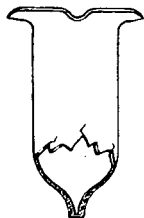


## APPARATUS FOR THE PRECIPITATION OF BARIUM SULPHATE.\*

BY A. G. MURRAY.

As is well known, the determination of sulphate by means of barium is subject to serious errors unless certain precautions are observed in the precipitation. Among other things it is necessary to have the sulphate solution boiling and to add the barium chloride solution slowly and preferably hot. The device illustrated is intended to facilitate this operation.



Apparatus for precipitation of barium sulphate.

It consists of a cup the bottom of which is drawn out to form a capillary opening of such size as to permit water to pass through at the rate of 2 or 3 drops a second. Depressions in the broad rim allow the escape of steam from a flask in the neck of which the device is placed.

A capacious Erlenmeyer flask containing the sulphate solution is placed over a flame and when the solution boils the requisite amount of dilute barium chloride solution is poured into the cup. By this means the barium chloride solution is introduced slowly and boiling hot into the sulphate solution. When the cold barium chloride solution is introduced into the cup the condensed steam trickles to its point, thus greatly diluting the first drops of barium chloride solution which fall into the sulphate solution.

## A NEW METHOD FOR THE PREPARATION OF STYRAX AND BALSAM OF TOLU FOR USE AS MICROSCOPIC MOUNTING MEDIA OF HIGH REFRACTIVE INDEX.

BY GEORGE H. NEEDHAM.

Mounting media of high refractive index are a necessity for the study of diatoms. As stated by Carpenter<sup>1</sup> "There is a marked increase of visibility in proportion as the mounting medium has a refractive index higher than the object mounted, . . . thus facilitating the discovery of obscure details."

Numerous substances have been used for this purpose ranging from styrax with a refractive index of 1.582 to artificial realgar with an index of 2.549.<sup>2</sup> The former, due to its resinous nature, has proved to be the best. To the writer's knowledge the majority of microscopists prepare styrax by just dissolving the commercial article in an organic solvent such as xylol or chloroform and filtering. Due to this quick method quite often a fine precipitate appears in the mount after several years. Balsam of Tolu, which has a higher refractive index of 1.618, has been discarded for this reason. In both cases, little thought seems to have been given to the complex nature of these two balsams and to the fact that the precipitate is either cinnamic or benzoic acid, or both.

\* Contribution from Bureau of Chemistry, U. S. Department of Agriculture. Demonstrated at the "Stunt Show" of the Scientific Section, A. Ph. A. meeting at Asheville, N. C., 1923.

<sup>1</sup> Wm. B. Carpenter, "The Microscope," London, 1891, p. 445.

<sup>2</sup> *Ibid.*, p. 1028.

It is necessary that the balsam used be examined carefully for adulteration. However, in the case of styrax nearly all of the commercial samples are adulterated. Therefore it should be light grayish brown in color, with an aromatic odor similar to benzoin, indicating oriental styrax (*Liquidambar orientalis*), and conform to the United States Pharmacopœial tests. Because of the scarcity of foreign styrax, American styrax (*Liquidambar styraciflua*) has been suggested and used as a substitute, due to its very similar composition. However, as yet the author has not used it in microscopic work.

For the reasons stated above and that quite recently<sup>1</sup> information was requested on the preparation of styrax for use as a mounting medium, the following simple method for preparing styrax and balsam of Tolu, practically free from cinnamic and benzoic acids and esters, is offered:

The styrax or balsam of Tolu is dissolved in warm absolute alcohol and filtered. The resultant liquid is poured slowly and with constant stirring into ten to twenty times its volume of distilled water. The precipitated resinous material is allowed to settle and the supernatant liquid decanted. The resins are freed as much as possible from adherent water and dissolved in alcohol again. The whole is poured into water as before. This process is repeated once more. Then the resins are in a fairly pure form and can be transferred to an evaporating dish and dried in a hot water oven. Finally the dried material is dissolved in sufficient chloroform so that the medium drops easily from a glass rod.

The prepared styrax or balsam of Tolu is used as follows: The diatoms on the cover glass or slide are cleared in xylol, the superfluous liquid drained off and two or three drops of the mounting medium added. The chloroform is volatilized by covering the slide with a small watch glass and keeping at 60° C. for half an hour. Then the mounting is finished as usual. However, after the cover glass is placed on the slide it is advisable to keep the mount at 60° C. for several hours. By doing this one may be sure that all of the volatile solvent has been driven off and hence secure the full benefit of the high refractive index.

The following refractive indices were determined after driving off the volatile solvent. The media were prepared by the method given. Water at 95° C. was run through the Abbé refractometer in order to melt the resin and secure contact between the prisms. The medium was then allowed to cool to 25° C. The indices given represent the average of four readings.

Styrax. Sample No. 1	Refractive Index 1.5595 at 25° C.
Styrax. Sample No. 2	Refractive Index 1.5923 at 25° C.
Balsam of Tolu. No. 1	Refractive Index 1.6035 at 25° C.

These results show that the refractive indices of the resins compare favorably with those given for the commercial material.

After three years no precipitation has occurred in any of the diatom slides mounted with media prepared as above. Of course, further time is necessary before one can draw definite conclusions as to permanence.

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<sup>1</sup> *Nature*, London, Vol. 113, 2831, p. 159, 1924.