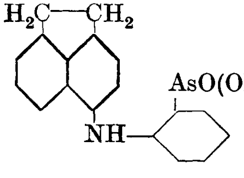
NOTES.

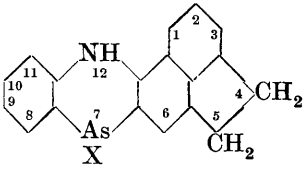
Substances of the Phenarsazine Type containing the Acenaphthene Nucleus. By CHARLES STANLEY GIBSON and JOHN DOBNEY ANDREW JOHNSON.

THE generality of the method previously described (J., 1927, 2499; 1928, 2204) for the preparation of organic arsenicals is further illustrated by the preparation of such compounds from 3-aminoacenaphthene.

3-Aminoacenaphthene (30 g.), *o*-bromophenylarsinic acid (50·4 g.), anhydrous potassium carbonate (34·2 g.), amyl alcohol (150 c.c.), and a trace of copper powder boiled for 5 hours gives a 17·5% yield of crude o-(3-*acenaphthylamino*)*phenylarsinic acid* (I). The crude acid, which is not easy to purify, is dissolved in dilute aqueous ammonia and treated with an equal volume of aqueous ammonia (*d* 0·880); the ammonium salt is then precipitated in small shining plates. From an aqueous solution of this salt the acid is recovered by means of acetic acid; and it now crystallises from dilute acetic acid in fine colourless needles, decomp. 180—181° (Found: As, 20·3. $C_{18}H_{16}O_3NAs$ requires As, 20·3%).

(I.)





(II.)

On reduction of the crude acid (7·2 g.), dissolved in a hot mixture of alcohol (40 c.c.) and concentrated hydrochloric acid (40 c.c.) containing a trace of iodine, with sulphur dioxide an orange-red precipitate is obtained. If the reaction follows the usual course, this compound is 7-*chloro*-12 : 7-*dihydro*iso*acenaphthabenzarsazine* (II, X = Cl). On crystallisation from toluene, it is obtained in deep orange-red, rhombic plates, m. p. 241° (decomp.) (Found: Cl, 10·0; As, 21·0. $C_{18}H_{13}NClAs$ requires Cl, 10·0; As, 21·2%).

7-*Bromo*-12 : 7-*dihydro*iso*acenaphthabenzarsazine* (II, X = Br), similarly prepared from the above acid, hydrobromic acid (38%) being used instead of hydrochloric acid, is obtained in deep orange-red, rhombic prisms, m. p. 244—246° (decomp.) (Found : Br, 20·1. $C_{18}H_{13}NBrAs$ requires Br, 20·1%).

The oxidation of the above chloro-compound by the methods generally employed does not appear to proceed smoothly.—GUY'S HOSPITAL MEDICAL SCHOOL (UNIVERSITY OF LONDON), LONDON, S.E.1. [*Received, June 12th,* 1929.]

The Reaction between Magnesium Phenyl Bromide and Phenyl Glycide. By D. R. BOYD and G. J. C. VINEALL.

*γ-Phenoxy-α-phenyl*iso*propyl Alcohol.*—A solution of phenyl glycide (14·5 g.) in 20 c.c. of ether was added in small quantities to a cooled Grignard reagent prepared from 2·4 g. of magnesium, 16 g. of bromobenzene, and 40 c.c. of ether. The mixture was heated for 2 hours in a water-bath, the ether removed, and heating continued at 100° until frothing occurred. Water was added to the cooled product, which was then subjected to steam distillation until the distillate was only slightly milky. The cooled residue was treated with hydrochloric acid and the precipitated oil was separated after solidification, and crystallised from alcohol and water. After recrystallisation from ether–light petroleum it formed very small plates, m. p. 92·5° (Found : C, 79·0; H, 7·0. $C_{15}H_{16}O_2$ requires C, 78·95; H, 7·0%).

When the reaction was carried out as described, 16·5 g. of crude product (75% of the theoretical yield) were obtained, and an ethereal extract of the steam distillate gave only bromobenzene and phenyl glycide. When the reaction mixture was not heated after the addition of the phenyl glycide, the yield was reduced. It was almost negligible if heating was omitted after removal of the solvent ether, and it was very much diminished if the heating was not continued until the residue frothed. An oil containing bromine, b. p. 165°/15 mm., probably α-bromo-γ-phenoxy*iso*propyl alcohol, was obtained from the steam distillate in those cases where the magnesium addition compound was not heated before decomposition with water. Ribas (*Anal. Fís. Quím.*, 1928, **26**, 122) obtained α-bromo-γ-methoxy*iso*propyl alcohol along with a 25% yield of γ-methoxy-α-phenyl*iso*propyl alcohol by the action of magnesium phenyl bromide on methyl glycide. γ-Phenoxy-α-phenyl*iso*propyl alcohol is readily soluble in the usual organic solvents, but difficultly so in light petroleum. It reacts with phosphorus trichloride to give a product which on decomposition with ice yields an ester of phosphorous acid soluble in ammonia. The ester is less stable towards hydrolysing agents than the corresponding derivative of αγ-diphenoxy*iso*propyl alcohol (Boyd, J., 1901, **79**, 1221), and attempts to crystallise it failed.—UNIVERSITY COLLEGE, SOUTHAMPTON. [*Received, June 1st,* 1929.]