

pyrites, being for each per cent. of sulphur present as pyrites, plus 9.6 calories in one case and minus 6.6 calories in the other.

Some of the products resulting from the oxidation of pyrites and the occurrence of organic sulphur in coal will be considered in another paper.

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THE RAPID DETERMINATION OF FAT BY MEANS OF CARBON TETRACHLORIDE.

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THE number of solvents which have been proposed for use in determining the fat in animal and vegetable tissues is considerable, including ether, petroleum ether, alcohol, carbon bisulphide, acetone, chloroform and carbon tetrachloride. Of these the most generally accepted is ether. This may, in fact, be considered as the official solvent in all cases of dispute or comparison. Petroleum ether is, however, preferred by many who consider the results obtained with this solvent as rather more trustworthy than those with ether.

It is not the purpose of this paper to enter into any historical resumé of the subject of the determination of fat, either as regards kinds of solvents or methods of manipulation; but rather to indicate the reasons which have led up to the adoption of carbon tetrachloride by the writer as a rapid and accurate reagent in the commercial determination of fat. In factory control work it becomes necessary to determine the proportion of fat in different products within a comparatively few hours. The usual duration of extraction with ether is about sixteen hours, although essentially all of the fat can be extracted in much less time. Petroleum ether is said to require about the same length of time as ether, although approximate results can usually be obtained in from four to six hours' extraction. Carbon bisulphide extracts to practical completion inside of four hours, and has been used quite frequently in the rapid determination of fat, giving quite satisfactory results as a rule. It has the advantage that it can be used on materials containing not more than 10 per cent. moisture without previous removal of the water. As just stated, extraction appears to be com-

plete in four hours so that the whole determination, including the final weighings can readily be completed within eight hours.

There are, however, several disadvantages in the use of carbon bisulphide, which have at times assumed such proportions as to make its further use undesirable. In the first place it is difficult to obtain a strictly chemically-pure product at reasonable price. Much of the carbon bisulphide on the market requires redistillation before it can be used and even then a slight decomposition may take place during the extraction with a deposit of sulphur in the oil flask and the consequent vitiating of results. This has been the chief objection which the writer has found with this solvent.¹

Another undesirable feature is the highly inflammable nature of carbon bisulphide. This objection is equally applicable to ether and petroleum ether. Recently a chemist was severely burned by an explosion of carbon bisulphide due to the spilling of a little on a superheated steam-pipe while taking off the fat flasks. In another case a bottle of carbon bisulphide exploded without warning. The disagreeable odor and the unhealthfulness of the vapors of carbon bisulphide are further objections to its use.

The ideal solvent for factory work should combine quick and complete extraction of fat with a high degree of safety in its use. These qualifications are found in carbon tetrachloride in a marked degree. While this solvent is well known, it does not appear to have been adopted to any great extent in the quantitative determination of fat.

Carbon tetrachloride is, as is well known, a heavy water-white liquid of a specific gravity of about 1.6, immiscible with water, unflammable and having a boiling-point of about 76° C. It is a very active solvent, two hours being sufficient for the complete removal of fat by the ordinary methods of extraction, the results being practically identical with those obtained by extracting with ether for sixteen hours.

The unflammability of carbon tetrachloride, its high boiling-point and the rapidity and thoroughness of extraction make it an ideal solvent. It has been found, moreover, that a commercial product can be purchased at a cost considerably less than that of carbon bisulphide and having a residue of not over 0.003 gram per 30 cc., the amount taken for one extraction, and that, after re-

¹ See article on "Chemical Studies on the Extraction of Fatty Substances by Means of Carbon Bisulphide," L. Fabre, *Oester. Chem. Ztg.*, 3, (1900), 370.

distillation, there is no danger of further residue. Owing to its comparatively small volatility there is in addition less loss of reagent during extraction than with ether, petroleum ether or carbon bisulphide.

Before adopting carbon tetrachloride in the determination of fats a number of comparisons were made between the results thus obtained and those obtained by use of ether and carbon bisulphide. These are shown in the following table. The values for ether extract were obtained by extracting the water-free material sixteen hours with anhydrous ether, those for carbon tetrachloride by extracting the air-dry material two hours with carbon tetrachloride, those for carbon bisulphide by extracting the air-dry material with this reagent for four hours. In all cases the Knorr apparatus was used.

The samples were all of vegetable origin with the exception of No. 9 which was a piece of air-dried pork.

COMPARISON OF QUANTITY OF MATERIAL EXTRACTED BY MEANS OF DIFFERENT SOLVENTS.

Sample.	Ether extract. Per cent.	Carbon tetrachloride extract. Per cent.	Carbon bisulphide extract Per cent.
(1)	0.12	0.12
(2)	0.14	0.15	0.25
(3)	0.17	0.17
(4)	4.06	4.06	4.20
(5)	4.53	4.57
(6)	9.37	9.48	9.10
(7)	11.3	11.6
(8)	41.35	41.40
(9)	71.3	72.6
(10)	10.7	10.1
(11)	11.0	10.8
(12)	11.0	10.7

The carbon tetrachloride was redistilled previous to using. The ether was chemically-pure anhydrous, manufactured by Merck. The carbon bisulphide was redistilled till it left no further residue.

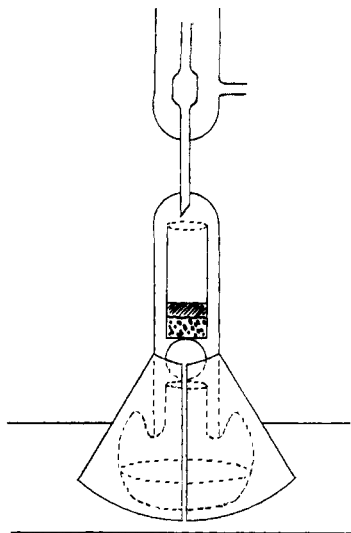
It will be observed that the agreement between the quantities extracted by ether and by carbon tetrachloride, in samples Nos. 1 to 5 and in No. 8, is practically identical, while in the other cases the ether removed slightly less than the carbon tetrachloride. This is particularly the case in sample No. 9 consisting of air-dried pork. This may be due either to imperfect extraction by ether, extraction of something not fat by the carbon tetrachloride or the rendering

partially insoluble of a portion of the fat during the preliminary drying before extraction with ether. Time did not permit of further investigation along this line. The carbon bisulphide in some cases gave higher and in some cases lower results than the carbon tetrachloride.

A few comparative tests were made with air-dry and water-free material with identical or practically identical results. Thus, in sample No. 4, which contained 6.9 per cent. water, identically the same amounts of oil were obtained by extracting the air-dry and the water-free material, in spite of the fact that it contained 14 per cent. of water-soluble material. In a sample similar to Nos. 6 and 7 there was 0.10 per cent. more extract from the air-dry substance, containing 12 per cent. soluble material and 10 per cent. water. No. 8, on the other hand, gave a slightly higher extract on the air-dry material, although this contained but 2.5 per cent. of water and practically no water-soluble material. There was probably a slight oxidation during drying, even at 100° *in vacuo*. The values in the table were obtained on the water-free material.

Method and Manipulation.—As stated above, the Knorr apparatus is used, the fat flasks being 2.5 inches in diameter and about the same height. Three grams of material are taken for extraction in each case, except where there is a very large percentage of fat, when 1 or 2 grams may be used. The flasks are placed in holes in the top of a steam-bath, the edge of the holes being covered with cloth to protect the flasks from resting directly upon the metal. Live steam is introduced into the bath in sufficient quantity to maintain brisk ebullition of the solvent. In order to prevent the condensation of the carbon tetrachloride in the lower part of the apparatus half cones of copper are placed around the flasks, reaching above the neck of the flask and as far as the base of the extraction tube or thimble. This keeps the whole flask surrounded by steam and drives the vapors of the tetrachloride into the condenser before condensation, from whence the solvent drops back into the extraction tube. The arrangement is illustrated in the following diagram. The condensers in use have four bulbs, but a very short two-bulb condenser would doubtless furnish ample condensing surface. In case the solvent in one flask boils to hard, the copper shields can be slightly opened and ebullition regulated.

After two hours of actual extraction, steam is turned off and the extraction tube allowed to drain a few minutes, when the con-



denser is lifted and a collection tube substituted in its place. These latter are essentially thick-walled test-tubes with triangular grooved bottom to prevent closure of the top of the fat flask. The solvent is then distilled into the collection tube, the heat being maintained at a point sufficient to volatilize the condensed portion which collects between the neck of the fat flask and the lower portion of the condenser. Even if the heat causes ebullition in the collection tube during this final period the vapors all condense and run back into the tube. In this way the solvent can be coincidentally recovered from a whole battery of flasks.

The amount of carbon tetrachloride required for each extraction is about 30 cc. Of this one-third to one-half will usually be lost: (*a*) in the extraction tube when taken off; (*b*) between the flask and the neck of the condenser when the extraction process is finished; (*c*) and in final incomplete collection in the tube.

The final drying of the flasks can be accomplished in a hot air or vacuum oven at 100° C. in about two hours. The last traces of carbon tetrachloride appear to be driven off with some difficulty. It is at this point that the only objection to the method has been found, and, judging from the comparative results shown above, this will prove no material objection. By actual tests it has been

found practicable to raise the temperature to 120° for a short time without oxidizing the fat.

Conclusions.—The use of carbon tetrachloride as a solvent in the estimation of fats in foods and feeding-stuffs appears to give very satisfactory results and has several points which make it especially desirable.

(1) It is very rapid, two hours apparently sufficing for complete extraction in all cases.

(2) It is unflammable, thus reducing the danger of explosion and fire to a minimum.

(3) It is inexpensive.

THE GLUCOSE SUGAR REFINING CO.,
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NOTE.

The Use of Aniline Oil in the Determination of Weighting in Aniline Colors.—It is perhaps not generally known that aniline oil is an excellent solvent for many basic aniline colors, when it is desired to determine the amount of weighting or letting down, especially in the case of methylene blues, which are not very soluble in strong alcohol.

It is cheap, easily obtained pure, and has a high boiling-point. It dissolves the color readily and has scarcely any solvent action on the substances generally used for weighting, such as dextrine, sugar, salt, etc.

The color to be tested is extracted with small portions of hot aniline oil until the filtrate is colorless, using a balanced filter or Gooch crucible. The aniline oil is finally removed from the filter with a little strong alcohol and the residue dried as usual.

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NEW BOOKS.

PHYSIKALISCH-CHEMISCHES CENTRALBLATT. Vollständiges internationales Referatenorgan für die physikalische Chemie und die angrenzenden Gebiete der Chemie und Physik. Edited by DR. MAX RUDOLPHI.

The descriptive part of the title of this new periodical suffices to indicate its general purpose of gathering together in a single place