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1,3,5,7-Tetraselenocane

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Abstract. $(CH_2Se)_4$, orthorhombic, $Pna2_1$, a = 10·713 (8), b = 6·904 (4), c = 11·662 (3) Å, Z = 4, D_c = 2·86 g cm^{-3}, V = 862·55 Å^3, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 182·0 cm^{-1}. The structure, solved by Patterson–Fourier methods, was refined to R = 0·041. The molecule assumes a twist-chair conformation.

Introduction. The compound was prepared by Russo, Mortillaro, Credali & De Checchi (1966) and the crystals were grown from light petroleum.

The intensities were collected from a crystal of approximate dimensions 0·1 × 0·25 × 0·4 mm, on a Philips PW 1100 four-circle diffractometer operating in the $\theta/2\theta$ scan mode (scan width = 1·2°, scan speed = 0·04° s^{-1}) with Mo $K\alpha$ radiation monochromatized by a graphite crystal. 795 independent reflections up to θ = 25° were measured, of which 604 had $I \geq 3\sigma(I)$, $\sigma(I)$ being calculated from counting statistics. During the data collection two standard reflections were measured every 90 min to check the stability of the crystal and electronics. Intensities were corrected for Lorentz and polarization factors; an experimental absorption correction was applied following the method suggested by North, Phillips & Mathews (1968).

Approximate positions of the four Se atoms were deduced from two-dimensional Patterson maps. Structure factor calculations based on refined positions of these heavy atoms yielded a conventional R factor of 0·12. A three-dimensional difference electron density map showed the positions of all C atoms, which were included in the subsequent cycles of full-matrix least-squares refinement. The coordinates of the H atoms were calculated and included in the last cycle of refinement, but not varied. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $1/w = 0·55 \times \{1 + [(F_o - 43)/15]^2\}$. The correction for the real and imaginary parts of the anomalous dispersion was applied for Se only. The final R and R_w values for all observed reflections were 0·041 and 0·052 respectively.

The scattering factors were obtained from *International Tables for X-ray Crystallography* (1968). All the calculations were carried out on the Cyber 76 computer of the 'Centro di Calcolo Interuniversitario dell'Italia Nord Orientale' with the XRAY system of crystallographic programs (Stewart, Kundell & Baldwin, 1970).

Discussion. Polyselenomethylene can be obtained in two crystalline forms, hexagonal and orthorhombic, by cationic polymerization in bulk of triselenane and tetra-

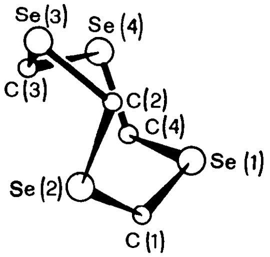

Fig. 1. A perspective view of 1,3,5,7-tetraselenocane.

Table 1. *Final positional parameters with their standard deviations for non-hydrogen atoms*

	x	y	z
Se(1)	0·3482 (2)	0·3584 (3)	0·0795 (2)
Se(2)	0·1194 (1)	0·2710 (3)	0·2720 (2)
Se(3)	0·0364 (2)	0·7253 (3)	0·2648 (3)
Se(4)	0·1488 (2)	0·7029 (3)	0·0
C(1)	0·269 (2)	0·183 (3)	0·188 (2)
C(2)	0·164 (2)	0·541 (3)	0·302 (2)
C(3)	0·011 (2)	0·656 (3)	0·108 (2)
C(4)	0·208 (2)	0·439 (3)	−0·013 (2)

Table 2. *Atomic coordinates for hydrogen atoms*

	x	y	z
H′(C1)	0·243	0·055	0·140
H″(C1)	0·339	0·145	0·251
H′(C2)	0·186	0·555	0·393
H″(C2)	0·247	0·576	0·253
H′(C3)	−0·010	0·503	0·106
H″(C3)	−0·069	0·736	0·078
H′(C4)	0·131	0·346	0·010
H″(C4)	0·233	0·416	−0·101

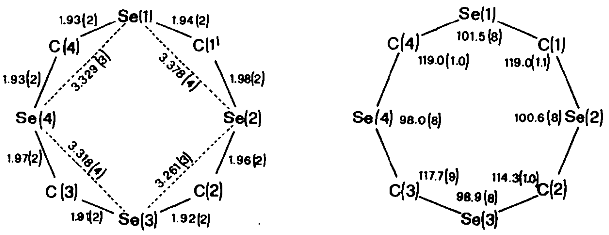

Fig. 2. Interatomic distances (Å) and angles (°) in 1,3,5,7-tetraselenocane.

Table 3. *Torsion angles* (°)

The torsion angle of the bonded group *A—X—Y—B* is the angle between the planes *A—X—Y* and *X—Y—B*. Sign according to Klyne & Prelog (1960).

Se(1)–C(1)–Se(2)–C(2)	38·5
C(1)–Se(2)–C(2)–Se(3)	131·5
Se(2)–C(2)–Se(3)–C(3)	−55·4
C(2)–Se(3)–C(3)–Se(4)	−66·2
Se(3)–C(3)–Se(4)–C(4)	103·0
C(3)–Se(4)–C(4)–Se(1)	−82·5
Se(4)–C(4)–Se(1)–C(1)	69·6
C(4)–Se(1)–C(1)–Se(2)	−129·1

selenocane respectively (Russo, Mortillaro, Credali & De Checchi, 1966). The radiation-induced solid-state polymerization of triselenane single crystals yielded also twinned crystals of orthorhombic polyselenomethylene (Carazzolo & Mammi, 1967). In contrast we have not been able to induce polymerization in the crystals of cyclic 1,3,5,7-tetraselenocane, $(CH_2Se)_4$, the structure of which is reported here (Fig. 1).

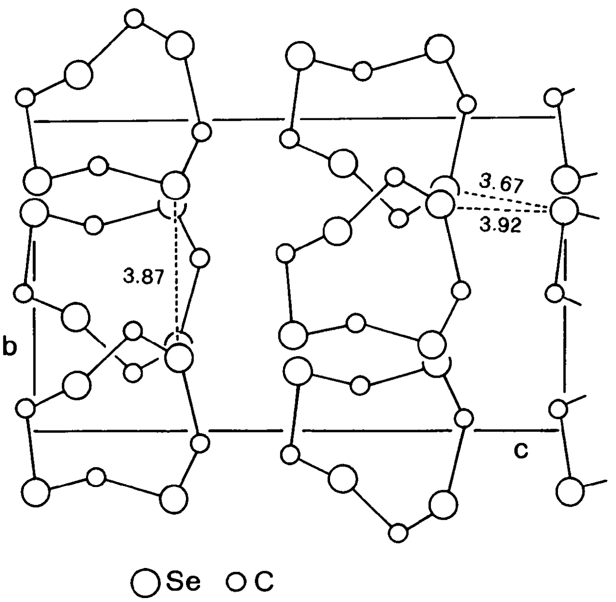

Fig. 3. The projection of the structure down *a*; some relevant intermolecular contact distances are marked.

The final positional parameters with their e.s.d.'s in parentheses for non-hydrogen and H atoms are listed in Tables 1 and 2 respectively; a schematic drawing of the molecule with distances and angles is shown in Fig. 2.* The C—Se bond distances, which are in the range 1·91–1·98 Å (mean = 1·94 Å), and the C—Se—C valence angles (mean = 99·8°) are in good agreement with those of analogous Se compounds (Mammi, Carazzolo, Valle & Del Pra, 1968; Valle, Busetti & Mammi, 1973; Valle, Zanotti & Del Pra, 1975). On the other hand, the carbon valence angles (mean = 117·5°) are larger than in the previously quoted compounds, but compare well with those found in analogous S-containing rings (Frank & Degen, 1973). The deviations of bond lengths and valence angles from the mean value are not significant.

The molecular conformation, described by the torsion angles listed in Table 3, can be defined as twist-chair (Fig. 1), being slightly distorted from the boat-chair predicted by Hendrickson (1967) for cyclooctane and found in the crystal of 1,3,5,7-tetrathiacyclodecane (Frank & Degen, 1973); a boat-like conformation has been also found in the crystal of 1,3,5,7,9-pentathiacyclodecane (Valle, Piazzesi & Del Pra, 1972).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33503 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The rather rigid molecular packing is shown in Fig. 3: each molecule is surrounded by twelve first-nearest neighbours, with Se–Se intermolecular contact distances ranging from 3·6 to 4·1 Å; some distances shorter than the sum of the van der Waals radii (Pauling, 1960) are indicated on the figure.

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Acide Ethacrynique

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Abstract. $C_{13}H_{12}Cl_2O_4$, triclinic, $P\bar{1}$, $a = 13{\cdot}244$ (3), $b = 15{\cdot}162$ (3), $c = 7{\cdot}434$ (2) Å, $\alpha = 105{\cdot}86$, $\beta = 98{\cdot}81$, $\gamma = 92{\cdot}92°$, $V = 1412$ Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, μ(Cu $K\alpha$) = 42 cm^{-1}, m.p. 215 °C. The structure was solved by direct methods and refined by least-squares calculations to a final $R = 0{\cdot}06$ for 2483 observed reflexions. The asymmetric unit contains two independent molecules.

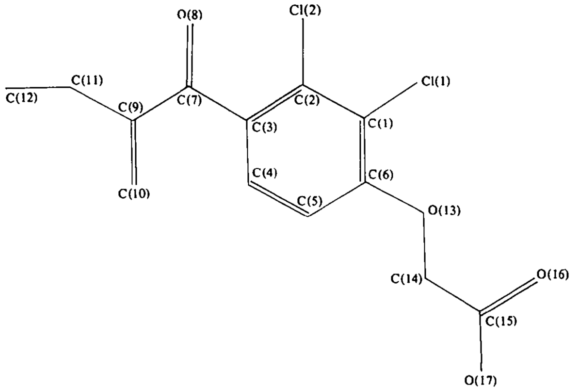

Fig. 1. Acide éthacrynique.

Introduction. Dans le cadre de nos recherches sur les diurétiques, nous avons déterminé la structure cristalline du principal représentant des acides carboxyliques diurétiques: l'acide éthacrynique (nomenclature commerciale: Edecrin®) (Fig. 1).

3534 réflexions uniques ont été mesurées à l'aide d'un diffractomètre à quatre cercles Hilger & Watts à la longueur d'onde $K\alpha$ du cuivre. Parmi celles-ci, 2543 ont été considérées comme observées [$I > 2\sigma(I)$]. La structure a été résolue en utilisant le programme *MULTAN* (Declercq, Germain, Main & Woolfson, 1973) et affinée par moindres carrés dans l'approximation des blocs diagonaux (Ahmed, Hall, Pippy & Saunderson, 1967) avec des facteurs de température anisotropes pour tous les atomes non-hydrogène. Les coordonnées des hydrogènes correspondent à des valeurs calculées, maintenues fixes sauf celles de H(1) et H(2) que l'on a laissé varier dans les deux derniers cycles d'affinement.