The preparation of iodomethylmercuric iodide

A recent report by Seyferth[1] on the reactions of iodomethylmercuric iodide has caused a resurgence of interest in this simple compound first prepared by Sakurai[2] and subsequently by Baldoni and Miyashiro[3]. Our interest in this compound was aroused by the successful methylene-transfer reagent resulting from the reaction of methylene iodide with zinc[4]. Several workers have privately noted difficulties in following the literature procedure for the preparation of iodomethylmercuric iodide. In this note we report our procedure for its preparation and its spectral properties.

Experimental

Iodomethylmercuric iodide. In a 1-liter three-necked flask equipped with mechanical stirrer and condenser was charged mercury (60.3 g, 0.3 mole) and methylene iodide (402 g, 1.5 mole) (Matheson, Coleman and Bell). The mixture was stirred and irradiated with a G.E. sunlamp kept at a distance of *ca.* 4–6" from the flask. After 3.5 h the mercury puddle had disappeared. The resulting solution was cooled and treated

SPECTRAL DATA

Compound	*IR*	*UV* (λ_{max}, ϵ)	*NMR* [$(CD_3)_2SO$]
ICH_2HgI	3000 cm^{-1} (w) 1050 cm^{-1} (s) 656 cm^{-1} (s)	227 mμ, 22,000 244 mμ, 27,700	7.32τ
$IHgCH_2HgI$	2940 cm^{-1} (w) 943 cm^{-1} (s) 663 cm^{-1} (s)	237 mμ, 5200 262 mμ, 4400	8.15τ

with 200 ml of benzene. The precipitate was collected on a filter and air dried to leave 97 g (69%) of product as a pale yellow powder. Recrystallization from hot (93°) methylene iodide (28 ml) gave 67.6 g of material as pale yellow crystals, m.p. 113–116° (lit.[2] m.p. 108–109°). Pure methylene iodide to which is added a few crystals of iodine may be used in place of the commercial material.

Methylenebis(mercuric iodide). Iodomethylmercuric iodide (2.0 g) was dissolved in THF (25 ml) and heated at reflux for 2.5 h. Filtration left 0.52 g of the subject compound as a colorless powder, m.p. 232–234° (lit.[2] m.p. 230°).

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