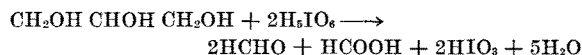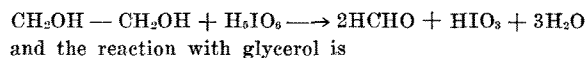# Determination of Glycerol by Oxidation With Periodic Acid

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Malaprade's (1) investigation of the oxidation of polyalcohols with periodic acid and periodates has made available new and additional tools for the analytical and research chemist. The reactions have since been applied to a number of organic compounds. Fleury, et al, studied the action of periodic acid on hydroxy acids and sugars (2) (3) (4), glycerophosphoric acid (5), glycerol in the presence of sugar (6), lactic and pyruvic acid (7) and tartaric acid (8). Hudson and co-workers, (9) (10) (11) have made use of the reaction in their studies of sugars, and Nicolet and Shinn (12) showed that periodic acid reacted rapidly and quantitatively with alpha-amino alcohols. The kinetics of the periodic oxidation of 1, 2 glycols have been investigated by Price and associates (13) (14). Recently Allen, Charbonnier and Coleman (15) have shown that glycerol, ethylene glycol and diethylene glycol could be determined in the presence of each other by the use of the periodic acid and the potassium dichromate methods. Hoepe and Treadwell (16) have also presented a procedure for determining glycerol, ethylene glycol and 1, 2 propylene glycol in the presence of one another.

Previous investigators have shown that at room temperatures periodic acid oxidizes polyalcohols containing two or more adjacent hydroxyl groups, splitting the compound between the carbon atoms attached to these hydroxyl groups. The end hydroxyl groups are oxidized to aldehydes and the inner HCOH groups are oxidized to formic acid. The reaction with ethylene glycol can be represented by the equation —

$$CH_2OH - CH_2OH + H_5IO_6 \longrightarrow 2HCHO + HIO_3 + 3H_2O$$

and the reaction with glycerol is

$$CH_2OH\ CHOH\ CH_2OH + 2H_5IO_6 \longrightarrow$$
$$2HCHO + HCOOH + 2HIO_3 + 5H_2O$$

Trimethylene glycol and other polyalcohols in which the hydroxyl groups are not adjacent are not oxidized at normal temperatures.

In the reaction with glycerol, the betahydroxyl group is oxidized to formic acid and the adjacent groups are oxidized to formaldehyde. Therefore, the amount of formic acid produced is a measure of the glycerol present. The specificity of this reaction suggests that it might be applicable to the determination of glycerol in the presence of substances which interfere with present accepted methods such as the acetin and dichromate procedures. Compounds containing only two adjacent hydroxyl groups, such as ethylene glycol, react with periodic acid but not to produce formic acid. Trimethylene glycol does not interfere since the hydroxyl groups are not on adjacent carbons. Ordinary oxidizable materials, normally associated with glycerin, which would be acted upon by potassium dichromate do not react with periodic acid to give formic acid. The acetin method, in which the sample is acetylized with acetic anhydride, is not specific for glycerol because all alcoholic hy-

droxyl groups are reacted upon by this reagent. The dichromate procedure, while it should be a precise method for pure glycerol, is not so in the presence of other oxidizable substances. Since the periodic acid reaction gave promise of being specific except for impurities which would not normally be associated with glycerin, our object was to attempt to apply it to the determination of glycerol in commercial glycerin, especially glycerin in process such as soap lyes, soap lye crudes, saponification crudes, and sweet waters.

Periodic acid may be titrated directly with sodium hydroxide. A graphical representation of this titration is shown in Figure 1 a. This curve was obtained
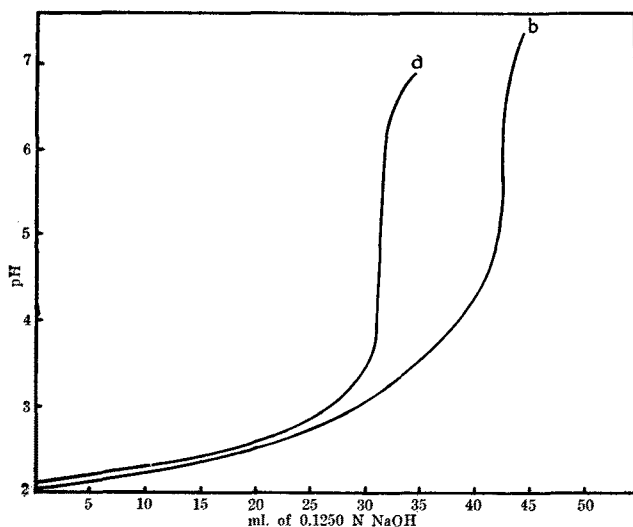

FIG. 1. Potentiometer titration curves of periodic acid and glycerol.

by plotting the data from the titration of 50 ml. of periodic acid solution (approximately 20 gm. per liter) with standard sodium hydroxide, using a glass electrode pH-meter. Examination of this curve reveals a sharp inflection between pH 4.0 and 6.2 with its maximum slope between 4.9 and 5.6. This represents the neutralization of the first hydrogen ion. It has been shown (15) that there is a second inflection at a higher pH value but this has no significance insofar as this problem is concerned. Thus periodic acid may be determined as a monobasic acid by direct titration with sodium hydroxide using methyl red indicator or a glass electrode.

The titration curve of the reaction products resulting from the oxidation of glycerol with periodic acid (Figure 1 b) has a maximum slope at pH 6.0 to 6.3. Because the resulting mixture from this reaction is slightly buffered by salts of iodic acid, formic acid and unreacted periodic acid and because more accurate results are desired than can be obtained with an indicator, it is necessary to use a glass electrode and to titrate both the sample and the blank to a definite

end-point. The glycerol in a sample is calculated from the difference between the amount of alkali required to titrate the reaction mixture and the amount required to titrate a blank. This difference represents the amount of formic acid formed which in turn is equivalent to the amount of glycerol oxidized.

From the titration curve, the end-point for the titration of the oxidized sample was located at pH 6.20. In like manner, the end-point range for the titration of periodic acid was found to be pH 5.1-5.4. Determinations of known samples have indicated that more accurate results are obtained by titrating this blank to pH 5.4.

As in all oxidation reactions, a certain minimum excess of the oxidizing agent is necessary to be sure that the reaction will go to completion. In this reaction results have indicated that there is also a maximum excess of periodic acid which must not be exceeded. The significance of this is best understood by reference to Table I and Figure 2. These data were

TABLE I

Influence of Size of Sample on Glycerol Analyses

| Gm. glycerol taken	Gm. glycerol determined	Ml. of alkali used to titrate formic acid	Ratio of glycerol determined to glycerol in sample
0.0135	0.0169	1.28	1.270
0.0271	0.0306	2.32	1.125
0.0542	0.0567	4.38	1.045
0.0691	0.0709	5.35	1.025
0.0813	0.0830	6.28	1.020
0.1084	0.1090	8.25	1.005
0.1360	0.1359	10.26	1.000
0.1634	0.1628	12.29	0.996
0.1905	0.1891	14.30	0.993
0.2176	0.1894	14.31	0.913
0.2844	0.1729	13.06	0.608
0.7107	0.1263	9.55	0.178
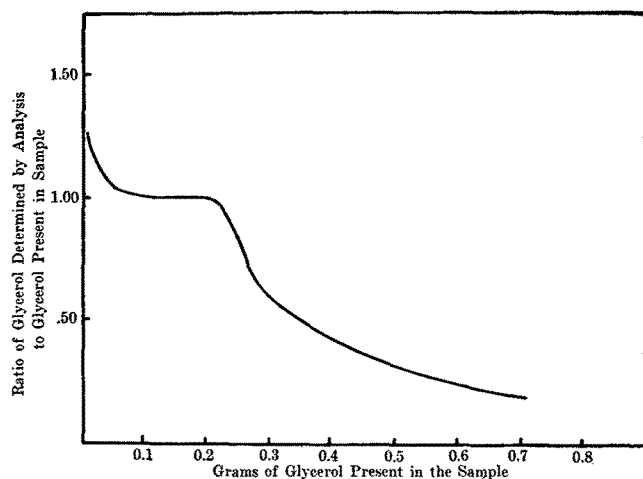
FIG. 2. Influence of size of sample on glycerol analyses.

obtained by varying the size of sample, using a constant amount of periodic acid. In effect, this resulted in varying the amount of excess reagent. It may be observed from the curve that when a sample containing 0.1200 to 0.1500 gm. of glycerol was oxidized with 50 ml. of periodic acid, stoichiometric results were obtained. Therefore the size of sample selected for oxidation should be such as to contain an amount of glycerol between these limits.

When the correct size of sample is taken, the titration of the formic acid produced will be not less than 30 and not more than 40 per cent of the blank titration (10.5 to 13.0 ml. of 0.125 N NaOH). Figure 3

shows that it is possible to obtain false results with a titration within the accepted limits by having a large excess of glycerol present. In such a case, repetition of the determination with a larger sample will give a still lower alkali titration.
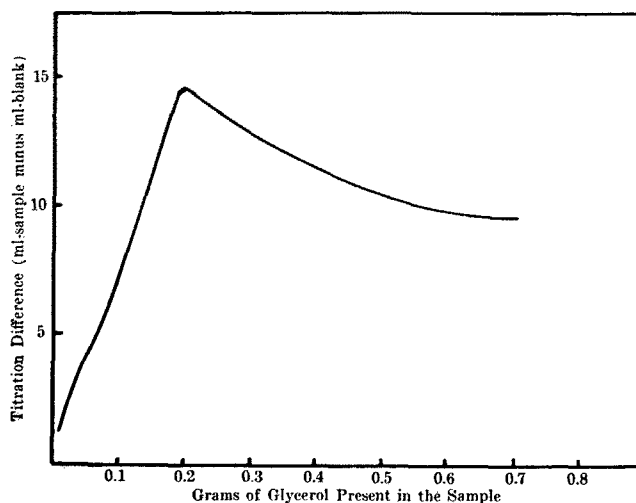

FIG. 3. Influence of size of sample on the titration difference.

Since results are dependent upon an acidimetric titration it is essential that the reaction be started at some definite pH level. This is conveniently done by adjusting to the yellow color of methyl red indicator which corresponds to a pH of approximately 6.2. When the color of the sample solution masks the color change of the indicator, this adjustment is performed with the pH-meter. The pH-meter is also necessary in the case of some samples which contain buffering material.

The per cent of glycerol in commercial C. P. glycerin, as determined by oxidation with periodic acid has been found to agree closely with the per cent of glycerol calculated from the specific gravity, using the Bosart and Snoddy tables (18). Results on several different samples are shown in Table II.

TABLE II

Per cent glycerol as calculated from sp. gr.	Per cent glycerol as determined by oxidation with periodic acid
95.31	95.4
95.31	95.6
95.31	95.2
95.31	95.2
95.31	95.4
95.45	95.5
95.16	95.1
95.16	95.3
95.16	95.1

The quantity of sodium chloride in the sample has to be taken into consideration in the analysis of many samples of glycerin in process, such as spent soap lyes and soap lye crudes, as well as evaporated salts. Salt does not affect the oxidation reaction but its presence does decrease the pH to which the sample must be titrated. The influence of various amounts of salt on the end-point was determined by adding pure sodium chloride to the reaction mixture of glycerol and periodic acid after the usual titration to pH 6.2 and noting the final pH values. These data were checked by adding salt to samples containing known amounts of glycerol and determining the glycerol present, titrat-

ing the samples to the end-point corrected for the amount of salt. The end-point for the alkali titration of solutions containing sodium chloride is given in Table III. Usually the salt content can be estimated sufficiently close for accurate analysis.

To determine the effect of the usual impurities found

TABLE III

The End-Point for the Alkali Titration of Solutions Containing Sodium Chloride

Grams of Sodium Chloride in the sample titrated	pH to which the titration should be carried
0.0	6.20
0.1	6.20
0.2	6.19
0.3	6.17
0.4	6.16
0.5	6.15
1.0	6.08
2.0	6.02
4.0	5.96
6.0	5.85
10.0	5.75
15.0	5.63
20.0	5.54
25.0	5.46

in spent soap lyes, glycerol-free synthetic lyes were prepared to which known amounts of glycerol were added. A mixture of approximately one part tallow and one part yellow grease was saponified with aqueous sodium hydroxide and then acidified with sulfuric acid. The fatty acids thus liberated were extracted with petroleum ether and washed several times with water to remove all glycerol and sulfuric acid. The petroleum ether was then removed by evaporation and the fatty acids resaponified. The soap thus produced was salted out (grained) from the aqueous solution with sodium chloride. Small amounts of protein, lykopon, and lactic acid were added to the lye removed as additional impurities. Similar lyes were prepared starting with coconut oil. Further treatment was given to portions of these lye samples so as to be comparable with processing operations. Ferric chloride and hydrochloric acid were added after which the lyes were filtered, made alkaline with sodium hydroxide, and refiltered. Known percentages of glycerol were then added to each of these portions. Finally, the glycerol was determined with periodic acid. The results appear in Table IV.

TABLE IV

Determination of Glycerol in Prepared Soap Lyes

Sample	Per cent glycerol added	Per cent glycerol found
Untreated Lyes:		
From grease and tallow soap	4.53	4.54
From grease and tallow soap	4.25	4.24
From cocoanut oil soap	4.53	4.53
Lyes After Treatment With Ferric Chloride and Hydrochloric Acid:		
From grease and tallow soap	4.28	4.27
From grease and tallow soap	4.22	4.21
From cocoanut oil soap	4.28	4.27
From cocoanut oil soap	4.22	4.21
Treated Lyes, Filtered, Made Alkaline and Refiltered:		
From grease and tallow soap	4.28	4.27
From grease and tallow soap	4.22	4.23
From cocoanut oil soap	4.28	4.27
From cocoanut oil soap	4.22	4.20

To check the effect of some of the usual impurities found in crude glycerin, known percentages of glycerol were added to trimethylene glycol (Tech. grade E. K. Co.). This sample of glycol contained 0.93 per cent glycerol as determined with periodic acid. This was taken into account in the calculation of the glycerol

present in the sample. The glycerol was determined and results appear in Table V. We have no explanation for the fact that the determined glycerol was 0.2 to 0.3% lower than the glycerol present in each case, however, there is no reason to believe that it is in any way due to the trimethylene glycol.

TABLE V

Determination of Glycerol in the Presence of Trimethylene Glycol and Polyglycerols

	Per cent glycerol	
	Present	Determined
Trimethylene Glycol	......	0.93
Trimethylene Glycol + Glycerol	7.3	7.0
Trimethylene Glycol + Glycerol	13.1	12.9
Trimethylene Glycol + Glycerol	25.7	25.5
Trimethylene Glycol + Glycerol	36.3	36.0
Trimethylene Glycol + Glycerol	71.6	71.3
Polyglycerol + Glycerol	93.8	93.8
Polyglycerol + Glycerol	75.7	76.0

Glycerin foots may be assumed to contain large portions of polymerized glycerol. The determination of glycerol in this residue without treatment has not been found to be satisfactory due to the presence of substances which have a very marked buffering action. To remove some of the buffering material, glycerol foots were dissolved in water, acidified with hydrochloric acid, filtered, treated with an excess of calcium hydroxide, refiltered and evaporated under reduced pressure. The residue was extracted with alcohol and the alcohol removed by evaporation. The alcohol-soluble material contained 8.5% glycerol as determined with periodic acid. The glycerol was then removed in a manner similar to the A. O. C. S. procedure for the determination of total residue (17). In this way the glycerol content of this material was reduced to 0.9%. The remainder could be assumed to be predominantly polyglycerols. To these polyglycerols were added known percentages of glycerol and the glycerol determined with periodic acid. These results appear in Table V. In the determination of glycerol in the presence of polyglycerols it must be remembered that although periodic acid will not oxidize polyglycerols with the formation of formic acid some periodic acid is reduced by any adjacent hydroxyl groups of the polyglycerols.

In the event that the polyglycerols present in any sample are of such nature or such amount that they reduce the periodic acid below that concentration necessary for the stoichiometric conversion of the glycerin to formic acid, the method is not satisfactory.

Method. Reagents and Solutions:

Periodic Acid Solution: Dissolve 20 gm. of periodic acid in one liter of water. If the solution is not clear filter through sintered glass filter. Store the solution in a dark, glass stoppered bottle. The oxidizing power of this solution decreases slowly with time. A blank must be run each day analyses are made.

Sodium hydroxide 0.1250 N. Standardize with potassium acid phthalate using phenolphthalein indicator.

Methyl red. Dissolve 0.1 gm. in 100 ml. 95% alcohol.

Sodium hydroxide: Approximately 0.05 N.

Sulfuric acid: Approximately 0.2 N.

Apparatus:

    50 ml. burrette, accurately calibrated

    50 ml. pipette, accurately calibrated

    Variable speed stirrer

    Glass electrode pH-meter.

Procedure:

The amount of excess periodic acid in the glycerol-periodic acid reaction is critical. Therefore it is necessary to know the approximate concentration of the sample to be analyzed. Some-

times trial tests must be made to locate the approximate range. Refer to Table VI for the size sample to be weighed.

All weighing must be accurately and rapidly made.

Weighings are conveniently made in a small beaker, pouring from this into a flask or beaker.

If the glycerin content is 30-100% weigh into a 2-liter volumetric flask, make to volume with distilled water, mix well, pipette 50 ml. into 600-ml. beaker, and cover with watch glass.

If glycerin content is 10-30% follow the same procedure, using 500-ml. volumetric flask instead of a 2-liter.

If the glycerin content is below 10% pour directly from weighing beaker into 600-ml. beaker, add about 50 ml. distilled water, and cover with watch glass.

Add one drop of the methyl red indicator to the sample in the 600-ml. beaker, acidify with the 0.2 N sulfuric acid. Neutralize with the 0.05 N sodium hydroxide to the yellow color which corresponds with a pH of about 6.2. If the color of this solution interferes with the detection of the color changes of the indicator, use the pH-meter and in this case adjust to the pH to which the final titration is to be carried. Add with a pipette 50 ml. periodic acid, shake gently to effect thorough mixing, cover with a watch glass and allow to stand for one hour at room temperature. At the same time prepare a blank containing only 50 ml. water, with no glycerol, and run along with the sample.

After allowing the sample to stand for one hour dilute to 240-250 ml. and titrate with the 0.125 N sodium hydroxide using a glass electrode pH-meter to determine the end-point. The sample should be agitated with a stirrer during the titration. The blank is titrated to a pH of 5.4 and the sample to a pH of 6.2. If the actual fraction of sample being titrated contains more than 0.1 gm. salt, titrate to the corrected end-point shown in Table III.

When the titration approaches the end-point add the alkali in increments of 0.1 ml. to and past the equivalence point. Record the volume of sodium hydroxide and the corresponding pH when within 0.1-0.2 ml. of the end-point and do likewise after passing the end-point. From these data calculate the actual alkali required to titrate to the proper end-point.

Calculation of end-point:

V_1 = volume of alkali before end-point — pH_1 corresponding to V_1

V_2 = volume of alkali after end-point — pH_2 corresponding to V_2

V_x = volume of alkali at end-point — pH_x corresponding to V_x and obtained from Table II

$$V_x = V_1 + (V_2 - V_1) \frac{pH_x - pH_1}{pH_2 - pH_1}$$

Calculation of per cent of glycerol:

X = ml. sodium hydroxide to titrate sample
Y = ml. sodium hydroxide to titrate blank
N = normality of sodium hydroxide
W = weight of sample

% glycerol equals:

$$\frac{(X - Y) \times N \times 0.09206 \times 100}{W}$$

Notes on Method:

1. If the sample contains an appreciable amount of buffering material always adjust the pH with pH-meter to the end-point to which the sample will be titrated. In some instances the buffering action may be sufficiently great to tend to prevent good reproducibility of results.

2. In order to produce accurate results extreme care is necessary in all manipulations. The burette should be read to 0.01 ml., using a magnifier. This analysis must be carried out in an atmosphere free from acid or alkaline vapors. A suitable temperature is from 25 to 31° C.

3. When analyzing samples of salt containing more than 2% glycerol, dissolve in 50 ml. of water. For samples containing less than 2% glycerol dissolve in 100 ml. of water.

4. Use 25 gm. of salt for all samples containing less than 0.5% glycerol.

5. Cork should not be used to stopper any of the flasks used in this determination or in any other way be allowed to come in contact with any of the materials used in or for the analysis.

6. The titration for the formic acid (titration of the sample minus the titration of the blank) must be not less than 30% and not more than 40% of the blank titration. This is to insure the proper ratio of periodic acid to sample, that is, the correct excess of reagent.

Very careful manipulation is required throughout the periodic acid method. This is readily appreciated when it is realized that the glycerol in the sample is equivalent to approximately 10 ml. of the standard alkali and that 0.01 ml. of the standard alkali is equivalent to 0.1% of the actual glycerol. The determination must be carried out in an atmosphere free from acid or alkaline vapors which might be absorbed and influence the titration. In some cases the buffering action of some samples, especially glycerin foots, has been found to be so great that it was impossible to obtain accurate results.

The determination of glycerol in widely varying samples has been found to agree within 0.4% or less of the actual glycerol present. The glycerol content of crude glycerins as determined by the periodic acid method has, in general, been found to be higher than that as determined by the acetin procedure.

In all of this work the criterion of purity of the known samples has been the specific gravity determination of chemically pure glycerin (18) which in turn was checked by moisture determination using the Fischer Volumetric Method (19) (20).

This method has and is serving the very excellent purpose of making possible the accurate determination of glycerol in the presence of impurities which interfere with other procedures. The authors do not propose to submit it as a substitute for either the dichromate or acetin methods but rather as an added tool to fill a previous gap.

Summary

An acidimetric procedure for the determination of glycerol following oxidation with periodic acid has been described. This procedure has been successfully applied to C. P. grade glycerin, spent soap lyes, soap lye crudes, half crudes, saponification crudes, evaporator salt, and mixtures of glycerol with trimethylene glycol and polyglycerols. Factors that influence the determination, such as size of sample, presence of salt and the presence of organic compounds containing hydroxyl groups, have been discussed.

TABLE VI

Weight of Sample to be Taken for Analysis Based Upon the Glycerin Content

Per cent glycerol in product to be analyzed	Size of sample to be selected if entire sample is used for analysis		Size of sample to be selected if same is to be diluted to 2 liters and 50 ml. withdrawn for analysis	Size of sample to be selected if same is to be diluted to 500 ml. and 50 ml. withdrawn for analysis
100	.1200 to	.1500	4.8 to 6.0	
90	.1330 to	.1670	5.3 to 6.7	
80	.1500 to	.1880	6.0 to 7.5	
70	.1720 to	.2180	6.9 to 8.6	
60	.2000 to	.2500	8.0 to 10.0	
50	.2400 to	.3000	9.6 to 12.0	
40	.3000 to	.3750	12.0 to 15.0	
30	.4000 to	.5000	16.0 to 20.0	
25	.4800 to	.6000		4.0 to 5.0
20	.6000 to	.7500		4.8 to 6.0
15	.8000 to	1.000		6.0 to 7.5
10	1.200 to	1.5		8.0 to 10.0
8	1.5 to	1.88		12.0 to 15.0
7	1.72 to	2.18		15.0 to 18.8
6	2.0 to	2.5		
5	2.4 to	3.0		
4	3.0 to	3.75		
3	4.0 to	5.0		
2	6.0 to	7.5		
1	12.0 to	15.0		
0.9	13.3 to	16.7		
0.8	15.0 to	18.8		
0.7	17.2 to	21.9		
0.6	20.0 to	25.0		
0.5	24.0 to	30.0		
0.4	30.0 to	37.5		
0.3	40.0 to	50.0		
0.2	60.0 to	75.0		

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Smoke, Fire, and Flash Points of Cottonseed, Peanut, and Other Vegetable Oils*

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Introduction

The smoke, fire, and flash points of a fat or oil are measures of its thermal stability. Their relation to thermal stability may be stated as follows: (a) The *smoke point* is that temperature at which appreciable decomposition begins, when a body of the oil is heated in contact with the atmosphere. It is the temperature at which volatile decomposition products are evolved in sufficient quantity for them to become visible. (b) The *flash point* is that temperature at which the decomposition products are evolved in such quantity as to be capable of ignition but not of continued combustion. (c) The *fire point* is that temperature at which decomposition products are evolved in such quantity and at such a rate that they will support continued combustion.

In any operation involving the heating of an oil to a high temperature, the thermal stability of the oil is a matter of manifest importance. The degree to which an oil or fat may be heated without undergoing undue breakdown or evolution of volatile substances determines not only the temperature range within which it may be effectively used, but also the fire hazard attendant upon its use. Among the commercial operations which require the use of fats or oils and which are conducted at temperatures within the range of their thermal instability, are deep fat frying of food, the manufacture of "bodied" oils, varnishes and similar products, and the manufacture of tin plate, and of tin and terne-plated articles.

Since both cottonseed and peanut oils have long been prized for their edible qualities, their industrial use has for many years been insignificant. Consequently, little interest was attached to their high temperature behavior except under the conditions of food frying. Recent dislocations in the world supply of fats and oils, however, have given a new interest to the possible technical uses of these and other domestic oils. It does not seem likely that oils of foreign origin will be available in this country in any quantity for an extended period of time. As a consequence, the more expensive, domestically produced edible oils, such as cottonseed and peanut, may be expected to find increasing use in the future in technical or non-edible fields.

From time to time, proponents of various cooking oils have made claims of superiority of one oil over another with regard to its smoke point. Olive oil has long been widely used for cooking purposes and one of the chief claims made for it was its high smoke point. Later, cottonseed oil came into general use for deep fat frying and again one of its chief advantages was said to be its high smoke point. More recently, peanut oil has appeared on the market in appreciable volume and again a high smoke point has been advanced as one of its principal virtues.

It has, however, been generally known that the smoke point of any fat or oil is dependent on the degree of refinement of the product, and especially on its freedom from fatty acids (1). In fact, it has been considered that the initial free fatty acid content of a fat or oil, and the rate at which additional free fatty acids are formed under conditions of use, are the determining factors affecting the smoking temperature of a fat or oil.

A survey of the literature revealed little pertinent information on the smoke, flash, and fire points of peanut oil, and relatively few references to the smoke, flash, and fire points of cottonseed oil. Blunt and Feeney (1) mention two smoke points for peanut oil, namely, 325° F. and 300° F., but they make no mention of the flash or fire points of this oil. Dickhart (3) reported the determination of the smoke point (464° F.), flash point (632° F.), and fire point (692° F.) on a single sample of peanut oil.

No data on the smoke, flash, and fire points of peanut oil are reported in the monographs by Jamieson (6) and Lewkowitsch (8), or in the International Critical Tables (5). Detwiler and Markley (2) did not include peanut oil in the series of oils which they examined with respect to the smoke, flash, and fire points.

The data on cottonseed oil are somewhat more extensive. The International Critical Tables record the flash and fire points of cottonseed oil as 582° F. and 644° F., respectively. Jamieson (6) and Lewkowitsch (8) report a range of temperatures for these properties. Vollertsen (12) also reported data on the smoke points of various oils, including cottonseed. None of these authors reported the determination of the free fatty acid content of the same sample of oil on which the smoke, flash, and fire points were determined.