

Isolation of an Arsenic Compound of Pyridine and Some Observations Concerning the Phase System Arsenic Trichloride-Pyridine.—During the past twenty years many articles have appeared describing reactions that take place between pyridine and inorganic salts. Most of the studies that have been made in this country trace back, directly or indirectly, to Dr. L. Kahlenberg. The studies have usually been accompanied by phase rule interpretations.

In so far as the author knows, no study has been made of the system arsenic trichloride-pyridine, though evidence of reaction between these compounds is recorded in the well-known studies of P. Walden.¹ Walden predicted from conductivity studies that a compound containing one molecule of pyridine and one molecule of arsenic trichloride was formed from interaction between these chemicals, but he made no attempts to isolate or describe any of the properties of such a compound. While a compound of the formula predicted by Walden was not isolated in this work, a compound giving the theoretical arsenic content for the compound $\text{AsCl}_3 \cdot 2\text{C}_5\text{H}_5\text{N}$ was isolated.

Experimental.—The best grade of pyridine produced by Kahlbaum was distilled from potassium permanganate, then from fused barium oxide and the fraction coming over between 114.9 and 115.3° at 750 mm. was stored over recently fused potassium hydroxide. Arsenic trichloride of c. p. grade was distilled from recently sublimed metallic arsenic. Upon taking an excess of the purified pyridine (*i. e.*, about 75 mole per cent.), and thoroughly mixing it in a dry container with the prepared arsenic trichloride, there was much heat evolved. Upon cooling in a dry atmosphere distinct rosetts of crystals were produced. These crystals were freed from mother liquor by thorough centrifuging. Since the crystals were highly hygroscopic, it was impractical to wash them free of mother liquor by use of organic solvents. The compound was analyzed for arsenic by decomposing it with alkali and titrating with iodine solution. The iodine solution had been carefully standardized against arsenious acid made up from purified and resublimed arsenious oxide. The following analyses were obtained: Sample I, As, 22.11%; II, 21.91%; III, 22.16%. The theoretical amount of arsenic in the compound $\text{AsCl}_3 \cdot 2\text{C}_5\text{H}_5\text{N}$ is 22.08%.

Phase rule applications to this system are particularly difficult due to undercooling and to tendency toward obtaining abnormal time-temperature cooling curves. While an arsenic trichloride-pyridine mixture of 66.67 mole per cent., with reference to pyridine, gives a distinct freezing point rise, roughly fixing the melting point for the compound $\text{AsCl}_3 \cdot 2\text{C}_5\text{H}_5\text{N}$ at 64°, the curve may or may not be continuous in its rise.

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¹ P. Walden, *Z. physik. Chem.*, **43**, 445 (1903).