XX.—*Purification of Phosphoric Oxide.*

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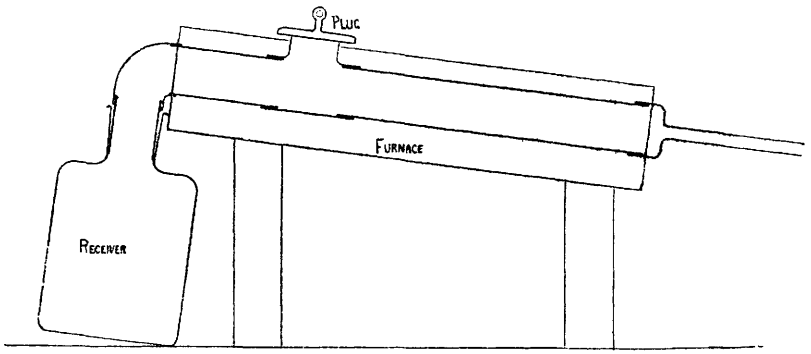
THE method for the purification of phosphoric oxide as devised by Finch and Peto (J., 1922, **121**, 692) has been employed in these laboratories for the last 4 years. In the light of the experience thus gained, several modifications in the original apparatus have been adopted which permit of the attaining of greater yields and increased rapidity of working. The modified form of apparatus consists of two iron tubes of 2-inch bore, the one 24 and the other 12 inches in length. These tubes are coaxially joined together by means of a $2\frac{1}{4}$-inch bore T-piece, the 1-inch long upper branch of which is machined flat and closed by the plug P, the lower surface of which is also machined. This 36-inch tube is supported in an inclined, gas-fired combustion furnace. Oxygen is led from a cylinder through glass wool into the lower end of the tube, the attachment being made by means of a length of $\frac{1}{4}$-inch iron tubing and a screwed adaptor. The cylinder oxygen employed in these laboratories is made by fractional distillation of liquid air and therefore needs no preliminary drying. The glass wool serves to retain dust. The upper and condensing end of the tube consists

of a $2\frac{1}{4}$-inch smoothly rounded, right angle bend, into the lower end of which a 2-inch length of 2-inch bore tubing is screwed. The receiver in which phosphoric oxide is condensed consists of a $1\frac{1}{2}$ litre glass-stoppered bottle, the neck of which is just sufficiently wide to admit the 2-inch bore iron tube. The right angle bend as far as the neck of the bottle is well lagged with asbestos cloth and heated by means of a large Téclu burner in order to prevent condensation in the tube itself.

The method of operation is as follows. The main, 36-inch tube is first brought to a bright red heat. A rapid stream of oxygen (3 litres per minute) is then admitted, and 10 to 15 g. of the impure phosphoric oxide are fed in, in one lot, through the short branch of the T-piece into the main tube, whereupon the plug is immediately replaced. The viscous phosphoric acid soon formed on the

FIG. 1.



machined surfaces of the plug and T-piece forms an effective seal. Rapid volatilisation of the oxide occurs, and a dense white cloud of pure crystalline phosphoric oxide is seen to condense in the receiver. A fresh charge of impure oxide is introduced as soon as the condensation due to the previous charge subsides. Glassy phosphoric acids formed in the main tube flow down towards the oxygen inlet end. They may be removed when the tube is cold by unscrewing the $\frac{1}{4}$-inch adapter and hammering the tube. The necessity for this does not arise until after several pounds of oxide have been distilled. The 24-inch section of the main tube serves to preheat the oxygen and to distil the volatile portions of the viscous oxides. The distillation section of the main tube must be kept at a bright red heat, and the right angle bend and the short piece of tubing attached to it and entering the receiver must be kept at a sufficiently high temperature to prevent condensation anywhere in the iron tube itself. At a normal rate of working,

about 500 g. of oxide can be distilled in this manner in 1 hour. The yield obtained varied from 70 to more than 80%, according to the nature of the original impure oxide and the skill of the operator. Owing to the rapid rate and the high temperature (above 800°) at which distillation occurs, a uniform crystalline product of a volume about 5 times that of the original oxide, and thus well suited for drying operations, is obtained. In order to obtain rapidly a good yield of pure product, distillation should not be commenced until the main tube is at a bright red heat with a large excess of oxygen passing through it. The receiver serves as the storage bottle, the stopper being lubricated with phosphoric oxide. The pure product obtained and stored in this manner will keep indefinitely.

The suitability of a phosphoric oxide for use in the experimental study of heterogeneous gas reactions or the attainment of a Bakerian degree of dryness depends on its vapour pressure, voluminous nature, and freedom from impurities, such as lower oxides of phosphorus and traces of organic matter. Smits and Rutgers (J., 1924, **125**, 2573) have shown that heating at temperatures above 400° yields that form of phosphoric oxide which exhibits the minimum vapour pressure. The rapid distillation carried out in the manner described above yields a crystalline product of great voluminousness. Traces of organic impurities may be detected by gently heating the moistened product in a test-tube; discoloration occurs if organic matter is present. The absence of lower oxides of phosphorus is best confirmed by the silver nitrate test described by Finch and Peto (*loc. cit.*). The mercuric chloride test also mentioned by them and advocated by Whitaker (J., 1925, **127**, 2219) has been found by us to be less sensitive than the silver nitrate test and therefore is not to be recommended.

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SOUTH KENSINGTON, S.W. [*Received, December 18th*, 1925.]