water. Transfer filter with precipitate back into same beaker in which precipitation was made. Add 25 cc. hydrogen peroxide solution (10-50 cc. hydrogen peroxide U. S. P. strength to the liter, plus 50 cc. concentrated nitric acid). Stir until dissolved, add 15 cc. nitric acid, (1.20 sp. gr.) plus 75-100 cc. distilled water and titrate the excess of the hydrogen peroxide by standard potassium permanganate of the strength indicated above. For instance, if 25 cc. H_2O_2 blank requires 80.0 cc. $KMnO_4$ and the sample 34.5 cc. then the lead equals $80.0-34.5 \div 0.455$ per cent. If 25 cc. of the H_2O_2 solution should fail to dissolve the lead peroxide, add another 25 cc. and double the blank. The accompanying samples taken from practice, show that the new method checks exactly with the long chromate method:

	Ericson method	Gravimetric(a) as chromate
Refined spelter.....	0.052 per cent lead	0.055 per cent lead
Refined spelter.....	0.082	0.083
Refined spelter.....	0.043	0.044
Prime Western spelter....	0.530	0.538
Prime Western spelter....	0.533	0.531
Special spelter.....	{ 1.272 }	1.279
	{ 1.283 }	

(a) Filitz, *Praktische Leitfaden für Zinkhütten-laboratorien*, p. 27.

For spelter rather high in lead, it will be advisable to check the lead factor against a standard spelter, under identical conditions.

CADMIUM DETERMINATION

Boil the ammoniacal filtrate from lead until nearly neutral and a white precipitate appears, then take off hot plate and add 40 cc. dilute sulfuric acid (1 : 3) and boil about ten minutes. Add water to make the volume about 200 cc. and saturate with hydrogen sulfide gas, adding a little water occasionally. Allow the precipitate to settle, filter through double filters and wash a few times with water. Redissolve on the filter the cadmium sulfide contaminated with zinc sulfide, in as little warm dilute hydrochloric acid as possible and wash with warm water. If copper is present, the sulfide remains insoluble on the filter. It may be ignited and weighed as CuO or colorimetrically.

The filtrate containing the chlorides of cadmium and zinc is nearly neutralized with dilute ammonia and about 8 grams of trichloroacetic acid dissolved in water added, or enough to dissolve the CdS formed by neutralization. Add distilled water to about 200 cc. volume and reprecipitate Cd by H_2S . Allow to settle, filter and determine by any of the usual methods, preferably as sulfate or phosphate. Duplicate determinations usually agree within a few hundredths of 1 per cent.

Iron is determined in a separate sample by dissolving 10 grams in 75 cc. dilute sulfuric acid and allowing it to stand until practically all dissolved; a few drops of platonic chloride added to effect complete solution; then titrate with the same standard permanganate solution used for lead. Each 0.1 cc. $KMnO_4$ solution equals 0.001 per cent. iron on a 10-gram sample basis.

The method outlined above takes care of the usual constituents called for in spelter analysis, such as lead,

iron and cadmium. Zinc is taken by difference. Copper, tin and antimony are indicated and may also be determined, whenever present. The lead method is a marvel of accuracy and gives very concordant results. In making this claim, I am aware of recent criticism in regard to shortcomings of most analytical methods proposed. This one will be found, on investigation, to be a notable exception, and its daily use for several years has fully demonstrated its reliability.

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FACTORY METHOD FOR THE DETERMINATION OF TOTAL FATTY ACIDS IN COTTON-SEED FOOTS¹

By F. N. SMALLEY

The method herein described is proposed for a rapid method for the determination of the total fatty acids in cotton-seed foots, or soap stock, and similar materials, by a determination of the iodine number of the same, and its conversion into terms of fatty acids by means of an appropriate factor.

Cottonseed oil soap stock is the sludge removed from crude oil in the process of refining with caustic soda, and consists of the soda soaps of the fatty acids of the oil, together with some neutral oil, coloring and albuminous matters which are characteristic of crude oil, caustic soda and water.

The most accurate method of the determination of the total fatty acids is the Official Method of the Interstate Cotton-seed Crushers Association, which consists in complete saponification of the fat by means of alcoholic soda, evaporation of the alcohol, solution of the soap in the hot water, decomposition of the soap with dilute mineral acids, melting of the liberated fatty acids, chilling to form a cake, filtering and washing free from mineral acid, extraction of the cake with warm petroleic ether, evaporation of the solvent and weighing of the residual fatty acids.

The method is so long that it is almost useless for factory control, and for that reason after trials of a number of various proposed methods, the iodine method was selected in the summer of 1905, as the best fitted to obtain the most accurate results in the shortest time. Since then it has been in continuous use in the refineries of the Southern Cotton Oil Company.

The method is carried out as follows: After the sample has been well mixed, weigh out accurately 0.5 gram on to a small square of fat-free paper. This is then introduced into a ground glass stoppered bottle with neck so arranged that the joint can be sealed with the potassium iodid solution. Five cc. of carbon tetrachloride and 20 cc. of Wijs' iodine solution are added. The bottle is stoppered and the contents gently shaken until the soap stock is in solution. Seal with potassium iodid solution and allow to stand for thirty minutes.

The determination of the iodine number is now carried out in the usual Wijs' method, and the resultant number is converted by division by 1.24 into per cent fatty acids.

The factor was arrived at by comparison of the

¹ Paper presented at the Eighth International Congress of Applied Chemistry, New York, September, 1912.

iodin number with the total fatty acids as determined by the official method as before outlined. Some one hundred and fifty samples were analyzed by both methods before the final selection of the factor was made.

The following table shows the closeness of the two methods.

Sample number	F. F. A. by official method	Iodine No.	F. F. A. by calculated for I No.
2171	43.39	53.53	43.17
72	40.70	50.90	41.05
75	47.35	58.68	47.32
76	46.70	57.64	46.48
77	49.34	60.71	48.96
78	47.13	58.16	46.90
79	46.51	57.13	46.07
81	48.48	58.88	47.48
84	51.52	61.19	47.31
87	49.30	61.0	49.20
88	46.67	47.34	38.18
90	47.45	58.66	47.31
91	49.31	61.48	49.58
92	47.03	58.16	46.90
94	49.29	61.19	49.35

The above represented one day's work, and the results are typical of all work done. The three samples 2181-84-88, which show the greatest difference, were several weeks old and fermentation was in an advanced stage.

The results showed that the conversion factor could not be depended on if the soap stock was more than a week old, but as the whole purpose was to obtain results as soon as possible after the refining was made, this was no serious objection.

The six years' use has proven that the method, as used, gave, on the whole, extremely satisfactory results, and it has only received minor modifications, consisting in using special strength of sodium thio-sulfate and iodine solution, so as to simplify the calculation.

SAVANNAH, GEORGIA

THE MODIFIED BABCOCK FOR FAT IN SWEETENED DAIRY PRODUCTS—ICE CREAM¹

By J. O. HALVERSON²

DIFFICULTIES IN TESTING SWEETENED DAIRY PRODUCTS BY THE BABCOCK TEST

The Babcock test is not applicable to sweetened dairy products on account of the charring action of the acid on the sugar. This causes a black charred mass to rise in the neck of the test bottle. Nor can the Adams-Soxhlet method be depended upon as the sugar particles enclose fat.³ Leach³ overcomes this by pipetting off this solution containing sugar after precipitating the proteids with the fat by Fehling's copper sulfate solution. He then centrifuges in the cold, washes twice with water. The chances for errors are in thrice pipetting off the sugar solutions. Errors are also increased three-fold by multiplying the final reading by three.

¹ Paper presented at the Annual meeting of the American Chemical Society, Milwaukee, March, 1913. A preliminary report was read at the Washington meeting of the A. O. A. C., September 17, 1912.

² Formerly Deputy Food and Drug Commissioner, Acting Analyst, Missouri Department of Food and Drug Inspection, Columbia, Mo. Patents dedicated to the public are pending on the novel features of the test bottle described in this article.

³ Leach, *J. Am. Chem. Soc.*, 1900, p. 589.

Later, in 1900, Farrington² published a method of coagulating the proteids with small portions of 3 cc. of sulfuric acid, centrifuging at 1000 revolutions for six minutes with the tester heated to a temperature of 200° F. This gathered the proteids with the fat into a firm lump, so the sugar solution could be poured out the neck. Ten cc. of water are then added and the curd shaken up, 3 cc. of acid added and the process continued as above. After most of the sugar is removed, 10 cc. of water are added and continued as in the Babcock test.

These are the modified Babcock methods used to remove sugar from sweetened condensed milk. Where the per cent. of fat runs high, as in ice cream, these methods do not precipitate all the fat, part of which rises to the surface. Hence these methods are inapplicable.

GRAVIMETRIC AND VOLUMETRIC METHODS IN USE ON ICE CREAM

For ice cream gravimetric methods are used as the Roesse-Gottlieb Extraction Method² and Paul's Method,³ a combination of the Leach and the extraction method. The fat obtained is finally weighed in both these methods.

Of the volumetric methods, none remove the sugar entirely, but attempt to minimize its interference the most possible. Such a method is that of Holm.⁴ This consists of a mixture of equal parts of glacial acetic and hydrochloric acid on a nine-gram sample. This acid used is less liable to char the sugar.

Howard⁵ uses 10 cc. of Fehling's Copper Sulphate Solution, shakes and centrifuges 3 minutes to precipitate the proteids. First chloroform is added, which dissolves the fat. He then removes the supernatant liquor. The chloroform is then driven off by running in steam.

The elimination of sugar in sweetened dairy products would make the Babcock test available as a rapid, easy method for the determination of fat.

It was thought if the sugar could be drained off after all the curd was practically dissolved by the ordinary acid used in testing cream and after the fat was centrifuged to the top, the Babcock could still be used as a rapid volumetric method for determining fat in ice cream and other sweetened dairy products.

It was observed that in the Babcock method in testing ice cream, an appreciable interval of time elapses on adding sulfuric acid slowly before action on the sugar begins. This is the basis of the proposed Modified Babcock Method and the essential point in this method. The acid-sugar solution is readily drained off by using a modified cream test bottle, which consists in fusing a small glass stopcock (1 mm. bore) on the side of a modified 6-inch 30 per cent cream test bottle. To drain off the acid containing the sugar, after centrifuging, equal parts of water and acid are added up to near the neck. Two minutes more centrifuging drives the fat and undissolved curd

¹ Wis. Exp. Sta., *Annual Report*, 1900, pp. 86-89.

² *Landsw. Vers.*, 40, 1-27 (1892).

³ *Bur. Chem., Circ.* 90, p. 10.

⁴ *Ann. Report, State Food Com., Illinois*, 1906, p. 80.

⁵ *J. Am. Chem. Soc.*, 1907, 1622.