Molecular and Crystal Structure of *cyclo*-Undecasulphur, S_{11}

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Single crystals of S_{11} (space group $Pca2_1$) are shown to consist of cyclic molecules of approximately C_2 symmetry with bond lengths of 203.2—211.0 pm, bond angles of 103.8—108.4°, and torsion angles of 69—140°.

cyclo-Undecasulphur, S_{11}, is a minor component of liquid sulphur and of certain synthetic sulphurs[1] and has recently been prepared as bipyramidal crystals from $(C_5H_5)_2TiS_5$ and S_6Cl_2 at 0 °C.[2] This is the first report of the crystal and molecular structure of an eleven-membered homocyclic molecule.

Crystal data: S_{11}, $M = 352.7$, orthorhombic, space group $Pca2_1$, $a = 1493(1)$, $b = 832.1(5)$, $c = 1809(1)$ pm, $Z = 8$, $D_c = 2.08$ g cm^{-3} (−110 °C, Mo-K_α, $\lambda = 71.069$ pm, $\mu = 19.3$ cm^{-1}). Least squares refinement, based on 1982 observed reflections [$I > 2\sigma(I)$; 2042 measured reflections] recorded on a Syntex $P2_1$ diffractometer at −100 °C, converged to $R = 0.032$ (anisotropic thermal parameters, no absorption correction).†

The crystal lattice consists of molecules in general positions but with approximately C_2 symmetry and with only van der Waals' type interactions (shortest intermolecular distance 337 pm). The asymmetric unit contains two molecules of identical conformation but with slightly differing molecular parameters (maximum difference in bond lengths 3.3 pm). The data for only one molecule are given below and in Figure 1.

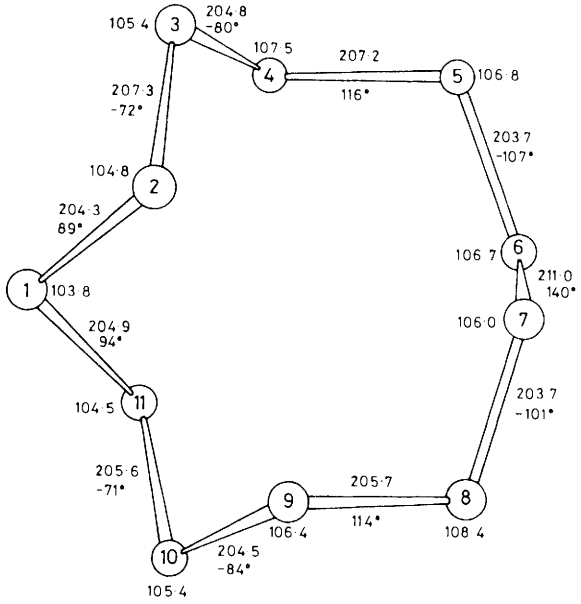

Figure 1. Molecular structure of S_{11} [numbering of atoms, bond lengths (pm), bond angles (°), and torsional angles of molecule 1].

The conformation of S_{11} can be characterized by the motif +−−+−+−−++−− which represents the order of signs of the torsion angles around the ring. Formally, the molecules can be generated from an S_5 unit cut from the S_8

† The atomic co-ordinates for this work are available on request from Prof. Dr. G. Bergerhoff, Institut für Anorganische Chemie, Universität, Gerhard-Domagk-Str. 1, D-5300 Bonn 1, F.R. Germany. Any request should be accompanied by the full literature citation for this communication.

molecule (motif $+-+-+\ldots$) and an S_6 unit cut from S_{12} (motif $--++--++\ldots$). The odd-numbered ring size results in a low molecular symmetry; the C_2 axis passes through atom 1 and the middle of bond 6–7 which is the longest in the molecule owing to the exceptionally large torsion angle 5–6–7–8 (140°). This is the largest torsional angle so far observed for a cyclic molecule of type S_n.

The bond distances show an alternation around the ring which is characteristic of homocyclic sulphur molecules of low symmetry (*cf.* S_7[3] and S_{10}[4]). The bond lengths are in agreement with the S–S stretching wavenumbers observed in the Raman spectrum[2] which are correlated to the distances.[5] The mean bond length of 205.7 pm is almost identical with those observed in S_8 (205.1), S_{10} (205.6), and S_{12} (205.2 pm[6]). Therefore, the enthalpy of formation of S_{11} from S_8 should be quite small. The bond angles found are in the normal range of 103.8—108.4° (S_8 108.2°, S_{10} 106.2°, and S_{12} 106.6°).

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