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dition the Society has about \$700.00 worth of standard reagents on hand, but less than \$50.00 with which to start the next year's work. If the new committee is to have any funds for advertising it will be necessary either to sell more of the standard reagents or borrow from the Society's treasury.

We used to think we realized the great service Dr. Smalley was rendering the Society in handling this work, but no one who has not had the actual responsibility can appreciate the size of the job he unselfishly and efficiently performed. So long as this co-operative check work is continued by our Society it will be remembered by our older members as a memorial to Dr. Smalley. That it may always be linked with his personality even in the years to come when those who loved him are gone, it is suggested that this henceforth be known as the Smalley Foundation.

SEPARATION AND DETERMINATION OF SATURATED AND UNSATURATED FATTY ACIDS BY MEANS OF THE LEAD SALT-ETHER METHOD.

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Chemists apparently avoid the separation and determination of the saturated and unsaturated acids of fats and oils, probably because these determinations seem tedious when compared with those usually made in the routine examination of an oil or fat, such as saponification value and iodine number. The information to be gained from these determinations and from the iodine number of the unsaturated acids, however, is of great assistance in establishing the identity of an oil or fat because these values are more characteristic for certain oils than some of the other values usually determined, such as saponification value, specific gravity and refractive index, as an inspection of the table will show.

The lead salt-ether method for effecting this separation was first proposed by Gusserow¹ and Varrentrapp². Many other investigators have since suggested modifications and changes, but it still seems to be the general impression that the results thus obtained are not very accurate.

¹Liebigs Annal., 27, 153 (1828).

²Ibid., 35, 197 (1840).

This method is based on the solubility of the lead salts of the liquid unsaturated acids, oleic, linolic, linolenic and clupanodonic, in ether, in which the lead salts of the solid saturated acids, myristic, palmitic, stearic, arachidic, lignoceric, etc., are practically insoluble.

Satisfactory results cannot be obtained with coconut, palm kernel and similar oils, which contain lauric, capric, caprylic and caproic acids, because the lead salts of these acids are appreciably soluble in ether.

The authors, who have had a wide experience with this method during the past several years, have developed several modifications which it is believed improve the results. It is impossible to effect a complete separation. The saturated acid fraction always absorbs iodine, showing the presence of unsaturated acids. There is also the possibility of the results being vitiated by the slight solubility in ether of the saturated acid lead salts but this can be avoided by not excessively washing the precipitated lead salts of the saturated acids.

To test the purity of the unsaturated acid fractions, 50 g. samples, prepared from various oils, including peanut and corn oils, were solidified by immersion in a freezing mixture. When completely solidified they were placed in an ice box and allowed to liquify slowly. The small amount of flocculent precipitate that remained was removed by quickly filtering through a pre-cooled filter, using suction. The melting points proved that the precipitates were saturated acids but in no case did they weigh more than 2 or 3 mg.

Reducing the washing, of course, increases the amount of unsaturated acids remaining with the saturated acid fraction. However, if the assumption that the precipitated lead salts of the saturated acids exert no selective attraction for the lead salts of the unsaturated acids is correct, this amount can be calculated from the iodine numbers of the saturated and unsaturated acid fractions and the proper correction made. It is important, therefore, to determine the iodine numbers of the two fractions, first, in order to make this correction and, second, because the iodine number of the unsaturated acid fraction is an important characteristic and indicates what unsaturated acids are present. In the literature are found widely varying values for the saturated and unsaturated acid contents of oils and in a great many cases the iodine number of the saturated acid fraction is not given; consequently there is no way of knowing how complete the separation was, and the results are not comparable. The procedure recom-

DATA ON THE DETERMINATION OF SATURATED AND UNSATURATED FATTY ACIDS.¹

	Iodine Number of Oil	Saturated Acids Per Cent.	Unsaturated Acids Per Cent.	Iodine Number of Unsaturated Acids
Cottonseed Oil ²	103—111.2	20.9—25.3	69.7—73.9	144.2—148.0
Peanut Oil ³ (Runner Peanuts).....	89—90.2	15.8—16.5	79.0—79.5	109.0—113.2
Peanut Oil ⁴ (Spanish Peanuts).....	93.1—94.5	18.7—20.4	75.0—76.6	120.2—125.8
Corn Oil ⁵	117.2	11.2	82.5	137.2
Soya Bean Oil.....	128.0	11.4	84.1	148.2
Sunflower Seed Oil	130.8	7.1	87.8	147.5
Rapeseed Oil ⁶	92.5—101.3	0.1—1.4	94.1—95.4	100.5—105.1
Mustard seed Oil ⁷	98.4—110.4	1.0—4.0	91.5—94.5	103.1—119.8
Linseed Oil	179.5	3.9	91.6	196.0
Olive Oil ⁸ (California)	78.5—89.8	13.0—20.0	82.5—93.5	88.9—96.6
Olive Oil ⁹ (Italian)	79.2—86.1	5.0—17.7	77.8—90.5	89.8—98.4

¹ The analyses of the first six samples were made by the authors. The figures for the percentages of sat. and unsat. acids are the corrected figures as explained in the text. The analyses of the last five samples were taken from Bureau of Chemistry Bulletin No. 77 by Tolman and Munson. The figures for sat. and unsat. acids have not been corrected.

² 41 samples.

³ 6 samples.

⁴ 5 samples.

⁵ This sample also contained 1.7% unsaponifiable matter having an iodine number of 113.5.

⁶ 4 samples.

⁷ 5 samples.

⁸ 39 samples.

⁹ 18 samples.

mended for the separation and determination of the saturated and unsaturated acids is as follows:

Weigh such a quantity of oil as contains about 2 g. of saturated acids, in no case using more than a 20 g. sample, into a 200 cc. Erlenmeyer flask, add 30 cc. of 95% alcohol and a 25% excess of a 1-1 aqueous solution of potassium hydroxide. Heat on the steam bath until saponification is complete (about one-half hour). Add a slight excess of dilute acetic acid, using phenolphthalein as an indicator, and bring back to a faint pink color with a dilute potassium hydroxide solution. Measure a quantity of 20% lead acetate solution (60 cc. if a 10 g. sample is used, 120 cc. for a 20 g. sample) into a liter Erlenmeyer flask, add the same amount of water, heat to boiling and wash the neutralized soap solution carefully into the boiling lead acetate solution. Continue the boiling for about five minutes, shake thoroughly and cool under the tap, rotating the flask continuously so as to cause the lead soap to adhere to the sides and bottom of the flask. Pour off the cold aqueous solution, taking care to lose none of the lead soap. Usually the solution is only slightly cloudy with basic lead acetate and no particles of lead soap are to be seen. Wash two or three times with cold tap water, drain and remove the last drops of water by means of a thin roll of filter paper or a wad of cotton held by the forceps, or by shaking the end of a clean towel in the flask. Do not expose the lead salts to the air any longer than necessary as they slowly absorb oxygen. Add a quantity of ether (100 cc. if a 10 g. sample is used, 200 cc. for a 20 g. sample) to the flask, shake and warm cautiously until the lead soap is entirely loosened from the flask and completely disintegrated. Rinse down the sides of the flask with a small quantity of ether, cork loosely and place in the ice box over night. Place a 7 cm. ordinary filter paper in a 7.5 cm. diameter Büchner funnel; then fit in a 9 cm. hardened paper as snugly as possible, fitting in wet and drying with alcohol and ether. Decant the lead soap mixture through this filter, using just enough suction to draw the liquid through. Too much suction causes the moisture in the mixture to freeze and clog the filter, owing to the lowering of the temperature caused by the rapid evaporation of the ether. Transfer as much as possible of the precipitate to the filter, rinse out the flask and wash the precipitate four or five times with ether, using a small horn spoon to stir it. Care should be taken that the lead soap remain slightly moist; otherwise it is difficult to remove it from the filter paper. Remove the filter paper with the adhering precipitate. Separate as much as possible of the lead soap with a spatula and transfer it to a 500 cc. separatory funnel. Wash the adhering lead soap into the separator with a stream of ether, then drop the filter into the flask. Shake the contents of the separator until the lumps of lead soap are disintegrated. Then add about 20 cc. of concentrated hydrochloric acid and shake until the soap is decomposed. Add a few cc. of hydrochloric acid and water to the flask in order to decompose the soap that continues to adhere to the flask and filter paper; then wash into the separator with alternate portions of ether and water until no more particles of lead chloride or soap remain. Again shake the contents of the separator and after settling draw off the aqueous layer, being careful not to lose any particles of undecomposed lead soap. If the soap is not all decomposed shake again with acid. Wash the ether solution with successive portions of distilled water until the washings are neutral to methyl orange. Dehydrate the ether solution with 6 or 7 grams of anhydrous sodium sulphate. Then pour as much as possible of the solution from the neck of the separator into a weighed 300 cc. Erlenmeyer flask and run the remainder from the stem through a small filter into the flask.

Wash the separator and sodium sulphate with several portions of ether and run through the small filter into the flask. Distill off the ether, heat in an oven at about 110° until the weight is constant and weigh the saturated acid fraction thus obtained.

Transfer the ether solution of the soluble lead soap to a 500 cc. separator, shake with 20 cc. concentrated hydrochloric acid until the lead soap is decomposed, add 75 cc. of water and again shake. After settling run aqueous layer into a beaker. Often drops of the ether solution are trapped in the lead chloride precipitate and run off with the aqueous layer. If drops of fatty acid float on the surface of the solution in the beaker, add a few cc. of ether and pour back into the separator, being careful to retain the bulk of the lead chloride precipitate in the beaker. Wash the ether solution with successive portions of distilled water until the washings are neutral to methyl orange.

Dehydrate with anhydrous sodium sulphate and transfer to a 300 cc. weighed Erlenmeyer flask according to the directions for the saturated acid determination. Distill off most of the ether and remove the remainder by heating in an oven at 115°-120° C. for at least an hour while passing a stream of carbon dioxide through the flask. Cool in an atmosphere of carbon dioxide. Then remove the carbon dioxide from the flask by placing it under a bell jar connected with a vacuum system and exhausting the jar a number of times. Weigh the unsaturated acid fraction thus obtained.

Determine the iodine numbers of both saturated acid and unsaturated acid fractions. The iodine number of the saturated acid fraction usually is between 5 and 10, but it is sometimes as high as 15.

The correction for the unsaturated acids that contaminate the saturated acid fraction is calculated as follows:

$$\frac{\text{Iodine No. of Sat. Acids} \times 100 = A}{\text{Iodine No. of Unsat. Acids}} \quad \left(\begin{array}{l} \text{percentage of unsaturated} \\ \text{acids contaminating the} \\ \text{saturated acid fraction} \end{array} \right)$$

The proper correction is then obtained by means of the formula $\frac{A \times B}{100}$ in which B is the percentage of

impure saturated acids. This correction is subtracted from the percentage of impure saturated acids and added to the percentage of unsaturated acids actually determined.

R. D. OILAR GOES TO SOUTH AMERICA

R. D. Oilar sailed in April for South America to build, install and put into operation (lard) compound, winter oil, butterine, oleo oil and stearine, and refrigerating plants.

He expects to correct the present practice of the local slaughterers who now produce no edible fats except hog lard sending practically all the fats to the inedible tank for soaps, much of which should be made into edible products.

After completing the work in Peru, he expects to continue on to Chili, Argentine, Brazil and Venezuela.

He would be pleased to get in touch with any of the readers friends in that section and can be addressed care Larribure Hnos. Lima, Peru, S. A.

CORRECTION

Determination of Moisture and Volatile Matter in Coconut Oils. By A. Edeler. April Chemists'

Section

On page 35, 2d column, 5th paragraph, the figure in the middle of the 4th line should be 0.03% instead of 0.3% as printed. In the 2d and 3d tables on page 36, the "Loss during — hour heating" should have been stated as in the 1st table, i.e., Loss during 2d hour, 3d hour, etc., and not 2 hours etc.