10,000. With careful work and attention to details it should be accurate to a much greater degree than this, certainly to 0.03 part. It can hardly be said, however, that amounts of carbon dioxide varying from 0 to 40 parts in 10,000, can be determined without modification of the method. The barium hydroxide must always be present in considerable excess, a condition which is most readily attained by the use of a smaller sample of bad air. With a little practice, the method was found to be rapid and easy of execution and preferable to the Pettenkofer method.

BOSTON, MASS.,
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LIQUID BATHS FOR MELTING-POINT DETERMINATIONS.

BY HEYWARD SCUDDER.

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A MIXTURE prepared by boiling together for five minutes seven parts by weight of sulphuric acid (sp. gr. 1.84) and three parts by weight of potassium sulphate remains a transparent liquid at ordinary temperatures and can be heated up to 325° without boiling. If the proportions are changed to six parts by weight of acid and four parts of the sulphate the mixture forms a soft mass at ordinary temperatures (though after boiling and then cooling down it will usually remain liquid for half an hour or more) melting from 60°-100° and boiling above 365°. Acid potassium sulphate can be used instead of potassium sulphate. In this case the amount should be calculated to give the same ratio of potassium sulphate to sulphuric acid.

These mixtures are self-clearing and remain permanently white (turning slightly yellowish at about 230°) unless much organic matter gets in. They can be cleared, if brown, by boiling with a few drops of concentrated nitric acid or with a small crystal of potassium nitrate. The vapor is so slightly acid that the capillary can always be fastened to the thermometer by a rubber band, provided the band is 1 to 2 cm. above the surface of the bath. Platinum wire need never be used. In preparing the bath, after the potassium sulphate has melted the two layers should be mixed or explosive boiling may occur.

Most melting-points given accurately are below 325°. The first mixture is therefore suitable for all ordinary purposes. It

does not have to be renewed frequently as does the ordinary sulphuric acid bath nor does it become permanently discolored as does paraffin after short use, even though prepared by distillation under diminished pressure.

If the bath has solidified (which happens sometimes under unknown conditions though very rarely), boiling for a minute or two will bring it back to its original condition. If it has not been used for a number of weeks, it should be boiled for a minute or two before using for temperatures above 300°.

For temperatures from 360° to 600° Mr. Booth, working under the direction of Dr. Mulliken of this laboratory, has found that the most satisfactory bath is fused zinc chloride. This melts to a clear transparent liquid at about 250°. After use it must be poured out on a tile while liquid, since it expands on solidification and will crack any flask or beaker in which it is allowed to grow cold. It can be cleared from organic impurities by heating with a small crystal of potassium nitrate. Unless the zinc chloride used is pure, the bath will gradually become opaque after a short time and can not be made transparent again.

The temperatures given here are uncorrected. They were obtained in a melting-point apparatus made by inserting a test-tube in a round-bottomed flask (150-250 cc.). The bottom of the tube is about 1 cm. from the bottom of the flask. The bath is placed both in the test-tube and in the flask. In this apparatus fresh sulphuric acid (sp. gr. 1.84) can be used up to 280°-300°. In using the sulphate mixtures, if the upper part of the tube becomes cloudy from a film of solid, it can be cleared by boiling with a little concentrated nitric acid. In using such an apparatus it is not safe to heat it much above the temperatures given here for the various baths since the boiling is apt to start very violently. It is safe to heat it till large bubbles begin to rise moderately frequently.

When these baths are used in open beakers, it is not possible with sulphuric acid or the sulphate mixtures to obtain temperatures as high as those previously mentioned, without having disagreeable amounts of acid vapor given off. The zinc chloride bath can be used in a beaker up to about 600°.

The sulphate mixtures seem to contain some compound in a persistent state of supersaturation. Although possessed of considerable oxidizing power when hot, it is a curious fact that they

are markedly less caustic than hot concentrated sulphuric acid when dropped on the skin.

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THE PREVENTION OF BUMPING.

BY HEYWARD SCUDDER.

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A SINGLE glass capillary tube when used according to the following directions will stop most cases of bumping that occur in ordinary laboratory work. The method is simple, effective and introduces into the liquid no foreign substance except glass and one small bubble of air. In order to carry it out successfully the following details of procedure must be carefully observed.

The origin of the use of a capillary is obscure. The earliest published reference that I have found is by Gernez.[1] Since these references are only incidental and therefore difficult to find, it is quite possible that there may be an earlier one. I have tried all the methods published (most of which are only for special cases), and have found none so simple or of such wide application as the one presented here.

The capillary is made by drawing out a piece of glass tubing until the internal diameter is about 0.5 to 1 millimeter. A seal is then made by holding the tube horizontally in the edge of the flame of a Bunsen burner, until the walls have melted together. The tube is bent, if necessary, or held horizontally till cold. For most purposes the seal should be about 1 cm. from the open end of the tube. The tube is cut off at the desired length and the other end sealed to prevent the entrance of liquid. When cold, the tube is placed open end down in the liquid to be boiled. The open end should rest on the bottom of the vessel containing the liquid and should remain there during use. When liquids of high specific gravity are being boiled, it is necessary, therefore, to have the capillary so heavy that it will not be thrown off the bottom. This weight can be obtained by drawing out the tube from which the capillary is made only near the seal, or by using a very thick walled tube.

In a general way the theory of the action of such a tube is that

[1] *Compt. Rend.*, **86**, 472.