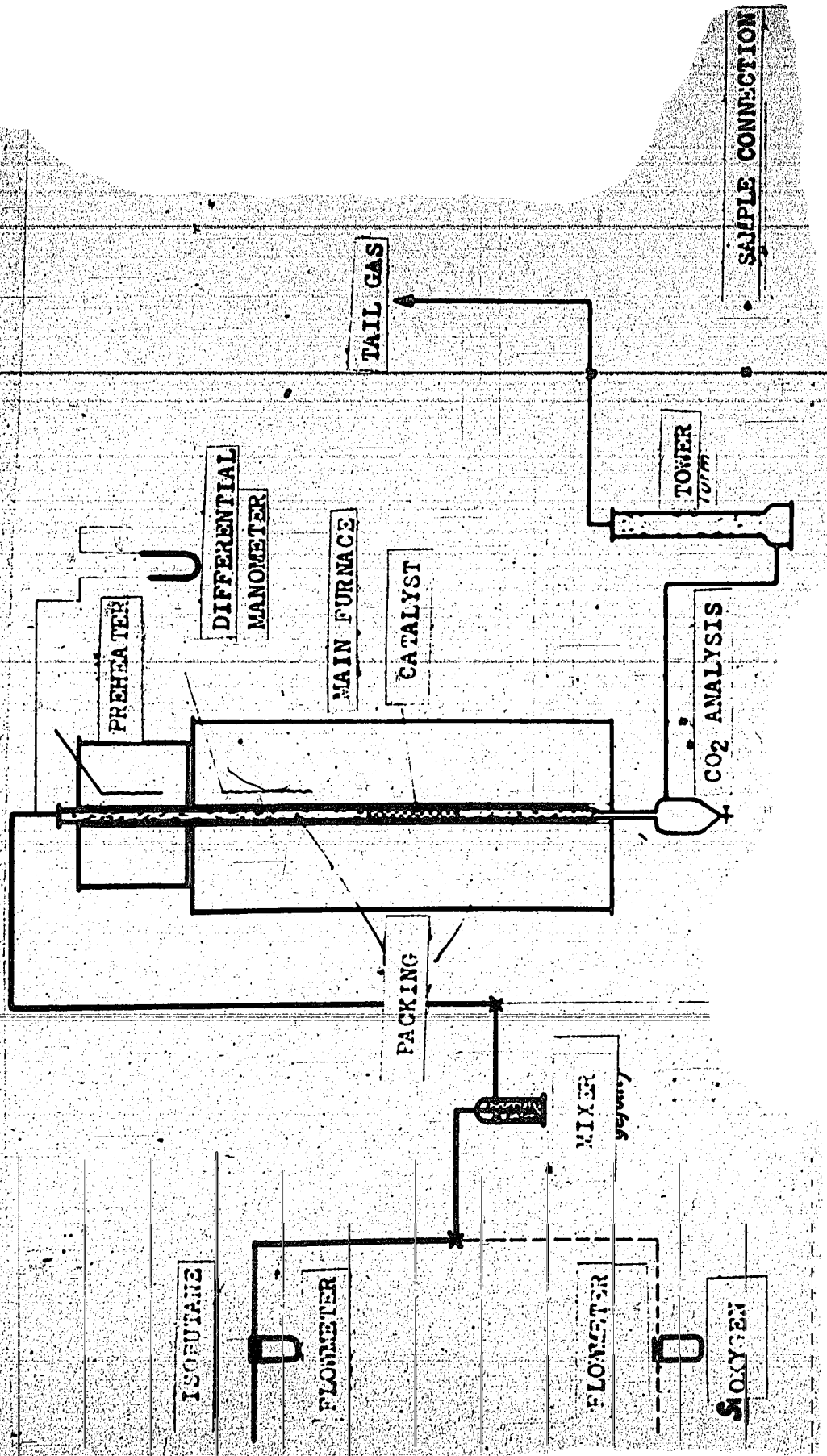
Temperature Of	Pressure psia.	O ₂ %	Residence Time (seconds) (cold gas)	Space velocity vol. isoC ₄ H ₁₀ / vol. catalyst/ hour	Total Conversion of isoC ₄ H ₁₀ Charged %	Conversion of isoC ₄ H ₁₀ Reacted	
						Dehydro- genation %	Cracking %
842	14.7	-	3.0	266	11.75	90.0	10.0
932	14.7	-	3.0	266	22.24	89.4	10.6
1022	14.7	-	3.0	266	20.28	87.5	12.5
1112	14.7	-	3.0	266	35.44	78.8	21.2
842	3.7	-	3.0	66	17.05	91.2	8.8
932	3.7	-	3.0	66	17.7	93.8	6.2
1022	3.7	-	3.0	66	15.5	92.4	7.6
1112	3.7	-	3.0	66	24.35	86.4	13.6
842	11.0	15	3.2	133	13.64	94.5	5.5
932	11.0	15	3.2	133	21.52	90.2	9.8
1022	11.0	15	3.2	133	23.11	84.6	15.4
1112	11.0	15	3.2	133	31.46	87.1	12.9



APPARATUS FOR ISOBUTANE DEHYDROGENATION AT REDUCED PRESSURE WITH AND WITHOUT O₂

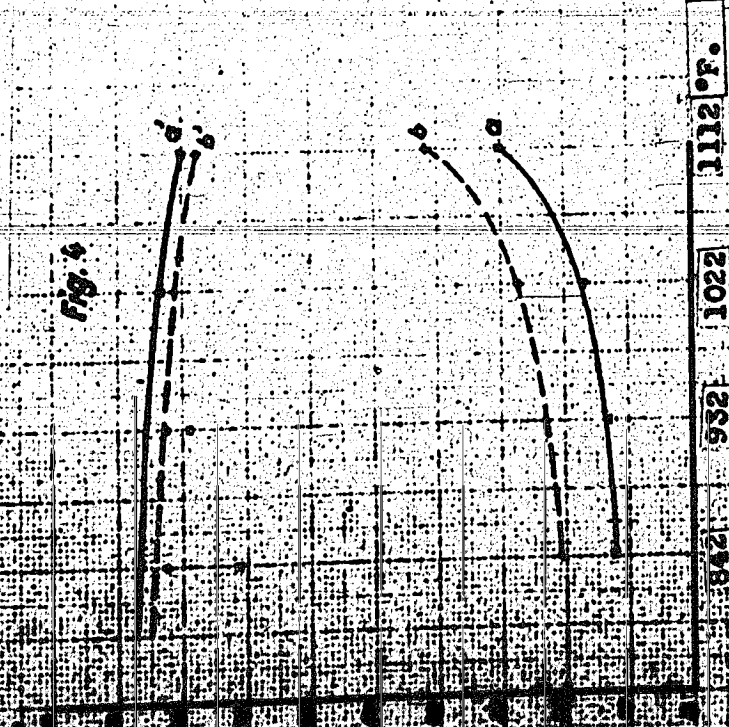


APPARATUS FOR ISO-BUTANE DEHYDROGENATION AT ATMOSPHERIC PRESSURE WITH AND WITHOUT O₂



EXPERIMENTS WITH VARIOUS CATALYSTS

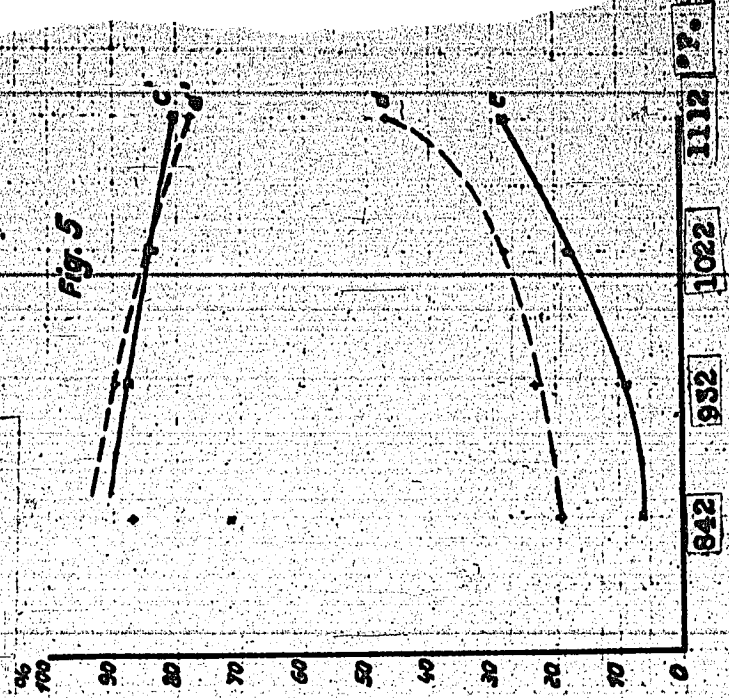
Fig. 6



CATALYST 2 W.S.M.S.
 C₂O₃ 5% CH₂Cl₂

a, b, c, d = CONVERSION, % ISO-C₄H₁₀ CHANGED
 a', b', c', d' = YIELD ISO-C₄H₁₀ (2 ISO-C₄H₁₀ CONVERTED)

Fig. 5



CATALYST 3 M.S. MoS₃
 C₂O₃ 5% CH₂Cl₂

a, b, c, d = CONVERSION, % ISO-C₄H₁₀ CHANGED
 a', b', c', d' = YIELD ISO-C₄H₁₀ (2 ISO-C₄H₁₀ CONVERTED)

EXPERIMENTS WITH DIFFERENT CHROMIUM OXIDE CATALYSTS

Fig 1

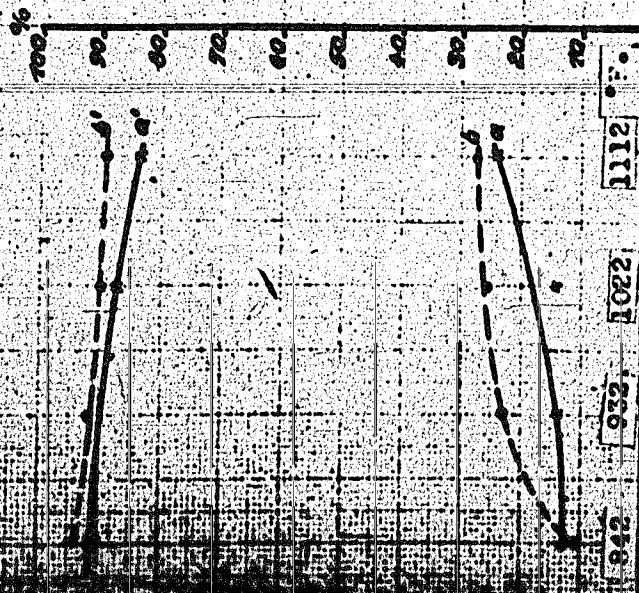
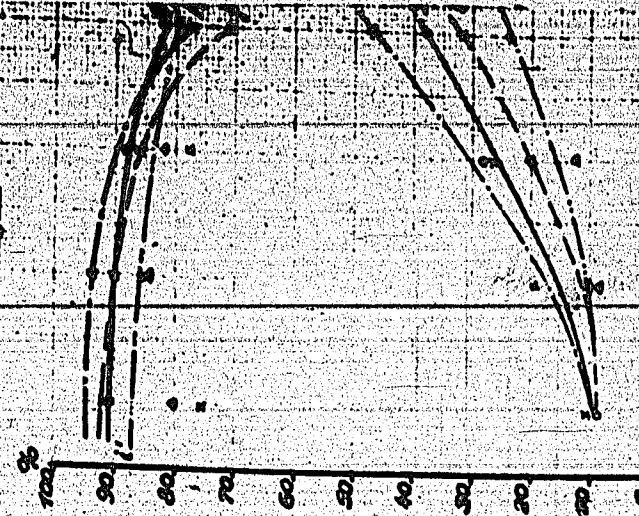


Fig 2



Fig 3



CURVE	PRESSURE (PSI)		VOL. ISO-C ₄ H ₁₀ (VOL./HOUR)		CATALYST
	932	1022	932	1022	
a	3.7	60	60	60	Cr ₂ O ₃
b	3.7	60	60	60	Cr ₂ O ₃ 5% SnO ₂

932 1022 1112 842

Cr₂O₃: 5% SnO₂
 d - Cr₂O₃: 20% SnO₂
 e - Cr₂O₃: 5% SnO₂ (ZnO)
 f - Cr₂O₃: 5% ZnO
 g - Cr₂O₃: 5% Al₂O₃
 h - Cr₂O₃: 5% Al₂O₃
 i - Cr₂O₃: 5% Al₂O₃

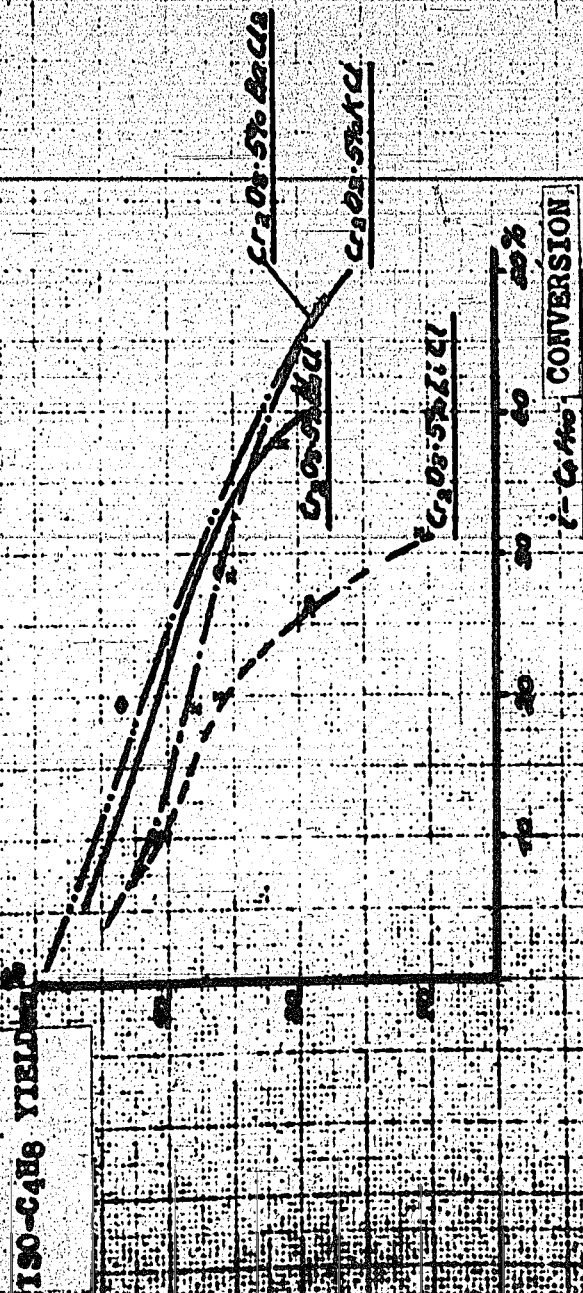
842 932 1022 1112

AT 133 VOL. ISO-C₄H₁₀/VOL./HOUR
 1497 PSI

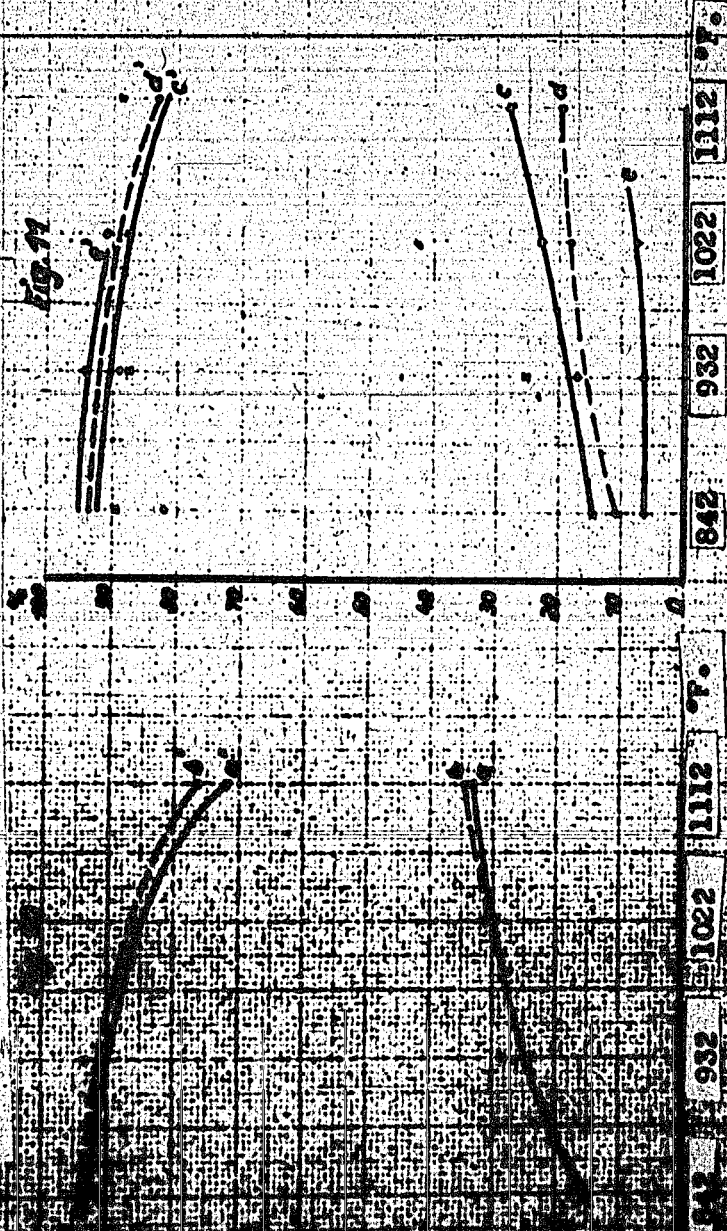
g - i CONVERSION (% OF ISO-C₄H₁₀ CHARGED)
 g - i YIELD (% OF ISO-C₄H₁₀ REACTED)

EXPERIMENTS WITH DIFFERENT CHROMIUM OXIDE CATALYSTS
 RELATION OF $i\text{-C}_4\text{H}_6$ YIELD ON $i\text{-C}_4\text{H}_6$ CONVERSION

Fig. 2



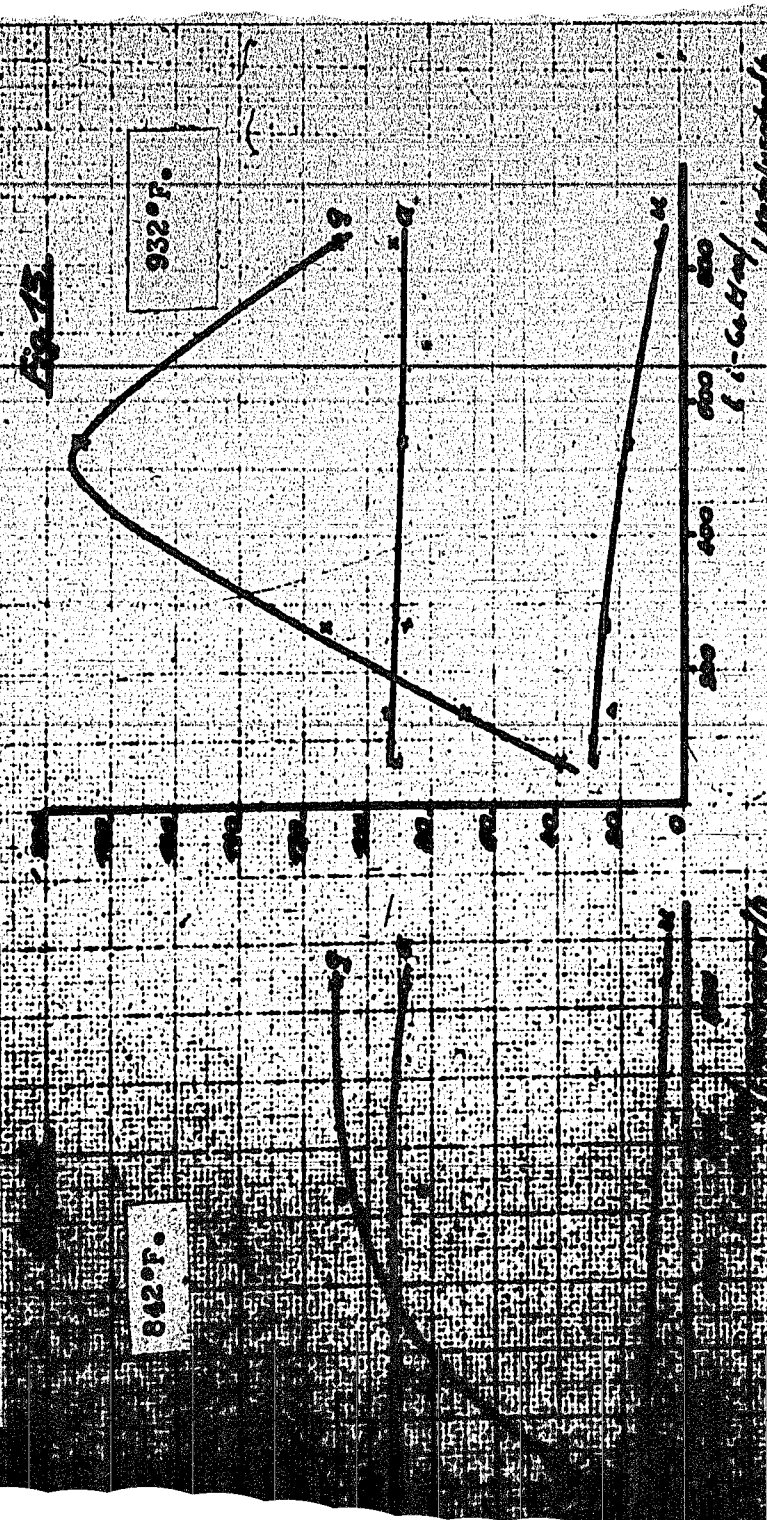
EXPERIMENTS AT REDUCED PRESSURE WITHOUT THE ADDITION OF OXYGEN
(11 psi. pressure)



180-C₂H₅O CHARGED 1111-044/CATALYST/HOUR

Temp. °F.	1022	932	842
1112	•	•	•
1022	•	•	•
932	•	•	•
842	•	•	•

EXPERIMENTS AT REDUCED PRESSURE WITHOUT THE ADDITION OF OXYGEN
 (11 psi. pressure)



21 - CONVERSION, % ISO-C₄H₁₀ CHARGED

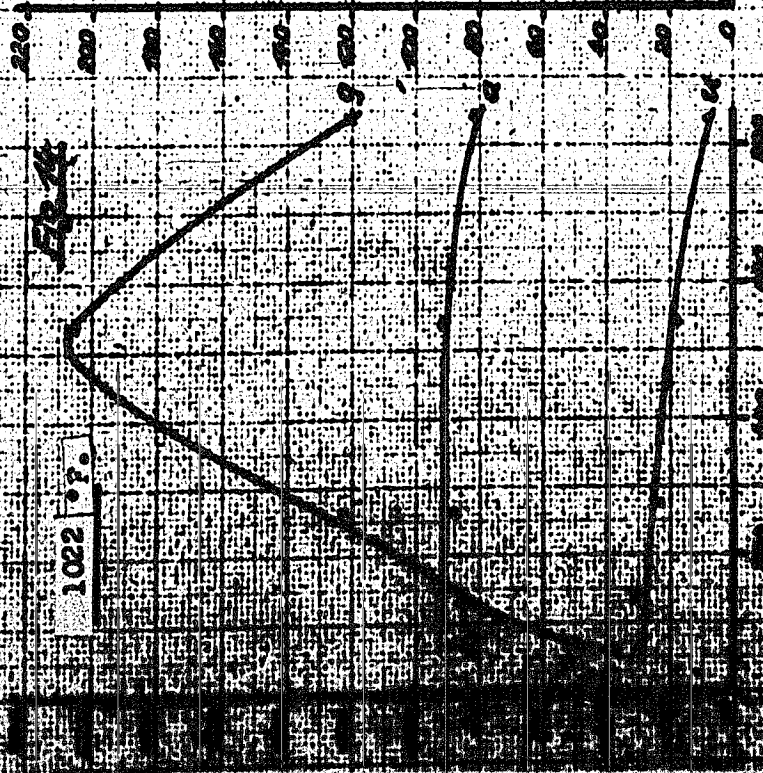
22 - YIELD OF ISO-C₄H₈, % ISO-C₄H₁₀ CONVERTED

23 - GRAMS ISO-C₄H₈/LITER OF CATALYST/HOUR

EXPERIMENTS AT REDUCED PRESSURE WITHOUT THE ADDITION OF OXYGEN
(11 psi. pressure)

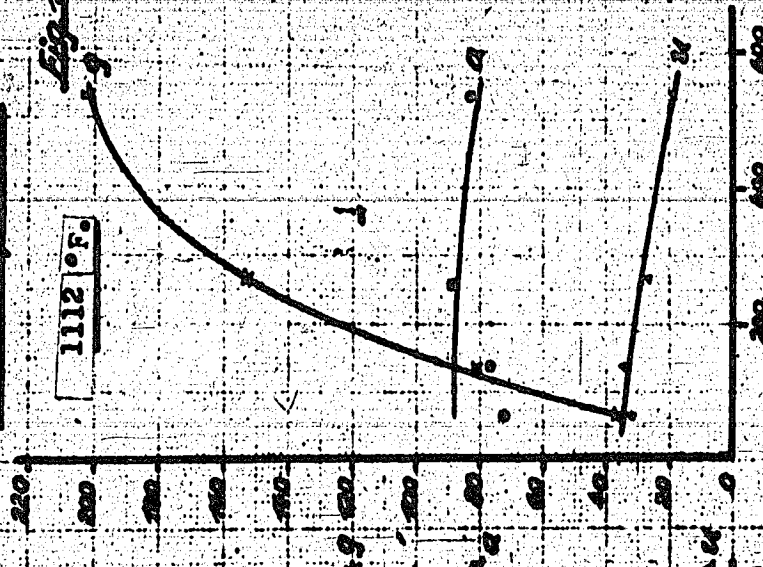
1022 °F.

Fig. 14



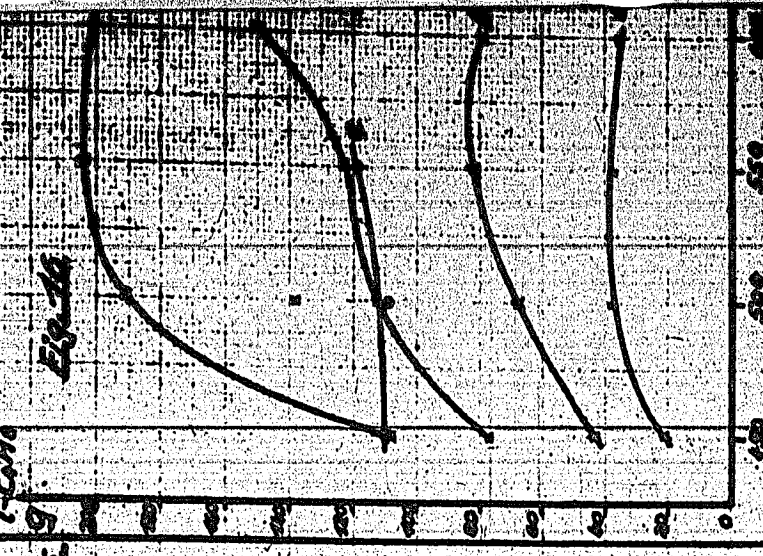
1112 °F.

Fig. 15



1400 °F.

Fig. 16



VOL. ISO-C₄H₁₀/VOL. CATALYST/HOUR

CONVERSION, % OF ISO-C₄H₁₀ CHARGED

YIELD OF ISO-C₄H₁₀, % OF ISO-C₄H₁₀ CONVERTED

YIELD OF ISO-C₄H₁₀/LITER OF CATALYST/HOUR

Common 2-Catalyst/Run/h

a - 600 L. Catalyst/h

b - 100 L. Catalyst/h

c - 200 L. Catalyst/h

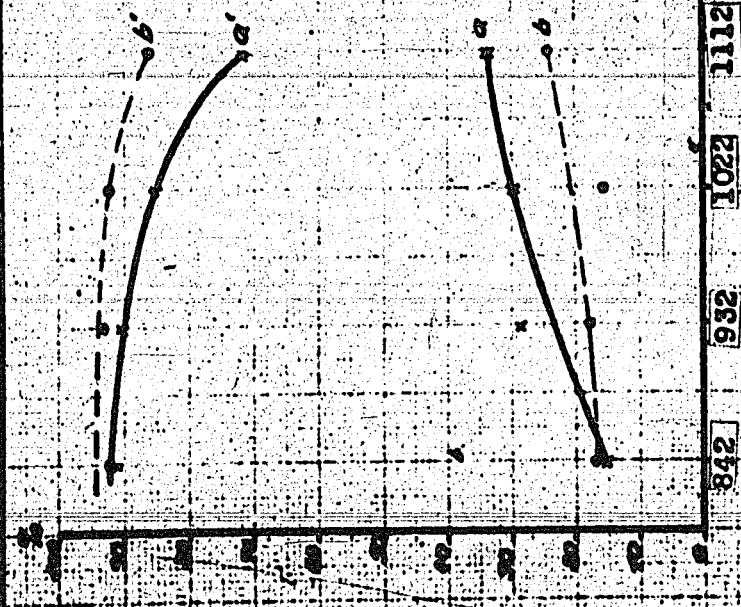
d - 500 L. Catalyst/h

e - 1000 L. Catalyst/h

10000355

EXPERIMENTS AT REDUCED PRESSURE WITHOUT OXYGEN

Fig. 17

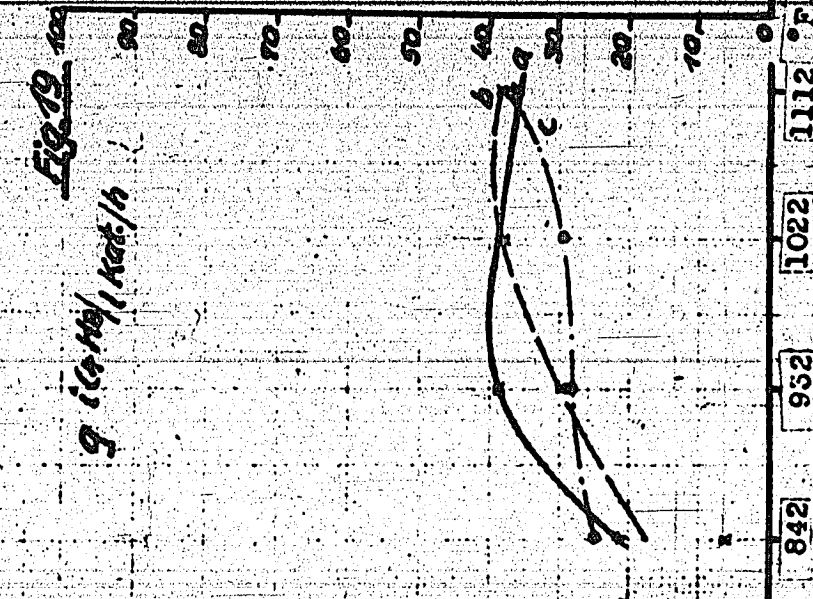
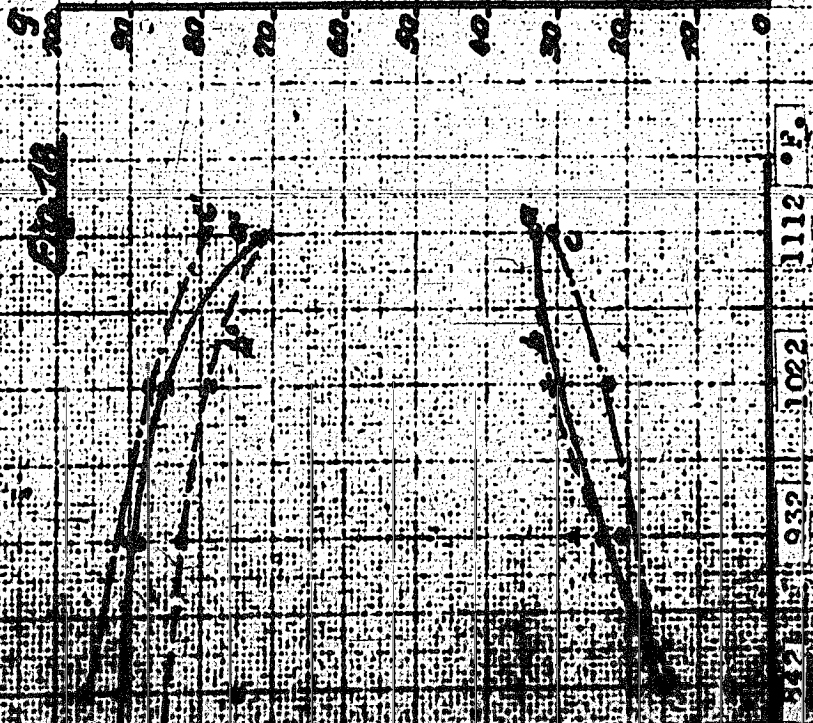


a) 11 psi } 66 VOL. ISO-C₄H₁₀/VOL. CATALYST/HOUR
 b) 3.7 psi }

a - CONVERSION (% ISO-C₄H₁₀ CHARGED)
 b - YIELD OF C₄H₈ (% ISO-C₄H₁₀ REACTED)

C-770

EXPERIMENTS AT REDUCED PRESSURE WITH AND WITHOUT OXYGEN (11 PSI)

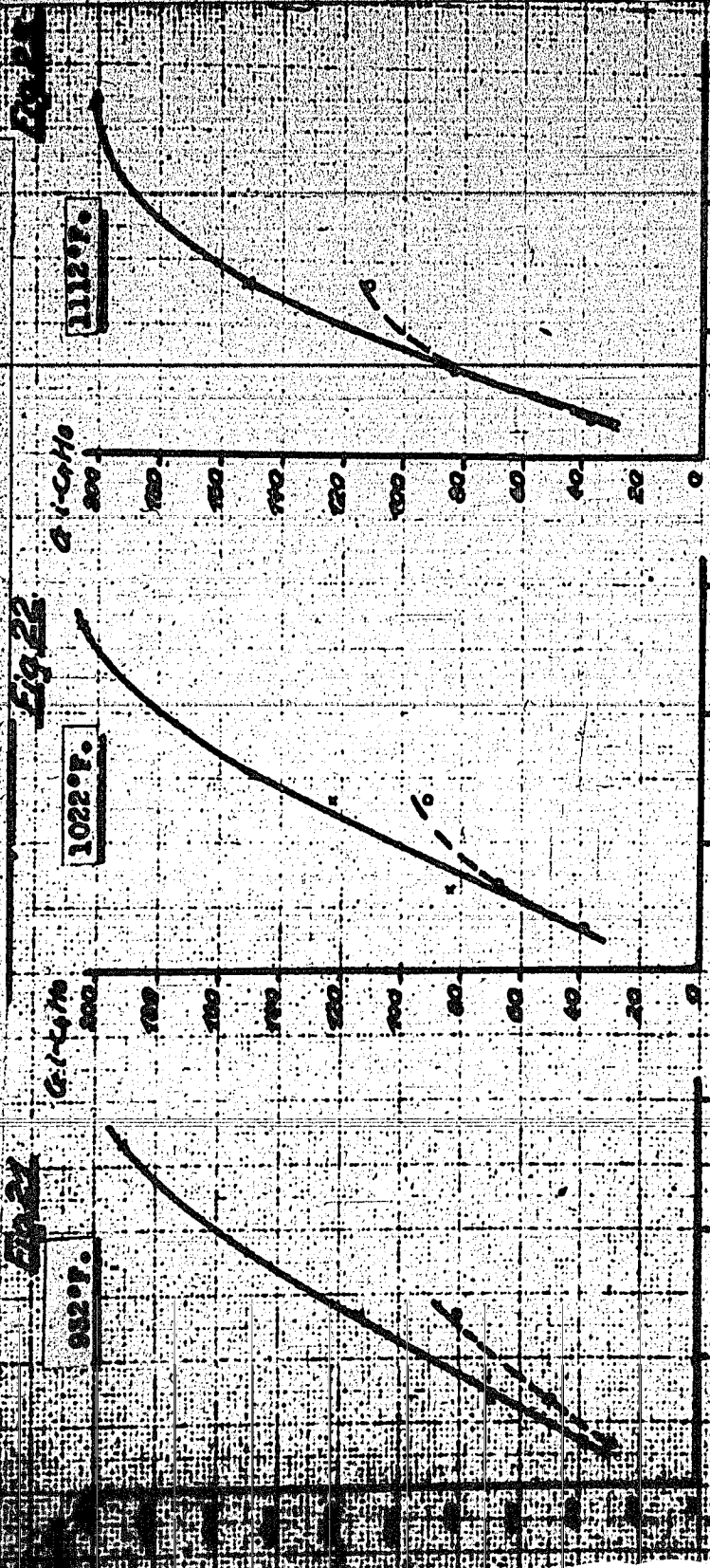


CONVERSION % ISO-C ₄ H ₁₀ CHARGED AT 66 VOL.	ISO-C ₄ H ₁₀ VOL./HOUR	WITHOUT C ₂	WITH 15% O ₂
66	"	"	"
66	"	"	"
133	"	"	"
133	"	"	"

CORRESPONDING ISO-C₄H₆ YIELDS (% OF ISO-C₄H₁₀ REACTED) (C₄H₆)

932 1022 1112 F. 842 932 1022 1112 F. 842 932 1022 F.

EXPERIMENTS AT REDUCED PRESSURE WITH AND WITHOUT THE ADDITION OF OXYGEN (11 psi. pressure)



ISOBUTYLENE YIELD

EXPTS. WITHOUT O₂

EXPTS. WITH 15% O₂

EXPERIMENTS AT ATMOSPHERIC PRESSURE WITH AND WITHOUT O₂

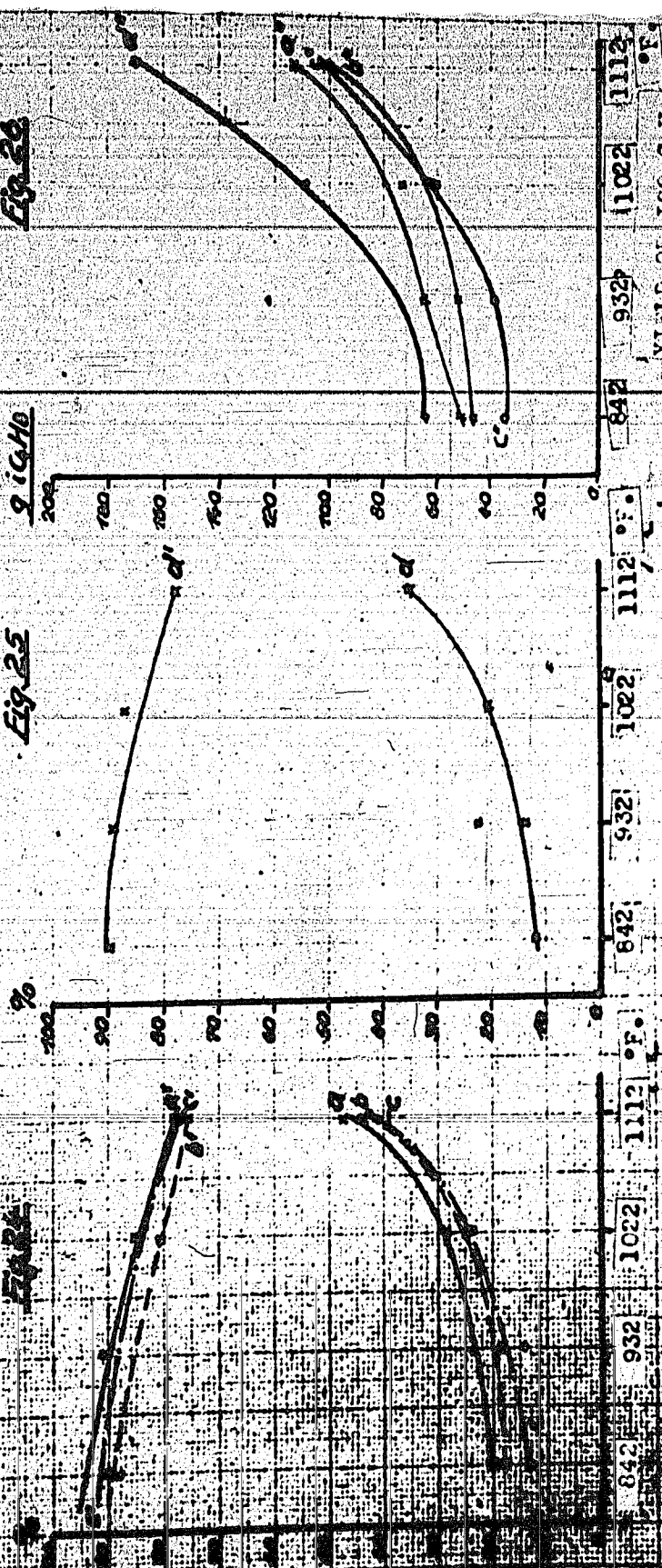
Vergleichen

Ammoniakwerk Merseburg
 Gruba, bei der Eisenhütte Merseburg
 Louis-Weber (Königsteiner)

Fig. 24

Fig. 25

Fig. 26



TEMPERATURE °F.	CONVERSION AT 15% VOL. ISO-C ₄ H ₁₀ /VOL. HOUR WITHOUT O ₂	CONVERSION AT 15% VOL. ISO-C ₄ H ₁₀ /VOL. HOUR WITH 10% O ₂	CONVERSION AT 15% VOL. ISO-C ₄ H ₁₀ /VOL. HOUR WITH 15% O ₂
842	"	"	"
932	"	"	"
1022	"	"	"
1112	"	"	"

TEMPERATURE °F.	YIELD OF ISO-C ₄ H ₈	GRAMS ISO-C ₄ H ₈ /LITER OF CATALYST/FOUR
842	"	"
932	"	"
1022	"	"
1112	"	"

Katalysatoraktivität

EXPERIMENTS WITH RESIDENCE TIMES OF 5.36-6.5 SEC.

(7024)

Fig. 21

g.i.-C₄H₁₀

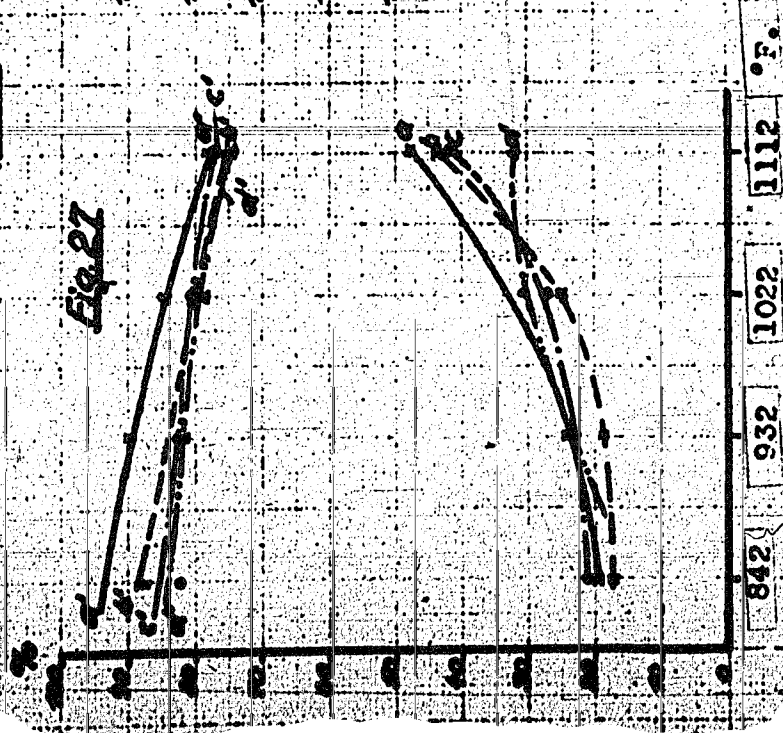
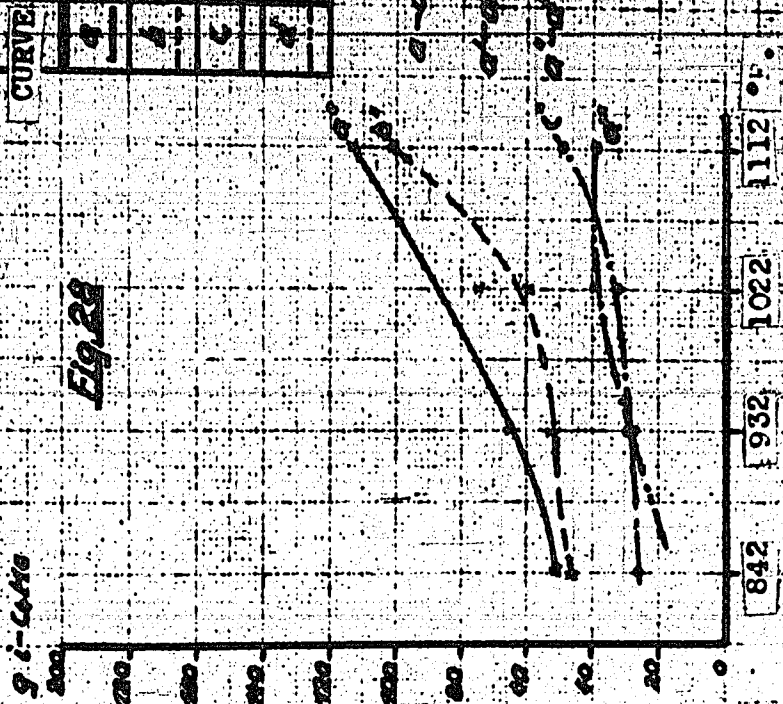


Fig. 22



CURVE	YIELD OF ISO-C ₄ H ₁₀ CHARGED	YIELD OF ISO-C ₄ H ₁₀ REACTED
a	14.7	14.7
b	14.7	14.7
c	7.8	7.8
d	11.0	11.0

41000000

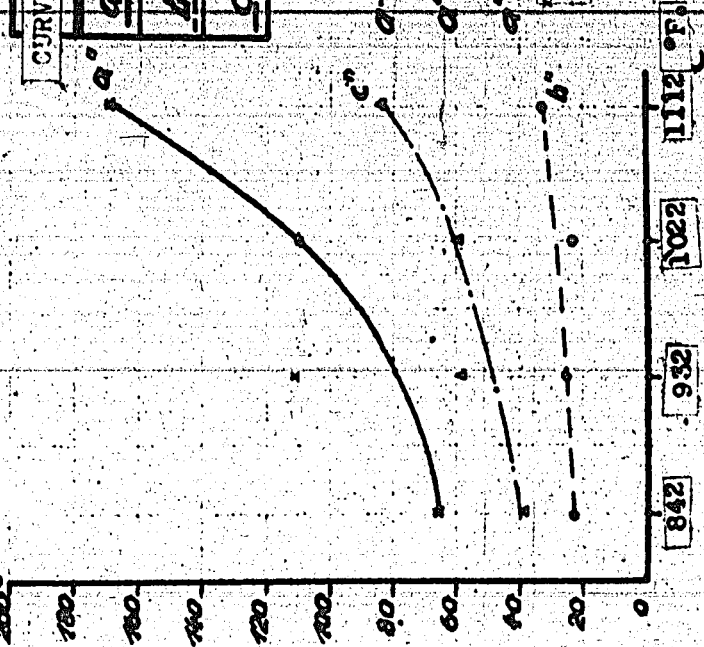
EXPERIMENTS WITH RESIDENCE TIMES OF 3.0 - 3.2 SEC.

(Table 5)

Fig. 29

2009 C₄H₈

Fig. 30



CURVE	4 PSI	133
a'	14.7	—
b'	3.7	—
c'	11.0	45

66	66
—	—
—	—
—	—

GRAMS ISO-C₄H₈/LITER OF CATALYST/HOUR

Fig. 29

