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APPENDIX A

EXPERIMENTAL DATA

Table A1 Selection of the optimum temperature on the hydrogenation of rubber seed oil :

Type of catalyst G95E
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm
 Hydrogenation time 5 hours

120°C		140°C		160°C		180°C	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)
0	0.0	0	0.0	0	0.0	0	0.0
5	0.0	5	0.5	5	10.5	2	2.5
10	0.5	10	1.0	10	25.0	8	34.0
15	1.5	15	2.5	15	32.5	12	48.0
20	1.5	20	4.0	20	39.0	17	50.5
25	4.5	25	5.0	25	43.0	22	50.5
30	6.0	30	7.0	30	47.0	32	52.5
35	9.0	35	9.5	35	49.5	37	53.5
40	12.0	40	10.0	40	51.5	42	54.5

120°C		140°C		160°C		180°C	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)
45	14.0	45	12.0	45	54.0	47	58.0
50	17.5	50	15.0	55	56.0	52	58.5
55	19.0	55	16.0	65	57.5	60	58.5
60	22.5	70	19.0	75	59.0	70	58.5
65	25.0	80	24.0	85	59.5		
70	27.0	90	27.5	95	60.5		
75	29.0	100	29.5	105	61.0		
80	31.5	110	32.0	115	61.5		
85	33.5	120	34.0	125	62.0		
90	35.0	130	36.0	145	62.5		
95	38.5	140	37.0	170	62.5		
105	40.0	160	41.5	200	63.0		
110	41.0	180	45.0	230	63.0		
125	42.5	200	48.0	260	63.0		
135	44.5	220	50.5				
145	45.5	240	52.0				
155	47.0	260	54.0				
175	49.5	300	56.0				
195	51.5						
215	53.5						
235	55.0						
265	56.5						
300	57.0						



Table A2 Selection of the suitable commercial catalyst on the hydrogenation of rubber seed oil :

Reaction temperature 160°C
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm
 Hydrogenation time 5 hours

Kawaken		G53D		G95E		Nysel HK-4	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)
0	0.0	0	0.0	0	0.0	0	0.0
5	0.0	5	0.5	5	10.5	2	1.0
10	0.0	10	1.5	10	25.0	4	3.0
20	1.5	15	4.0	15	32.5	6	7.0
25	4.5	20	6.5	20	39.0	8	14.0
30	5.5	25	9.0	25	43.0	10	20.5
35	7.5	35	14.0	30	47.0	12	24.0
40	8.5	40	15.5	35	49.5	14	26.5
45	9.5	45	18.0	40	51.5	16	28.5
50	11.0	50	20.5	45	54.0	18	30.0
55	12.0	55	22.5	55	56.0	20	32.0
60	12.5	60	24.5	65	57.5	25	35.0
70	14.0	65	27.0	75	59.0	30	38.5
80	15.0	70	29.0	85	59.5	35	41.0
90	15.5	75	31.0	95	60.5	40	43.5

Kawaken		G53D		G95E		Nysel HK-4	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)	Time (min)	Hydrogen consumption (min)
100	16.5	80	33.0	105	61.0	45	45.5
110	17.0	85	35.0	115	61.5	50	47.5
120	17.5	90	36.5	125	62.0	60	50.5
140	18.5	95	38.5	145	62.5	70	52.5
160	20.0	100	40.0	170	62.5	80	54.5
180	21.5	105	42.0	200	63.0	100	58.0
200	22.5	110	43.5	230	63.0	120	60.0
230	23.0	115	43.5	260	63.0	150	62.0
260	23.5	120	44.5			180	63.5
300	24.0	130	46.5			210	64.5
		140	48.5			240	65.5
		150	50.0			270	66.5
		160	51.0			300	67.0
		170	52.0				
		180	53.0				
		200	55.0				
		220	56.5				
		240	57.5				
		270	58.5				
		300	59.5				

Table A3 Study of the reaction temperature effect on the hydrogenation of rubber seed oil :

Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm
 Hydrogenation time 5 hours

Run no.1		Run no.2		Run no.3		Run no.4	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
0	0.0	0	0.0	0	0.0	0	0.0
15	0.0	5	0.0	2	1.0	1	6.0
25	1.0	15	1.0	4	3.0	2	13.5
35	3.5	20	2.5	6	7.0	3	22.0
45	8.0	25	6.5	8	14.0	4	24.0
50	10.5	30	14.5	10	20.5	6	27.0
55	13.5	35	22.0	12	24.0	8	30.0
60	17.0	40	27.0	14	26.5	10	32.5
65	19.5	45	30.0	16	28.5	12	34.5
70	22.5	50	32.0	18	30.0	15	38.0
75	25.0	55	33.5	20	32.0	20	41.5
80	27.0	60	35.5	25	35.0	25	45.5
85	28.5	65	37.5	30	38.5	30	49.0
95	31.0	70	39.0	35	41.0	35	51.5
105	33.0	75	40.0	40	43.5	40	54.0
120	35.0	80	41.0	45	45.5	45	56.0

Run no.1		Run no.2		Run no.3		Run no.4	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
130	36.5	85	42.0	50	47.5	50	57.5
140	37.5	95	44.0	60	50.5	60	59.0
150	38.5	105	45.5	70	52.5	70	60.5
160	39.5	115	47.0	80	54.5	90	62.5
170	40.5	125	49.0	100	58.0	110	63.5
180	41.5	135	49.5	120	60.0	140	64.5
200	43.5	145	51.0	150	62.0	180	65.0
220	45.0	165	52.5	180	63.5	240	65.5
240	46.5	185	54.0	210	64.5	300	66.0
270	48.0	205	55.5	240	65.5		
300	49.5	235	57.0	270	66.5		
		265	59.0	300	67.0		
		300	60.0				

Table A4 Study of the hydrogen pressure effect on the hydrogenation of rubber seed oil :

Reaction temperature 160°C
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm
 Hydrogenation time 5 hours

Run no.5		Run no.6		Run no.7		Run no.3	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
0	0.0	0	0.0	0	0.0	0	0.0
5	2.0	5	0.5	2	1.0	2	1.0
10	8.5	10	3.5	4	3.5	4	3.0
15	12.5	15	15.0	6	7.0	6	7.0
20	16.5	20	23.5	8	12.5	8	14.0
25	21.0	25	27.5	10	19.0	10	20.5
30	24.5	30	30.0	12	24.0	12	24.0
35	26.5	35	33.0	14	27.0	14	26.5
40	29.0	40	36.5	16	29.5	16	28.5
45	30.5	45	39.0	18	31.0	18	30.0
50	32.0	50	41.0	20	32.5	20	32.0
55	34.0	55	42.5	25	36.0	25	35.0
60	35.0	60	44.5	30	38.0	30	38.5
70	38.0	65	46.0	40	42.5	35	41.0
80	40.0	70	47.5	50	46.0	40	43.5
90	42.0	75	48.5	60	49.0	45	45.5
100	43.5	80	49.5	70	51.0	50	47.5

Run no.5		Run no.6		Run no.7		Run no.3	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
110	45.0	90	51.5	80	53.5	60	50.5
120	46.0	100	53.5	90	55.0	70	52.5
140	48.5	110	55.0	100	56.5	80	54.5
160	50.5	120	56.5	120	59.0	100	58.0
190	53.0	130	57.5	140	60.5	120	60.0
220	54.5	150	60.0	160	62.0	150	62.0
240	56.0	170	62.0	180	63.0	180	63.5
270	57.0	190	63.0	210	64.5	210	64.5
300	58.5	210	64.5	240	65.5	240	65.5
		240	65.5	270	66.0	270	66.5
		270	67.0	300	66.0	300	67.0
		300	67.5				

Table A5 Study of the catalyst concentration effect on the hydrogenation of rubber seed oil :

Reaction temperature 160°C
 Hydrogen pressure 150 psig
 Agitation 700 rpm
 Hydrogenation time 5 hours

Run no.8		Run no.9		Run no.3		Run no.10	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
0	0.0	0	0.0	0	0.0	0	0.0
5	0.0	5	1.0	2	1.0	3	14.0
10	0.0	10	2.5	4	3.0	5	25.5
15	1.0	15	7.0	6	7.0	7	30.5
21	3.0	20	15.0	8	14.0	13	36.0
26	5.0	25	20.0	10	20.5	15	38.0
30	7.0	30	23.5	12	24.0	20	43.0
35	9.0	35	26.0	14	26.5	25	47.0
40	11.0	40	27.0	16	28.5	30	50.5
45	12.5	45	28.5	18	30.0	35	53.0
50	13.5	50	30.0	20	32.0	40	55.5
55	14.5	55	31.0	25	35.0	45	58.0
65	15.5	60	32.0	30	38.5	50	59.0
75	17.5	70	33.5	35	41.0	55	60.0

Run no.8		Run no.9		Run no.3		Run no.10	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
85	18.5	80	35.0	40	43.5	60	61.0
95	19.0	90	36.5	45	45.5	70	63.5
115	20.0	100	37.0	50	47.5	80	64.5
140	21.0	110	37.5	60	50.5	90	65.0
155	22.0	130	39.0	70	52.5	110	66.0
175	22.5	150	40.5	80	54.5	140	66.5
195	23.0	170	41.5	100	58.0	170	67.0
240	23.5	190	42.5	120	60.0	210	67.5
270	24.0	210	43.5	150	62.0	240	67.5
300	24.5	240	44.5	180	63.5	300	67.5
		270	45.5	210	64.5		
		300	46.5	240	65.5		
				270	66.5		
				300	67.0		

Table A6 Study of the agitation effect on the hydrogenation of rubber seed oil :

Reaction temperature 160°C
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Hydrogenation time 5 hours

Run no.11		Run no.12		Run no.13		Run no.3	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
0	0.0	0	0.0	0	0.0	0	0.0
5	2.5	5	1.0	5	2.0	2	1.0
10	5.5	11	5.0	10	6.5	4	3.0
15	9.0	15	13.0	13	14.5	6	7.0
20	12.0	20	19.5	16	23.0	8	14.0
25	15.0	25	24.0	19	26.5	10	20.5
30	18.5	30	27.0	22	29.5	12	24.0
35	21.0	35	30.5	25	31.5	14	26.5
40	24.0	40	33.5	30	35.5	16	28.5
45	27.0	45	35.5	36	40.0	18	30.0
50	29.0	50	38.0	40	42.0	20	32.0
55	31.0	55	40.0	45	43.5	25	35.0
60	32.5	60	41.5	50	45.0	30	38.5
70	35.5	70	44.5	55	46.5	35	41.0

Run no.11		Run no.12		Run no.13		Run no.3	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
80	38.5	80	47.0	60	48.0	40	43.5
90	40.5	90	49.0	65	49.5	45	45.5
100	42.5	100	50.5	70	51.0	50	47.5
110	44.5	110	52.0	80	53.0	60	50.5
120	46.5	120	53.5	90	55.0	70	52.5
130	47.5	140	55.5	110	58.0	80	54.5
140	49.0	160	57.5	130	60.0	100	58.0
150	50.0	180	59.0	150	61.5	120	60.0
160	51.0	210	60.0	180	63.0	150	62.0
170	52.0	240	61.0	210	64.0	180	63.5
180	52.5	270	62.0	240	65.0	210	64.5
190	53.5	300	62.5	270	66.0	240	65.5
200	54.0			300	66.0	270	66.5
210	54.5					300	67.0
230	55.5						
250	57.0						
280	58.0						
300	58.5						

Table A7 Comparing the in-house catalysts to the best commercial catalyst :

Reaction temperature 160 C
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm
 Hydrogenation time 5 hours

Nysel HK-4		PC 1		PC 2		PC 3		IC	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
2	1.0	95	0.0	5	0.5	15	0.5	5	5.5
4	3.0	100	0.5	10	1.5	20	2.5	10	13.0
6	7.0	105	1.0	20	7.5	25	5.0	15	19.5
8	14.0	110	1.5	25	8.5	30	7.0	20	22.0
10	20.5	115	2.0	30	10.0	35	9.0	25	24.0
12	24.0	120	2.5	35	11.5	40	10.0	30	25.0
14	26.5	130	3.0	40	13.0	50	12.5	35	25.5
16	28.5	140	4.0	45	14.0	60	15.5	45	27.0
18	30.0	150	4.5	50	16.0	70	17.5	55	28.5
20	32.0	170	6.0	55	16.5	80	20.5	65	29.5
25	35.0	190	7.0	60	18.0	90	22.0	85	31.0
30	38.5	215	8.0	70	20.0	100	23.0	105	32.0
35	41.0	240	8.5	80	22.0	120	24.5	125	33.0
40	43.5	265	10.0	90	24.0	150	26.0	145	33.5
45	45.5	295	11.0	100	25.5	180	28.0	175	35.0
50	47.5	325	12.0	110	27.0	210	30.0	205	36.5

Nysel HK-4		PC 1		PC 2		PC 3		IC	
Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)	Time (min)	Hydrogen consumption (psi)
60	50.5	355	13.0	120	28.5	240	31.5	235	37.0
70	52.5			140	30.5	270	34.0	265	37.5
80	54.5			170	33.5	300	34.5	300	39.0
100	58.0			190	35.0				
120	60.0			220	36.5				
150	62.0			250	39.5				
180	63.5			280	41.0				
210	64.5			310	43.5				
240	65.5								
270	66.5								
300	67.0								

APPENDIX B

SAMPLE OF CALCULATION

B1 Calculation of Iodine Value

The calculation of iodine value are as follow:

- Let P_1 = hydrogen consumed, psi
 T_1 = average room temperature during operation,
 30°C or 303 K
 V_1 = volume of hydrogen stored tank, 6.7 L
 P_2 = hydrogen pressure at STP, 14.7 psi
 T_2 = temperature at STP, 0°C or 273 K
 V_2 = volume of hydrogen consumed at STP

$$\begin{aligned} \text{From ideal gas law: } \frac{P_1 V_1}{T_1} &= \frac{P_2 V_2}{T_2} \\ \text{Then, } V_2 &= \frac{V_1 P_1 T_2}{P_2 T_1} \quad \text{L} \\ &= \frac{6.7 \times P_1 \times 273}{14.7 \times 303} \quad \text{L} \\ &= 0.4107 P_1 \quad \text{L} \end{aligned}$$

At STP, the volume of 1 mole of gas is 22.414 L

- Let $\Delta P = P_1$
 $n =$ mole of hydrogen consumed

$$\begin{aligned} \text{Then, } n &= \frac{V_2}{22.414} \quad \text{mole} \\ \text{or } n &= \frac{0.4107 \times \Delta P}{22.414} \quad \text{mole} \\ &= 0.0183 \times \Delta P \quad \text{mole} \end{aligned}$$

From Bailey's Industrial Oil and Fat Products [15]:

To reduce 1000 kg of oil, one I.V. unit = 0.0795 kg of H₂

Let w = weight of oil, g

$$\begin{aligned} \text{Then, to reduce } w \text{ g of oil, one I.V. unit} &= \frac{0.0795 \times 10^3 w}{10^6} \text{ g of H}_2 \\ &= 7.95 \times 10^{-5} w \text{ g of H}_2 \end{aligned}$$

Molecular weight of H₂ = 2.016

$$\text{Then, to reduce } w \text{ g of oil, one I.V. unit} = \frac{7.95 \times 10^{-5} w}{2.016} \text{ mole of H}_2$$

For ΔP psi of hydrogen consumed, the I.V. reduce is:

$$\begin{aligned} \text{I.V. reduced} &= \frac{0.0183 \Delta P \times 2.016}{7.95 \times 10^{-5} w} \\ &= \frac{464.6 \Delta P}{w} \end{aligned}$$

From Run no. 3, at 8 min, $\Delta P = 14.158$ psi, $w = 203.8$ g

Then,

$$\begin{aligned} \text{I.V. reduced} &= \frac{464.6 \times 14.158}{203.8} \\ &= 32.4 \end{aligned}$$

At $t = 0$ min, I.V. = 141

Then, at $t = 8$ min:

$$\begin{aligned} \text{I.V. of oil} &= 141 - 32.4 \\ &= 108.6 \end{aligned}$$

B2 Test for the First Order Reaction of the Hydrogenation

From Table C3: Run no. 3

At $t = 0$ min, (I.V.)₀ = 141

At $t = 8$ min, I.V. = 108.6

$$\begin{aligned} \ln [(I.V.)_0 / I.V.] &= \ln \frac{141}{108.6} \\ &= 0.261 \end{aligned}$$

B3 Determination of the Activation Energy and the Frequency Factor.

From Arrhenius Equation:

$$k = A \exp(-E_{act}/RT)$$

In the logarithmic form:

$$\ln k = \ln A - E_{act}/RT$$

or

$$\log k = \log A - E_{act}/RT$$

Where k = the overall rate constant, min^{-1}
 A = the frequency factor, min^{-1}
 E_{act} = the activation energy, cal mole^{-1}
 R = the gas constant, $1.987 \text{ cal mole}^{-1} \text{ K}^{-1}$
 T = absolute temperature, K

From Figure 5.12:

The slope of the curve = -3167.51 K

Then,

$$\frac{-E_{act}}{2.303 R} = -3167.51 \text{ K}$$

$$E_{act} = 3167.51 \times 2.303 \times 1.987$$

$$= 14,495 \text{ cal mole}^{-1}$$

or $E_{act} = 14.5 \text{ kcal mole}^{-1}$

The y-intercept of the curve = 5.8

Then,

$$\log A = 5.8$$

or $A = 6.31 \times 10^5 \text{ min}^{-1}$

Table C2

Calculated iodine value at any time on the hydrogenation of rubber seed oil for the selection of the suitable commercial catalyst:

Reaction temperature 160 °C
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm

Kawaken		G56D		G95E		Nysel HK-4	
Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated
0	141.0	0	141.0	0	141.0	0	141.0
5	141.0	5	139.8	5	111.5	2	132.3
10	141.0	10	137.5	10	86.8	4	123.9
20	137.4	15	131.8	15	67.6	6	116.1
25	130.2	20	126.1	20	52.9	8	108.6
30	127.8	25	120.3	25	41.7	10	101.8
35	123.0	35	108.8	30	33.5	12	95.1
40	120.6	40	105.4	35	27.1	14	89.0
45	118.1	45	99.6	40	22.3	16	83.3
50	114.5	50	93.9	45	18.6	18	77.8
55	112.1	55	89.3	55	13.1	20	72.8
60	110.9	60	84.7	65	9.5	25	61.6
						30	52.0
						35	44.3
						40	37.9
						45	32.4
						50	28.3
						60	22.1

Table C3 Calculated iodine value at any time on the study of reaction temperature effect :

Type of catalyst Nysel HK-4
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm

Run no.1		Run no.2		Run no.3		Run no.4	
Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated
0	141.0	0	141.0	0	141.0	0	141.0
15	141.0	5	141.0	2	132.3	1	111.6
25	136.6	15	126.7	4	123.9	2	106.8
35	128.5	20	118.2	6	116.1	3	102.2
45	118.8	25	109.9	8	108.6	4	97.8
50	113.5	30	101.8	10	101.8	6	89.3
55	108.4	35	94.5	12	95.1	8	81.5
60	103.1	40	87.6	14	89.0	10	74.2
		45	80.9	16	83.3	12	67.5
		50	74.7	18	77.8	15	58.1
		55	68.9	20	72.8	20	44.8
		60	63.6	25	61.6	25	34.0
				30	52.0	30	25.3
				35	44.3	35	18.4
				40	37.9	40	12.9
				45	32.4	45	9.0
				50	28.3	50	6.0
				60	22.1	60	2.6

Table C4

Calculated iodine value at any time on
the study of hydrogen pressure effect :

Type of catalyst Nysel HK-4
Reaction temperature 160 °C
Catalyst concentration 0.2% Ni in oil
Agitation 700 rpm

Run no.5		Run no.6		Run no.7		Run no.3	
Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated
0	141.0	0	141.0	0	141.0	0	141.0
5	131.9	5	133.9	2	132.9	2	132.3
10	120.8	10	119.5	4	124.3	4	123.9
15	110.8	15	105.8	6	116.3	6	116.1
20	101.8	20	95.4	8	108.9	8	108.6
25	93.9	25	85.2	10	101.9	10	101.8
30	86.9	30	76.3	12	95.3	12	95.1
35	80.6	35	68.6	14	89.0	14	89.0
40	75.1	40	61.7	16	83.3	16	83.3
45	70.0	45	55.5	18	77.9	18	77.8
50	65.6	50	50.4	20	72.9	20	72.8
55	61.8	55	45.8	25	61.6	25	61.6
60	58.4	60	41.8	30	52.4	30	52.0
				40	38.6	35	44.3
				50	29.7	40	37.9
				60	24.3	45	32.4
						50	28.3
						60	22.1

Table C5 Calculated iodine value at any time on
the study of catalyst concentration effect :

Type of catalyst Nysel HK-4
Reaction temperature 160 °C
Hydrogen pressure 150 psig
Agitation 700 rpm

Run no.8		Run no.9		Run no.3		Run no.10	
Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated
0	141.0	0	141.0	0	141.0	0	141.0
5	137.0	5	127.9	2	132.3	3	126.5
10	133.1	10	116.4	4	123.9	5	117.6
15	129.3	15	106.1	6	116.1	7	109.2
21	124.9	20	97.2	8	108.6	13	87.3
26	121.5	25	89.3	10	101.8	15	80.9
30	118.8	30	82.4	12	95.1	20	66.9
35	115.6	35	76.5	14	89.0	25	55.3
40	112.6	40	71.3	16	83.3	30	45.8
45	109.7	45	66.9	18	77.8	35	38.1
50	107.1	50	63.1	20	72.8	40	32.1
55	104.6	55	59.8	25	61.6	45	27.4
65	100.1	60	57.1	30	52.0	50	23.8
				35	44.3	55	21.2
				40	37.9	60	19.3
				45	32.4		
				50	28.3		
				60	22.1		

Table C6 Calculated iodine value at any time on
the study of agitation effect :

Type of catalyst Nysel HK-4
Reaction temperature 160 °C
Hydrogen pressure 150 psig
Catalyst concentration 0.20% Ni in oil

Run no.11		Run no.12		Run no.13		Run no.3	
Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated	Time (min)	I.V. Calculated
0	141.0	0	141.0	0	141.0	0	141.0
5	132.3	5	126.1	5	120.5	2	132.3
10	124.1	10	112.7	10	102.6	4	123.9
15	116.4	15	100.6	13	93.1	6	116.1
20	109.1	20	89.7	16	84.3	8	108.6
25	102.2	25	80.0	19	76.3	10	101.8
30	95.8	30	71.3	22	69.1	12	95.1
35	89.8	35	63.6	25	62.4	14	89.0
40	84.2	40	56.7	30	52.7	16	83.3
45	78.9	45	50.5	36	42.9	18	77.8
50	74.0	50	45.1	40	37.4	20	72.8
55	69.4	55	40.2	45	31.5	25	61.6
60	65.1	60	35.9	50	26.5	30	52.0
				55	22.4	35	44.3
				60	18.9	40	37.9
						45	32.4
						50	28.3
						60	22.1

Table C7

Calculated iodine value at any time of comparing the in-house catalysts to the best commercial catalyst.

Reaction temperature 160 °C
 Hydrogen pressure 150 psig
 Catalyst concentration 0.2% Ni in oil
 Agitation 700 rpm

Nysel HK-4		PC 1		PC 2		PC 3		IC	
Time (min)	I.V. calculated	Time (min)	I.V. calculated	Time (min)	I.V. calculated	Time (min)	I.V. consumption	Time (min)	I.V. calculated
0	141.0	0	141.0	0	141.0	0	141.0	0	141.0
2	132.3	95	141.0	5	139.9	10	138.7	5	128.2
4	123.9	100	139.8	10	137.6	15	137.6	10	110.8
6	116.1	105	138.7	20	133.5	20	133.0	15	95.7
8	108.6	110	137.5	25	121.6	25	127.3	20	89.9
10	101.8	115	136.3	30	118.2	30	122.7	25	85.2
12	95.1	120	135.2	35	114.7	35	118.1	30	82.9
14	89.0	130	134.0	40	111.3	40	115.8	35	81.7
16	83.3	140	131.7	45	109.0	50	110.1	45	78.2
18	77.8	150	130.5	50	104.5	60	103.2	55	74.7
20	72.8	170	127.0	55	103.3	70	98.6	65	72.4
25	61.6	190	124.7	60	99.9	80	95.2	85	68.9
30	52.0	215	122.4	70	95.3	90	91.8	105	66.6
35	44.3	240	121.2	80	90.8	100	88.3	125	64.3
40	37.9	265	117.7	90	86.2	120	86.0	145	63.1

Nysel HK-4		PC 1		PC 2		PC 3		IC	
Time (min)	I.V. calculated	Time (min)	I.V. calculated	Time (min)	I.V. calculated	Time (min)	I.V. consumption	Time (min)	I.V. calculated
50	28.3	325	113.1	110	79.3	150	79.2	205	56.1
60	22.1			120	75.9	180	74.61	235	55.0
				140	71.4	210	70.0	265	53.8
				170	64.5	240	66.6	295	50.3
				190	61.1	270	60.9		
				220	57.6	300	59.7		
				250	50.8				
				280	47.4				
				310	41.7				

Table C8 Test for the first order reaction of
the hydrogenation at various temperatures :

Type of catalyst Nysel HK-4
Hydrogen pressure 150 psig
Catalyst concentration 0.20% Ni in oil
Agitation 700 rpm

Run no.1		Run no.2		Run no.3		Run no.4	
Time (min)	$\frac{\ln[(I.V.)_o]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_o]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_o]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_o]}{I.V.}$
0	0.000	0	0.000	0	0.000	0	0.000
15	0.000	5	0.000	2	0.064	1	0.234
25	0.032	15	0.107	4	0.129	2	0.278
35	0.093	20	0.176	6	0.194	3	0.322
45	0.171	25	0.249	8	0.261	4	0.366
50	0.217	30	0.326	10	0.326	6	0.457
55	0.263	35	0.400	12	0.394	8	0.548
60	0.313	40	0.476	14	0.460	10	0.642
		45	0.555	16	0.526	12	0.737
		50	0.639	18	0.595	15	0.887
		55	0.716	20	0.661	20	1.146
		60	0.796	25	0.828	25	1.422
				30	0.997	30	1.718
				35	1.158	35	2.036
				40	1.314	40	2.391
				45	1.471	45	2.751
				50	1.606	50	3.157
				60	1.853	60	3.993

Table C9 Test for the first order reaction of
the hydrogenation at various pressures :

Type of catalyst Nysel HK-4
Reaction temperature 160 °C
Catalyst concentration 0.20% Ni in oil
Agitation 700 rpm

Run no.5		Run no.6		Run no.7		Run no.3	
Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$
0	0.000	0	0.000	0	0.000	0	0.000
5	0.067	5	0.052	2	0.059	2	0.064
10	0.155	10	0.165	4	0.126	4	0.129
15	0.241	15	0.287	6	0.193	6	0.194
20	0.326	20	0.391	8	0.258	8	0.261
25	0.406	25	0.504	10	0.325	10	0.326
30	0.484	30	0.614	12	0.392	12	0.394
35	0.559	35	0.720	14	0.460	14	0.460
40	0.630	40	0.826	16	0.526	16	0.526
45	0.700	45	0.932	18	0.593	18	0.595
50	0.765	50	1.029	20	0.660	20	0.661
55	0.825	55	1.124	25	0.828	25	0.828
60	0.881	60	1.216	30	0.990	30	0.997
				40	1.295	35	1.158
				50	1.558	40	1.314
				60	1.758	45	1.471
						50	1.606
						60	1.853

Table C10 Test for the first order reaction of the hydrogenation at various catalyst concentration :

Type of catalyst Nysel HK-4
 Reaction temperature 160 °C
 Hydrogen pressure 150 psig
 Agitation 700 rpm

Run no.8		Run no.9		Run no.3		Run no.10	
Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$	Time (min)	$\ln\left[\frac{(I.V.)_0}{I.V.}\right]$
0	0.000	0	0.000	0	0.000	0	0.000
5	0.029	5	0.097	2	0.064	3	0.108
10	0.058	10	0.192	4	0.129	5	0.181
15	0.087	15	0.284	6	0.194	7	0.256
21	0.121	20	0.372	8	0.261	13	0.479
26	0.149	25	0.457	10	0.326	15	0.556
30	0.171	30	0.537	12	0.394	20	0.746
35	0.199	35	0.611	14	0.460	25	0.936
40	0.225	40	0.682	16	0.526	30	1.124
45	0.251	45	0.746	18	0.595	35	1.308
50	0.275	50	0.804	20	0.661	40	1.480
55	0.299	55	0.858	25	0.828	45	1.638
65	0.343	60	0.904	30	0.997	50	1.779
				35	1.158	55	1.895
				40	1.314	60	1.989
				45	1.471		
				50	1.606		
				60	1.853		

Table C11 Test for the first order reaction of
the hydrogenation at various agitations :

Type of catalyst Nysel HK-4
Reaction temperature 160 °C
Hydrogen pressure 150 psig
Catalyst concentration 0.20% Ni in oil

Run no.11		Run no.12		Run no.13		Run no.3	
Time (min)	$\frac{\ln[(I.V.)_0]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_0]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_0]}{I.V.}$	Time (min)	$\frac{\ln[(I.V.)_0]}{I.V.}$
0	0.000	0	0.000	0	0.000	0	0.000
5	0.064	5	0.112	5	0.157	2	0.064
10	0.128	10	0.224	10	0.318	4	0.129
15	0.192	15	0.338	13	0.415	6	0.194
20	0.256	20	0.452	16	0.514	8	0.261
25	0.322	25	0.567	19	0.614	10	0.326
30	0.386	30	0.682	22	0.713	12	0.394
35	0.451	35	0.796	25	0.815	14	0.460
40	0.516	40	0.911	30	0.984	16	0.526
45	0.581	45	1.027	36	1.190	18	0.595
50	0.645	50	1.140	40	1.327	20	0.661
55	0.709	55	1.255	45	1.499	25	0.828
60	0.773	60	1.368	50	1.672	30	0.997
				55	1.840	35	1.158
				60	2.010	40	1.314
						45	1.471
						50	1.606
						60	1.853

APPENDIX D

FITTING POLYNOMIAL EQUATION

Program polyfit is used for fitting the suitable curves in order to determine the overall rate constant. The program is shown below.

PROGRAM POLYFIT

```
1 IF PEEK (104) = 64 THEN GOTO 5
2 P$ = "RUN POLYFIT":D$ = CHR$ (4)
3 POKE 104,64: POKE 16383,0: POKE 103,0: PRINT D$;P$
4 STOP
5 REM
60 POKE 232,0: POKE 233,09
70 D$ = "": REM CONTROL-D CHAR
80 PRINT D$;"BLOAD SMALL CHARACTERS,A$0800"
85 REM THE SMALL CHARACTERS BINARY FILE WAS PRODUCED BY MINNESOTA EDUCATIONAL COMPUTING CONSORTIUM AND IS AVAIL
   ABLE ON THE PROGRAMMERS AID VOL.1 DISKETTE
87 HOME
90 SCALE= 2:SC = 2: ROT= 0:YO = 90:Y0 = 50:L$ = "POLYFIT": HCOLOR= 2: HGR : 60SUB 3500
93 FOR I = 1 TO 1000: NEXT I
95 SCALE= 1:SC = 1: TEXT
100 HOME : PRINT : PRINT : PRINT "          POLY-FIT"
110 PRINT : PRINT "A PROGRAM FOR FITTING VARIOUS POLYNOMIALEQUATIONS TO REAL DATA"
115 PRINT : PRINT "COPYRIGHT (C) 1981                      ADDISON-WESLEY PUBLISHING COMPANY, INC."
120 PRINT : PRINT "DEVELOPED FOR THE TEXT:                      BASIC MICROCOMPUTER MODELS IN BIOLOGY BY JAMES D. S
   PAIN."
127 VTAB 22: INPUT "FOR INTRODUCTION...PRESS <RETURN>";Q$: HOME
130 PRINT : PRINT "THE PROGRAM ALLOWS THE USER TO ENTER A NUMBER OF X,Y DATA POINTS WHICH MAY BE FITTED BY LEA
   ST SQUARES TO DIFFERENT POLYNOMIALS."
140 PRINT : PRINT "THE COEFFICIENTS ARE PROVIDED ALONG WITHSTATISTICS NECESSARY TO JUDGE HOW WELL THE EQUATION
   FITS THE DATA."
160 VTAB 22: INPUT "PRESS RETURN TO CONTINUE";A$
161 DIM A(6,12),S(9),Z(12),C(6)
162 DIM X(100),Y(100)
163 HOME : PRINT "YOU MAY ENTER DATA IN EITHER OF THE FOLLOWING WAYS:                      1. FROM THE
   KEYBOARD.                      2. FROM A PREVIOUSLY SAVED DISK FILE."
165 PRINT : PRINT : INPUT "WHICH MODE OF DATA ENTRY (1 OR 2) ? ";A$:A$ = LEFT$ (A$,1)
166 IF A$ = "1" OR A$ = "K" THEN GOTO 190
168 IF A$ < > "2" THEN PRINT "PLEASE TYPE EITHER (1) OR (2)": INPUT A$: GOTO 166
170 PRINT : INPUT "NAME OF TEXT FILE TO BE USED? ";N$
172 D$ = CHR$ (4)
174 PRINT D$;"OPEN";N$: PRINT D$;"READ";N$: INPUT N: FOR I = 1 TO N: INPUT X(I),Y(I): NEXT : PRINT D$;"CLOSE";N$
```



```

176 XM = 0:YM = 0: FOR T = 1 TO N: IF X(T) > XM THEN XM = X(T)
178 IF Y(T) > YM THEN YM = Y(T)
179 NEXT
180 GOTO 292
190 PRINT
200 INPUT "HOW MANY DATA POINTS ARE YOU GOING TO ENTER (AT LEAST 7 PLEASE) ? ";N
220 HOME : PRINT "IF YOU MAKE AN ERROR TYPE IN 'E' AND PRESS <RETURN>"
223 PRINT
225 PRINT "BECAUSE OF THE EXPONENTIATION REQUIRED IN THIS PROGRAM, ALL DATA SHOULD BE NORMALIZED TO NUMBERS
    BETWEEN -100 AND +100. PARTICULARLY IF YOU ARE FITTING HIGHER POLYNOMIALS."
226 PRINT : PRINT "NOTE...THERE IS NO LIMITATION AGAINST ZERO OR NEGATIVE NUMBERS IN THIS PROGRAM"
227 PRINT
230 FOR C = 1 TO N: REM ENTERING N DATA POINTS
240 PRINT "ENTER (X,Y) DATA POINT NUMBER "C" ";: INPUT " ";X$,Y$
242 IF LEFT$(X$,1) = "E" OR LEFT$(Y$,1) = "E" THEN C = C - 1: PRINT " ": GOTO 240
250 X = VAL (X$)
260 Y = VAL (Y$)
270 REM IF X OR Y IS TO BE TRANSFORMED, PUT TRANSFORM EQUATIONS HERE
280 LET X(C) = X:Y(C) = Y
283 IF X > XM THEN XM = X
285 IF Y > YM THEN YM = Y
290 NEXT C
292 PRINT : INPUT "DO YOU NEED TO CORRECT ANY OF THE DATA ?";Q$
294 IF LEFT$(Q$,1) = "Y" THEN GOSUB 6000
300 HOME : PRINT "CALCULATING THE SUMS OF SQUARES FOR DATA...PLEASE STAND BY"
310 FOR C = 1 TO N
320 LET Z(1) = Z(1) + X(C)
330 LET Z(2) = Z(2) + X(C) ^ 2
340 LET Z(3) = Z(3) + X(C) ^ 3
350 LET Z(4) = Z(4) + X(C) ^ 4
360 LET Z(5) = Z(5) + X(C) ^ 5
370 LET Z(7) = Z(7) + X(C) ^ 7
380 LET Z(6) = Z(6) + X(C) ^ 6
390 LET Z(8) = Z(8) + X(C) ^ 8
400 LET Z(9) = Z(9) + X(C) ^ 9
410 LET Z(10) = Z(10) + X(C) ^ 10
420 LET S(1) = S(1) + Y(C)
430 LET S(2) = S(2) + X(C) * Y(C)
440 LET S(3) = S(3) + Y(C) * X(C) ^ 2
450 LET S(4) = S(4) + Y(C) * X(C) ^ 3
460 LET S(5) = S(5) + Y(C) * X(C) ^ 4
470 LET S(6) = S(6) + Y(C) * X(C) ^ 5
480 LET S(9) = S(9) + Y(C) ^ 2
490 NEXT C
500 TEXT : HOME : PRINT "THE FOLLOWING POLYNOMIAL EQUATIONS MAY BE FITTED TO THE DATA:"
502 PRINT
503 PRINT "1. STRAIGHT LINE Y = A0 + A1*X"
504 PRINT
505 PRINT "2. QUADRATIC Y = A0 + A1*X + A2*X^2"
507 PRINT
510 PRINT "3. CUBIC Y = A0 + A1*X + A2*X^2 + A3*X^3"
515 PRINT : PRINT "4. QUARTIC Y = A0 + A1*X + A2*X^2
    + A3*X^3 + A4*X^4"
517 PRINT
520 PRINT "5. QUINTIC Y = A0 + A1*X + A2*X^2
    + A3*X^3 + A4*X^4 + A5*X^5"

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530 PRINT : INPUT "ENTER THE NUMBER OF THE EQUATION TO BE FITTED. ";R
540 IF R + 2 > N OR R > 5 THEN PRINT "SORRY...THE ";R;" ORDER POLYNOMIAL CAN NOT BE FITTED TO ..... ";N;" DAT
    A POINTS ! ": GOTO 530
550 REM SETTING MATRIX TO ZERO
560 FOR I = 1 TO 6
570 FOR J = 1 TO 12
580 LET A(I,J) = 0
590 NEXT J
600 NEXT I
610 REM LOADING THE MATRIX WITH THE SUMS OF SQUARES
620 LET A(1,1) = N:A(1,2) = Z(1):A(2,1) = Z(1):A(2,2) = Z(2)
630 IF R = 1 THEN GOTO 730
640 LET A(3,1) = Z(2):A(1,3) = Z(2):A(2,3) = Z(3):A(3,2) = Z(3):A(3,3) = Z(4)
650 IF R = 2 THEN GOTO 730
660 LET A(4,1) = Z(3):A(1,4) = Z(3):A(2,4) = Z(4):A(4,2) = Z(4):A(3,4) = Z(5):A(4,3) = Z(5):A(4,4) = Z(6)
670 IF R = 3 THEN GOTO 730
680 LET A(5,1) = Z(4):A(1,5) = Z(4):A(2,5) = Z(5):A(5,2) = Z(5):A(3,5) = Z(6):A(5,3) = Z(6):A(4,5) = Z(7):A(5,4)
    = Z(7):A(5,5) = Z(8)
690 IF R = 4 THEN GOTO 730
700 LET A(6,1) = Z(5):A(1,6) = Z(5):A(2,6) = Z(6):A(6,2) = Z(6):A(3,6) = Z(7):A(6,3) = Z(7):A(4,6) = Z(8):A(6,4)
    = Z(8)
710 LET A(5,6) = Z(9):A(6,5) = Z(9):A(6,6) = Z(10)
720 REM SETTING UP THE IDENTITY MATRIX NEEDED FOR INVERSION
730 LET M = R + 1
740 FOR I = 1 TO M
750 LET A(I,I + M) = 1
760 NEXT I
770 IF PF = 0 THEN PRINT "CURVE FITTING CALCULATIONS IN PROGRESS."
780 REM INVERSION OF MATRIX...A
790 FOR T = 1 TO M
800 FOR B = 1 TO M
810 LET F(B) = A(B,T)
820 NEXT B
830 FOR J = T TO 2 * M
840 LET A(T,J) = A(T,J) / F(T)
850 NEXT J
860 IF T = M THEN GOTO 930
870 LET C = T + 1
880 FOR I = C TO M
890 FOR J = 1 TO 2 * M
900 LET A(I,J) = A(I,J) - A(T,J) * F(I)
910 NEXT J
920 NEXT I
930 NEXT T
940 FOR I = M TO 2 STEP - 1
950 FOR B = M TO I STEP - 1
960 LET F(B) = A(I - 1,B)
970 NEXT B
980 FOR B = M TO I STEP - 1
990 FOR J = I TO 2 * M
1000 LET A(I - 1,J) = A(I - 1,J) - A(B,J) * F(B)
1010 NEXT J
1020 NEXT B
1030 NEXT I
1040 FOR I = 1 TO M

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1050 FOR J = M + 1 TO 2 * M
1060 LET K = J - M
1070 LET A(I,K) = A(I,J)
1080 NEXT J
1090 NEXT I
1100 REM MULTIPLICATION OF MAT C = A * S
1110 FOR I = 1 TO 6
1120 LET C(I) = 0
1130 NEXT I
1140 FOR I = 1 TO M
1150 FOR J = 1 TO M
1160 LET C(I) = C(I) + A(I,J) * S(J)
1170 NEXT J
1180 NEXT I
1182 IF PF = 0 THEN GOTO 1187
1183 PRINT : PRINT "THE BEST FITTING POLYNOMIAL EQUATION IS:"
1184 PRINT "   Y = A0 + A1*X "; IF R > 1 THEN PRINT "+ A2*X^2 "; IF R > 2 THEN PRINT "+ A3*X^3 "; IF R > 3
THEN PRINT "+ A4*X^4 "; IF R > 4 THEN PRINT "+ A5*X^5";
1185 PRINT "   ": GOTO 1200
1187 HOME
1190 PRINT "FOLLOWING ARE THE COEFFICIENTS OF THE POLYNOMIAL OF ORDER ";R
1195 PRINT
1200 PRINT "A0 = ";C(1):A0(R) = INT (C(1) * 10000) / 10000
1205 PRINT
1210 PRINT "A1 = ";C(2):A1(R) = INT (C(2) * 10000) / 10000
1215 PRINT
1220 IF R > 1.5 THEN PRINT "A2 = ";C(3):A2(R) = INT (C(3) * 10000) / 10000
1225 PRINT
1230 IF R > 2.5 THEN PRINT "A3 = ";C(4):A3(R) = INT (C(4) * 10000) / 10000
1235 PRINT
1240 IF R > 3.5 THEN PRINT "A4 = ";C(5):A4(R) = INT (C(5) * 10000) / 10000
1245 PRINT
1250 IF R > 4.5 THEN PRINT "A5 = ";C(6):A5(R) = INT (C(6) * 10000) / 10000
1255 PRINT
1260 LET R2 = C(1) * S(1) + C(2) * S(2) + C(3) * S(3) + C(4) * S(4) + C(5) * S(5) + C(6) * S(6) - (S(1) ^ 2) / N
1270 LET R2 = R2 / (S(9) - (S(1) ^ 2) / N)
1280 LET CR = SQR (R2)
1290 PRINT "THE COEFFICIENT OF DETERM.,R^2 IS:"; INT (R2 * 1000) / 1000
1295 PRINT
1300 PRINT "THE CORRELLATION COEFFICIENT IS:"; INT (CR * 1000) / 1000
1310 LET RC(R) = INT (CR * 1000) / 1000
1312 IF PF = 1 THEN GOTO 1330
1315 PRINT
1320 INPUT "TO COMPARE REAL DATA WITH MODEL DATA ....PRESS <RETURN>";Q$: HOME
1330 PRINT : PRINT "REAL X   REAL Y   MODEL Y   RESIDUAL": PRINT
1340 LET D2 = 0
1350 FOR C = 1 TO M
1360 LET Y1 = C(1) + C(2) * X(C) + C(3) * X(C) ^ 2 + C(4) * X(C) ^ 3 + C(5) * X(C) ^ 4 + C(6) * X(C) ^ 5
1370 LET D = Y(C) - Y1
1380 LET D2 = D2 + D ^ 2
1390 PRINT X(C);: HTAB 8: PRINT Y(C);: HTAB 18: PRINT INT (Y1 * 1000) / 1000;: HTAB 32: PRINT INT (D * 1000) /
1000
1400 NEXT C
1405 PRINT : PRINT
1407 IF PF = 1 THEN GOTO 1420

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1410 INPUT "FOR ADDITIONAL STATISTICS..PRESS RETURN";Q$
1415 HOME
1420 PRINT "THE RESIDUAL SUM OF SQUARES IS:"; INT (D2 * 1000) / 1000
1430 LET RM(R) = INT (D2 * 1000 / (N - M)) / 1000
1435 PRINT
1440 PRINT : PRINT "THE RESIDUAL MEAN SQUARE IS:";RM(R)
1450 PRINT : PRINT "THE STANDARD ERROR OF THE RESRESSION ESTIMATE:"; INT ( SQR (RM(R)) * 10000) / 10000
1455 PRINT
1460 LET F = R2 * (S(9) - (S(1) ^ 2) / N) / (D2 / (N - M))
1470 PRINT : PRINT "THE VALUE OF F IS:"; INT (F * 100) / 100
1475 IF FF = 1 THEN GOTO 2500
1480 LET VF(R) = INT (F * 100) / 100
1483 PRINT : INPUT "DO YOU WANT TO SEE THE MODEL GRAPHED IN RELATION TO THE DATA POINTS ? ";Q$
1485 IF LEFT$ (Q$,1) = "Y" THEN GOTO 2500
1490 INPUT "DO YOU WANT TO FIT ADDITIONAL EQUATIONS (YES OR NO) ";Q$
1500 IF LEFT$ (Q$,1) = "Y" THEN GOTO 500
1545 TEXT : HOME
1550 PRINT "SUMMARY OF CURVE FITTING RESULTS"
1560 PRINT : PRINT "EQUATION          F VALUE"
1570 PRINT : PRINT "1. STR. LINE          "; INT (VF(1))
1575 PRINT : PRINT "2. QUADRATIC          "; INT (VF(2))
1580 PRINT : PRINT "3. CUBIC              "; INT (VF(3))
1585 PRINT : PRINT "4. QUARTIC           "; INT (VF(4))
1590 PRINT : PRINT "5. QUINTIC           "; INT (VF(5))
1600 PRINT : PRINT : INPUT "WOULD YOU LIKE HARD-COPY OF THE SUMMARY ? ";Q$
1610 IF LEFT$ (Q$,1) < > "Y" THEN GOTO 1715
1620 PR# 1
1630 PRINT "SUMMARY OF RESULTS OF FITTING POLYNOMIAL EQUATIONS TO A SET OF DATA:"
1640 PRINT : PRINT "   LINE","QUADRATIC","CUBIC","QUARTIC","QUINTIC"
1645 PRINT : PRINT "A0   ";A0(1),A0(2),A0(3),A0(4),A0(5)
1650 PRINT "A1   ";A1(1),A1(2),A1(3),A1(4),A1(5)
1655 PRINT "A2   ";A2(2),A2(3),A2(4),A2(5)
1660 PRINT "A3   "; " ",A3(3),A3(4),A3(5)
1665 PRINT "A4   "; " " " ",A4(4),A4(5)
1670 PRINT "A5   "; " " " " " ",A5(5)
1675 PRINT
1680 PRINT "R   ";RC(1),RC(2),RC(3),RC(4),RC(5)
1685 PRINT
1690 PRINT "F   ";VF(1),VF(2),VF(3),VF(4),VF(5)
1695 PRINT
1700 PRINT "RMS ";RM(1),RM(2),RM(3),RM(4),RM(5)
1710 PR# 0
1715 TEXT : HOME : INPUT "DO YOU WANT HARD-COPY OF THE STATISTICS ON THE BEST FITTING MODEL ? ";Q$
1720 IF LEFT$ (Q$,1) = "Y" THEN GOTO 1730
1722 PRINT : INPUT "DO YOU WANT TO FIT ANOTHER EQUATION TO DATA ? ";A$
1723 IF LEFT$ (A$,1) = "Y" THEN HOME : GOTO 200
1725 PRINT : INPUT " WOULD YOU LIKE TO SAVE YOUR DATA IN A TEXT FILE? ";B$
1726 IF LEFT$ (B$,1) = "Y" THEN GOTO 7000
1728 PRINT : INPUT "DO YOU WANT TO FIT ANOTHER SET OF DATA ? ";Q$: IF LEFT$ (Q$,1) = "Y" THEN TEXT : HOME : RUN
161
1729 PRINT : PRINT "HOPE POLYFIT WAS USEFUL.....PROGRAM START POINTER RESET TO STANDARD LOCATION.": POKE
103,1: POKE 104,B: POKE 2048,0: STOP
1730 FOR C = 1 TO 5
1733 IF VF(C) > VF THEN R = C:VF = VF(C)
1735 NEXT C

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1740 PF = 1: PR# 1: GOTO 550
2497 REM GRAPH OF DATA ALONG WITH MODEL CURVE
2500 X$ = "X VALUE":Y$ = "Y VALUE": HCOLOR= 3: HGR : GOSUB 3000
2505 ZF = 1:ZG = 0
2510 FOR I = 0 TO 260 STEP 2
2520 X = I * XM / 260
2530 Y = C(1) + C(2) * X + C(3) * X ^ 2 + C(4) * X ^ 3 + C(5) * X ^ 4 + C(6) * X ^ 5
2540 GOSUB 4000
2545 IF YO < 0 OR YO > 149 THEN ZG = 0
2550 NEXT I
2555 ZG = 0
2560 FOR C = 1 TO N
2570 ZC = 1:X = X(C):Y = Y(C): GOSUB 4000
2580 NEXT C
2585 IF PF = 1 THEN PR# 0: VTAB 21: PRINT "TYPE...EXEC HARD COPY...PRESS <RETURN>": PRINT : PRINT "AFTER HARD
COPY IS OBTAINED.....TYPE ....GOTO 1490 ...PRESS <RETURN>": TEXT : STCP
2590 VTAB 22
2600 GOTO 1490
3000 REM AXES AND UNITS FOR GRAPHS
3010 REM SUBROUTINE DEVELOPED BY J. SPAIN, MICHIGAN TECH UNIV., AND B.J. WINKEL, ALBION COLLEGE
3020 REM DEFINE X$=VARIABLE PLOTTED ON X AXIS
3030 REM DEFINE Y$=VARIABLE PLOTTED ON Y AXIS
3040 REM DEFINE YM =MAXIMUM UNITS ON THE Y AXIS
3045 REM DEFINE XM = MAXIMUM UNITS ON THE X AXIS
3050 HCOLOR= 3: SCALE= 1:SC = 1: ROT= 0
3055 REM LIST OF RESERVED VARIABLES: X,XO,XM,X$,Y,YO,YM,Y$,Z,ZF,ZG,ZP,ZO,I,L$,SC
3060 HPLOT 23,0 TO 23,149
3070 HPLOT 25,149 TO 279,149
3075 FOR I = 9 TO 116 STEP 35
3080 HPLOT 26,I TO 23,I: NEXT I
3085 FOR I = 55 TO 279 STEP 32
3090 HPLOT I,146 TO I,149: NEXT I
3095 REM WRITE VARIABLE NAME ON X-AXIS
3100 ZO = 0:L$ = X$:XO = 175:YO = 150
3110 GOSUB 3500
3120 REM WRITE VARIABLE NAME ON Y-AXIS
3130 ZO = 1:L$ = Y$:XO = 10:YO = 60
3140 GOSUB 3500
3150 REM WRITE UNITS ON THE X-AXIS
3155 YO = 150:ZO = 0
3160 L$ = LEFT$ ( STR$ (XM),3):XO = 260: GOSUB 3500
3165 L$ = LEFT$ ( STR$ (XM / 2),3):XO = 146: GOSUB 3500
3170 L$ = LEFT$ ( STR$ (XM / 4),3):XO = 82: GOSUB 3500
3172 REM WRITE UNITS ON Y-AXIS
3175 XO = 1
3180 L$ = LEFT$ ( STR$ (YM),3):YO = 6: GOSUB 3500
3185 L$ = LEFT$ ( STR$ (YM / 2),3):YO = 76: GOSUB 3500
3190 L$ = LEFT$ ( STR$ (YM / 4),3):YO = 111: GOSUB 3500
3195 DRAW 48 AT 15,150
3200 RETURN
3500 REM ALPHANUMERIC CHARACTERS FOR HGR
3510 REM THE FOLLOWING MUST BE DEFINED
3511 REM BEFORE ENTERING THE SUBROUTINE
3512 REM L$ = "CHARACTER STRING"
3513 REM YO = THE INITIAL Y POSITION

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3514 REM X0 = THE INITIAL X POSITION
3515 REM SET Z0 = 0 IF PRINTING HORIZONTAL
3516 REM SET Z0 = 1 IF PRINTING VERTICALLY
3520 FOR Z = 1 TO LEN (L$):Z3 = ASC ( MID$ (L$,Z,1))
3530 IF Z0 < > 0 THEN GOTO 3570
3540 IF Z3 > 64 THEN DRAW Z3 - 64 AT X0 + (Z - 1) * 7 * SC,Y0: GOTO 3600
3550 DRAW Z3 AT X0 + (Z - 1) * 7 * SC,Y0: GOTO 3600
3570 ROT= 48: IF Z3 > 64 THEN DRAW Z3 - 64 AT X0,Y0 - (Z - 1) * 7: GOTO 3600
3580 DRAW Z3 AT X0,Y0 - (Z - 1) * 7
3600 ROT= 0: NEXT Z
3610 RETURN
4000 REM PLOTTING X AND Y VALUES
4005 REM FOR LINE PLOT MAKE ZF=1
4006 REM TO PLOT (+) SET ZP = 1
4007 REM TO PLOT (O) SET Z0 = 1
4010 X0 = Z3 + X * 253 / XM
4020 Y0 = 149 - Y * 140 / YM
4030 IF X0 > 279 OR X0 < 0 OR Y0 > 149 OR Y0 < 0 THEN GOTO 4050
4033 IF ZP = 1 THEN DRAW 43 AT X0 - 2,Y0 - 3:ZP = 0: GOTO 4050
4034 IF Z0 = 1 THEN DRAW 79 AT X0 - 2,Y0 - 3:Z0 = 0: GOTO 4050
4035 IF Z6 = 1 THEN HPLDT TO X0,Y0: GOTO 4050
4040 HPLDT X0,Y0: IF ZF = 1 THEN Z6 = 1
4050 RETURN
6000 PRINT : INPUT " WHICH DATA POINT NUMBER DO YOU WANT TO CHANGE? ";AN
6005 IF (AN > N) OR (AN < 0) THEN PRINT " "; PRINT "THAT NUMBER DOESN'T EXIST IN YOUR FILE. TRY AGAIN.": GOTO 6000
6007 PRINT "CURRENT VALUES FOR DATA POINT NUMBER "AN" ARE "X(AN)", "Y(AN)".
6010 PRINT "ENTER NEW X AND Y VALUES FOR DATA POINT NUMBER ";AN;: INPUT X(AN),Y(AN)
6020 INPUT "MORE CORRECTIONS? (Y/N) ";AN$
6030 IF LEFT$ (AN$,1) = "Y" THEN 6000
6040 RETURN
7000 PRINT : INPUT "ENTER NAME OF NEW FILE ";N$
7010 D$ = CHR$ (4)
7015 PRINT D$;"MDN C,I,O"
7020 PRINT D$;"OPEN "N$
7025 PRINT D$;"DELETE";N$
7027 PRINT D$;"OPEN";N$
7030 PRINT D$;"WRITE "N$
7040 PRINT N
7050 FOR I = 1 TO N
7060 PRINT X(I);", ";Y(I)
7070 NEXT
7080 PRINT D$;"CLOSE "N$

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D1 Curve Fitting for Hydrogen Consumption

Experimental data of hydrogen consumption versus time are taken from Run No. 3. The conditions are:

Reaction temperature	160 °C
Hydrogen pressure	150 psig
Type of catalyst	Nysel HK-4
Catalyst concentration	0.2% Ni in oil
Agitation	700 rpm

The meaning of the variables of the fitting curve are:

Real X = Time, min

Real Y = Experimental hydrogen consumption, psi

Model Y = Calculated hydrogen consumption, psi

THE BEST FITTING POLYNOMIAL EQUATION IS:

$$Y = A_0 + A_1 * X + A_2 * X^2 + A_3 * X^3 + A_4 * X^4 + A_5 * X^5$$

A0 = -.0341608375

A1 = 1.9875464

A2 = -.0282014156

A3 = 1.93940055E-04

A4 = -6.18439705E-07

A5 = 7.33654707E-10

THE COEFFICIENT OF DETERM., R² IS: .988

THE CORRELLATION COEFFICIENT IS: .994

REAL X	REAL Y	MODEL Y	RESIDUAL
0	0	-.035	.034
2	1	3.829	-2.83
4	3	7.477	-4.478
6	7	10.916	-3.917
8	14	14.158	-.159
10	20.5	17.208	3.291
12	24	20.077	3.922
14	26.5	22.772	3.727
16	28.5	25.301	3.198
18	30	27.671	2.328
20	32	29.891	2.108

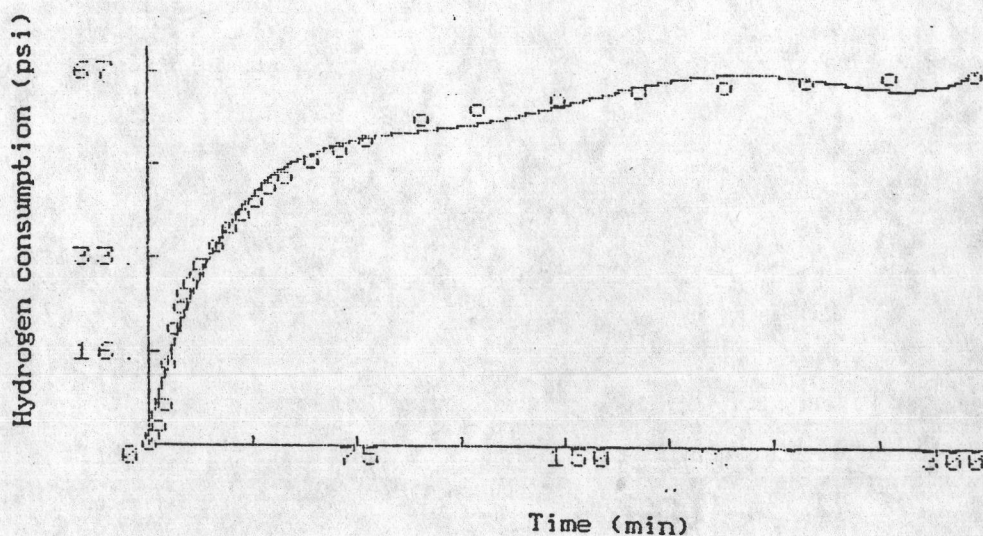
25	35	34.824	.175
30	38.5	38.964	-.465
35	41	42.408	-1.409
40	43.5	45.249	-1.75
45	45.5	47.569	-2.07
50	47.5	49.446	-1.947
60	50.5	52.14	-1.641
70	52.5	53.812	-1.313
80	54.5	54.85	-.351
100	58	56.138	1.861
120	60	57.515	2.484
150	62	60.74	1.259
180	63.5	64.472	-.973
210	64.5	66.631	-2.132
240	65.5	65.949	-.45
270	66.5	64.114	2.385
300	67	67.903	-.904

THE RESIDUAL SUM OF SQUARES IS:144.942

THE RESIDUAL MEAN SQUARE IS:6.588

THE STANDARD ERROR OF THE REGRESSION ESTIMATE:2.5667

THE VALUE OF F IS:1860.8



D2 Curve Fitting for testing of the First Order Reaction and Determination of Overall Rate Constant, k.

The data of calculated hydrogen consumption from D1 are used to calculate iodine value and $\ln [(I.V.)_0/I.V.]$, as shown in B1 and B2.

The meaning of the variables of the fitting curve are:

Real X = Time, min

Real Y = Real $\ln [(I.V.)_0/I.V.]$

Model Y = Model $\ln [(I.V.)_0/I.V.]$

From the fitting curve, k is the slope of the curve which is equal to A1. Then

$$k = 0.0318 \text{ min}^{-1}$$

THE BEST FITTING POLYNOMIAL EQUATION IS:

$$Y = A_0 + A_1 * X$$

$$A_0 = .0140555871$$

$$A_1 = .0318582264$$

THE COEFFICIENT OF DETERM., R^2 IS: .998

THE CORRELATION COEFFICIENT IS: .999

REAL X	REAL Y	MODEL Y	RESIDUAL
0	0	.014	-.015
2	.064	.077	-.014
4	.129	.141	-.013
6	.194	.205	-.012
8	.261	.268	-8E-03
10	.326	.332	-7E-03
12	.394	.396	-3E-03
14	.46	.46	-1E-03
16	.526	.523	2E-03
18	.595	.587	7E-03
20	.661	.651	9E-03
25	.828	.81	.017
30	.997	.969	.027
35	1.158	1.129	.028
40	1.314	1.288	.025
45	1.471	1.447	.023
50	1.606	1.606	-1E-03
60	1.853	1.925	-.073

THE RESIDUAL SUM OF SQUARES IS:9E-03

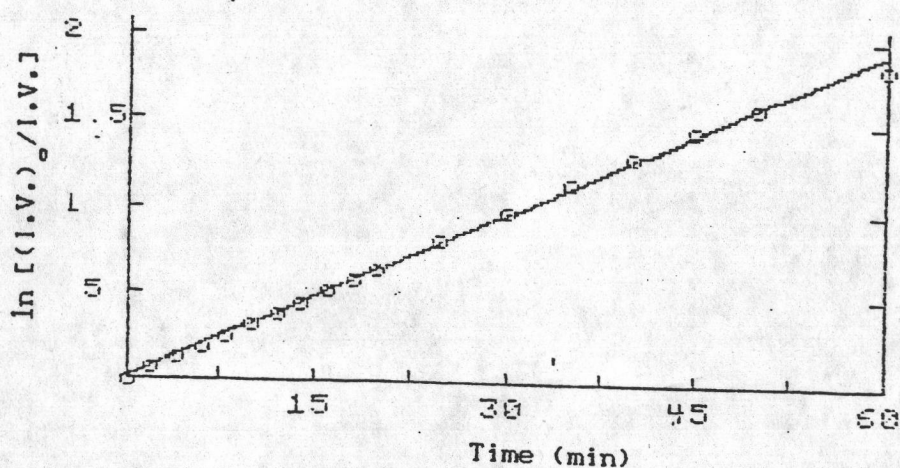
THE RESIDUAL MEAN SQUARE IS:0

THE STANDARD ERROR OF THE REGRESSION ESTIMATE:0

THE VALUE OF F IS:93531.6

SHAPE DISPLAY.....

SELECT SHAPE NUMBER :



D3 Curve Fitting for Arrhenius Plot.

The experimental data are taken from Run No. 1, 2, 3 and 4. The variable of these data is temperature. They are 120°C, 140°C, 160°C and 180°C, respectively. The other conditions are as described in D1.

The overall reaction rate constants from D2 are used to fit the curve. The activation energy, E_a , and frequency factor, A , can be determined from this curve. The calculation of E_a and A are shown in Appendix B.

The meaning of the variables of the fitting curve are:

$$\text{Real X} = 1/T, \text{K}^{-1}$$

$$\text{Real Y} = \text{Real} (-\log k)$$

$$\text{Model Y} = \text{Model} (-\log k)$$

THE BEST FITTING POLYNOMIAL EQUATION IS:

$$Y = A0 + A1 * X$$

$$A0 = -5.80166219$$

$$A1 = 3167.51079$$

THE COEFFICIENT OF DETERM., R^2 IS: .998

THE CORRELLATION COEFFICIENT IS: .999

REAL X	REAL Y	MODEL Y	RESIDUAL
2.54E-03	2.255695082	2.243	.011
2.42E-03	1.853859411	1.863	-.01
2.31E-03	1.496778411	1.515	-.019
2.21E-03	1.215020861	1.198	.016

THE RESIDUAL SUM OF SQUARES IS: 0

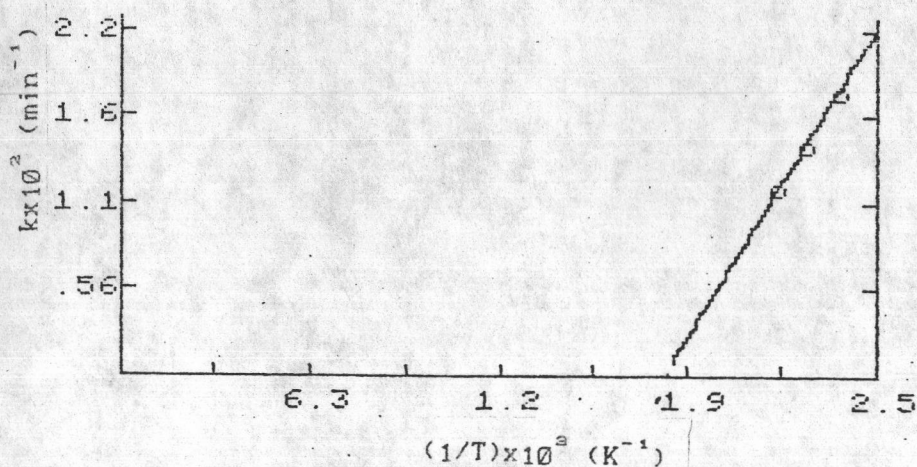
THE RESIDUAL MEAN SQUARE IS: 0

THE STANDARD ERROR OF THE REGRESSION ESTIMATE: 0

THE VALUE OF F IS: 14262.97,

SHAPE DISPLAY....

SELECT SHAPE NUMBER :



APPENDIX E

A.O.C.S. METHOD

E1 Iodine Value

A. O. C. S. Official Method Cd1-25

Definition: The iodine value is a measure of the unsaturation of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample (% iodine absorbed).

Scope: Applicable to all normal fats and oils not containing conjugated systems.

A. Apparatus:

1. Glass stoppered bottles or wide-mouth Erlenmeyer flasks, 500 ml. wide-mouth bottles or flasks are essential if stirring is done by mechanical means.

2. Glass stoppered volumetric flask, conforming to Bureau of Standards tolerances and accurately calibrated to contain 1000 ml.

3. Pipet, 20 ml.

4. Two pipets, 25 ml.

Note: One 25-ml. pipet is reserved for use with the standard potassium dichromate soln. This pipet must conform to Bureau of Standards tolerances and be accurately calibrated to deliver 25 ml.

5. Bottles, pyrex, actinic, glass stoppered. 1000 ml.

6. Filter paper, Whatman No. 41 H.

B. Reagents:

1. Glacial acetic acid, A.C.S. grade. The permanganate test should be applied to be sure that this specification is met.

Test: Dilute 2 ml. of the acid with 10 ml. of distilled water and add 0.1 ml. of 0.1 N KMnO_4 . The pink color must not be entirely discharged within 2 hours.

2. Potassium iodide, A.C.S. grade.

3. Chlorine, 99.8%. Satisfactory commercial grades are available in cylinders, but this gas must be dried by passing through sulfuric acid (sp. gr. 1.84) before introducing into the iodine soln.

Chlorine may be prepared by allowing hydrochloric acid (sp. gr. 1.19) to drop onto potassium permanganate or on a mixture of potassium permanganate and manganese dioxide. The gas thus generated is led through a glass tube into sulfuric acid (sp. gr. 1.84) and then into the iodine soln.

4. Carbon tetrachloride, A.C.S. grade.

5. Hydrochloric acid, A.C.S. grade, sp. gr. 1.19.

6. Soluble starch.

Test for Sensitivity. Make a paste with 1 g. of starch and a small amount of cold distilled water. Add, while stirring, 200 ml. of boiling water. Place 5 ml. of this soln. in 100 ml. of water and add 0.05 ml. of 0.1 N iodine soln. The deep blue color produced must be discharged by 0.05 ml. of 0.1 N sodium thiosulfate.

7. Potassium dichromate, A.C.S. grade. The potassium dichromate is finely ground and dried to constant weight at ca 110 °C. before using.

Note: A standard sample of potassium dichromate with a certificate of analysis may be obtained from the National Bureau of Standards at Washington, D.C. This sample is strongly

recommended as the primary standard for this method. Treat as directed in the certificate of analysis accompanying the sample.

8. Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$), A.C.S. grade.
9. Iodine, A.C.S. grade.
10. Iodine monochloride, technical grade, Eastman or Baker.
11. Sulfuric acid, A.C.S. grade, sp. gr., 1.84.

C. Solutions:

1. Potassium iodide soln., dissolve 150 g. in distilled water and make up to 1 liter.
2. Starch indicator soln., make a homogeneous paste of 10 g. of soluble starch in cold distilled water. Add to this 1 liter of boiling distilled water, stir rapidly and cool. Salicylic acid (1.25 g. per liter) may be added to preserve the indicator. If long storage is required, the soln. must be kept in a refrigerator at 4 ° to 10 °C. (40 ° to 50 °F.). Fresh indicator must be prepared when the end-point of the titration from blue to colorless fails to be sharp.
3. Standard potassium dichromate solution, 0.1 N, dissolve 4.9035 g. of finely ground and dried potassium dichromate in distilled water in the 1000-ml. volumetric flask and make to volume at 25 °C.
4. Sodium thiosulfate soln., 0.1 N, dissolve 24.8 g. of sodium thiosulfate in distilled water and dilute to 1 liter.

Standardization. Pipet 25 ml. of the standard dichromate soln. into Erlenmeyer flask or bottle. Add 5 ml. of hydrochloric acid, 10 ml. of potassium iodide soln. and rotate to mix. Allow to stand for 5 minutes and then add 100 ml. of distilled water. Titrate with sodium thiosulfate soln., shaking continuously, until the yellow color has almost disappeared. Add

1 to 2 ml. of indicator and continue titration adding the thiosulfate soln. slowly, until the blue color has just disappeared. The strength of the sodium thiosulfate soln. is expressed in terms of its normality.

$$\text{Normality of Na}_2\text{S}_2\text{O}_3 \text{ soln.} = \frac{2.5}{\text{Ml. sodium thiosulfate soln. required}}$$

5. Wijs soln., dissolve 13.0 g. of iodine in 1 liter of glacial acetic acid. Gentle heat may be necessary to promote soln. Cool and remove a small quantity (100 to 200 ml.) and set aside in a cool place for future use. Pass dry chlorine gas into the iodine soln. until the original titration is not quite doubled. A characteristic color change takes place in the Wijs soln. When the desired amount of chlorine has been added. This may be used to assist in judging the end-point. A convenient procedure is to add a small excess of chlorine and bring back to the desired titration by addition of some of the original iodine soln. which was taken out at the beginning. The original iodine soln. and finished Wijs soln. are both titrated with $\text{Na}_2\text{S}_2\text{O}_3$ soln. as directed in D, 6 and 7.

The Wijs solution may be prepared from commercial iodine monochloride as follows:

Stock Solution:

(a) Add 317 ± 0.1 g. of iodine monochloride to one liter of glacial acetic acid and filter through Whatman No. 41 H filter paper into a clean and dry actinic glass bottle. Filter rapidly to prevent contamination with moisture and store in a cool place. Discard the solution if a precipitate forms on standing.

(b) Wijs Solution: Using a graduate pour 117.0±0.1 ml. of the stock solution into a standard five-pound bottle of glacial acetic acid and mix well by shaking.

6. The I/Cl ratio of the Wijs solution shall be within the limits of 1.10±0.1. The procedure for determining the ratio is as follows:

Iodine Content:

(a) Pour 150 ml. of saturated chlorine water into a 500 ml. Erlenmeyer flask and add some glass beads.

(b) Pipet 5 ml. of the Wijs solution into the flask containing the saturated chlorine water. Shake and heat to boiling.

(c) Boil briskly for 10 minutes, cool, and add 30 ml. of 2% sulfuric acid and 15 ml. of 15% potassium iodide solution.

(d) Mix well and titrate immediately with 0.1 N sodium thiosulfate solution to a starch end-point.

Total Halogen Content:

(a) Pour 150 ml. of recently boiled distilled water into a clean, dry 500-ml. Erlenmeyer flask.

(b) Add 15 ml. of 15% potassium iodide solution

(c) Pipet 20 ml. of Wijs solution into the flask and mix well.

(d) Titrate immediately with 0.1 N sodium thiosulfate solution to a starch end-point.

Calculations of Halogen Ratio:

$$\text{Halogen ratio, } R = \frac{2A}{3B - 2A}$$

- A = Titration of iodine content as ml. sodium thiosulfate.
B = Titration of total halogen content as ml. sodium thiosulfate.

D. Procedure:

1. Melt the sample (see F, 1) if it is not already completely liquid (the temperature during melting and filtering should not exceed 10° to 15° C. above the melting point of the sample), and filter through filter paper to remove any impurities and the last traces of moisture. The sample must be absolutely dry.

Note: All glassware used must be absolutely clean and completely dry.

2. Weigh the sample accurately into 500-ml. flask or bottle to which have been added 20 ml. of CCl_4 . The weight of sample must be such that there will be an excess of Wijs solution of 50 to 60% of the amount added, i.e. 100 to 150% of the amount absorbed. The following table is a convenient guide to the size of sample to weigh (see F, 2):

3. Pipet 25 ml. of the Wijs soln. into flask containing the sample and swirl to insure an intimate mixture.

4. Prepare and conduct at least 2 blank determinations with each group of samples simultaneously and similar in all respects.

5. Store the flasks in a dark place for 30 minutes (see F, 3) at a temperature of $25^{\circ} \pm 5^{\circ}$ C.

6. Remove the flasks from storage and add 20 ml. of KI soln., followed by 100 ml. of distilled water.

7. Titrate with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ soln., adding it gradually and with constant and vigorous shaking (see F, 4). Continue the titration until the yellow color has almost disappeared. Add 1 to 2 ml. of starch indicator soln. and continue the titration until the blue color has just disappeared.

Iodine Value	Sample Weight (g.)		Weighing
	100% Excess	150% Excess	Accuracy
Less than 3	10	10	±0.001
3	10.576	8.4613	0.005
5	6.346	5.0770	0.0005
10	3.1730	2.5384	0.0002
20	1.5865	0.8461	0.0002
40	0.7935	0.6346	0.0002
60	0.5288	0.4231	0.0002
80	0.3966	0.3173	0.0001
100	0.3173	0.2538	0.0001
120	0.2644	0.2115	0.0001
140	0.2266	0.1813	0.0001
160	0.1983	0.1587	0.0001
180	0.1762	0.1410	0.0001
200	0.1586	0.1269	0.0001

E. Calculation:

$$\text{The iodine value} = \frac{(B - S) \times N \times 12.69}{\text{Weight of sample}}$$

B = titration of blank

S = titration of sample

N = normality of $\text{Na}_2\text{S}_2\text{O}_3$ soln.

F. Notes:

1. When the iodine value is required on the fatty

acids, the preparation and separation are performed as directed in A.O.C.S. Official Method Cd 6-38.

2. In the case of tung oil, weigh 0.1 to 0.2 g. of sample, using an excess of 55+3% Wijs soln. and allow the absorption to proceed for 1 hour at 20 ° to 25 °C.

3. In the case of dehydrated castor oils and dehydrated castor oil fatty acids, weigh 0.11 to 0.13 g.

4. Store samples with an iodine value above 150, such as linseed and perilla oils for 1 hour in a dark place at 25°±5°C.

5. Mechanical stirring is very satisfactory for agitating during the addition of thiosulfate.

6. All Wijs solutions are sensitive to temperature, moisture, and light. Store them in a cool and dark place and never allow them to come to a temperature much above 30 °C.

E2 Acid Value

A. O. C. S. Tentative Method Cd 3a-63

Definition: The acid value is the number of milligrams of potassium hydroxide necessary to neutralize the free fatty acids in 1 gram of sample. With samples that contain virtually no free acids other than fatty acids, the acid value may be directly converted by means of a suitable factor to percent free fatty acids.

Scope: Applicable to crude and refined animal, vegetable, and marine fats and oils, and various products derived from them.

A. Apparatus:

1. Erlenmeyer flasks, 250 or 300 ml.

B. Reagents:

1. Standard potassium hydroxide, 0.1 N. Add 6 g. c.p. KOH to 1 liter of water in a 2 liter Erlenmeyer flask, boil 10 minutes with stirring, add 2 grams c.p. $\text{Ba}(\text{OH})_2$, again boil 5 to 10 minutes, cool, stopper flask and let stand several hours, filter through sintered glass funnel, store in an alkali-resistant bottle protected from CO_2 standardize by titration with pure potassium acid phthalate using phenolphthalein indicator.

2. Solvent mixture consisting of equal parts by volume of isopropyl alcohol (A.O.C.S. specification H 18-58) and toluene (A.O.C.S. Specification H 19-58). The mixture must give a distinct and sharp end point with phenolphthalein in the titration procedure (Section C-5)

3. Phenolphthalein indicator soln., 1.0% in isopropyl alcohol.

C. Procedure:

1. Add indicator solution to the required amount of solvent in ratio of 2 ml. to 125 ml. and neutralize with alkali to a faint but permanent pink color.

2. Determine size of sample from the following table:

Acid Value	Wt. of sample ($\pm 10\%$), grams	Weighing accurately grams
0 to 1	20	0.05
1 to 4	10	0.02
4 to 15	2.5	0.01
15 to 75	0.5	0.001
75 and over	0.1	0.0002

3. Weigh the specified amount of well mixed liquid sample into an Erlenmeyer flask.

4. Add 125 ml. of the neutralized solvent mixture. Be sure that the sample is completely dissolved before titrating. Warning may be necessary in some cases.

5. Shake the sample vigorously while titrating with standard alkali to the first permanent pink color of the same intensity as that of the neutralized solvent before the latter was added to the sample. The color must persist for 30 seconds.

D. Calculation:

$$\begin{aligned} &\text{The acid value, mg. KOH per g. of sample} \\ &= \frac{\text{ml. alkali} \times N \times 56.1}{\text{Weight of sample}} \end{aligned}$$

To express in terms of free fatty acids as percent oleic, lauric, or palmitic, divide the acid value by 1.99, 2.81, or 2.19, respectively.

E. Precision:

Single determinations performed in two different laboratories shall not differ by more than 0.22 for values less than 4 nor by more than 0.36 for values in the range 4 to 20.

E3 Preparation of Methyl Esters of Long-Chain Fatty Acids

A. O. C. S. Official Method Ce 2-66

Definition: This method provides a means for preparing methyl esters of long-chain fatty acids for further analysis by methods such as gas liquid chromatography (Method Ce 1-62) and

infrared spectroscopy (Method Cd 14-61)

Scope: This method is applicable to common fats, oils and fatty acids (Note 1). Unsaponifiables are not removed and, if present in large amounts, they may interfere with subsequent analysis. The procedure will result in partial or complete destruction of the following groups; epoxy, hydroperoxy, cyclopropenyl, cyclopropyl and possibly hydroxyl, and is not suitable for the preparation of methyl esters of fatty acids containing these groups.

A. Apparatus:

1. 50 ml. and 125 ml. flat bottom boiling flasks or Erlenmeyer flasks with ST 19/38 or 24/40 outer necks.
2. Water cooled condensers, Liebig or West design, 20 or 30 cm, jacket, with ST 19/38 or 24/40 inner joint.
3. 250 ml. separatory funnels.
4. 200 ml. boiling flask for solvent removal.
5. Boiling chips-free of fat.

B. Reagents:

1. BF_3 - methanol reagent (125 g. per liter of methanol), available commercially or may be prepared using the gas and methanol (see section 3(d) of ASTM Method D-1983-64T, or Anal. Chem. 38, 514 (1966)).
2. Sodium hydroxide 0.5 N. in methanol
3. Sodium chloride, saturated solution in water.
4. Petroleum ether, redistilled, b.p. 30-60 °C.
5. Heptane, gas chromatographically clean.
6. Sodium sulfate, anhydrous, analytical reagent, A.C.S. grade.

7. Methyl red indicator, 0.1% in 60% ethanol.
8. Nitrogen gas, high purity.

C. Procedure:

Accurate weighing is not required. Sample size need be known only to determine the size of flask and amounts of reagents that should be used according to the following table:

Sample mg	Flask ml	NaOH 0.5 N ml	BF ₃ -methanol Reagent ml
100-250	50	4	5
250-500	50	6	7
500-750	125	8	9
750-1000	125	10	12

1. For Fatty Acids

Introduce the fatty acids into the 50 or 125 ml. reaction flask. Add the specified amount of BF₃-methanol reagent, attach a condenser and boil for two minutes. Add 2 to 5 ml. of heptane through the condenser and boil for one minute longer. Remove from heat, remove condenser and add enough saturated salt solution to float the heptane solution of the methyl esters into the neck of the flask. Transfer about 1 ml. of the heptane solution into a test tube and add a small amount of anhydrous sodium sulfate. The dry heptane solution may then be injected directly in a gas chromatograph.

To recover dry esters, transfer the salt solution and heptane phase to a 250 ml. separatory funnel. Extract twice with 50 ml. portions of redistilled petroleum (b.p. 30-60 °C). Wash the combined extracts with 20 ml. portions of water until free of acids (test water with methyl red indicator), dry with sodium sulfate, and evaporate the solvent under a stream of nitrogen on a steam bath (Note 1, Note 2)

2. For Fats and Oils

Introduce the fat into the 50 or 125 ml. reaction flask. Add the specified amount of 0.5 N methanolic sodium hydroxide and add a boiling chip. Attach a condenser and heat the mixture on a steam bath until the fat globules go into solution. This step should take 5-10 minutes. Add the specified amount of BF_3 -methanol reagent through the condenser and proceed as described under the fatty acid section.

D. Notes:

1. There is danger of losing some of the more volatile esters if the solvent removal step is prolonged or if too vigorous a stream of nitrogen is used. For infrared spectroscopy, this step should be terminated as soon as all solvent is removed. For gas-liquid chromatography the method may be extended to fatty acids with 8-carbon atoms if the solvent is not completely removed.

2. The methyl esters should be analyzed as soon as possible. They may be kept in an atmosphere of nitrogen in a screw cap vial at 2 °C for 24 hours. For longer storage they should be sealed in a glass ampule under vacuum and placed in a freezer.

E4 Fatty Acid Composition by Gas Chromatography

A. O. C. S. Official Method Ce 1-62

Definition: Methyl esters of fatty acids are separated and determined quantitatively by gas chromatography.

Scope: The method is applicable to methyl esters of fatty acids having 8 to 24 carbon atoms and to animal fats, vegetable oils, and fatty acids after their conversion to methyl esters. The method permits quantitative separation of mixtures containing saturated and unsaturated methyl esters. The conditions specified in this method are not suitable for determining epoxy or oxidized fatty acids nor to fatty acids that have been polymerized.

A. Apparatus:

1. The gas chromatographic instrument should have a minimum the following characteristics. These are available commercially.

(a) Column oven, capable of constant temperature operation to $\pm 1.0^{\circ}\text{C}$, at a temperature between 170°C and 210°C .

(b) Sample inlet port, independently heated to about 50°C higher temperature than the column oven.

(c) Columns, 5 to 10 feet long, 1/8 inch to 1/4 inch in outside diameter, made of stainless steel, glass, copper, or aluminum tubing. Columns of 1/8 inch O.D. are packed with 8 to 12% polyester liquid phase coated on 80 to 100 mesh acid washed chromosorb W or the equivalent. Column of 1/4 inch O.D. are packed with 15 to 20% polyester liquid phase coated on 60 to 80 mesh acid washed chromosorb W or the equivalent. Diethylene glycol succinate (DEGS) is the recommended liquid phase although for specific separations other liquid phases are employed. The

1/8 inch columns are usually employed with a flame ionization detector and the 1/4 inch columns with a thermal conductivity detector. It is recommended that the analysis be made on two columns, each with different separating properties (polyesters of differing polarity) to check for the presence of coincident peaks.

(d) Detectors, thermal conductivity (T.C.) or flame ionization (F.I.D.), if separately thermostated, should be maintained at column temperature or up to 50 °C hotter.

(e) Recorder, 0 to 1.0, 2.5 or 5.0 mv range, 1 second full scale deflection with a chart speed of 1/2 to 1 inch per minute. Attenuator switch to change recorder range as required. The 0-1.0 mv recorder is used when a thermal conductivity detector is employed; any one of the three may be used with the flame ionization detector.

(f) Gases

(1) Carrier

[a] T.C. : Helium, minimum purity 99.95 mol%

[b] F.I.D. : Helium, nitrogen, or argon, minimum purity 99.95 mol%

(2) F.I.D. : Hydrogen, minimum purity 99.95 mol%

Air, dry, dew point -75 °F maximum.

2. Syringe for injecting sample. A 1 to 5 μ l capacity with known and reproducible volume. A Hamilton 7000 Series syringe or equivalent is recommended.

B. Preparation of Methyl Esters:

1. A. O. C. S. Method Ce 2-66 is recommended.

C. Procedure:

1. With carrier gas flowing through the apparatus, adjust to operating temperature and record a base line to check



for stability of the instrument. With F.I.D., it is recommended that the apparatus be operated at medium sensitivity rather than near its maximum sensitivity. A new column should be conditioned by holding it about 10 °C above operating temperature with helium flowing at least 24 hours. The column should be disconnected from the detector during the conditioning period.

2. Proper gas flow rate and temperature will permit elution of methyl linolenate, other C₁₈'s and shorter chain length esters in less than 30 minutes. When esters of fatty acids of greater chain length are present, the gas flow and/or temperature should be increased so that the retention time of the last component is reduced. In addition, the largest sample size consistent with attenuation requirements (see C.3, below) should be used in order to better detect and quantitate these slow moving esters. A constant gas flow should be maintained throughout the analysis. When using a T.C. detector, the gas flow should be measured at the exit with a soap bubble flow meter or other suitable device. It is more difficult to measure the flow in an instrument equipped with a flame ionization detector. It will probably be best to follow the manufacturer's instructions.

3. Measure the methyl ester sample in a syringe. When using a T.C. detector, the usual sample size is between 0.5 and 4 µl. For F.I.D., about 0.1 to 0.01 µl of actual esters are measured but this is usually in a suitable solvent so that 0.5 to 4 µl of solution is injected. Pierce the septum of the sample inlet port and quickly discharge the sample. Withdraw the needle and note on the recorder chart the small peak due to air when using T.C. or solvent when using F.I.D., which marks the sample introduction reference point. Sample size must be adjusted so that the major peak is not attenuated more than eight times, preferably less.

4. Watch the recorder pen to see that peaks do not go off scale. Change the setting of the attenuator as necessary to keep

the peaks on the chart paper. Mark attenuator setting on the chart.

5. After all the peaks have been traced and the pen has returned to base line, remove the chart for calculation.

D. Calculations:

1. Identify the peaks by relative position on the chart. The esters appear on the chromatogram in order of increasing number of carbon atoms and of increasing unsaturation for the same number of carbon atoms. That is, C_{16} is ahead of C_{18} and the C_{18} esters appear in the order stearate, oleate, linoleate, and linolenate. The C_{20} saturated (arachidic) ester usually appears before $C_{18:3}$ (linolenic) ester but may be coincident or appear after $C_{18:3}$ on some columns. (Coincident peaks are usually revealed by repeating the analysis at a different column temperature or employing a second column with a different polyester.) With constant operating conditions, the retention times (or chart distances) from the air peak to various sample component peaks can be used for identification of the peaks. However, relative retentions are more reproducible. Relative retentions are determined by dividing the observed retention time for each peak by the retention time observed for the peak of methyl palmitate (or other peak if some other basis is desired). Compare the observed retention times or relative retention times with those calculated from known mixtures run periodically on the same column under the same conditions.

2. Determine the area of each peak. If the instrument is equipped with an electro-mechanical or electronic integrator, the area is best measured by following the manufacturer's instructions. When an integrator is employed, the base line must remain constant or the integrator must be equipped with a base line corrector. Otherwise, the area is obtained by drawing lines tangent to the sides of the peak and intersecting the base line.

Determine the area of the resulting triangle by multiplying the height by one-half the base. For an attenuated peak the outer sides of the peak must be full chart span and the tangents drawn intersecting the base line should utilize the upper two-thirds to obtain the peak width. Determine the area by multiplying the height by one-half the base. The height must be corrected for attenuation including the correction required if the base line is not the recorder zero. Divide the area of each component by its calibration factor. The percentage of each component is calculated from the ratio of each area to the sum of the areas under all of the component peaks and reported as per cent by weight.

3. Calibration factors are determined relative to methyl palmitate to correct for nonlinearity of instrument response and for molecular weight differences. Such factors are determined by analyzing known mixtures preferably having composition similar to that of the unknown sample. Divide the area of each peak by the true weight percentage of that component; then by dividing each value by the value for methyl palmitate, the calibration factors are obtained.

Note: Reference mixtures simulating most fats and oils may be obtained from:

Applied Science Laboratories, Inc., P.O. Box 440,
State College, Pa. 16801;

Supelco, Inc., Supelco Park, Bellefonte, Pa 16823;

Lipids Preparation Laboratory, The Hormel Institute,
Austin, Minnesota 55912; and

Analabs, Inc., 80 Republic Drive, North Haven,
Connecticut 06473

4. Instrument and column performance are monitored by noting the separation of the oleate and stearate ester peak which is expressed as peak resolution.

$$\text{Peak Resolution} = \frac{2Y}{S+O}$$

Where: Y is the distance between the peak maxima for stearate and oleate esters.

S is the base width of the stearate peak.

O is the base width of the oleate peak.

These values should be determined on a sample containing approximately equal quantities of oleate and stearate esters using a sample size such that peak heights are 25-50% of the chart width. If the Peak Resolution is equal to or greater than 1.0, the column and instrument are in satisfactory condition. All columns when used will show a gradual loss in peak resolution; when the value becomes less than 1.0, a new column should be installed.

E. Precision:

1. Two single determinations of major components (>5%) performed in one laboratory shall not differ by more than 1.0 percentage unit.

2. Two single determinations performed in different laboratories shall not differ by more than 3.0% percentage unit .

Note: It is recommended that chromatographers read "Tentative Recommended Practice for General Gas Chromatography Procedures."

ASTM Designation: E 260-65T issued 1965.

E5 Softening Point or Open Tube Melting Point

A. O. C. S. Official Method Cc 3-25

Definition: The softening point is an index of the temperature at which fat softens or becomes sufficiently fluid to slip or run.

Scope: This method is applicable to such fats as coconut oil, stearin, hydrogenated fats and hard tallow. It is less satisfactory for lard, soft tallow, and animal greases. It is unsatisfactory for lard compounds and mixtures of hard and soft fats or emulsions.

A. Apparatus:

1. Softening point tubes, capillary glass tubing, inside diameter 1 mm., outside diameter 3 mm. maximum, convenient length 50 to 80 mm.
2. Thermometer, A.O.C.S. Specification H 6-40 or H 7-45.
3. Glass beaker, 600 ml.
4. Heat source, gas burner or electric hot plate with rheostat control.

Note: A convenient and suitable assembly of apparatus is shown in the illustration with A.O.C.S. Official Method Cc 1-25.

B. Procedure:

1. Melt the sample and filter through filter paper to remove any impurities and the last traces of moisture. The sample must be absolutely dry.

2. Dip at least 3 clean capillary tubes into the

completely liquid sample so that the fat stands ca 10 mm. high in the tubes. Chill the sample at once by holding the ends of the tubes that contain the sample pressed against a piece of ice until the fat has solidified.

3. Place the tubes in a container with a tight cover and hold in a refrigerator at 4° to 10°C (40° to 50°F) over night (16 hours).

4. Remove the tubes from the refrigerator and attach with a rubber band or by any other suitable means to the thermometer so that the lower ends of the tubes are even with the bottom of the mercury bulb of the thermometer.

5. Suspend the thermometer in a 600-ml. beaker of clear distilled water. The bottom of the thermometer is immersed in the water to a depth of ca 30 mm.

6. Adjust the starting temperature of the bath to 8° to 10°C below the softening point of the sample. Agitate the water bath with a small stream of air or other suitable means, and apply heat so as to increase the bath temperature at the rate of 1°C per minute, slowing down to 0.5°C per minute as the softening point is approached.

7. Continue heating until the fat column rises in each tube. Observe the temperature at which each column rises and calculate the average of all tubes. Report this average as the softening point.

C. Notes:

1. Palm oil and palm oil products should be tempered at $10 \pm 1^{\circ}\text{C}$ for 16 hours. A thermostatted water bath is recommended for closer temperature control.

APPENDIX F

PROPERTIES OF COMMERCIAL HYDROGENATED PRODUCTS

Table F1 Properties of hydrogenated oils :

F1.1 Partially hydrogenated

<i>Oil</i>	<i>M.P.</i> °C	<i>Con- gelation Point</i> °C	<i>Butyrene- fractometer Reading</i> (40°C)	<i>Acid No.</i>	<i>Saponi- fication No.</i>	<i>Iodine No.</i>
Coconut	44.5	27.7	35.9	0.4	254.1	1.0
Sesame (Techn.)	62.1	45.3	38.4	4.7	188.9	25.4
Whale	45.1	33.9	49.1	1.2	192.3	45.2
Arachis	51.2	36.5	50.1	1.0	188.7	47.4
Sesame	47.8	33.4	51.5	0.5	190.6	54.8
Cottonseed	38.5	25.4	53.8	0.6	195.7	69.7

F1.2 Completely hydrogenated

<i>Hydrogenated Oil or Fat</i>	<i>M.P.</i> °C	<i>Iodine No.</i>	<i>Saponification No.</i>	<i>Fatty Acid, M.P., °C</i>
Tallow	62.0	0.1	197.7	64.0
Lard	64.0	1.0	196.8	62.0
Cacao Butter	63.5-64	0.0	193.9	65.5
Arachis	64-64.5	0.0	191.6	67.0
Cod Liver	65.0	1.2	186.2	59.0
Linseed	68.0	0.2	189.6	70.5
Sesame	68.5	0.7	190.6	69.5
Olive	70.0	0.2	190.9	71.0
Poppy	70.5	0.3	191.3	71.0
Almond	72.0	0.0	191.8	71.0

Table F2 Properties of selected commercial products

F2.1 Characteristics of hydrogenated fatty acids

Chemical	Predominant Chain Length	Titer °C	Iodine Value	Acid Value	Sap. Value	Color Lovibond 51* Max	Calculated Molecular Weight	Acetyl Value Min	Hydroxyl Value Min	Specific Gravity 100/25°C	C ₈ & C ₁₀	Approximate Composition %						12-Hyd. max. Unsaturated Content %
												C ₁₂	C ₁₄	C ₁₆	C ₁₈	C ₂₀	C ₂₂	
Hydrofol Acid 630	40% C ₁₂	31-37	1 max	245-255	246-256	10Y/1P ¹⁾	220-228			0.838	10	40	17	13	20			
Hydrofol Acid 150	80% C ₁₈	62-64	3 max	195-201	196-202	15Y/1.5R	279-286			0.839			T	20	80			
Distal 42	65% C ₁₈	40-43	40-60	200-205	201-206	40Y/4R	273-280			0.892			5	30	65		50	
Hydrofol Acid 405	65% C ₁₈	56-60	6 max	200-205	201-206	25Y/2.5R	273-280			0.835			5	30	65			
Hydrofol Acid 410	65% C ₁₈	58-61	1 max	201-206	202-207	10Y/1R	272-278			0.835			5	30	65			
Hydrofol Acid 420	53% C ₁₈	54-56	1 max	204-209	205-210	10Y/1R	268-274			0.834			7	40	53			
Hydrofol Acid 46	38% C ₁₈	45-48	20-30	190-203	198-204	30Y/3R	276-284			0.839			10	38	25	17	10	
Hydrofol Acid 51	38% C ₁₈	51-53	6 max	197-202	198-203	25Y/2.5R	277-284			0.832			10	38	25	17	10	
Soodotte Acid	30% C ₁₈	54 min	3 max	193-198	194-199	10Y/1R	282-290			0.832			10	30	20	25	15	
Hydrofol Acid AB	55% C ₁₈ -C ₂₂	60-63	5 max	178-185	179-186	25,Y/2.5R	302-314			0.828			2	13	30	30	25	
Erucic Acid Hydrofol Acid 565	80% C ₂₂	26-30	78-85	163-170	164-171	30Y/3.5R	329-343			0.837					8	12	80	98
Behenic Acid Hydrofol Acid 560	80% C ₂₂	69-73	4 max	168-174	169-175	25Y/2.5R	322-333			0.828					8	12	80	
12-Hydroxystearic Acid Hydrofol Acid 200	88% 12-Hydroxy C ₁₈	70-74	4 max	172-182	182-187	Light	300-309	133	147	0.880				2	10			88

¹ Gardner 5 max after 1 hour @ 204°C.

Compositions shown are based on gas chromatographic data. They are approximate and may vary ±10% of the values given depending upon the raw materials available.

F2.2 Characteristics of hydrofol glycerides

Chemical	Predominant Chain Length	Titer °C	FFA Max as Oleic %	Acid Value Max	Iodine Value	Sap. Value	Melting Point Open Tube		Color Lovibond 5½" Max	Acetyl Value Min	Hydroxyl Value Min	Ac. Flash Point °F	Ac. Fire Point °F	Sp. Gravity 100/25°C	Approximate Composition					12-hydroxyl Unsaturated Content %
							°C	°F							C14	C16	C18	C20	C22	
Hydrofol Glyceride 42	34% C18	41-44	3	6	32-45	188-195	44-47	111-117	40Y/4R					0.859	9	34	26	16	15	43
Hydrofol Glyceride 45	34% C18	44-47	3	6	22-35	188-195	47-50	112-122	30Y/3R					0.859	9	34	26	16	15	28
Hydrofol Glyceride 50-51	35% C18	50-53	3	6	6 max	188-195	53-57	127-135	25Y/2.5R					0.856	8	35	27	17	13	
Hydrofol Glyceride 150	92% C18	63-66	2	4	3 max	188-193	65-68	149-154	25Y/2.5R					0.858	8	92				
Hydrofol Glyceride T-57-L	75% C18	59-62	0.25	0.5	1 max	193-196	61-65	142-149	5Y/0.5R					0.847	1	24	75			
Hydrofol Glyceride T-57-N	70% C18	57-61	0.5	1	1 max	193-198	59-64	138-147	10Y/1R					0.846	2	28	70			
Hydrofol Glyceride T-57	70% C18	57-60	7	15	7 max	190-197	58-64	136-147	50Y/5R						2	28	70			
Hydrogenated Castor Oil Hydrofol Glyceride 200	88% 12-Hydroxy C18	70-74	2	4	3 max	177-181	86-88	187-190	50Y/5R	138	155			0.899	2	10				88
Heat Transfer Mediums																				
Hydrofol Tinfat 25		46-50	1	2	11 max	175-185	49-54	120-129				530	600	0.894						
Hydrofol Tinfat 50		49-53	1	2	12 max	187-194	49-55	120-131				585	655	0.854						

Table F3 Properties of hydrogenated coconut and cottonseed oils

Hydrogenated Oil	M.P. °F	Iodine No.	Saponification No.	Reichert-Meisssl No.	Polenske No.	Refractive Index
Coconut	107.2	2.2	250	8.5	15.5	1.4486 (40°C)
Cottonseed	140.7	3.9	195	0.5	0.7	1.4468 (60°C)

Hydrogenated vegetable oils have dielectric constants of 20-25²

¹ Langwill, *Mfg. Confectioner* 21, 15 (1941).

² Valter, *J. Tech. Phys.* (U.S.S.R.) 10, 1970 (1941).

Table F4 Properties of hydrogenated sardine oil and tallow

	45°		53°		Fully Hydro-	
	Incompletely Hydro- generated Sardine Oil Glycerides	Acids	Fully Hydro- generated Sardine Oil Glycerides	Acids	generated Tallow Glycerides	Acids
Titer	—	44-46	—	52-54	—	56-58
Melting Point (open tube)°C.	43	44.5-46.5	54	52.5-54.5	57	56.5-58.5
Iodine Number	27-34	28-35	2-4	2-4	2-6	2-6
Free Fatty Acids as Oleic	2-5%	95-97%	2-5%	95-97%	3-5%	96-98%
Acid Number	4-10	189-193	4-10	189-193	6-10	191-195
Saponification Number	185-189	193-199	185-189	193-199	190-193	199-201
Unsaponifiable	0.8-1.0%	0.8-1.0%	0.8-1.0%	0.8-1.0%	0.6-1.0%	0.6-1.0%
Average Molecular Weight	896	286	896	286	878	280
Specific Gravity 60°/15°	0.873	0.895	0.876	0.854	0.856	0.881
Flash Point °F.	530	405	530	385	515	405
Fire Point °F.	580	440	585	425	595	430
Unsaturated Content	37%	38%	3%	3%	4%	4%
Lovibond* Color	15-35 Y	10-15 Y	10-35 Y	10-15 Y	10-35 Y	10-15 Y
	2.5-5 R	2-3 R	2-5 R	2-3 R	2-5 R	2-3 R
Thiocyanogen No.	No data. Should be same as iodine on 53° and hydrogenated tallow, only slightly lower than iodine on 45°.					

* Lovibond readings on glycerides, 5¼ in. column; fatty acids, 1 in. column.

Table F5 Properties and composition of commercial stearic acids

	<i>Single- Pressed</i>	<i>Double- Pressed</i>	<i>Triple- Pressed</i>
Titer	52.2-53.0°C	53.5-53.9°C	54.6-55.2°C
Melting Point (Open Tube)	52.8-53.6°C	54.1-54.5°C	55.2-55.8°C
Iodine Number (Wijs)	10-14	7-9	2-4
Free Fatty Acids (as Oleic)	103-104%	104-105%	104-105%
Acid Number	205-207	207-209	207-209
Saponification Number	207-209	208-210	208-210
Unsaponifiable Matter	0.50-0.60%	0.20-0.30%	0.10-0.20%
Average Molecular Weight	272-274	269-272	269-272
Specific Gravity 60°/15°	0.853	0.855	0.857
Flash Point	400°F	400°F	405°F
Fire Point	430°F	425°F	425°F
Unsaturated Content	12-16%	8-10%	2-4%
Lovibond Color (Max.)	16 Y-3.0 R	4 Y-1.0 R	2 Y-0.5 R
Lovibond Color (Min.)	5 Y-0.6 R	2 Y-0.2 R	1 Y-0.1 R
Myristic Acid Composition	1-3%	1-3%	1-3%
Palmitic Acid	47-48%	49-50%	46-47%
Stearic Acid	28-33%	34-37%	46-48%
Palmitoleic Acid	trace	trace	nil
Oleic Acid	12-16%	8-10%	2-4%

Properties of Pure Stearic Acid

Melting Point	69.3°C
Iodine Number	0.0
Acid Number	197.5
Refractive Index, 80°C	1.4300
Specific Gravity 69.3°C	0.847
Boiling Point	232°C (15 mm)
Molecular Weight	284.28
Dipole Moment, 25°C	0.77



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