point of chloroform —62°, of carbon disulfide —111°, and boiling point of fresh liquid air —193°. The true melting point of chloroform is —63.7°, and of carbon disulfide —111.6°. All temperatures are in degrees centigrade.

Summary.

A method of separation of a natural gas into its paraffin hydrocarbons is shown. At a temperature of —185° to —190° the methane can be removed. The separation of the methane from the propane, butane, etc., is conducted at temperatures ranging from —150° to —140°. The propane is separated from the butanes, etc., at temperatures ranging from —135° to —120°.

This method of separating a gaseous mixture into its constituents while somewhat involved is the only known method in the case of some hydrocarbons. It can be extended to the separation of other gaseous mixtures. The authors have separated other natural gases but the example given herein is sufficient to describe the method.

[FROM THE CHEMICAL LABORATORY OF THE HOSPITAL OF THE ROCKEFELLER INSTITUTE FOR MEDICAL RESEARCH.]

DETERMINATION OF ARSENIC IN ORGANIC MATTER.

BY MARIAM VINOGRAD.

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Oxidation of the Tissue for the Determination of Arsenic.—The use of minimum quantities of reagents in oxidizing tissues for the Marsh test is important, because it is very difficult to get certain reagents, such as nitric acid, absolutely free from arsenic. Another important consideration is the prevention of the escape of the arsenic at the moment of carbonization of the tissue, since, as is well known, arsenic is volatile at the temperature of the oxidation. Both of these difficulties were avoided by Bertrand[1], who showed that dried substance could be conveniently burned in a Bertholet bomb with pure oxygen under 25 to 30 atmospheres pressure. The only disadvantage of Bertrand's method for general use is that analytical laboratories are not ordinarily equipped with the Berthelot apparatus.

Because of this fact, apparently, investigators have continued experimenting with methods of oxidation which could be carried out with the facilities usually available.

Chittenden and Donaldson, in 1880, published a paper describing in detail a simple method for partial oxidation of tissue with relatively small amounts of nitric and sulfuric acids, the carbonaceous residue being extracted with water and used for the Marsh test.[2] This method added

[1] Bertrand, *Ann. de l'Inst. Pasteur*, **1903**, 581.

[2] *Am. Chem. J.*, **2**, 255.

greatly to the reliability of the arsenic determinations in tissue. It required 100 grams of material, however, and the incomplete oxidation rendered the subsequent extraction of the charred residue rather laborious. Consequently other methods for the oxidation have continued to appear, not all of which can be regarded as improvements.

Karl Ulmann,[1] following Gautier's method, used a mixture of nitric and sulfuric acids in quantities twenty-fold the amount of tissue used. The tissue examined is heated for 6 to 36 hrs., according to its character, on a sand bath. A somewhat different method was used by Lockemann.[2] The tissue was first disintegrated by heating with an equal weight of a nitric-sulfuric acid mixture. Oxidation was completed by fusion with a mixture of sodium and potassium nitrates. These methods involve both the use of relatively large amount of reagents and the danger of volatilization of arsenic during the oxidation.

The application of the ordinary Carius' technique for organic analyses obviates both difficulties and we have found that it gives excellent results. With a sealed tube, loss by volatilization is impossible, and the application of high temperature and pressure enables one to complete the oxidation with a small amount of nitric acid. Thus, 1 cc. of nitric acid is sufficient to oxidize 3 cc. of blood serum, or 100 cc. of spinal fluid, or 0.5 g. of dry tissue. The method described in this communication has been used chiefly in the determination of arsenic in blood, tissues, and spinal fluid, after intravenous injections of salvarsan. The exact technique employed in testing these various substances is as follows:

The Oxidation of the Blood.—In the experiment under consideration 1 cc. of blood is put into a small test tube (about 6 cm. long and 5 mm. wide.) This tube is carefully lowered into a regular bomb tube, which contains 1 cc. of fuming nitric acid. The bomb tube is then sealed in the ordinary way and heated slowly to 260°. At this temperature the heating is continued for an hour or two, at the end of which time the oxidation is complete. The tube then contains a clear liquid of yellowish color, due to the nitrous oxides. Any arsenic present will have been completely converted into arsenic acid. The combustion oven with the tube is cooled to room temperature, and the pressure is released in the usual manner by melting the capillary point. The tube is then opened and the contents washed with distilled water into a 200 cc. Jena Kjeldahl flask. The liquid is evaporated until only about 15 cc. are left. This is then further heated with 4 to 5 cc. of sulfuric acid until all traces of nitric acid are driven out, as nitric acid appears to be a disturbing agent in the process

[1] Karl Ulmann, "Zur Frage der Parasitotropie und Toxizität des Salvarsans," *Wien. klin. Wochschr.*, **1913**, No. 6.

[2] Lockemann, "Uber den Arsennachweis mit dem Marschschen Apparat," *Z. angew. Chem.*, **18**, 416 (1905).

of the determination of arsenic. In order to dilute this sulfuric acid, it is washed with a known quantity of distilled water (about 10 cc.) into a small graduated cylinder. Distilled water is then added to make up the total volume to seven times the volume of the original sulfuric acid.

Oxidation of the Tissues.—The different parts, such as lungs, liver, kidneys and parts of the muscles, are dried thoroughly in porcelain dishes on a water bath, and are then ground to a powder. Up to 0.5 g. of dry tissue is taken for oxidation by the method described above. In case the tissue sample is small enough to go into the test tube, drying is unnecessary.

The Oxidation of Spinal Fluid.—In the determination of arsenic in the spinal fluid, 100 cc. of the fluid are put into a small, round-bottom crystallizing dish and evaporated nearly to dryness on a water bath. The semi-solid residue is transferred, as completely as possible, to a small tube (about 6 cm. long and $^1/_2$ cm. wide). The remaining traces are washed with 1 cc. of fuming nitric acid into the bomb tube. The small tube is lowered into the bomb tube, which is then sealed. The rest of the process is identical with the treatment of the blood, described above.

Determination of Arsenic in the Solution by Sanger and Black's Method.—The application of Gutzeit's color reaction, of hydrogen arsenite with mercuric chloride paper, to the determination of small amounts of arsenic has been carefully worked out by Sanger and Black.[1] We will describe briefly the use of the method with solutions of oxidized tissues prepared as above described.

The apparatus is shown in Fig. 1. A bottle with a wide opening, of 40 cc. capacity, is stoppered with an arsenic-free, two-hole rubber stopper (prepared by boiling for an hour in 5% NaOH solution). A 5 mm. glass funnel-tube, drawn out to 1 mm. at the lower end, passes through one perforation of the stopper and reaches to the bottom of the bottle. Through the other perforation passes a bent piece of glass tubing, through which the gases can escape. This tube is connected by means of a small calcium chloride tube, in which lies, next to the stopper, a rolled strip of filter paper saturated with lead acetate to absorb hydrogen sulfide. The bulb of the tube is filled with absorbing cotton, dried in a desiccator. The long part of the tube, the diameter of which is 5 mm., carries a strip of drawing paper, about 5 mm. wide and 7 cm. long, saturated with a 5% solution of mercuric chloride. These strips are prepared by immersing them in the mercury solution and then drying at room temperature. They are kept in a test tube containing, on the bottom, several pieces of calcium chloride, covered with absorbing cotton. In order to make the generation of hydrogen continuous, a sheet of platinum is placed on the bottom of the apparatus. On top of the platinum are placed 3 g. of zinc,

[1] Sanger and Black, *Proc. Am. Acad.*, **43**, 297 (1907); *J. Soc. Chem. Ind.*, **26**, 1115 (1907); *Z. anorg. Chem.*, **58**, 121 (1908).

then 10 cc. of special diluted sulfuric acid (see above) are poured in. Ten minutes after this, the solution to be tested for arsenic is poured in. The presence of arsenic is determined by the coloring of the end of the strip nearest the bulb. The quantity of arsenic is determined by the depth to which the yellowish orange color attains. The method is sensitive to less than 0.001 mg. of arsenic.

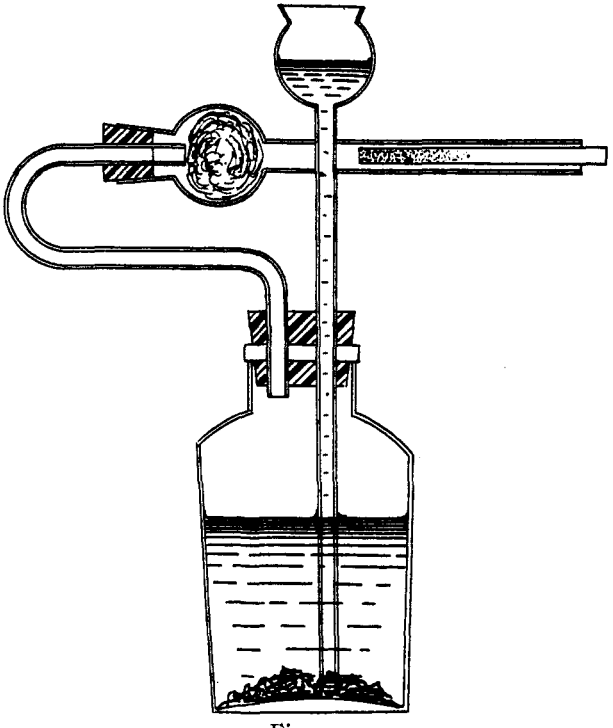

Fig. 1.

All the glass and porcelain apparatus used must be tested for arsenic, and only such taken as proves to be free from it. The sulfuric acid is usually found to be free from arsenic. The greatest difficulty was encountered with the zinc, but the brand called "Bertha Spelter," from the New Jersey Zinc Company of New York, recommended by Sanger and Black, proved to be arsenic-free. The zinc was delivered to us in large pieces. These were melted in French clay crucibles and the molten mass poured into water, where it solidified in small pieces suitable for the experiments.

NEW YORK, N. Y.

PARTITION OF THE NITROGEN OF PLANT, YEAST, AND MEAT EXTRACTS.

BY F. C. COOK.[1]

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Introduction.

An investigation of the nitrogenous and other constituents of meat extracts, bouillon cubes, and plant extracts was recently reported by Micko.[2] In previous papers he[3] discussed the purine bases of meat and

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[2] *Z. Nahr. Genussm.*, **26**, 321 (1913).

[3] *Ibid.*, **5**, 193 (1902); **6**, 781 (1903).