

By the interaction of potassium iodide with diazotized *p*-anisidine a 75–80% yield of pure *p*-iodo-anisole can be obtained.

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Note on the Purification of Brucine.¹—Brucine has come to be the alkaloid most commonly used in resolving optically active acids. The free base can be recovered after the resolution and used over and over. If one tries to obtain the alkaloid by precipitating it with ammonium hydroxide from a solution of the hydrochloride, the result is a paste which is difficult to filter and impossible to wash adequately.

The following procedure gives a crystalline product of a high degree of purity. To the solution of brucine hydrochloride is added about one-fifth its volume of ethyl alcohol. The solution is then made definitely alkaline with ammonium hydroxide. After standing for several hours crystals begin to appear. Sometimes twenty-four to forty-eight hours or even longer must elapse before crystallization starts. In about a week the precipitation is complete. The crystals are filtered, washed and air-dried. They are then ready for use again in resolution.

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A Variation of the Carius Method for the Determination of Sulfur.—Among the various modifications of the Carius method for the determination of sulfur, it is frequently suggested that bromine¹ or a few crystals of potassium bromide² be added to the fuming nitric acid. This modification probably arose through the observation that halogenated compounds are more readily oxidized.³ Rupp⁴ introduced the variation of placing a barium salt in the tube with a marked shortening of the time required to gather the resulting sulfate. He was aware of Carius' observation as to the greater ease of oxidation of chlorine containing compounds but did not correlate the use of barium chloride with any observed

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¹ Angeli, *Gazz. chim. ital.*, **21**, [1] 163 (1891).

² Thorpe and Whiteley, "Students Manual of Organic Chemical Analysis," Longmans, Green and Co., London, 1926, p. 59.

³ Carius, *Ann.*, **116**, 19 (1860).

⁴ Rupp, *Chem.-Ztg.*, **32**, 984 (1908).