Methyliodine(III) Difluoride

By James Andrew Gibson and Alexander F. Janzen*
(*Department of Chemistry, University of Manitoba, Winnipeg, Canada*)

Summary Methyliodine(III) difluoride may be prepared in quantitative yield by the reaction of xenon difluoride with methyl iodide at room temperature.

Several reports have appeared concerning the synthesis of perfluoroalkyliodine(III and V) fluorides.[1–3] These syntheses involve the use of powerful fluorinating agents such as ClF_3, BrF_3, and F_2 and would not be applicable for the synthesis of alkyliodine(III or V) fluorides owing to destructive fluorination of alkyl groups. The use of xenon difluoride as a mild, selective fluorinating agent has been reported by this [4] and other laboratories.[5] We now report a convenient synthesis of methyliodine(III) difluoride by the reaction of xenon difluoride with an excess of methyl iodide.

The reaction proceeds smoothly during 20 min, in the absence of a solvent at room temperature, in essentially quantitative yield; no reaction was observed below -40 °C. The n.m.r. spectrum of the product, dissolved in an excess of methyl iodide, gave a triplet δ_H $-$ 4·23 p.p.m. (external Me_4Si) and a quartet δ_F + 176·4 p.p.m. (external $CFCl_3$), with J_{HCIF} 7 Hz, confirming the structure of CH_3IF_2. The fluorine chemical shift is close to that of CF_3IF_2 (+172·7 p.p.m.[3]).

CH_3IF_2 was found to be surprisingly stable in solution, 50% remaining after 4 h at 20°, suggesting that the product is at least as stable as CF_3IF_2.[2,3] Excess of methyl iodide may be removed *in vacuo* from the involatile product. The product reacts slowly with glass and rapidly with mercury, while decomposition at room temperature gives iodine and oily material.

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