

PREPARATION AND SPECTRA OF
PURE BROMOiodoacetylene and MONOiodoacetylene

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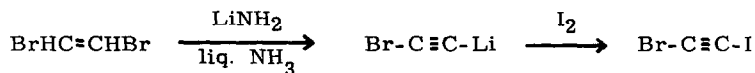
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In recent papers we reported the preparation (2) and vibrational spectra (3) of bromochloroacetylene and chloriodoacetylene, hitherto unknown, and of dichloro-, dibromo- and diiodoacetylene. Several attempts to synthesize bromoiodoacetylene via the same route failed.

We now present the successful preparation of this "missing link" in the dihaloacetylene series. Furthermore, we give a simple synthetic route to the almost undescribed monoiodoacetylene, which was needed in extended spectroscopic investigations of haloacetylenes. (4).

Bromoiodoacetylene (m. p. $15,3 - 16,5^{\circ}$, v. p. $8,5$ torr/ $17,5^{\circ}$, yield ca. 30%) was prepared as follows. 1,2-Dibromoethylene was reacted with lithium amide in liquid ammonia to produce lithium bromoacetylide (5), which by subsequent reaction with molecular iodine yielded bromoiodoacetylene.



The product was identified by its mass spectrum (m/e 230/232 (M^+), 151 (C_2I^+), 139 (CI^+), 127 (I^+), 115/116 (M^{++}), 103/105 (C_2Br^+), 91/93 (CBr^+), 79/81 (Br^+), 24 (C_2^+), and by its infrared spectrum, which showed weak absorption bands in the gas phase at 2158 and 785 cm^{-1} . These bands were assigned to the $-\text{C}\equiv\text{C}-$ str. and $\text{X}-\text{C}\equiv\text{C}-\text{Y}$ asym. str. modes, respectively, in accordance with the infrared absorption bands of dibromo- and diiodoacetylene (3).

Monoiodoacetylene (m. p. $-14,0 - -13,5^{\circ}$, v. p. 66 torr/ 0° , yield ca. 1%) was prepared by rapidly bubbling acetylene through a solution of iodine in liquid ammonia (6). Its infrared spectrum was in agreement with the literature (4a). The mass spectrum exhibited

peaks at m/e 152 (M^+), 151 (C_2I^+), 139 (CI^+), 127 (I^+), 76 (M^{++}), 25 (C_2^+) and 12 (C^+).

The gas chromatographed products (Apiezon L, 40 - 90°) were isolated on a gram scale. Collected at low temperatures, they crystallized as long needles. No impurities were observed in the mass spectra.

Detailed descriptions of the preparative techniques, the vibrational spectra, photoelectron spectra, mass spectra and ultraviolet spectra will be published elsewhere.

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References and Notes

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4. a) J. K. Brown and J. K. Tyler, Proc. Chem. Soc. **1961**, 13. b) W. J. Jones, B. P. Stoicheff and J. K. Tyler, Can. J. Phys. **41**, 2098 (1963).
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6. Owing to the ready iodination of both acetylenic hydrogens, the reaction yields diiodoacetylene preponderantly. See T. H. Vaughn and J. A. Niewland, J. Amer. Chem. Soc. **54**, 787 (1932), for the preparation of diiodoacetylene.