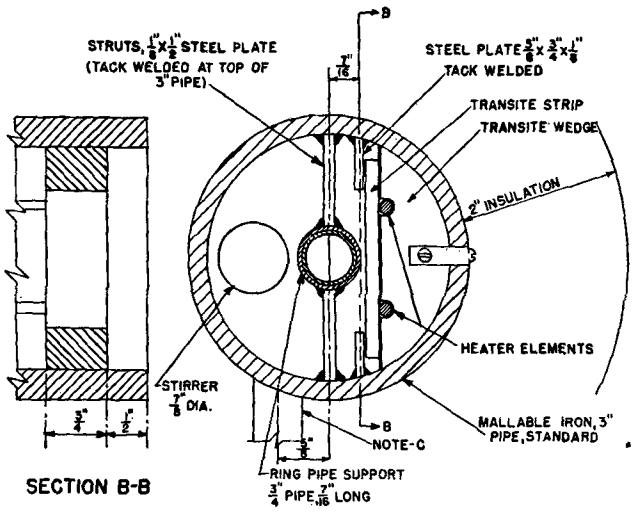

FIG. 4. Top view of oil bath.

the heat-treating portion of the system. This likewise caused intermittent ejection of soapstock that was not uniformly heat-treated. By use of a positive-displacement, diaphragm pump to force the soapstock through the system these difficulties were avoided. Rates were determined by collecting and weighing the heat-treated product.

Table I is a compilation of several test runs in which the soapstock passed through the apparatus once. Total and free gossypol were reduced to values as low as 0.03%. Cottonseed oil soapstock passed through the apparatus with a holding time of approximately 12 min. (attained by recycling treated material) showed a total gossypol content of 0.003%. Reduction of gossypol is dependent upon the holding time in the apparatus, the temperature of the heat treatment, and the type of soapstock being treated (1). Total fatty acid content of the soapstock is unaffected by the heat treatment. It is of some interest to note that the free and total gossypol content approached a common value, following heat treatment, despite initial differences.

Summary and Conclusions

Successful operation of the apparatus, as described in this paper, was conducted at rates of feed that ranged from 1.89 to 5.51 lbs. per hour. The data show that cottonseed oil soapstock can be heat-treated continuously on a pilot-plant scale so that both the free and total gossypol content, as measured by the *p*-anisidine method (2, 3), are reduced to values as low as 0.003%. In the work reported, the heat transfer medium was oil, heated electrically. Commercially a direct, gas-fired, heat exchanger would be more practical. These experiments indicate that additional work on a larger or plant scale is justified, provided, of course, that the marketing economics involved are favorable.

Acknowledgment

The authors take this opportunity to express their appreciation to H. L. E. Vix of the Engineering and Development Section and L. A. Goldblatt of the Oilseed Section for technical assistance and to V. Cirino and H. P. Pastor of the Analytical Section for the analytical data reported.

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[Received July 30, 1956]

Report of F.A.C. Monoglyceride Subcommittee—1956

THE MONOGLYCERIDE SUBCOMMITTEE of the Fat Analysis Committee was established in 1953 for the purpose of selecting a method for the determination of monoglycerides. The determination of monoglycerides is based upon the original work of Malaprade (1) on oxidation of polyalcohols. Fleury and Paris were the first to report on the reaction of periodic acid on glycerol phosphoric acid, a compound similar to the monoglycerides. The first method for fatty acid monoglycerides was reported by Pohle, Mehlenbacher, and Cook (2). This method was improved by Handschumaker and Linteris (3), and further improvement was made a little later by Pohle and Mehlenbacher (4) and by Krutz, Segur, and Miner (5).

Three methods have been tested comparatively by the subcommittee:

1. *Miner Method* (5). The sample is dissolved in a solution of 5% dimethylformamide in chloroform. The total monoglyceride and glycerol are determined on a 25-ml. portion by oxidation with a methanol solution of periodic acid. When the reaction is complete, a solution of sodium bicarbonate and potassium iodide is added, and the liberated iodine is titrated with a standard sodium arsenite solution, using starch indicator. The glycerol is determined by adding 100 ml. of water to the 25 ml. of the chloroform solution, then 25 ml. of an aqueous periodic acid solution. The monoglyceride is calculated from the difference in the titration for total monoglyceride and glycerol and the titration for the glycerol.

TABLE I

Repeated Analysis of the Same Sample

	Mean	Standard deviation	Coefficient of variation
Miner Method			
1954	38.5	0.41	1.07
1955	38.7	0.92	2.37
1955 (known)	38.6	0.30	0.78
1956 (known)	38.6	0.20	0.52
1956	38.7	0.62	1.60
Average	38.6	0.45	1.27
Extraction Method			
1954	38.4	0.65	1.69
1955	38.6	0.44	1.14
1955 (known)	38.2	0.39	1.02
1956 (known)	38.4	0.25	0.65
1956	38.4	0.41	1.06
Average	38.4	0.43	1.11
Partition Method			
1954	38.5	0.17	0.44
1955	38.8	0.63	1.62
1955 (known)	38.8	0.12	0.31
1956 (known)	38.8	0.23	0.59
1956	38.8	0.41	1.06
Average	38.7	0.31	0.80

TABLE II

Average-Collaborative Analyses—1956

	Sample 1		Sample 2		Sample 3	
	Analyst		Analyst		Analyst	
	1	2	1	2	1	2
Miner Method						
Collaborator 1	2.45	2.39	38.5	38.9	92.7	93.3
2	2.41	2.41	38.5	37.9	92.1	91.8
3	2.44	2.46	37.6	39.7	89.2	93.4
4	2.64	....	38.4	....	91.4	....
5	2.36	2.40	38.0	38.0	91.4	90.8
Extraction Method						
Collaborator 1	2.86	2.79	38.3	39.1	91.7	92.1
2	2.87	2.92	38.7	38.3	91.8	91.6
3	2.30	2.72	37.9	37.8	88.7	91.4
4	2.71	....	38.1	....	91.7	....
5	2.84	2.79	38.4	38.2	92.3	91.9
Partition Method						
Collaborator 1	2.91	2.74	38.4	39.2	91.8	91.1
2	2.86	2.80	38.8	38.8	92.4	92.9
3	2.23	2.81	38.8	38.9	89.1	92.9
4	2.90	....	39.1	....	93.0	....
5	2.90	....	39.2	38.5	92.9	92.6

TABLE III

Summary Indicating Over-all Precision—1956 Results

	Mean	Standard deviation	Coefficient of variation
Sample 1			
Miner Method	2.45	0.12	4.9
Extraction Method	2.80	0.17	6.1
Partition Method	2.80	0.18	6.4
Sample 2			
Miner Method	38.5	6.62	1.6
Extraction Method	38.4	0.41	1.1
Partition Method	38.8	0.41	1.1
Sample 3			
Miner Method	91.9	1.36	1.5
Extraction Method	91.5	1.15	1.3
Partition Method	92.2	1.12	1.2

2. *Extraction Method* (4). The sample is dissolved in chloroform, and the glycerol is removed by extraction with water. Periodic acid in an acetic acid-water solution is added. The periodic acid consumed by oxidation of the monoglyceride is determined from an iodimetric titration, using a sodium thiosulfate solution and starch indicator.

3. *Partition Method* (a modification of the extraction method developed in Swift and Company Research Laboratories). The sample is dissolved in chloroform; an equal volume of water is added, shaken, and allowed to separate into two phases; an aliquot of the chloroform solution is pipetted for the monoglyceride determination. The monoglyceride is determined in the same manner as in the extraction method.

A single sample was analyzed by the subcommittee in 1954, using the three methods under trial. The same sample was resubmitted to the subcommittee as an unknown in 1955 and 1956, and duplicate portions were also submitted as "knowns." The purpose of the "known" was to serve as a primary standard for over-all determination. The results obtained on this sample in three consecutive years appear in Table I.

Three additional samples were submitted to the subcommittee in 1955 and again in 1956. The latest results obtained on these samples appear in Table II.

All of the data were statistically processed by H. P. Andrews. The summary appearing in Table IV indicates the 95% probability limits for a number of analytical situations.

TABLE IV

Precision of the Partition Method at Various Monoglyceride Levels

% Monoglyceride present, approx.	2 x Standard deviation	Difference between		
		Single detn. by same analyst on different days	Single detn. by different analysts in the same labs.	Single analyses in different laboratories
3.0	±0.36	0.27	0.51	0.51
40.0	±0.8	0.6	1.2	1.2
90.0	±2.2	2.0	3.2	3.2

After due consideration the committee recommends the Partition Method on the basis of superior precision: more simple than the Extraction Method and more precise than the Miner Method.

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[Received February 27, 1957]

Studies on Castor Oil. II. Hydrogenation of Castor Oil[1]

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THE HYDROGENATION OF CASTOR OIL, also of methyl ricinoleate, has been studied by several investigators (2, 6–13, 15, 20–23). In spite of wide variations in conditions of hydrogenation with respect to type and amount of catalyst, temperature and pressure, practically all the investigators have assumed that the products formed will be essentially esters of 12-hydroxy stearic acid and/or stearic acid; only the relative proportions will be dependent on the external factors. Thus a) at low temperatures and low pressures the rate of hydrogenation is slow; b) at low temperatures and low pressures, saturation of the double bond is the predominant reaction; c) at high temperatures and low pressures, dehydroxylation is marked; and d) at high temperatures and high pressures, decomposition reactions predominate. These conclusions are based mainly on the chemical constants of the hydrogenated product and, in particular, on the iodine and acetyl or hydroxyl values. Only in certain cases have attempts been made to confirm the above conclusions by separating some of the constituent fatty acids. Thus Grün and Woldenberg (5) and Thoms and Deckert (19) isolated 12-hydroxy stearic acid from hydrogenated methyl ricinoleate and hydrogenated castor oil, respectively.

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