

## NOTES.

*Note on an Improved Method of Preparing Selenophen.* By HENRY VINCENT AIRD BRISCOE, JOHN BUTTERY PEEL, and PERCY LUCOCK ROBINSON.

IN the preparation of selenophen by heating selenium at  $400^{\circ}$  in a current of acetylene, considerable quantities of benzene and other hydrocarbons are also formed (Briscoe and Peel, this vol., p. 1741). By the following modification, however, the proportion of these inconvenient by-products may be greatly diminished. Selenium, in portions of 5 g., is heated in an inclined Pyrex tube, closed at the lower end, and provided with an axial inlet tube delivering acetylene some inches above the selenium and well below a side tube leading to a condenser. The middle of the tube is heated to redness, and the selenium is slowly vaporised by heating it from the surface downwards. The first evidence of reaction is the flashing of the acetylene (compare Peel and Robinson, this vol., p. 2068) with deposition of carbon, and thereafter a liquid condensate is formed at the rate of 3—4 c.c. in 4 hours. As in the previous experiments, the reaction proceeds more smoothly if the selenium is mixed with the carbonaceous residue from previous runs.

About 135 c.c. of the crude brown oil thus produced were fractionally distilled through a 4-foot, glass, bead-filled column and a 1-foot, glass, spiral column for the more volatile and the less volatile fractions respectively, and yielded (i) 20 c.c. of benzene, b. p.  $80^{\circ}$ ; (ii) 80 c.c. of selenophen, b. p.  $110^{\circ}$ ; (iii) 10 c.c. of liquid hydrocarbons, b. p.

110—120°; (iv) 10 c.c. of liquid hydrocarbons, b. p. 150—180°; (v) 10 g. of naphthalene; and (vi) 4 c.c. of a green liquid containing selenium.

The last fraction, on microfractional distillation, gave as the middle fraction a sky-blue liquid, b. p. about 240—250°, containing about 20% of selenium, which was probably an impure specimen of selenophthen, the analogue of thiophthen.

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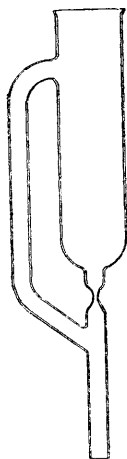
*The Preparation of Acetonecyanohydrin.* By KENNETH NORMAN WELCH and GEORGE ROGER CLEMO.

ACETONECYANOHYDRIN has been prepared from anhydrous hydrogen cyanide and acetone (Urich, *Annalen*, 1872, **164**, 255), and also by the action of potassium cyanide on acetone sodium bisulphite (Bucherer and Grolée, *Ber.*, 1906, **39**, 1225). The latter method is commonly given in text-books, but it has not been found possible to obtain the yields claimed, on account of the decomposition during distillation of the highly coloured, crude, reaction product. This decomposition appears to be due mainly to impurities which catalyse the following balanced reaction in the forward direction:  $\text{CMe}_2(\text{OH})\text{CN} \rightleftharpoons \text{CMe}_2\text{O} + \text{HCN}$ . Moreover, a considerable amount of a solid residue always remains in the distillation flask.

The following simple method does not need anhydrous hydrogen cyanide, and is also free from the above-mentioned objection. Acetone (29 g.) is added to a solution of potassium cyanide (31 g.) in water (100 c.c.). Sulphuric acid (167 g.; 30%  $\text{H}_2\text{SO}_4$  by weight), is then added slowly with constant stirring, the temperature being kept below 20° by the addition of ice, which also, by diluting the solution, prevents it from becoming unstirrable on account of the separation of potassium hydrogen sulphate. The reaction mixture is then extracted three times with ether, the extract dried over sodium sulphate, the bulk of the ether removed on the water-bath, and the residue rapidly distilled in a vacuum, whereupon acetonecyanohydrin (32 g.) passes over at 81°/15 mm. The compound thus prepared is much more stable than that obtained by Bucherer's method (*loc. cit.*), but even so, it is advantageous to distil it rapidly.—ARMSTRONG COLLEGE, NEWCASTLE-ON-TYNE. [Received, July 7th, 1928.]

*Apparatus for Chromic Anhydride Oxidations.* By WALLACE FRANK SHORT.

DURING the course of some experiments on the oxidation of diphenylmethane derivatives in acetic acid solution, it was found that addition of a concentrated aqueous solution of chromic acid caused the separation of unoxidised material from solution and retarded the reaction. On the other hand, addition of solid chromic anhydride resulted in poor yields of the substituted benzophenones, since they were partly destroyed under these conditions by further oxidation. Addition of an acetic acid solution of chromic anhydride produced the desired result, but the volume of the solution was inconveniently large on account of the comparatively small solubility of the anhydride. This difficulty was overcome by allowing the anhydride to dissolve in the acetic acid returning to the reaction flask from the reflux condenser. For this purpose, a modification of the apparatus designed by Walker (J., 1892, **61**, 717) for the preparation of ethyl iodide was employed. The apparatus (figured in  $\frac{1}{4}$  natural size) was mounted in a cork in the flask containing an acetic acid solution of the substance to be oxidised, and was provided with a reflux condenser. The constriction in the chromic anhydride reservoir had an internal diameter of approximately 2 mm., and was loosely plugged with glass wool. The introduction of the oxidising agent may be controlled by regulating the boiling of the acetic acid solution in the flask.



The apparatus may also be employed for the preparation of alkyl iodides by Walker's method (*loc. cit.*) and for the oxidation of organic substances by potassium permanganate in aqueous or acetone solution.—UNIVERSITY COLLEGE, AUCKLAND, NEW ZEALAND. [Received, August 2nd, 1928.]

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