

ANALYSIS OF PHOSPHOR BRONZE, PHOSPHOR COPPER, PHOSPHOR TIN, ETC.

BY MAX WICKHORST.

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Determination of Phosphorus Alone.—The following is a method which may be used where phosphorus alone is wanted, in phosphor bronze, phosphor copper, phosphor tin, etc.

Treat one gram of the sample with twenty cc. aqua regia, consisting of fifteen cc. nitric acid and five cc. hydrochloric acid and warm. When the reaction is complete, add water, then excess of ammonia, and make up to 200 cc. Pass in hydrogen sulphide until the copper, lead, etc., are all precipitated, and filter. To 100 cc. add ten cc. of magnesia mixture and ammonia in the usual manner. The phosphorus in the metal has been oxidized to the pentoxide by the aqua regia, and it is here obtained as the usual magnesium ammonium phosphate precipitate. After a few hours, filter off the precipitate into a Gooch crucible and wash with diluted ammonia water, containing a little ammonium sulphide. Dissolve the precipitate in a little dilute hydrochloric acid, add a little magnesia mixture and reprecipitate with ammonium hydroxide. Collect on a Gooch filter, ignite, and weigh. From this calculate the phosphorus. For most purposes the double precipitation may be dispensed with and the first precipitate ignited and weighed.

Complete Analysis of Phosphor Bronze.—Treat one-half gram of borings with five cc. of strong nitric acid, applying heat. After the reaction is over, rub the residue well with a rod, add a little water, filter off the residue on a nine cm. filter and wash with water containing a little nitric acid. Put the moist filter with contents into a weighed porcelain crucible, apply heat, at first very gently, and finally with a blast-lamp. The weight of the residue gives stannic oxide plus phosphorus pentoxide. Fuse the residue with one-half gram of sodium carbonate and one gram of sulphur, with cover on the crucible. Heat with Bunsen burner until excess of sulphur is all driven off. Allow to cool and dissolve the mass in hot water, add excess of ammonia, then one gram of ammonium chloride, and when cold precipitate with magnesia mixture. Collect the precipitate in the same manner as above, dissolve in a little dilute hydrochloric acid,

add ammonia and magnesia mixture, and re-collect and weigh the precipitate. This gives magnesium pyrophosphate, from which calculate phosphorus.

To obtain tin calculate magnesium pyrophosphate to phosphorus pentoxide, subtract this from stannic oxide plus phosphorus pentoxide. This gives stannic oxide. Calculate this to metallic tin.

However, by the treatment of the bronze with nitric acid, the phosphorus is not all rendered insoluble in combination with the tin. A little of it goes into the filtrate, and in order to get this phosphorus, a duplicate sample may be weighed out with the first, treated in the same way and filtered. The filtrate is precipitated with molybdate solution and the phosphorus determined in this by one of the usual methods. This phosphorus is added to that obtained above.

Frequently the tin and phosphorus are found together as above and the phosphorus determined in a separate portion of the sample. This is then calculated to phosphorus pentoxide, which is subtracted from the stannic oxide plus phosphorus pentoxide to give stannic oxide. But such a method causes results for tin a little too low, because, as explained above, the phosphorus is not all retained by the tin.

To the filtrate, from the stannic oxide plus phosphorus pentoxide, add ammonium hydroxide until just about neutral, then add five cc. nitric acid, make up to about 150 cc. with water, transfer to a large platinum dish and suspend in this a platinum foil about two inches square. Pass electric current through so as to precipitate the lead as dioxide on the dish, and the copper as metallic copper on the foil. Test whether action is complete by taking out a drop and treating with ammonia. If no blue color develops, the copper is all precipitated, and so also the lead, as its precipitation is complete long before all the copper has come down. Siphon off the liquid from the precipitates, pouring in fresh water meanwhile, until most of the acid is gone. Then disconnect the current and wash the precipitates well with water. Dry in an oven and weigh. This gives metallic copper direct, and the lead can be calculated from the lead dioxide.

The liquid may still contain iron and zinc. To determine these, warm the liquid somewhat, add an excess of ammonium

hydroxide and then ammonium sulphide. Allow to stand in a warm place until the precipitate has all settled, collect the precipitate on a small filter, ignite in a porcelain crucible, very cautiously at first, and finally with a blast-lamp. This gives ferric oxide and zinc oxide. Dissolve the ferric oxide and zinc oxide in hydrochloric acid, add water, heat, precipitate with ammonium hydroxide, collect ferric hydroxide, and calculate to metallic iron. Subtract ferric oxide from ferric oxide plus zinc oxide. This gives zinc oxide. Calculate to metallic zinc

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A NEW FORM OF CONDENSER FOR THE DISTILLATION OF LIQUIDS HAVING LOW BOILING-POINTS.

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THE device to which I desire to call attention is one that will be of interest to all persons who find it necessary to distil large quantities of highly volatile liquids, during the warm months of the year. The apparatus is a comparatively simple one, consisting of a block-tin condensing worm surrounded by two copper jackets. Through the upper one of these, hydrant water is circulated for the preliminary cooling of the vapor; the lower one is filled with ice water for the complete condensation of the vapor and the thorough cooling of the distillate. In the apparatus that we have in use, this ice-water chamber is closed at the top and the ice water is prepared in a separate tank and allowed to flow into the jacket of the condenser at such a rate as the thermometer indicates to be necessary. Dr. Brown, formerly of this laboratory, has suggested that the ice-water chamber be made open and hopper-shaped at the top in order that the ice may be introduced directly into the cooling chamber. This method of construction would doubtless lessen the amount of ice as well as the attention necessary for the satisfactory working of the apparatus. When chloroform or other liquids of moderately high boiling-points are being distilled, and during cold weather, we use hydrant water in both chambers.

The accompanying illustration shows the general form of the apparatus and the device for supporting it. A condenser