

The yields of the alkazones are quite good from the three, four, and five carbon sugars. The yield from six carbon sugars is very poor, and the reaction in this case is quite slow. The reason for this difference between the five and six carbon sugars is not clear, but it appears to be associated with side reactions in the slower oxidations of the hexoses.

The complete oxidation of sugars by 1-methylphenylhydrazine explodes a myth based on intuition rather than fact. It also opens new vistas of theoretical and practical consequence and provides a facile synthetic entry to a unique class of highly unsaturated compounds.

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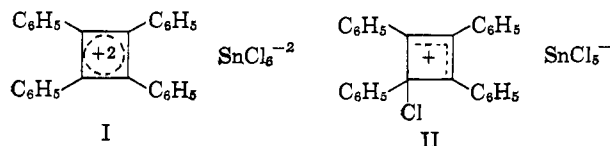
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The Crystal and Molecular Structure of a Carbonium Ion. 3-Chloro-1,2,3,4-tetraphenylcyclobutenium Pentachlorostannate

Sir:

The synthesis of the supposed dicarbonium ion salt, tetraphenylcyclobutenium hexachlorostannate (I), has recently been reported.¹ A three-dimensional structure analysis, by X-ray diffraction methods, of crystals of this material² has been carried out with the result that, at any rate in the solid state,³ the compound in question is the isomeric monocation salt 3-chloro-1,2,3,4-tetraphenylcyclobutenium pentachlorostannate (II). Since, as far as the author is aware, the literature contains no reports of structure determinations either of carbonium ions⁴ or of pentachlorostannate anions, some details of the structure of this salt may be of interest.



3-Chloro-1,2,3,4-tetraphenylcyclobutenium pentachlorostannate crystallizes in the orthorhombic system, in space group $Pbca$, with $a = 19.50 \pm 0.03$, $b = 27.42 \pm 0.05$, $c = 10.55 \pm 0.02$ Å. There is one molecule per asymmetric unit containing 35 atoms, excluding hydrogen.

Some 3560 reflections, representing three-dimensional data to about 1.1 \AA^{-1} , were measured from a single crystal, sealed in a thin-walled glass capillary, with $\text{Mo K}\alpha$ radiation. The intensity measurements were converted to structure amplitudes by means of a program specially written to handle the punched card output of the Hilger-Watts linear diffractometer used. No absorption corrections were applied.

The position of the tin atom in the unit cell was found from a study of the three-dimensional Patterson function and the rest of the structure determined from successive Fourier approximations to the electron density. The final electron-density distribution is shown in Fig. 1. Refinement was carried through in eight cycles of full matrix least-squares minimalization of the dif-

(1) H. H. Freedman and A. M. Frantz, *J. Am. Chem. Soc.*, **84**, 4165 (1962).

(2) Crystals suitable for X-ray work were kindly supplied by Dr. H. Freedman.

(3) H. H. Freedman and A. E. Young, *J. Am. Chem. Soc.*, **86**, 734 (1964).

(4) NOTE ADDED IN PROOF.—See, however, M. Sundaralingam and L. H. Jensen, *ibid.*, **85**, 3302 (1963).

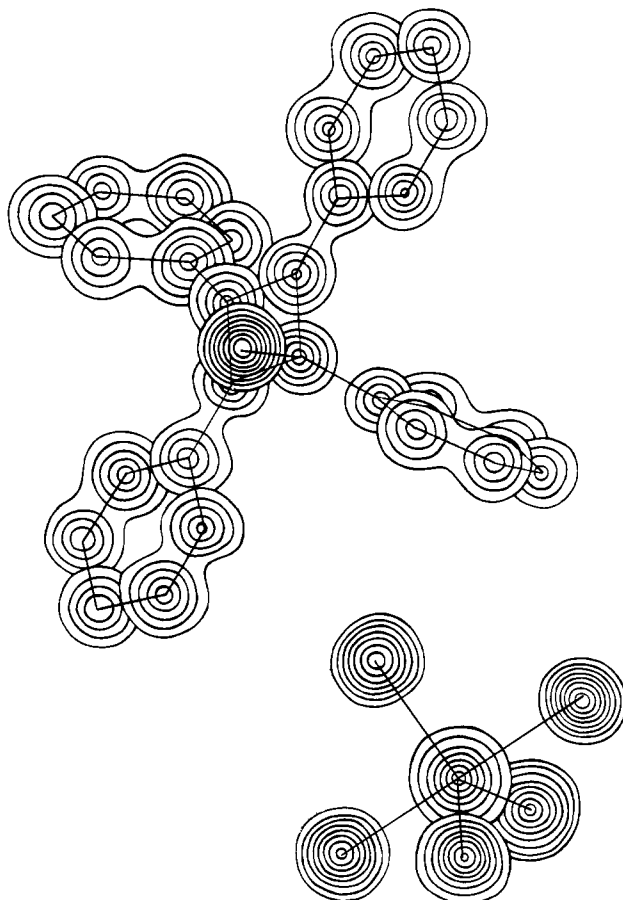


Fig. 1.—The electron density distribution over one formula unit in 3-chloro-1,2,3,4-tetraphenylcyclobutenium pentachlorostannate. The contours are at arbitrary, but equal, intervals for the various atomic species.

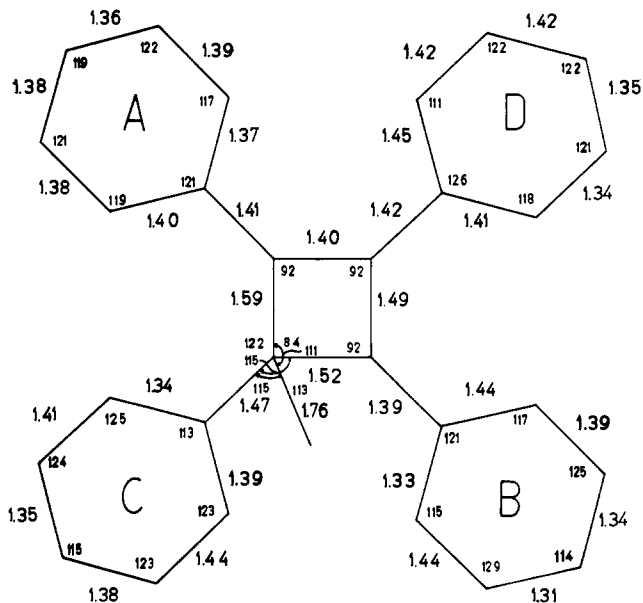


Fig. 2.—Bond lengths and angles in the cation of 3-chloro-1,2,3,4-tetraphenylcyclobutenium pentachlorostannate.

ferences between observed and calculated structure amplitudes. All 105 positional parameters, 35 isotropic thermal parameters, and 3 scale factors were varied in alternate cycles to give a value for R of 0.202 for all reflections. The values of the bond lengths and angles derived from the final positional coordinates of the cation are shown in Fig. 2. The standard deviation in