Hexachloropentalenyl Bis(hexachloroantimonate)

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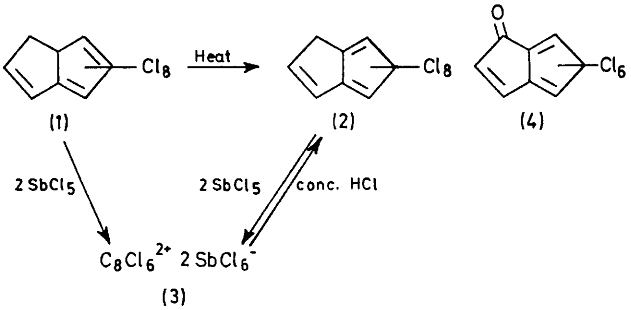
Summary Octachlorobicyclo[3,3,0]octa-1,3,7-triene (δ-C_8Cl_8) and octachlorobicyclo[3,3,0]octa-1,4,6-triene, (ε-C_8Cl_8) react with antimony pentachloride to give hexachloropentalenyl bis(hexachloroantimonate).

We have investigated the reactions of δ-C_8Cl_8 (**1**), m.p. 85—86 °C, and ε-C_8Cl_8 (**2**), m.p. 111—112 °C,[1,2] both having a [3,3,0]-carbon skeleton, with Lewis acid metal halides, in relation to our interest in perchlorinated non-benzenoid aromatic compounds and the hexachloropentalenyl dication.

Treatment of (**2**) with $SbCl_5$ (2 mol. equiv.) in dry CH_2Cl_2 under dry nitrogen resulted in immediate precipitation at room temperature. The mixture was heated to reflux for 5 min to complete the reaction and then cooled, to give yellow crystals (**3**), which were washed repeatedly with small portions of dry dichloromethane, ν_{max} (CH_2Cl_2) 1600m, 1370m, 1320s, 1155vw, 1105m, 1093m, 1062vw, 1030vw, 805m, 685vw, 570w, and 345m cm^{-1}. The i.r. spectrum of the complex is different from those of each component, and those of physical mixtures of the components. The absorption at 345 cm^{-1} appears to be due to $SbCl_6$.[3] (**1**) reacted with $SbCl_5$ essentially under the same conditions, to give a transient violet colour which disappeared within 5 min giving the same compound (**3**). Quenching of (**3**) in conc. HCl gave (**2**) and (**4**).

Reaction of (**2**) with anhydrous $AlCl_3$ (2 mol. equiv.) afforded a similar 1:2 complex, whose i.r. spectrum was essentially identical with that of (**3**) above 570 cm^{-1}, indicating the presence of the same type of organic species in these complexes. A band due to $AlCl_4^-$ was observed at 480 cm^{-1}.[3]



These spectral data, and analytical evidence indicate that the organic fragment is a dication, $C_8Cl_6^{2+}$, with a [3,3,0]-carbon skeleton. Although we have no definite evidence for the structure of (**3**) we believe the simplicity of its i.r. spectrum, in comparison with those of (**1**) and (**2**), rules out structures with localized charges, which can exist in six forms.

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[1] A. Roedig and R. Kohlhaupt, *Tetrahedron Letters*, 1964, 1107; A. Roedig, R. Helm, R. West, and M. Smith, *ibid.*, 1967, 3137; R. Crigee and R. Huber, *Chem. Ber.*, 1970, **103**, 1862.

[2] R. West, *Accounts Chem. Res.*, 1970, **3**, 130.

[3] R. West and P. T. Kwitowski, *J. Amer. Chem. Soc.*, 1966, **88**, 5280; K. Kusuda, R. West, and V. N. Mallikarjuna Rao, *ibid.*, 1971, **93**, 3627.