## NOTES

The  $\beta$ -Chlorovinyl-arsines.<sup>1</sup>—The work of Miller and Witherspoon<sup>2</sup> has shown that the compound obtained by reacting  $\beta$ -chlorovinyl-dichloroarsine with diphenylamine is not 6- $\beta$ -chlorovinyl-phenarsazine, as reported in the above entitled article, but 6-chloro-phenarsazine. The compound is extremely difficult to obtain pure by crystallizing from solvents. Five crystallizations from xylene gave a product melting at 189° (uncorr.). By vacuum sublimation, Miller reports a melting point of 193–194°.

Similar correction should hold for the product obtained when phenyl- $\alpha$ -naphthylamine is condensed with  $\beta$ -chlorovinyl-dichloro-arsine. Thus in both cases the amines apparently condense with arsenic chloride, present as a result of decomposition or equilibrium, giving a chloro-arsine and not a chlorovinyl-arsine.

> W. LEE LEWIS H. W. STIEGLER

RECEIVED JUNE 23, 1930 PUBLISHED OCTOBER 6, 1930

**Preparation of Benzene-azo Derivatives of 8-Hydroxyquinoline.**— According to J. Mathëus,<sup>1</sup> the alkaline coupling of 8-hydroxyquinoline with diazobenzene-chloride gives 5-benzene-azo-8-hydroxyquinoline. In the present work, which was executed to obtain such a compound for use in the Skraup reaction with certain azo compounds, when diazobenzene-chloride and 8-hydroxyquinoline were coupled in equimolecular proportion in an alkaline medium with as small an amount of water as possible, the chief product obtained was found to be 5,7-benzene-disazo-8-hydroxy-quinoline, which after three recrystallizations from alcohol gave deep violet crystals of excellent purity; m. p. 205–206°; yield, 16 g. from 14.5 g. of 8-hydroxyquinoline. Its alcoholic solution assumes a red color, and the solution in concd. sulfuric acid is indigo blue, but on addition of alcohol the color changes to violet red.

Anal. Subs., 6.320: CO<sub>2</sub>, 16.558; H<sub>2</sub>O, 2.488. Subs., 3.115, 1.897: N<sub>2</sub>, 0.576 cc. (30°, 762 mm.) 0.344 cc. (31°, 756 mm.). Calcd. for C<sub>21</sub>H<sub>15</sub>ON<sub>5</sub>: C, 71.39; H, 4.25; N, 19.83. Found: C, 71.45; H, 4.37; N, 20.08, 19.41.

On the other hand, when the coupling was conducted in a very dilute solution, the mono-azo compound was the chief product, which after four recrystallizations from alcohol gave brownish-yellow needles (m. p. 185– 186°); its properties were identical with those of 5-benzene-azo-8-hydroxyquinoline given by Mathëus. The mono-azo compound, however, was

<sup>1</sup> "The  $\beta$ -Chlorovinyl-arsines and their Derivatives," W. Lee Lewis and H. W. Stiegler, THIS JOURNAL, 47, 2546 (1925).

<sup>2</sup> Unpublished, Chemical Division, Edgewood Arsenal, Maryland.

<sup>1</sup> Mathëus, Ber., 21, 1644 (1888).