HYDROGENATION OF COTTONSEED OIL WITH RANEY NICKEL AS CATALYST

by

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TABLE OF CONTENTS

PAGE

Summary 1

Introduction 3

Presentation of Theory and Factors Effecting the Course of Hydrogenation 4

Material and Equipment 6

Experimental Procedure 7

Figure 1 - - - Schematic Drawing of Hydrogenator . . . 8

Results 10

Figure 2 - - - Effect of Catalyst Concentration on Hydrogenation Rate - Series A 11

Figure 3 - - - Effect of Catalyst Concentration on Hydrogenation Rate - Series B 12

Figure 4 - - - Effect of Catalyst Concentration on Hydrogenation Rate - Series C 13

Figure 5 - - - Effect of Catalyst Concentration on Hydrogenation Rate - Series D 14

Table I - - - Calculated Reaction Rates at Different Time Intervals for Each Run 15

Discussion of Results 17

Conclusion and Recommendation 19

Bibliography 20

Appendix 21

Table II -- Original Data - Run 1A 22

Table III -- Original Data - Run 2A 24

Table IV -- Original Data - Run 3A 25

11 DE47 8 1.50

TABLE OF CONTENTS (CONTINUED)

PAGE

Table V - - Original Data - Run 1B 26

Table VI - Original Data - Run 2B 27

Table VII - Original Data - Run 3B 28

Table VIII- Original Data - Run 1C 29

Table IX - -Original Data - Run 2C 30

Table X - - Original Data - Run 3C 31

Table XI - Original Data - Run 1D 32

Table XII - Original Data - Run 2D 33

Table XIII- Original Data - Run 3D 34

Sample Calculations 35

Table XIV - Hydrogen Pressure Gage Calibration Data 37

Figure 6 - Calibration Curve for Hydrogen Pressure Gage 38

Approval Sheet 39

HYDROGENATION OF COTTONSEED OIL WITH RANEY NICKEL AS CATALYST

Summary

Studies were made of the hydrogenation rates of cottonseed oil with Raney nickel as catalyst. The hydrogenation apparatus was located in the West Laboratory of Estabrook Hall. The hydrogenation was carried out under the following conditions:

Series A -- Temperature - 200 °F, Hydrogen Pressure - 265 lbs. per sq. in. (Abs.), Catalyst Concentrations - 0.087, 0.175, 0.350 per cent by weight.

Series B -- Temperature - 300 °F, Hydrogen Pressure - 265 lbs. per sq. in. (Abs.), Catalyst Concentrations - 0.043, 0.087, 0.175 per cent by weight.

Series C -- Temperature - 300 °F, Hydrogen Pressure - 165 lbs. per sq. in. (Abs.), Catalyst Concentrations - 0.175, 0.350, 0.700 per cent by weight.

Series D -- Temperature - 300 °F, Hydrogen Pressure - 65 lbs. per sq. in. (Abs.), Catalyst Concentrations - 0.350, 0.500, 0.700 per cent by weight.

It was found that the hydrogenation rate varied directly as the catalyst concentration for all the series. In Series C, when the catalyst concentration was 0.175 per cent by weight, the hydrogenation occurred as a monomolecular reaction as

indicated by a constant reaction rate. In Series A, B, and D, the reaction approached a monomolecular reaction, but the reaction rates decreased as the process proceeded. This decreasing reaction rate may have been caused by slow poisoning of the catalyst by carbon monoxide as a test for carbon monoxide poisoning indicated this to be occurring. For the test runs in Series C where the catalyst concentrations were 0.350 and 0.700 per cent by weight, the reaction rates were found to increase with time. This deviation was explained by Bailey (1) where the hydrogenation rate is dependent upon the rate of hydrogen dissolving in the oil for high catalyst concentration at ordinary pressures.

It is recommended that further studies be made of hydrogenation of cottonseed oil under similar conditions with nickel catalyst that contain "promoter" metals. Also a study should be made of the effects of agitation upon hydrogenation rates.

HYDROGENATION OF COTTONSEED OIL WITH RANEY NICKEL AS CATALYST

Introduction

It is difficult to exaggerate the importance of the hydrogenation process in modern oil technology. It is employed on a vast scale in both the soap and edible fat industries, for converting liquid oils to hard or plastic fats, for converting soft fats to firmer products and for improving the resistance of fats and oils to deterioration through exidation or flavor reversion.

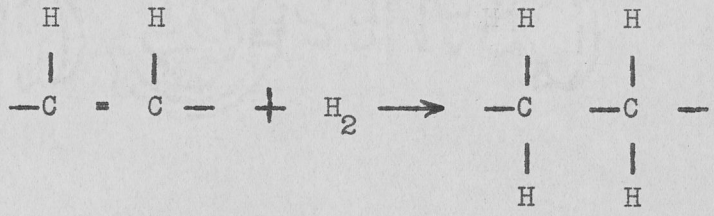
Although the hydrogenation of fatty oils was the first catalylic hydrogenation process to be adapted to commercial operation, and is yet by far the most important of such processes, there is singularly little known concerning its technology outside a relatively small group within the industry. Of the literature on catalytic hydrogenation, only a small portion is devoted to hydrogenation as applied to fats and oils, and most of this is so confused and contradictory as to be virtually worthless without benefit of expert and critical commentary.

Results of previous work on this subject were investigated in Bailey (1) and Groggins (3), but the remarks from these sources were general application and no specific results could be ascertained. In these studies, it was desired to determine the hydrogenation characteristics of cottonseed oil at different

temperatures and pressures, and using different amounts of Ramey nickel as catalyst.

Presentation of Theory and Factors Effecting the Course of Hydrogenation

Hydrogenation of an oil consists in the direct addition of hydrogen at double bonds in the fatty acid chains, according to the following scheme:



Thus, the degree of hydrogenation in an oil is directly related to the iodine value, the addition of one mol of hydrogen corresponding to the absorption of one mol of iodine. With this relationship, the degree of unsaturation of oils is classified by their iodine value.

The catalyst employed in commercial hydrogenation invariably consists basically of nickel, although minor amounts of copper, alumina, and several other metals which have catalytic properties may be incorporated with the nickel for their "promoter" action. The greatest amount of hydrogenation is carried out with powder catalysts composed of the metal or metals in a finely divided form, prepared by special methods. The catalyst is suspended in the oil during the operation of hydrogenation, and at the conclusion is removed by filtration.

For hydrogenation to take place, it is necessary to bring together at a suitable temperature gaseous hydrogen, liquid oil, and the solid catalyst. In ordinary practice it may be assummed that the hydrogen is first caused to dissolve in the oil, and the hydrogen-laden oil is then brought into contact with the catalyst by mechanical means. Agitation of the catalyst-oil mixture serves the double purpose of promoting solution of hydrogen in the oil, and continously renewing the oil at the catalyst surface. The reaction takes place upon the surface of the nickel catalyst, and the reaction is thought to proceed according to the following scheme as given by Bailey (1):

Catalyst + reactants $\underset{\text{desorption}}{\overset{\text{adsorption}}{\rightleftharpoons}}$ catalyst - reactants complex $\rightarrow$ reaction products + regenerated catalyst

Catalytic hydrogenation, like other chemical reactions, is accelerated by an increase in temperature. However, the effect of temperature on the reaction rate is somewhat less than in ordinary chemical reactions in a homogenous system, and is also variable. Over the ordinary range of hydrogenation temperatures, increasing the temperature has a relatively greater effect upon the conversion of linoleic acid to oleic acid than upon the conversion of oleic to stearic acid. Hence selectivity is enhanced by increasing the temperature.

Since hydrogen must first become dissolved in the oil before it can reach the catalyst-oil interface where hydrogenation occurs, any factor influencing the rate of solution will have

a corresponding effect on the rate of hydrogenation. The factors specifically affecting rate of solution are pressure of hydrogen and agitation of the catalyst-oil charge.

Under most conditions of hydrogenation, the velocity of the reaction is more or less proportional to the concentration of the catalyst. It is possible, however, to use so much catalyst that the speed of hydrogenation will be governed entirely by the rate at which hydrogen can dissolve in the oil. Under such conditions the time required to reduce the oil to a specific iodine value may not vary with considerable variation in the catalyst concentration.

Material and Equipment

The oil used in this test was Armour's Star Salad Oil, obtained from Armour and Co., Knoxville, Tennessee. It was classified as pure, refined cottonseed oil. The hydrogen used was purchased in regular hydrogen cylinders from National Cylinder Gas Co., Knoxville, Tennessee. Raney nickel was used as the catalyst and was obtained from the Lookout Oil and Refining Co., Chattanooga, Tennessee. The catalyst was in powdered form, immersed in water to prevent loss of activity.

The complete hydrogenation apparatus was obtained from the American Instrument Co., Silver Springs, Maryland. This apparatus included the reaction vessel (No.-D9774), the shaking mechanism (No.406-O1DA), and a one-third horsepower motor

(General Electric-Model 5KC47AB802B). A gear box (No. B-13976), with a thirty to one ratio, was connected between the motor and shaking mechanism. The necessary heating was accomplished by electric coils around the reaction vessell and was controlled by a selector switch. Temperatures were taken with a potentiometer (Leeds and Northrop No - 242420). The thermocouple leads were copper and constantan.

A vacuum pump (No. 531) was used to pull a vacuum above the oil while the apparatus was being heated or cooled.

The hydrogenation apparatus was set up and operated in the West Laboratory of Estabrook Hall at the University of Tennessee.

Experimental Procedure

The oil-catalyst mixtures were prepared by using a volume of oil sufficient to give the desired catalyst concentrations with given weights of nickel catalyst, this volume being approximately 500 ml. The changes in oil density for temperature changes were considered for these calculations.

The oil-catalyst mixture was placed in the reaction vessel, which in turn was inserted into the heating jacket. The hydrogen supply was then connected to the reaction vessel. A vacuum was then pulled over the oil-catalyst mixture while the reaction was being heated. When the desired temperature was reached, hydrogen was bled into the reaction vessel until

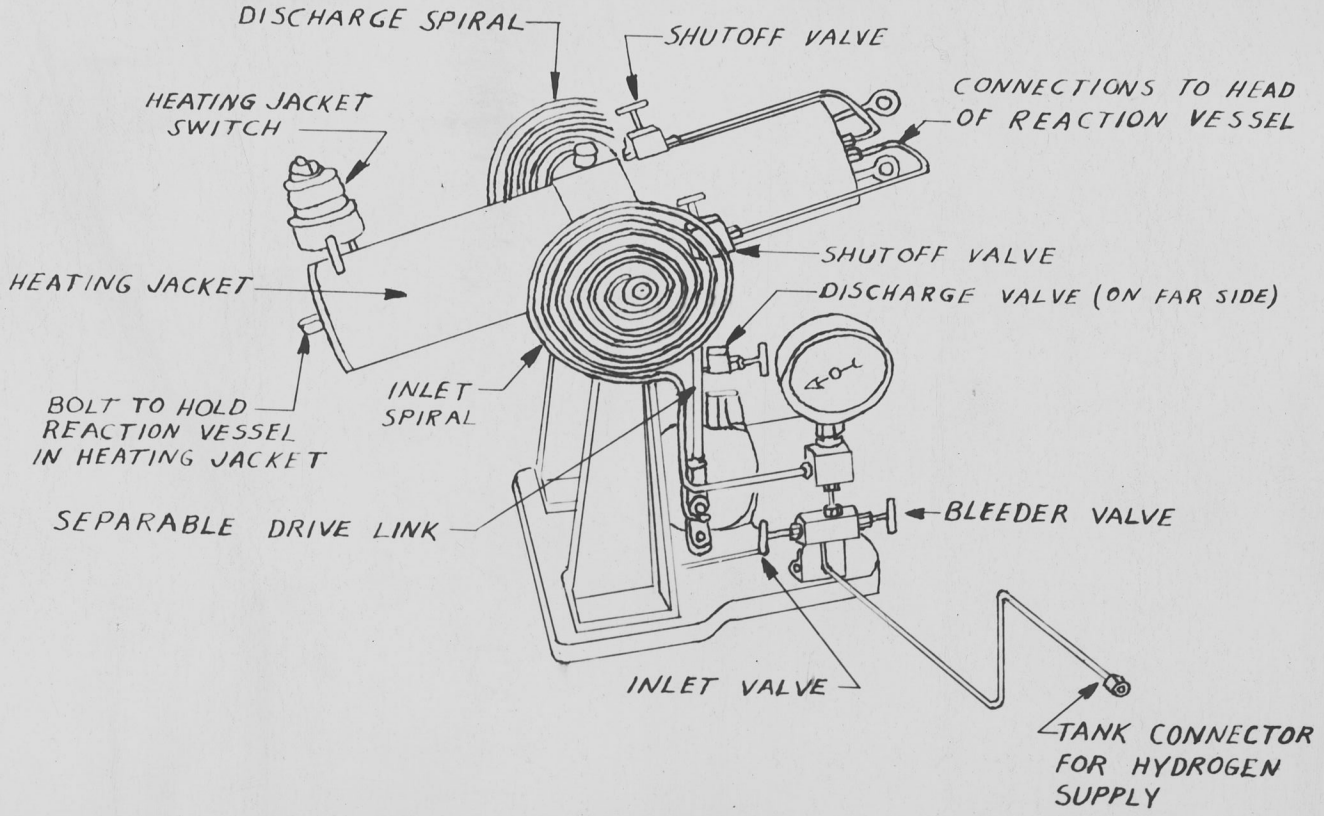


FIG. 1

SCHEMATIC DRAWING OF HYDROGENATOR

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desired pressure was obtained. At this point all tube connections were checked for hydrogen leaks. Agitation of reaction vessel was then started. Temperatures of the reaction mixture were recorded every fifteen minutes, and the hydrogen pressure was controlled by the inlet valve.

Hydrogenated oil samples were taken every sixty minutes during the runs of Series A, but for Series B, C, and D, samples were taken every forty-five minutes.

Hydrogenation runs were made under the following conditions:

Series A -- Temperature - 200 ^{o}F, Pressure - 265 lbs. per sq. in. (Abs.), Catalyst - varied from 0.087 per cent to 0.35 per cent by weight.

Series B -- Temperature - 300 ^{o}F, Pressure - 265 lbs. per sq. in. (Abs.), Catalyst - varied from 0.043 per cent to 0.175 per cent by weight.

Series C -- Temperature - 300 ^{o}F, Pressure - 165 lbs. per sq. in. (Abs.), Catalyst - varied from 0.175 per cent to 0.70 per cent by weight.

Series D -- Temperature - 300 ^{o}F, Pressure - 65 lbs. per sq. in. (Abs.), Catalyst - varied from 0.35 per cent to 0.700 per cent by weight.

The iodine value of the hydrogenated oil samples were determined with iodine monobromide solution. The preparation of this solution and the procedure used in determining the

iodine value of the oil were obtained from Mahin (4).

Curves were drawn for each run by plotting the logarithm of the iodine value against hydrogenation time. From these curves hydrogenation characteristics were determined for this test.

Results

The results of this test are shown graphically in Figures 2, 3, 4, and 5 for Series A, B, C, and D, respectively. These curves show the determined iodine value of the oil at the designated hydrogenation time. In Table I, the specific hydrogenation rates were calculated for the designated hydrogenation periods and from this table, the changing hydrogenation rates can be followed.

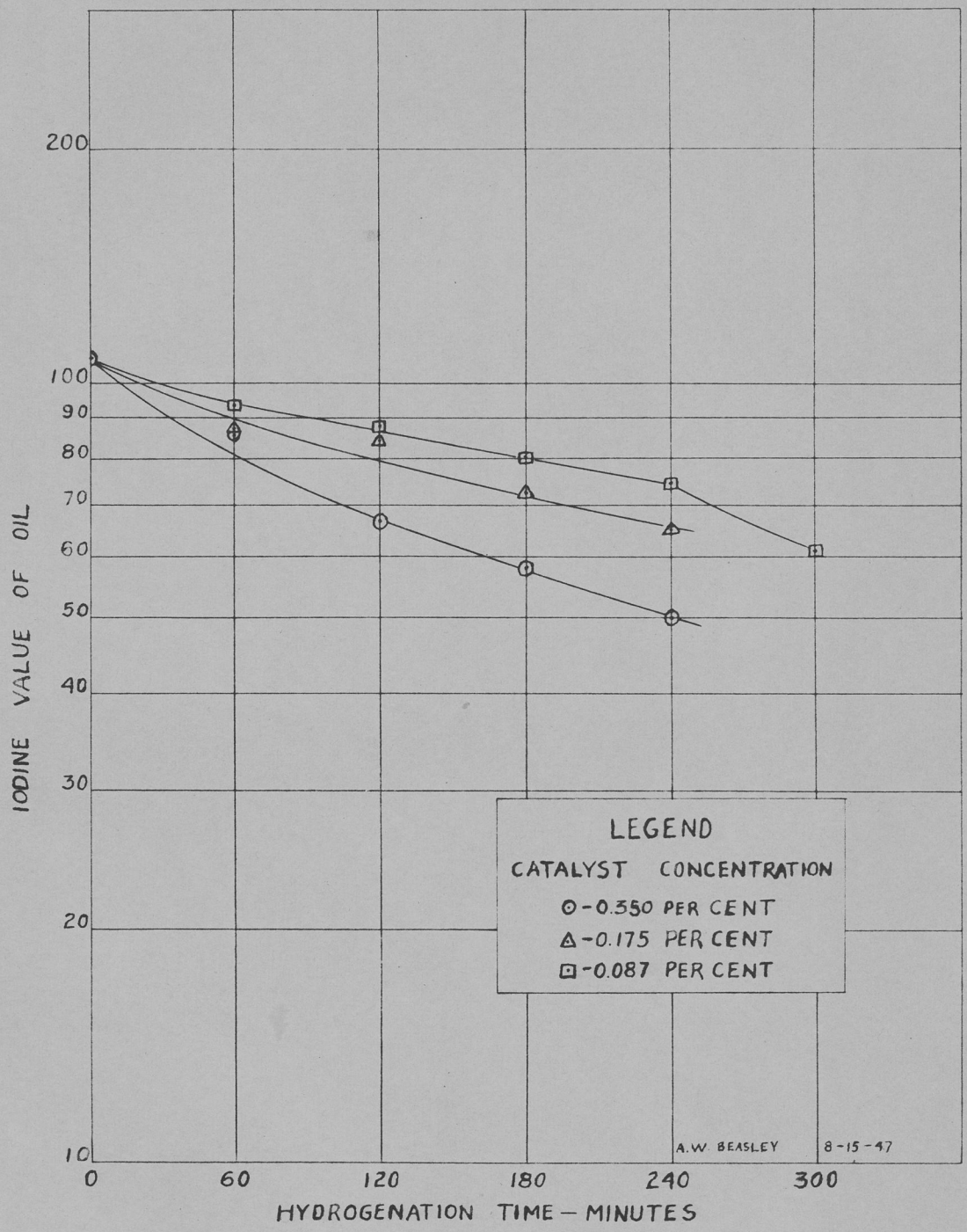

FIG. 2

EFFECT OF CATALYST CONCENTRATION ON HYDROGENATION RATE — SERIES-A

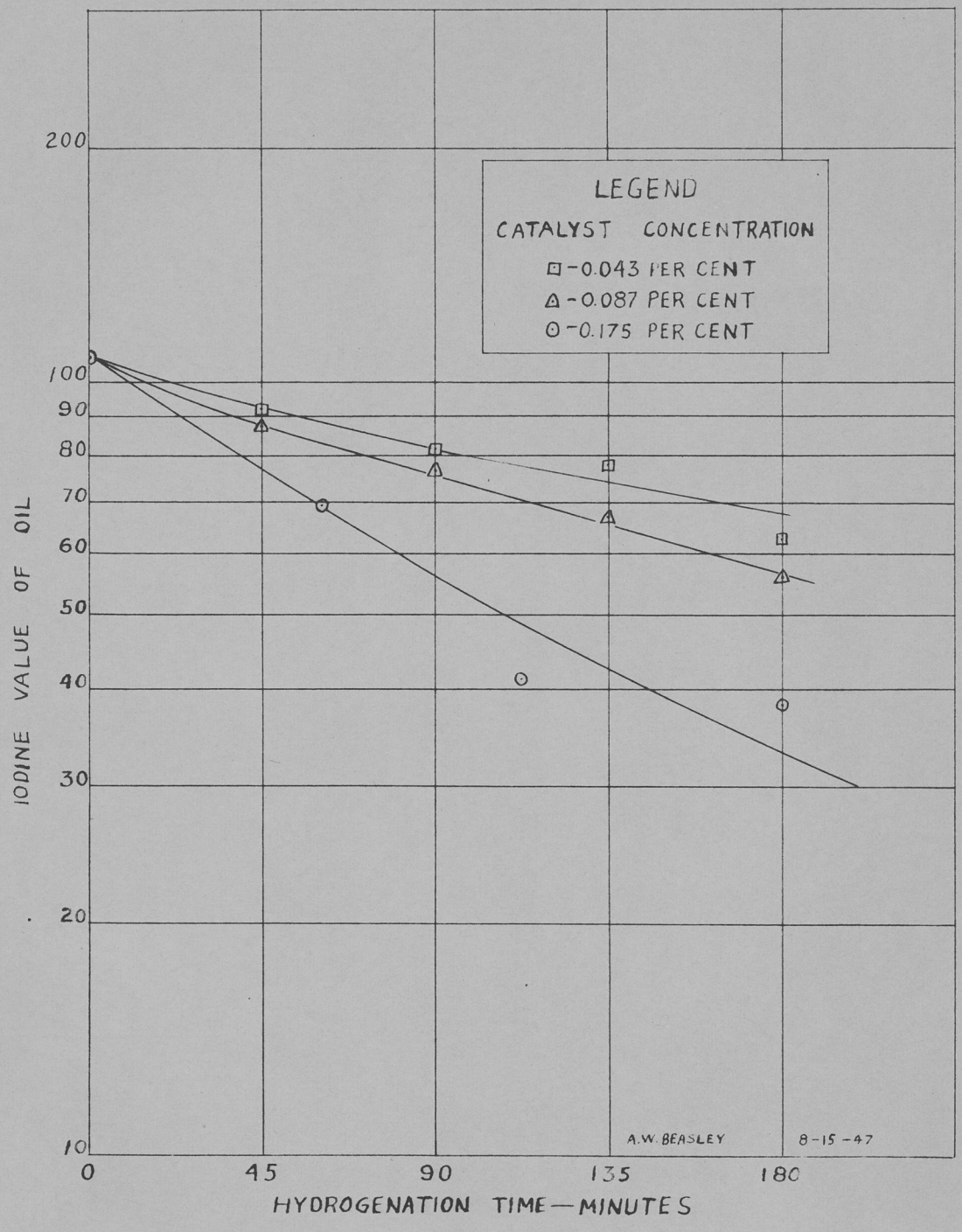

FIG. 3

EFFECT OF CATALYST CONCENTRATION ON HYDROGENATION RATE — SERIES-B

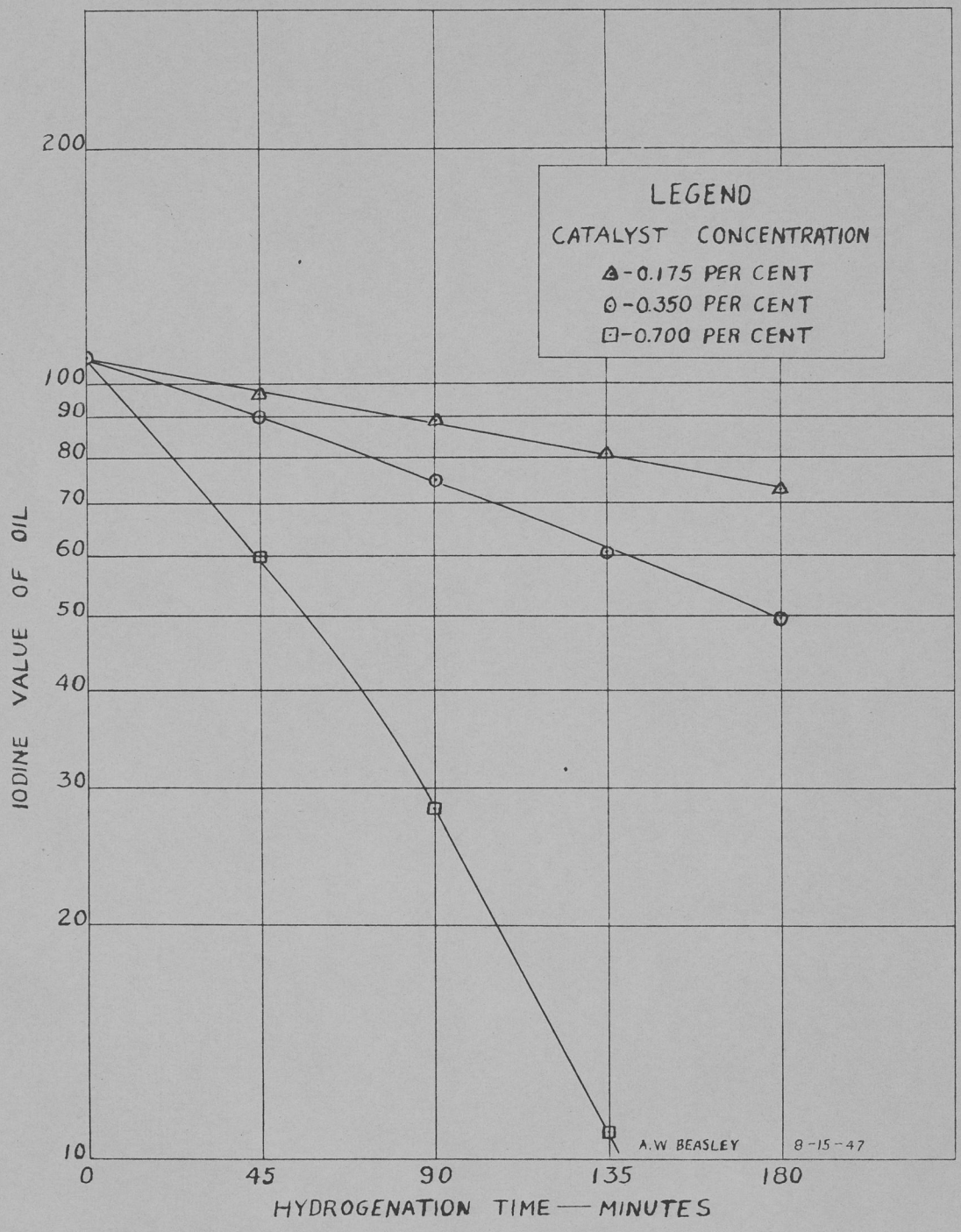

FIG. 4

EFFECTS OF CATALYST CONCENTRATION ON HYDROGENATION RATE — SERIES-C

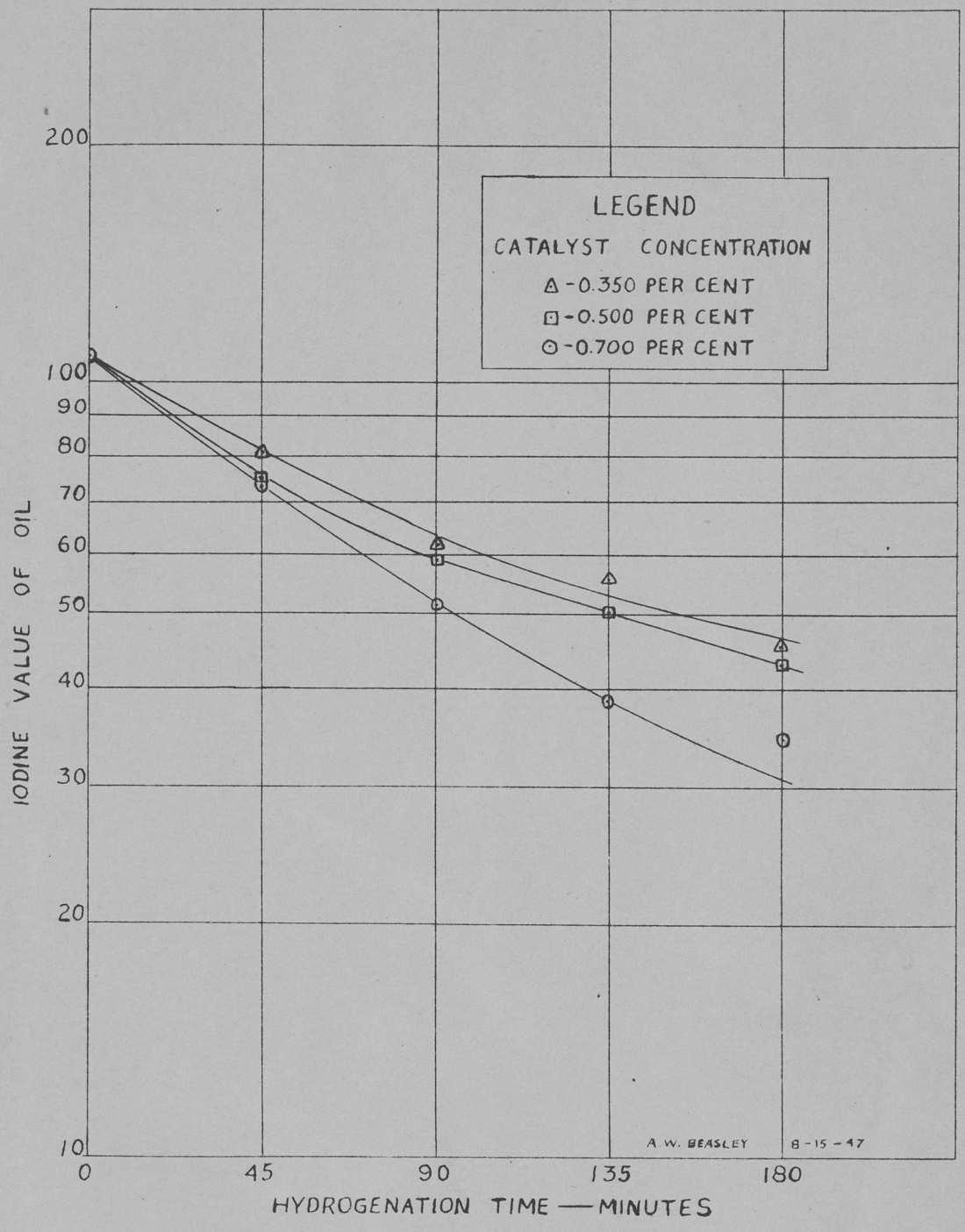

FIG. 5

EFFECT OF CATALYST CONCENTRATION ON HYDROGENATION RATE — SERIES-D

TABLE I

CALCULATED REACTION RATES AT

DIFFERENT TIME INTERVALS FOR EACH RUN

CATALYST CONCENTRATION % by weight	BEGINNING RATE $k \times 10^5$ sec^{-1}	RATE AFTER 45 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 90 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 135 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 180 MIN. $k \times 10^5$ sec^{-1}
		SERIES A			
0.087	4.11	2.76	2.34	2.06	1.82
0.175	4.11	3.59	3.59	3.39	2.98
0.350	7.84	6.56	5.29	3.85	3.53
		SERIES B			
0.043	6.40	4.56	4.04	3. 2	3. 2
0.087	9.90	6.03	6.03	6.03	6.03
0.175	13.05	11.78	10.87	9.87	9.00
		SERIES C			
0.175	3.59	3.59	3.59	3.59	3.59
0.350	5.56	7.18	7.21	7.46	8.05
0.700	16.80	27.90	25.5	28.90	-

TABLE I (CONTINUED)

CALCULATED REACTION RATES AT

DIFFERENT TIME INTERVALS FOR EACH RUN

CATALYST CONCENTRATION % by weight	BEGINNING RATE $k \times 10^5$ sec^{-1}	RATE AFTER 45 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 90 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 135 MIN. $k \times 10^5$ sec^{-1}	RATE AFTER 180 MIN. $k \times 10^5$ sec^{-1}
		SERIES D			
0.350	15.28	9.37	6.63	5.76	4.77
0.500	15.28	11.62	6.08	5.54	5.54
0.700	15.28	14.00	12.10	8.06	6.84

Discussion of Results

The results of this test indicate that the hydrogenation rate of cottonseed oil varies directly with the catalyst concentration, temperature, and hydrogen pressure. The increase in hydrogen pressure increased the solubility of the hydrogen gas in the oil.

The hydrogenation rate of cottonseed oil should result in a monomolecular reaction for the rate is dependent upon the degree of unsaturation of the oil. In only one test run was this found to be true. This occurred in Series C (Fig. 4) when the catalyst concentration was 0.175 per cent by weight. For the other two catalyst concentrations in Series C (Fig. 4), the hydrogenation rate increased as the process proceeded. This type of characteristic was explained by Bailey (1) to be due to the fact that the hydrogenation rate was determined by the rate of solution of hydrogen in the oil when the catalyst concentration is high. However, this did not occur in Series D, where the catalyst concentration was in the same range.

The decreasing hydrogenation rates may have resulted from slow poisoning of the catalyst. A test was made for carbon monoxide poisoning (Series A, catalyst concentration 0.087 per cent by weight) by agitating the oil-catalyst mixture under a vacuum for thirty minutes at 300 ^{o}F. The test indicated that the catalyst was being poisoned by carbon monoxide in the hydrogen gas.

There was some indication during the test that thorough agitation of the oil-catalyst mixture was not obtained. By drawing small portions of oil from the upper layer of the oil-catalyst mixture, a foamy oil was obtained. If a sample was taken from the lower portion of the oil-catalyst mixture, the oil was in liquid form with very small amounts of hydrogen in the oil in bubbles.

The specific reaction rates were calculated from the change in iodine value for a period of time since the iodine value conforms to the concentration of double bonds in the oil. These were calculated from the method described in Getman and Daniels (2).

Small errors may have been introduced in the determination of the iodine values of the oil samples due to small errors in weighing the oil samples, titration of the iodine monobromide solution, and slide rule errors.

Conclusion and Recommendations

It was found from these studies that the hydrogenation rate of cottonseed oil varies directly with the temperature, pressure, and catalyst concentration. No conclusions can be made from these studies concerning agitation, for in this test agitation was the same for all runs made.

It is recommended that further studies be made of the hydrogenation of cottonseed oil under similar conditions using different catalyst "promoters" with Raney nickel. A study of the effects of agitation should be made by using a variable speed motor to drive the agitation mechanism. Another study which can be made is the effect upon hydrogenation time which occurs by adding the amount of catalyst in portions over the hydrogenation run.

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APPENDIX

TABLE II

ORIGINAL DATA

RUN - 1A

HYDROGENATION CONDITION - AVERAGE TEMPERATURE - 201 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.) CALALYST, -
0.087 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN-PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
4:25	0	190	265	107.5
4:40	15	199	265	
4:55	30	206	265	
5:10	45	206	265	
5:25	60	202	265	94.0
10:10	60	203	265	94.0
10:25	75	205	265	
10:40	90	202	265	
10:55	105	199	265	
11:10	120	194	265	66.1
11:15	120	194	265	66.1
11:30	135	186	265	
12:00	165	192	265	
12:15	180	203	265	80.3
12:20	180	203	265	80.3
12:35	195	208	265	

TABLE II (CONTINUED)

ORIGINAL DATA

RUN - 1A

HYDROGENATION CONDITION - AVERAGE TEMPERATURE - 201 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.) CALALYST, - 0.087 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN-PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
12:35	195	208	265	
12:50	210	211	265	
1:05	225	209	265	
1:20	240	205	265	74.0
3:00	240	215	265	74.0
3:15	255	205	265	
3:30	270	195	265	
3:45	285	200	265	
4:00	300	210	265	60.7

TABLE III

ORIGINAL DATA

RUN 2A

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 204 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.175 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
4:00	0	200	265	107.5
4:20	20	198	265	
4:35	35	207	265	
4:50	50	209	265	
5:00	60	208	265	87.4
10:30	60	196	265	87.4
10:45	75	200	265	
11:00	90	204	265	
11:15	105	204	265	
11:30	120	200	265	66.2
11:33	120	200	265	66.2
11:48	135	190	265	
12:03	150	192	265	
12:18	165	200	265	
12:33	180	210	265	58.0
12:35	180	210	265	58.0
12:50	195	220	265	
1:05	210	220	265	
1:20	225	205	265	
1:35	240	198	265	49.9

TABLE IV

ORIGINAL DATA

RUN 3A

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 203 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.350 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
11:50	0	200	265	107.5
12:05	15	206	265	
12:20	30	210	265	
12:35	45	209	265	
12:50	60	206	265	88.1
1:55	60	186	265	88.1
2:10	75	188	265	
2:25	90	200	265	
2:40	105	208	265	
2:55	120	210	265	85.1
3:00	120	208	265	85.1
3:15	135	204	265	
3:30	150	198	265	
3:45	165	192	265	
4:00	180	196	265	73.1
4:05	180	196	265	73.1
4:20	195	198	265	
4:35	210	212	265	
4:50	225	214	265	
5:05	240	214	265	47

TABLE V

ORIGINAL DATA

RUN 1B

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 302 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.043 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
3:45	0	290	265	107.5
4:00	15	286	265	
4:15	30	288	265	
4:30	45	296	265	92.05
4:31	45	296	265	92.05
4:45	59	308	265	
5:00	74	310	265	
5:16	90	306	265	82.2
3:35	90	296	265	82.2
3:50	105	294	265	
4:05	120	300	265	
4:20	135	309	265	78.2
4:21	135	309	265	78.2
4:35	149	314	265	
4:50	164	314	265	
5:06	180	312	265	62.3

TABLE VI

ORIGINAL DATA

RUN 2B

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 302 °F
HYDROGEN PRESSURE 265 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.087 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
3:10	0	288	265	107.5
3:30	20	294	265	
3:45	35	301	265	
3:55	45	303	265	88.1
3:56	45	303	265	88.1
4:10	59	310	265	
4:25	74	310	265	
4:41	90	306	265	77.0
4:42	90	306	265	77.0
4:55	103	299	265	
5:10	118	297	265	
5:27	135	300	265	67.3
5:28	135	300	265	67.3
5:40	147	305	265	
5:55	162	305	265	
6:13	180	304	265	56.2

TABLE VII

ORIGINAL DATA

RUN 3B

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 301 °F
HYDROGEN PRESSURE - 265 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.175 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
12:40	0	295	265	107.5
12:55	15	290	265	
1:20	40	296	265	
1:40	60	310	265	69.6
1:45	60	310	265	69.6
2:00	75	313	265	
2:30	105	316	265	
2:45	120	305	265	43.7
2:50	120	305	265	43.7
3:10	140	296	265	
3:30	160	288	265	
3:50	180	290	265	37.3
4:00	180	284	265	37.3
4:15	195	296	265	
4:30	210	303	265	
5:00	240	300	265	24.8

TABLE VIII

ORIGINAL DATA

RUN 1C

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 303 °F
HYDROGEN PRESSURE - 165 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.175 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
3:45	0	314	165	107.5
4:00	15	303	165	
4:15	30	303	165	
4:30	45	303	165	96.9
4:31	45	303	165	96.9
4:45	59	300	165	
5:00	74	296	165	
5:16	90	294	165	89.5
11:20	90	293	165	89.5
11:35	105	290	165	
11:50	120	298	165	
12:05	135	306	165	80.9
12:06	135	306	165	80.9
12:20	149	310	165	
12:35	164	316	165	
12:51	180	310	165	72.4

TABLE IX

ORIGINAL DATA

RUN 2C

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 305 °F
HYDROGEN PRESSURE - 165 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.350 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs./sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
4:35	0	294	165	107.5
4:50	15	290	165	
5:05	30	296	165	
5:20	45	306	165	90.45
2:10	45	296	165	90.45
2:25	60	290	165	
2:40	75	300	165	
2:55	90	310	165	74.8
2:56	90	310	165	74.8
3:10	104	316	165	
3:25	119	316	165	
3:41	135	310	165	60.8
3:42	135	310	165	60.8
3:55	148	300	165	
4:10	163	294	165	
4:27	180	296	165	49.8

TABLE X

ORIGINAL DATA

RUN 3C

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 303 °F
HYDROGEN PRESSURE - 165 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.700 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
4:34	0	292	165	107.5
4:53	19	292	165	
5:05	31	300	165	
5:19	45	308	165	60.2
10:30	45	292	165	60.2
10:45	60	290	165	
11:00	75	300	165	
11:15	90	314	165	28.4
11:23	90	314	165	28.4
11:38	105	318	165	
11:53	120	318	165	
12:08	135	314	165	10.9
12:13	135	314	165	10.9
12:30	152	306	165	
12:45	167	296	165	
12:58	180	298	165	5.76

TABLE XI

ORIGINAL DATA

RUN 1D

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 303 °F
HYDROGEN PRESSURE 65 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.350 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
1:50	0	304	65	107.5
2:05	15	296	65	
2:20	30	300	65	
2:35	45	306	65	80
2:36	45	306	65	80
2:51	60	312	65	
3:06	75	311	65	
3:21	90	308	65	61.6
3:23	90	308	65	61.6
3:38	105	306	65	
3:53	120	304	65	
4:08	135	300	65	55.6
4:09	135	300	65	55.6
4:24	150	298	65	
4:39	165	294	65	
4:54	180	292	65	45.9

TABLE XII

ORIGINAL DATA

RUN 2D

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 304 °F
HYDROGEN PRESSURE - 65 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.50 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
11:30	0	308	65	107.5
11:45	15	297	65	
12:00	30	305	65	
12:15	45	310	65	73.8
12:17	45	310	65	73.8
12:32	60	305	65	
12:47	75	308	65	
1:02	90	304	65	58.7
1:05	90	304	65	58.7
1:20	105	302	65	
1:35	120	298	65	
1:50	135	300	65	50.9
1:51	135	300	65	50.9
2:06	150	306	65	
2:21	165	307	65	
2:36	180	303	65	45.1

TABLE XIII

ORIGINAL DATA

RUN 3D

HYDROGENATION CONDITIONS - AVERAGE TEMPERATURE - 303 °F
HYDROGEN PRESSURE - 65 LBS. PER SQ. IN. (Abs.)
CATALYST - 0.70 PER CENT BY WEIGHT

TIME	HYDROGENATION TIME - MIN.	TEMPERATURE °F	HYDROGEN PRESSURE lbs/sq.in.(Abs.)	DETERMINED IODINE VALUE OF OIL AT TIME INDICATED
2:00	0	298	65	107.5
2:15	15	288	65	
2:30	30	298	65	
2:45	45	308	65	73.4
2:46	45	308	65	73.4
3:01	60	316	65	
3:16	75	318	65	
3:31	90	316	65	51.5
3:32	90	316	65	51.5
3:47	105	308	65	
4:02	120	300	65	
4:17	135	294	65	38.7
4:18	135	294	65	38.7
4:33	150	290	65	
4:48	165	297	65	
5:03	180	295	65	34.4

SAMPLE CALCULATIONS

Determination of Iodine Value of Oil

Basis of Calculation - Oil Before Hydrogenation

	I	II
Weight of Oil Sample	0.2115 gms.	0.2019 gms.
ml. of 0.1005 N $Na_2S_2O_3$ Solution for Titration	4.85 ml.	5.75 ml.
ml. of 0.1005 N $Na_2S_2O_3$ Solution for Titration of blanks	22.7	22.9

Calculation of Iodine Value:

ml. of $Na_2S_2O_3$ for blank - ml. of $Na_2S_2O_3$ for oil Sample = ml. corresponding to absorbed iodine in oil

Iodine No. = ml. of $Na_2S_2O_3$ absorbed in oil sample X Normality of $Na_2S_2O_3$ solution X milli-equivalent weight of iodine X 100 gms. of oil ÷ weight of oil sample

Sample I - (22.80 - 17.95) X 0.1005 X 0.1269 X 100/.2115 = 107.7

Sample II - (22.80 - 5.75) X 0.1005 X 0.1269 X 100/.2019 = 107.3

Ave. Value - 107.5

Calculation of Reaction Rate

Basis of calculation : Series C - Catalyst Concentration - 0.175%

$$k = \frac{2.303}{t} \log \frac{C_o}{C}$$

SAMPLE CALCULATIONS (CONTINUED)

Where: t = time of reaction in seconds = 10,800 sec.

C_o = initial concentration or iodine value = 107.5

C = final concentration or iodine value = 73.0

$$k = \frac{2.303}{10{,}800 \text{ sec.}} \log \frac{107.5}{73}$$

$$= 0.000213 \log 1.474$$

$$= 0.000213 \times 0.1685$$

$$= 0.000359 \ \text{Sec}^{-1}$$

TABLE XIV

HYDROGEN PRESSURE GAGE
CALIBRATION DATA

DEAD WEIGHT READINGS LB/SQ.IN.	GAGE READINGS LB/SQ.IN.	DEAD WEIGHT READINGS LB/SQ.IN.	GAGE READINGS LB/SQ. IN.
5	5	240	231
10	9	260	250
15	14	280	268
25	23.5	300	288
35	34	280	268
50	50	260	250
60	59	240	231
80	79	220	213
100	98.5	200	195
120	118	180	176
140	137	160	157
160	157	140	137.5
180	176.5	100	99
200	195	60	59
220	213	50	50

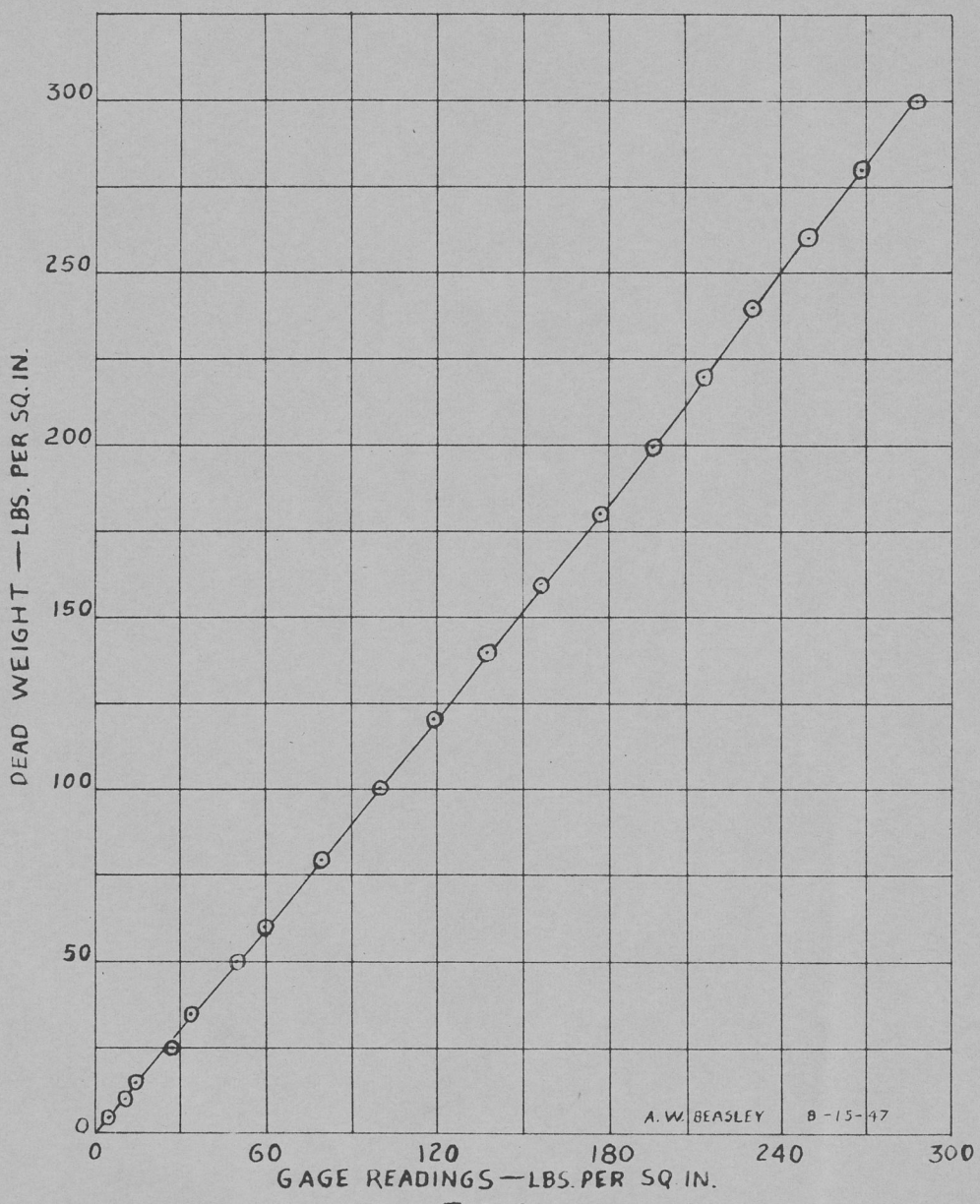

FIG. 6

CALIBRATION CURVE FOR PRESSURE GAGE

APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it is submitted.

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