The Molecular Structure of 1,6-Epoxy[10]annulene

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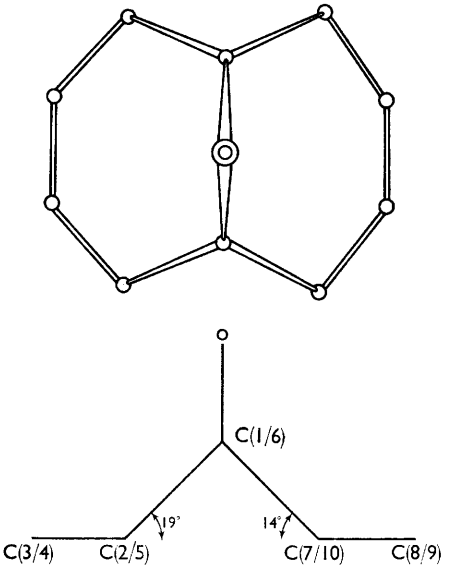
THE preparation and characterisation of 1,6-epoxy-[10]annulene has been reported by Sondheimer and Shani.[1] A detailed structural analysis of the closely related 1,6-methano[10]annulene-2-carboxylic acid[2] has been completed by Dobler and Dunitz.[3] Relatively poor quality crystals of epoxy[10]annulene were grown from petroleum ether (b.p. 30—40°); the crystals are orthorhombic, space group *Pbca*, with a unit cell, $a = 14{\cdot}16$ Å, $b = 9{\cdot}84$ Å, $c = 11{\cdot}28$ Å, and $Z = 8$. Three-dimensional, room-temperature, visually estimated intensity data consisting of only 278 independent reflexions were observed and the phases of 58 strong reflexions ($E_{hkl} \geqslant 1{\cdot}2$) were determined using the Σ_2 relationship of Karle and Karle.[4] A Fourier synthesis using the normalised structure factors, E_{hkl} as coefficients clearly showed the molecular skeleton. Least-squares refinement of positional and anisotropic vibrational parameters for the oxygen and carbon atoms has reduced R to 0·108; the positions of hydrogen atoms were approximately determined from difference Fourier syntheses and adjusted to give acceptable bond lengths and bond angles.



FIGURE

The structure (Figure) is very similar to the carboxylic acid derivative of 1,6-methano[10]-annulene although the present determination of bond lengths and bond angles is less precise [$\bar{\sigma}$(C–C) = 0·03 Å; $\bar{\sigma}$ (C–C–C) = 2°]. The eight carbon atoms not directly associated with the

bridge, that is C(2)–C(5) and C(7)–C(10), are essentially coplanar (the r.m.s. deviation from the mean plane is 0·025 Å) while the re-entrant carbon atoms, C(1) and C(6), each lie 0·35 Å above the plane and the oxygen atom is displaced 1·25 Å from this plane. The C–O–C angle is 102° compared with 99·6° for the related carbon atom in the methano-analogue.

The tendency towards bond alternation noted[3] in the methano[10]annulene is not seen in epoxy[10]-annulene, where all the bond lengths observed from our present analysis at room temperature are within 2σ of 1·39 Å. The results of a study of the molecular structure at 95° K, which is under way, are, however, necessary before any precise comparison can be made.

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[1] F. Sondheimer and A. Shani, *J. Amer. Chem. Soc.*, 1964, **86**, 3168.
[2] E. Vogel and H. D. Roth, *Angew. Chem. Internat. Edn.*, 1964, **3**, 228.
[3] M. Dobler and J. D. Dunitz, *Helv. Chim. Acta*, 1965, **48**, 1429.
[4] J. Karle and I. L. Karle, *Acta Cryst*, 1966, **21**, 849.