(b) AVERAGE SEMI-BITUMINOUS COAL (NOT OVER 25 % VOLATILE MATTER.)

½ gram $KClO_3$	0.040°
1 per cent. Sulphur	0.006
7 " " Ash	0.007
4 " " Hydroxyl	0.013
Fuse Wire	0.008
Total	0.074° C.
or,	0.123° F.

(c) AVERAGE BITUMINOUS COAL (OVER 25 % VOLATILE MATTER).

½ gram $KClO_3$	0.040°
2 per cent. Sulphur	0.012
10 " " Ash	0.010
10 " " Combined Water	0.033
Fuse Wire	0.008
	0.103° C.
	0.185° F.

Where the component parts vary widely from these average conditions the ultimate correction factor may readily be made up from the schedule of units as listed under No. 6 of the Summary, below.

Summary.

1. The heat of combustion of carbon is 73 per cent. of the heat of the reaction of the carbon with sodium peroxide.

2. The heat of combustion of hydrogen (to liquid water) is 73 per cent. of the heat of the reaction of hydrogen with sodium peroxide.

3. The sodium peroxide used must be free from absorbed moisture or the correction value of the same must be determined.

4. Standard conditions based upon theoretical data are available for checking material and apparatus.

5. The constants are the large elements in the case and practically govern the accuracy of the process.

6. The correction components may be readily reduced to a unit basis; thus,—

Sulphur	0.006°	for 1% in 0.5 g. of material
Ash	0.001°	" " " " " " "
Combined Water	0.0033°	" " " " " " "
$KClO_3$	0.040°	" 0.5 g. used
Fuse Wire	0.008°	" 10 mg. burned

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THE ANALYSIS OF ICE CREAM.

BY CHARLES D. HOWARD.

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With the recent enactment of standards for ice cream by several of the states, the analysis of this substance is just beginning to receive attention on the part of food inspection chemists. As the literature seems to contain no specific methods, the following (in part simple modifications of

general methods) are offered as having been in successful use at this laboratory.

Fat.—It is especially desirable to make this estimation as soon as possible after the sample has melted, as it not infrequently occurs that on short standing, even before appreciable souring has taken place, the butter-fat tends to separate in such a manner, and on mixing, returns to the surface so rapidly as to render the removal of a representative portion exceedingly difficult, while if care is not exercised in attempts at emulsifying, butter-clots are apt to be formed.

Except for purposes of approximation, or unless the sample is very largely diluted, the use of the plain Babcock method is not admissible. Leach's copper sulphate method for the estimation of fat in condensed milk is not directly applicable, because of the fact that in this case the fat is not carried down by the precipitated proteids, but persists in rising to the surface, and no other precipitant seems capable of affording any different result.

The procedure finally adopted consists of a modification of Leach's method, involving solution of the fat in chloroform, with the subsequent removal of the latter in a current of steam. Using a blunt-pointed pipette, eighteen grams of the sample are weighed into a Babcock cream bottle, preferably of the Bartlett, or bulb type graduated to 25 per cent. in fifths. Add 3 cc. chloroform and water to about three-fourths the capacity of the bottle. Agitate, add about 10 cc. of Fehling's copper sulphate solution, mix and centrifuge for three minutes, when all of the fat will be found in the underlying chloroform layer. In the presence of appreciable quantities of gum tragacanth or gelatin, the supernatant liquid will persist in remaining turbid; this difficulty may be obviated by the addition of two or three cc. of N/10 alkali, the resulting copper hydroxide serving to precipitate the gum very perfectly. Insert a glass tube of small bore, connect with an aspirator and remove most of the supernatant liquid. Repeat the washing once, then introduce a tube of small bore connected with a source of steam provided with a safety cock, and blow in live steam for two or three minutes, or until the chloroform has been completely expelled; the latter condition is essential. The use of a water bath for the removal of the chloroform is not feasible, as the precipitated proteids retain the latter with great persistency, and its removal is not complete even after prolonged heating in this manner. Cool the contents of the bottle, add water to a volume of 17.5 cc., and proceed as usual, being careful to secure complete solution in the acid of the somewhat hardened proteid substance.

A second method for fat, recommended by the Chicago Board of Health,[1] has been given only a brief trial in this laboratory. This method has

[1] Rep. State Food Commissioner, Illinois, 1906, 80.

the advantage of rapidity, and is said by those who have used it extensively to give good results. It would seem, however, that some little skill is required in its execution to avoid contamination of the fat column by casein on the one hand, and charring on the other. The procedure is as follows: "Run 9 g. of ice cream into the test bottle. Add 30 cc. of a mixture of equal parts by volume of conc. HCl and 80 per cent. acetic acid. Heat on a water-bath till well darkened, but short of charring. Whirl in a Babcock centrifuge and read the percentage of fat directly. If the cream is charred, ether may be added after whirling. The layer containing the fat is drawn off into another bottle and the ether evaporated. The bottle is then filled with water and again whirled and the fat read directly."

Character of the Fatty Matter. While the practice is apparently not a common one, there seems to be evidence that in certain sections at least, not a little "ice cream" is put upon the market, the fat of which consists in part or wholly of oleo or cotton-seed oil. For purposes of examination the fatty matter may be advantageously separated by removing 30-40 cc. of the cream layer to a fat-bottle, adding one cc. of strong mercuric nitrate solution and 20 cc. of petroleum ether and whirling in a centrifuge. The ethereal layer is removed, washed with water and the ether evaporated.

Chemical Preservatives.—The presence of borax may be detected by removing five cc. of the separated serum to a porcelain dish, rendering distinctly acid with ten per cent. hydrochloric acid, adding five drops of turmeric tincture and evaporating. For formaldehyde the hydrochloric acid test may be used as a preliminary, but as ice cream free from this body affords by this treatment a purplish brown color, the distillation method should be employed to confirm any positive indication noted. Saccharin and other bodies of this class are detected in connection with the test for gelatin.

Fillers and "Mechanical Preservatives."—The old-fashioned recipes for cheap ice cream call for corn-starch or eggs, the first being quickly detectable and the second indicated by the deep yellow color imparted by the yolks to the underlying serum separating from the sample on standing. At present there is a large number of patent, or secret preparations on the market, most of which consist essentially of gelatin or gum tragacanth. The writer has recently had his attention directed to two such, sold to manufacturers of ice cream under the names of "foamaline" and "cream-puff."

The use of compounds of this description is not altogether for the purpose of taking the place of butter-fat, since manufacturers of high grade cream allege that their addition in small quantity is necessary, in the case of goods intended to be shipped or to be kept over much more than twenty-

four hours, for the purpose of causing the ice cream to "stand up," that is, retain its homogeneity and obviate the tendency to marginal icing that occurs with storage. It appears that manufacturers are thus able to "hold over" goods under refrigeration for several days or even indefinite periods, in order to await favorable weather or market conditions.

Another substance that is being used to some extent as a filler of ice cream is commercial caesin. Such use in New Hampshire has been observed in several instances—the solidified, cheesy appearance of the caseinous stratum separating from the sample, affording an indication that was later confirmed outside the laboratory. Obviously, a method for detecting this class of goods might be based upon the determination of the increased albuminoid content.

For the detection of gelatin the method of Stokes may be followed, using 10–15 cc. of the mixed sample. Any faint cloudiness corresponding to less than 0.10 per cent. of gelatin is ignored. The main portion of the filtrate from the proteids is then extracted with chloroform and examined for saccharin and similar substances in the usual manner.

Where any considerable quantity of gum tragacanth or a similar substance has been used, such fact is determinable without difficulty. The writer has observed samples of melted ice cream that were fairly stiff from the presence of excessive amounts of gum. However, in those cases where the latter is used for its mechanical effect the quantity may be less than 0.5 per cent., or even as little as 0.25 per cent. In view of the remarkable similarity in the general behavior to reagents of casein and the vegetable gums, and the tendency of the latter to be carried down with the former under the influence of metallic precipitants, the development of a method for the detection of such a small quantity of this class of substances offers more difficulty than might be supposed.

The conversion of the gummy material into mucic acid might be used as a test, but it is of course necessary to first remove all lactose, and the subsequent procedure not only consumes some time, but the results are apt to be rather uncertain, especially in the presence of but 0.2–0.3 per cent. of gum. Reichl and Breinl's orcinol test (Allen, "Commercial Organic Analysis," Volume 1, page 422) would seem to be valuable in this connection, as it affords a striking reaction as applied to the alcohol-washed mixture of gum and casein; unfortunately, however, casein alone appears to give a similar reaction.

A preliminary test that serves well consists in precipitating 10 cc. of the separated serum with acetone, washing with two or three portions of dilute alcohol (using a centrifuge). The washed residue is boiled with 6–8 cc. of water, and 1 cc. sodium hydroxide (1–10) for about thirty seconds. This treatment results in the formation of albuminates largely soluble in hot alcohol. The gum is converted into pectates soluble in

water, but insoluble in hot alcohol, while any gelatin is decomposed into bodies not precipitable by alcohol. The liquid is cooled and allowed to stand a few minutes to facilitate the separation of any insoluble caseinous matter; filtered, filtrate heated to boiling, one and one-half volumes of warm alcohol added and mixed. In the presence of 0.2 per cent. of gum tragacanth or agar-agar, a characteristic flocculent precipitate will *immediately* separate. Any turbidity, or slight precipitation on standing a few moments, should be ignored. The precipitated pectate may be dissolved in cold water and the solution saturated with ammonium sulphate for the purpose of demonstrating the absence of any appreciable quantity of proteids.

A second procedure, involving more or less complete preliminary removal of proteids and bases, with conversion of the gum into pectic acids, consists in boiling 10 cc. of the separated serum to 5 cc. after addition of one half cc. of concentrated hydrochloric acid. Two volumes of alcohol are then added to the hot liquid and the resulting precipitate washed two or three times with alcohol. The residue is boiled with 5 cc. of water and one drop of strong acetic acid and filtered hot. The resulting filtrate is rendered just neutral or very faintly alkaline, boiled and filtered. Finally 1 cc. of sodium hydroxide (1–10) is added to the filtrate, the latter boiled for a few seconds, followed by the addition of one and one-half volumes of alcohol, noting any precipitation of alkaline pectate.

NEW HAMPSHIRE LABORATORY OF HYGIENE.

THE DETECTION AND DETERMINATION OF BENZOIC ACID IN KETCHUPS, FRUITS AND CIDERS.

BY HARRY S. REED.

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The following methods were worked out by the writer on occasion of being called upon to determine the presence of, and quantity of benzoic acid, in certain fruits, ketchups, ciders, etc. They are presented in the hope that some one may find them useful.

Detection of Benzoic Acid.—For this purpose a modification of Mohler's test is used. One hundred g. of the substance under examination is acidified with dilute sulphuric acid and thoroughly and repeatedly extracted with chloroform. The chloroform extract is separated from the water solution each time as far as possible by means of a separatory funnel. The last traces are then spun out with a centrifuge. The success of both qualitative and quantitative determinations depends largely on the care with which the extraction is made.

The chloroform extract is then placed in the reservoir (A) of the following apparatus:

(Capacity of A., 50 cc.)

(Capacity of B., half filled, 12 cc.)