according to the official method for determining unsaponifiable matter (1). The four extracts were combined to give two equal samples, which were then evaporated to dryness and each dissolved in 50 ml. of 90 per cent ethanol, a sufficient volume to assure solution. A digitonin solution (0.4-g. digitonin in 40 ml. of 90 per cent ethanol) was added to each unsaponifiable fraction and the mixture allowed to stand at room temperature for 24 hours. The crystalline digitonide was filtered on a sintered glass crucible under suction.

TABLE IX
Yield of Ragweed Oil Sterols

Wt. Oil (g.)	Digitonide (g.)	Sterols (g.)	Yield (Per cent by wt. of oil)
10.027	0.1795	0.0460	0.46
10.004	0.1909	0.0489	0.49

The yield obtained corresponds closely to that from soybean oil, suggesting ragweed oil as a potential source of sterols. Since stigmasterol is important in the syntheses of certain sex hormones, the possible presence of this sterol was investigated by brominating (10) the prepared acetates of ragweed sterols. To 0.301 g. of sterol acetate dissolved in 3 ml. of ethyl ether were added 3.8 ml. of a 5 per cent bromine in acetic acid solution. Upon standing for two hours 24 mg. of crystals were filtered. They were recrystallized twice from chloroform-methanol (methanol added to the chloroform solution until turbidity occurred); these plates melted from 190-191.5° C. Further purification was impossible because of a lack of sufficient material. Although stigmasteryl acetate tetrabromide melts slightly above 200° C., the crystals melting from 190-191.5° C. seemed to be impure stigmasteryl acetate tetrabromide or the tetrabromide of a related steryl acetate.

Conclusions

Ragweed seed contains approximately 19 per cent fat and 23 per cent protein. Large quantities of these seed can be readily obtained both from direct harvesting of the ragweed and from the cleaning of some commercial seeds.

The fatty acid distribution in ragweed seed oil is as follows: palmitic acid—5.5 per cent; stearic acid—4.8 per cent; oleic acid—19.9 per cent; linoleic acid—69.8 per cent; linolenic acid—possibly traces. The composition of this oil indicates that it would have slightly better drying properties than soybean oil. The results of preliminary drying and heat-bodying experiments suggest the limited use of ragweed seed oil in paints and varnishes.

No investigation has been made of the edible properties of ragweed seed oil but its relative freedom from linolenic acid indicates its use in the edible field.

Ragweed seed oil contains about 1.2 per cent of a wax mixture which is made up of 55 per cent hydrocarbons, 23 per cent high molecular weight acids, and 22 per cent high molecular weight alcohols. Sterols occur in ragweed seed oil to the extent of 0.48 per cent of the weight of the oil. The unsaponifiable matter also contains high molecular weight hydrocarbons and alcohols. Pure mixed sterols were separated from the accompanying materials by the use of an adsorption process. Bromination of the acetates of the mixed sterols gave evidence for the presence of stigmasterol.

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Determination of Carbon Dioxide in Soap and Soap Products by Loss in Weight

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Introduction

A large number of different forms of apparatus have been suggested for the gravimetric determination of carbon dioxide by loss in weight. The alkalimeter devised by Schrötter * combines simplicity of design with certain advantages that render it particularly suitable for the determination of combined carbon dioxide in soaps. Up to the present time none of the numerous alkalimeters described in the literature have been employed in the analysis of the latter product, their main use being in the analysis of baking powders and limestone. The following paper describes the application of the Schrötter alkalimeter to the analysis of combined carbon dioxide in soaps and soap products. It is quite possible that various of the other types of alkalimeters would prove suitable for use in this determination if it were attempted to employ them.

Principle

A weighed amount of the sample is placed in the clean, dry alkalimeter and 5 ml. of 1:2:4 trichlorbenzene added (1) (2). The trichlorbenzene is added to dissolve the fatty acids liberated in the course of the determination thus preventing bumping. Next dilute hydrochloric acid and concentrated sulfuric acid are placed in the appropriate bulbs and the apparatus is weighed. The hydrochloric acid is then allowed to flow down on the sample. The evolved gas passes through the strong sulfuric acid which

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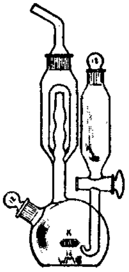
* Despite a diligent search of the literature the original description of this type of alkalimeter could not be found.

absorbs any accompanying moisture. After the carbon dioxide evolution has subsided the apparatus is placed on a hot plate and dry air slowly aspirated through the apparatus while the contents are brought to the boiling point. After cooling, the apparatus is again weighed and the loss of weight is taken as the carbon dioxide content of the sample.

Apparatus

The Schrötter alkalimeter is illustrated in various textbooks (3) (4). A slight modification of the apparatus facilitates the determination of carbon dioxide if there are a large number of samples to be analyzed. This modification consists in providing the top of the funnel designed to hold the hydrochloric acid with an inlet tube which can be connected to or sealed from the remainder of the apparatus by turning a hollow glass stopper similar to the manner in which glass stoppered U-tubes are fabricated and utilized. No further modifications of the apparatus are required or desirable.



Procedure

Thoroughly clean the alkalimeter with cleaning solution followed by water and finally alcohol. Draw a stream of dry clean air through the apparatus until it is thoroughly dry. Allow to stand for fifteen minutes to come to moisture and temperature equilibrium with the surrounding atmosphere.

Weigh the empty and clean alkalimeter. Remove from the balance and add 1 to 5 grams of the sample depending upon the expected sodium carbonate content. Table I will indicate an approximation of the weight of sample to be employed. Reweigh the alkalimeter to obtain the weight of the sample.

Add sufficient concentrated sulfuric acid to just cover the outlet of the vapor trap and thoroughly dry the ground surface at the top of the trap before inserting the small outlet tube plug. Add 5 ml. of 1:2:4 trichlorbenzene to the sample through the hydrochloric acid funnel. Close the stopcock and add 8 to 10 ml. of 1:1 hydrochloric acid to the acid funnel. Carefully wipe the ground surfaces with filter paper before inserting the glass stopper. Weigh the apparatus.

If the alkalimeter has been modified as described under apparatus connect the outlet tube to a drying tube filled with calcium chloride, to admit dry air to the apparatus, and allow the hydrochloric acid to come in contact with the sample by turning the stopcock in the funnel. Otherwise remove the ground glass stopper and replace with a rubber stopper and tube connected to a drying tube filled with calcium chloride before opening the stopcock and permitting the acid to come in contact with the sample.

Connect the vapor trap side of the apparatus to a vacuum line and adjust the vacuum to draw a steady stream of air bubbles through the concentrated sulfuric acid.

Place the alkalimeter on a hot plate and slowly heat until the soap is decomposed and the fatty acids are in solution with the trichlorbenzene. Occasionally swirl the contents of the alkalimeter to hasten liquefaction and solution of the fatty acids.

Finally bring the contents of the apparatus to the boiling point and immediately remove from the hot plate.

Continue aspiration for 15 minutes, disconnect the apparatus, allow to stand until cooled to room temperature and weigh.

The loss in weight (CO_2) may be calculated to any desired form of combination, usually sodium carbonate. In the latter case the calculation is as follows:

$$\frac{\text{Loss in weight} \times 2.4090 \times 100}{\text{weight of sample}} = \%\ Na_2CO_3$$

$$\%\ Na_2CO_3 \times 0.4151 = \%\ CO_2$$

$$\%\ Na_2CO_3 \times 0.5849 = \%\ Na_2O.$$

Results

The following tabulation (Table I) gives the results obtained in the analysis of various types of soap and soap products.

TABLE I

Type of Product	Weight of Sample	Analyst	% Na_2CO_3	% Na_2CO_3
			Alkalimeter Procedure	A.O.C.S. Train Method
	grams			
Washing Powder	2.2361	Boggie	41.80	41.75
Sprayed Soap	1.9622	Moore	4.65	4.78
Sprayed Soap	1.7330	Moore	9.38	9.64
Laundry Bar Soap	1.9632	Moore	2.18	2.10
Naphtha Bar Soap	3.7430	Moore	2.51	2.27
Sprayed Soap	5.4177	Peluso	1.70	1.93
Sprayed Soap	4.9307	Peluso	4.48	4.50
Sprayed Soap	5.6362	Peluso	1.99	1.96
Laundry Bar Soap	5.6876	Peluso	2.30	2.10

TABLE II

Determination No.	Weight of Sample	% Sodium Carbonate	Deviation
	grams		
1	4.0582	4.17	—0.053
2	4.0978	4.27	+0.047
3	4.4717	4.14	—0.083
4	3.6455	4.27	+0.047
5	5.5566	4.19	—0.033
6	4.5606	4.22	—0.003
7	6.1966	4.28	+0.057
8	5.2548	4.27	+0.047
9	5.4632	4.19	—0.033
10	5.2841	4.23	+0.007
		Mean 4.223	0.0410 Mean Deviation

$$\text{Average deviation of the mean is } \frac{0.0410}{\sqrt{10}} = \pm\ 0.013$$

$$\text{Most probable }\%\ Na_2CO_3 = 4.223 \pm 0.013.$$

Reproducibility of Results

The following tabulation (Table II) gives results obtained in the analysis of a sprayed soap. Eleven determinations were run by the same analyst and one determination was omitted in the tabulation as falling beyond the limits of the experimental error. The same sample gave a result of 4.23 per cent carbon dioxide by the standard train method of the A.O.C.S.

Conclusions

The method described above is rapid and gives reproducible values closely approximating those obtained by the gravimetric train method. It is not recommended as a substitute for the train method in accuracy but the procedure is admirably suited for either small laboratories that are not equipped with carbon dioxide trains or for the purpose of making quick checks.

The results tabulated in Table II show that a high order of precision is obtainable by an analyst skilled in the technic of the method. Table I shows what can be expected when the method is given to analysts previously unacquainted with the procedure. An inspection of the results given shows that satisfactory checks between the train and alkalimeter method were obtained in all cases.

Acknowledgments

The authors are indebted to Messrs. Boggie, Peluso, and Moore for their assistance in obtaining the analytical results incorporated in Table I.

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Report of the Soybean Analysis Committee 1941-42

In the 1941 report of the Soybean Analysis Committee (1), it was pointed out that the determination of moisture in biological materials is a purely empirical procedure, governed by three variables—temperature, pressure, and time—and that in determining moisture by oven loss-in-weight methods it is necessary to learn what combination of these three may be used to give the most valid results without the results being influenced by oxidation or decomposition or both. It has been experimentally demonstrated that a reliable referee procedure for the determination of moisture in ground soybeans (1,2) and soybean meals (1,3) is to dry the samples for 6 hours at 105°C. in a vacuum oven maintained at less than 25 mm. Hg pressure. It has also been shown that results agreeing with this vacuum oven procedure are obtained by heating for 2 hours at 130°C. in an air oven and by heating for 1 hour at 130°C. in a vacuum oven at less than 25 mm. Hg pressure. The time factor must be strictly observed in the latter procedure.

The collaborative work conducted by the committee was for the purpose of comparing these and some other oven loss-in-weight methods for the determination of moisture in soybean meals.

One sample of each of the four types of soybean meals was ground to pass a 1 mm. sieve in a Wiley mill and was sent to each of ten collaborators with the request that the moisture be determined by six temperature, time, and pressure combinations after the sample had been exposed to assume the equilibrium moisture content of the laboratory. The procedure specified was as follows:

Weigh a 2-gram sample into a tared aluminum moisture dish (A.O.C.S.), 2 inches in diameter and ¾ inches high, fitted with a close-fitting cover. Remove cover, place in oven, and dry under specified oven conditions. The temperature of the oven is to be taken at the level of the samples. Remove pan from oven, cover, cool in desiccator for 15 or 20 minutes, and weigh. Calculate loss in weight as moisture. The specified conditions of drying were:

Method

No. 1. Regular laboratory procedure of collaborator
No. 2. 105°C. for 3 hours in air oven
No. 3. 130°C. for 1 hour in air oven
No. 4. 130°C. for 2 hours in air oven
No. 5. 105°C. for 6 hours in vacuum oven, less than 25 mm. Hg
No. 6. 130°C. for 1 hour in vacuum oven, less than 25 mm. Hg

The regular procedures used by the various collaborators were found to be quite different. Their reports indicated the following temperature, time, and pressure combinations:

Collaborators

No. 1. 130°C., 40 minutes in air oven
No. 2. 110°C., 18 hours in air oven
No. 3. 100°C., 2½ hours in vacuum oven at less than 50 mm. Hg
No. 4. 100°C., 5 hours in vacuum oven at less than 25 mm. Hg
No. 5. 108°C., 2 hours in air oven
No. 6. 135°C., 2 hours in air oven
No. 7.
No. 8. 100°C., 5 hours in vacuum oven at less than 25 mm. Hg
No. 9. 130°C., 1 hour in air oven
No. 10. 130°C., 3 hours in air oven

Collaborators 1 and 6 used forced draft air ovens, while all others used air ovens of the conventional type. The vacuum ovens used were of the externally-heated vacuum chamber type with the exception of the one used by collaborator 9 who used an oven having an internal hot plate. The low pressure desired in the vacuum ovens was not maintained in all cases with the equipment on hand.

The purpose of the study was to compare the temperature, time, and pressure combinations in determining the moisture in the meal samples and not to check the technique of the collaborators. There was considerable variation in the relative values obtained by the collaborators with the several methods. These variations may be due in part to ovens and thermometers. Collaborator 9 used a vacuum oven with an internal hot plate and obtained low results. Some collaborators did not have equipment that permitted the low vacuum pressure desired.

It was hoped that with uniform technique, including time, temperature, and pressure control, the average values obtained by each collaborator for each