A MODIFICATION OF THE ELECTROLYTIC GUTZEIT APPARATUS FOR THE ESTIMATION OF ARSENIC IN BIOLOGICAL MATERIAL.

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The quantitative estimation of arsenic in biological material, particularly in blood, urine, and feces, has become of increasing importance because of the widespread use of arsenicals in chemotherapy, with resultant clinical manifestations of arsenical poisoning in many instances. Like that of other investigators our experience has run the gamut of the various modifications of the Gutzeit method, criticisms of which will not be offered here since Fink has excellently summarized them in his article. None has given complete satisfaction. The electrolytic apparatus described by Fink (1) is the most simple and compact of any type hitherto described and usually serves well for the estimation of arsenic in blood, urine, and feces following oxidation of the organic matter in these materials. The apparatus has, however, not been ideal in our hands. In many instances low results have been obtained because of the necessity for diffusion of the arsenous acid from the anode to the cathode chamber before reduction to arsine may take place. It seemed better if possible to have the anode and cathode chambers separated by some type of diaphragm, as suggested by Trotman (2) and used in the apparatus described by him. This has resulted in the production of the apparatus as shown in Fig. 1. In this apparatus the entire sample of 15 to 16 cc. is placed in the cathode chamber which is separated from the anode chamber by a sealed in alundum disk. This insures complete and rapid reduction of all arsenous acid since it is always present on the reducing side of the apparatus.
Electrolytic Gutzeit Apparatus

Description.

The apparatus is entirely of Pyrex glass and can be made by any amateur fairly skilled in glass blowing. The apparatus is in the shape of a V, the two arms, C and G, serving respectively as cathode and anode chambers. These are 16 cm. in length, the cathode chamber having an internal diameter of 25 mm. and the anode chamber a diameter of 10 mm.; the two chambers are separated at their junction by a sealed in disk of alundum. The bent
arm, \( B \), of 6 mm. bore and 22 cm. in length is sealed to the cathode chamber and is attached, by means of a ground glass joint, to the horizontal arm, \( A \), which is 10 cm. in length, and serves to hold the sensitized paper. To retain any hydrogen sulfide which may be formed, arm \( B \) is loosely packed with glass wool moistened with a lead acetate solution and partially dried by pressing between towels. The cathode consists of a rounded lead sheet, 4 by 9 cm.; the anode is a platinum foil 26 by 70 mm. The two electrodes are fastened to the glass stoppers, \( D \) and \( E \), in the arms, \( C \) and \( G \), by means of sealed in platinum wires, which make connection through mercury to a copper wire, cemented through the top of the stopper and connecting with the source of the current. It is necessary that the stopper of only the cathode chamber be a ground joint. The cathode chamber holds approximately 16 cc., the anode chamber approximately 8 cc. A current of 1 ampere and 12 volts is used and as many of these units may be connected in series as desired. To prevent heating during electrolysis the electrode chambers are immersed in a bath of cold water.

**Method.**

The method here described is that which we have adopted as a routine for urine and feces and may be adapted to other material frequently met with in toxicological examinations. The method which is used for the destruction of organic matter is essentially that employed by Lawson and Scott (3). 10 cc. of urine or feces suspension, 20 cc. of concentrated sulfuric acid, and a crystal of copper sulfate are digested in a Kjeldahl flask until all organic matter is oxidized. I have found it unnecessary usually to use potassium sulfate; if possible this is omitted in order to avoid the separation of as much inorganic material in the final volume as possible. The digestion mixture is diluted to 100 cc. and an aliquot part taken for estimation. The volume of the aliquot part is dependent of course on the amount of arsenic present. A sample containing from 0.002 to 0.03 mg. of arsenic gives excellent results. As a sample, 0.5 cc. of stannous chloride solution (80 gm. of stannous chloride, 5 cc. of HCl, 95 cc. of water) is added to 40 cc. of the solution; the mixture is boiled to a volume of approximately 15 cc. and then cooled. This entire sample is added to the cathode chamber of the apparatus, the anode chamber having been filled
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previously with an electrolyte of 12.5 per cent sulfuric acid. The two electrodes are then put in place and the electrolysis begun.

The sensitized paper strips have been prepared essentially as described by previous authors. In our hands the unsensitized paper strips measuring 120 by 2.5 mm., as furnished by commercial apparatus firms, give good results. The papers are sensitized by warming on a steam bath in a 5 per cent alcoholic solution of mercuric bromide and allowing them to soak for 24 hours or more. The papers are then air-dried. When used they are moistened slightly by momentarily holding them over a steam bath. The period of electrolysis is usually 1 hour, although a shorter period is ample in most instances. At the end of this time the papers are immersed in potassium iodide solution, then in melted paraffin, and compared with standards obtained by adding known amounts of arsenic to urine. The calculation of the total arsenic present in the sample is obvious and depends on the volume of the sample and the size of the aliquot portion which is used.

CONCLUSIONS.

A modification of the electrolytic Gutzeit apparatus for the estimation of arsenic in biological material is described. The cathode and anode chambers are separated by an alundum disk. This permits the entire sample to be placed in the cathode chamber and insures rapid and complete conversion of all inorganic arsenic to arsine.

BIBLIOGRAPHY.

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