

## NOTES.

*Tetrabromodimethylquinoxaline.* By GEORGE MACDONALD BENNETT and GERVASE HEWITSON WILLIS.

THE substance of m. p.  $234^{\circ}$  resulting from the bromination of 2:3-dimethylquinoxaline described by Henderson (J., 1929, 466) as the 5:6:7:8-tetrabromo-derivative had previously been isolated by us (J., 1928, 1960) by brominating the base in acetic acid solution, the product having m. p.  $228^{\circ}$  (uncorr.) or  $233^{\circ}$  (corr.).

Although the bromine atoms in this compound are not readily removed by aqueous hydrolysis, they are in fact situated in the side chains and not in the nucleus, the substance being 2:3-di-( $\omega$ -dibromomethyl)-quinoxaline, for the halogen reacts quickly with silver acetate in boiling acetic acid solution or with potassium acetate in boiling ethylene glycol solution. Attempts to isolate the organic product of these reactions or its condensation product with hydrazine have again been unsuccessful.

If further support were needed for our formulation of this compound, it would be supplied by our results in brominating other quinoxalines: 3-phenyl-2-methylquinoxaline, for example, furnished a dibromo-derivative.

3 M

It follows that the dibromo-compound of m. p.  $150^{\circ}$  described also by Henderson is presumably 2 : 3-di-( $\omega$ -bromomethyl)quinoxaline.—THE UNIVERSITY, SHEFFIELD. [*Received, June 13th, 1930.*]

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*Compounds of Arsenious Chloride and Pyridine.* By CHARLES STANLEY GIBSON, JOHN DOBNEY ANDREW JOHNSON, and (the late) DUDLEY CLOETE VINING.

SHIREY (*J. Amer. Chem. Soc.*, 1930, **52**, 1720) has isolated a compound of arsenious chloride and pyridine having the composition  $\text{AsCl}_3 \cdot 2\text{C}_5\text{H}_5\text{N}$ , whereas Walden's studies (*Z. physikal. Chem.*, 1903, **43**, 445) of the conductivities of solutions in solvents other than water indicated the probable formation of a compound  $\text{AsCl}_3 \cdot \text{C}_5\text{H}_5\text{N}$ . Such a compound was not isolated either by Walden or by Shirey.

Under experimental conditions different from those described by Shirey, we have isolated a *compound* of the composition  $\text{AsCl}_3 \cdot \text{C}_5\text{H}_5\text{N}$ . It is highly crystalline and, although readily attacked by moisture, is comparatively stable. Two typical preparations are described in each of which carefully purified and dried materials were used and moisture was rigidly excluded.

(a) Arsenious chloride (9 g.; 1 mol.) was mixed with chlorobenzene (20 c.c.) containing pyridine (4.4 g.; 1.1 mols.). The colourless crystals which separated after some hours were filtered off, washed with chlorobenzene, and recrystallised from warm chlorobenzene. The cooled filtrate deposited colourless, highly refractive, needle-shaped crystals, which were washed with chlorobenzene and ether and dried under reduced pressure over sulphuric acid. In a sealed tube, the crystals softened at  $120^{\circ}$  and melted at  $138$ – $139^{\circ}$ . The compound reacted with water, pyridine hydrochloride and arsenious oxide being formed. For analysis, the chlorine and arsenic were determined by titration in the usual manner, and the pyridine by distillation with excess of sodium hydroxide and absorption in a known excess of standard sulphuric acid (Found: Cl, 39.2; As, 27.5;  $\text{C}_5\text{H}_5\text{N}$ , 33.3.  $\text{AsCl}_3 \cdot \text{C}_5\text{H}_5\text{N}$  requires Cl, 40.85; As, 28.8;  $\text{C}_5\text{H}_5\text{N}$ , 30.4%).

(b) Arsenious chloride (29.2 g.; 1 mol.), pyridine (12.7 g.; 1 mol.), and chlorobenzene (80 c.c.) were mixed and after some hours the liquid became filled with colourless needle-shaped crystals. These were filtered off on a sintered glass filter, washed with chlorobenzene, and left on porous porcelain under reduced pressure over sulphuric acid. In a sealed tube, the material softened from  $115^{\circ}$  and melted at  $133$ – $136^{\circ}$ . The material analysed was not free from adhering chlorobenzene, which was identified in the distillate during the

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estimation of the pyridine [Found: Cl (ionised), 38.5; As, 26.7;  $C_5H_5N$ , 28.0.  $AsCl_3.C_5H_5N$  containing 6.9% of chlorobenzene requires Cl, 38.0; As, 26.8;  $C_5H_5N$ , 28.3%].

Additional evidence for the existence of a compound having the composition  $AsCl_3.2C_5H_5N$  as described by Shirey was also obtained. Pyridine (10.3 g.; 2 mols.), arsenious chloride (11.8 g.; 1 mol.), and chlorobenzene (35 c.c.) were mixed under the usual conditions. The amount of crystalline material which separated was small. In a sealed tube at  $65^\circ$  it melted to a milky liquid which became clear at about  $100^\circ$ . This would correspond to an approximate melting point of  $64^\circ$  as deduced from the freezing-point rise of a mixture of arsenious chloride (1 mol.) and pyridine (2 mols.) as indicated by Shirey.—GUY'S HOSPITAL MEDICAL SCHOOL (UNIVERSITY OF LONDON), LONDON, S.E.1. [*Received, June 5th, 1930.*]

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