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3,4-Dichloro-6-selenatricyclo[5.4.0.$0^{2,5}$]undeca-1(7),8,10-trien-2-yl Acetate, $C_{12}H_{10}Cl_2O_2Se$

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Abstract. $M_r = 336{\cdot}0$, monoclinic, $P2_1/c$, $a = 12{\cdot}223\,(3)$, $b = 21{\cdot}631\,(6)$, $c = 7{\cdot}272\,(4)$ Å, $\beta = 137{\cdot}88\,(2)°$, $V = 1289{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}73$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 3{\cdot}51$ mm^{-1}, $F(000) = 664$, $T = 294$ (2) K, final $R = 0{\cdot}043$ for 2430 unique reflections. The chlorinated cyclobutane fragment has a *trans* configuration. The Se—C bond lengths are 1·917 (7) and 1·965 (7) Å; the C—Se—C bond angle is 89·6 (3)°. The lone-pair electrons of the Se atom are not involved in bond conjugation.

Introduction. Different examples of photo cycloaddition have been studied for 1,2-dichloroethylene, and it has been found very difficult to establish unambiguously from proton NMR analysis the configuration of the cyclobutane obtained in the reactions.

In some cases, where the structures have been solved by X-ray diffraction, only one of the stereoisomers obtained has been identified (Dilling, Tabor, Boer & North, 1970; Tökes, Christensen, Cruz & Crabbe, 1971). During the photoaddition of 1,2-dichloroethylene to benzo[*b*]selenol-3-yl acetate, we always observed the same two stereoisomers (four are expected), whatever stereochemistry we used (Tran Quang Minh, Christiaens, Grandclaudon & Lablache-Combier, 1977). We know the structure of one of these (Dideberg, Dupont & Baiwir, 1977), but this is insufficient for an accurate knowledge of the photochemical addition.

The X-ray crystallographic study reported here supplies more information towards an understanding of the photoaddition processes in these compounds.

Experimental. Mixture of two stereoisomers obtained from photoaddition of 1,2-dichloroethylene (*cis* or *trans*) to benzo[*b*]selenol-3-yl acetate. Title compound isolated from mixture using chromatography. Crystals recrystallized in a 1:1 heptane/toluene solution (Tran Quang Minh *et al.*, 1977). D_m not determined. Single crystal 0·3 × 0·2 × 0·4 mm. Philips PW 1100 four-circle diffractometer. Lattice parameters from 25 reflections ($20° < \theta < 27°$). ω–2θ scan, scan width fixed at 1·0°. 3485 intensities with $\sin\theta/\lambda \le 0{\cdot}745$ Å^{-1}, $-12 \le h \le 12$, $0 \le k \le 32$, $0 \le l \le 7$; 1325 unobserved [$I < 3{\cdot}0\sigma(I)$]. Three standard reflections monitored at intervals of 2 h (3% variation). Lp correction; absorption and extinction ignored. *MULTAN* (Germain, Main & Woolfson, 1971) and Fourier methods. Anisotropic full-matrix refinement on F (Sheldrick, 1976). H from $\Delta\rho$ synthesis, isotropic not refined. $R_w = 0{\cdot}043$, unit weights. Max. $\Delta\rho$ in final map 0·2, $-0{\cdot}3$ e Å^{-3}. $(\Delta/\sigma)_{\max} = 0{\cdot}14$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965).

Discussion. Final atomic parameters are in Table 1,* bond distances and angles in Table 2. Of the four diastereoisomers in Fig. 1 we are concerned here with structure (1); a perspective view of the molecule with the numbering of the atoms is displayed in Fig. 2.

The geometry of the molecule of this diastereoisomer can be compared with that found by Dideberg, Dupont & Baiwir (1977).

All the atoms of the benzo[*b*]selenole are in the mean plane with a deviation less than 0·06 Å, except for atoms C(10) and C(7). The atoms of the lateral-chain fragment are perfectly in a plane which makes an angle of 81·9° with the benzo[*b*]selenole fragment, instead of 88·7° for the other isomer. The cyclobutane fragment has also undergone a tetrahedral deformation whose amplitude is 0·14 Å and its plane makes an angle of

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and details of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39378 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic parameters*

Fractional positional parameters are given $\times 10^4$. $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$. Values in angle brackets are anisotropicities defined by $[\sum(U_{eq} - U_{ii})^2/3]^{1/2}$.

	x	y	z	U_{eq}(Å$^2 \times 10^2$)
Se	5311 (1)	6446 (1)	3950 (1)	78 ⟨1⟩
Cl(2)	−1051 (3)	7694 (1)	3599 (2)	87 ⟨2⟩
Cl(1)	3305 (3)	7973 (1)	2880 (2)	102 ⟨2⟩
O(2)	−2158 (6)	6475 (1)	2170 (3)	44 ⟨4⟩
O(1)	−1807 (8)	5593 (2)	1363 (5)	75 ⟨6⟩
C(9)	1278 (9)	7286 (2)	3759 (6)	52 ⟨6⟩
C(11)	−3159 (10)	5932 (2)	1305 (6)	47 ⟨6⟩
C(10)	692 (9)	6591 (2)	3265 (5)	40 ⟨5⟩
C(1)	2558 (9)	6135 (2)	4652 (5)	45 ⟨6⟩
C(8)	1042 (10)	7412 (2)	2444 (6)	59 ⟨6⟩
C(12)	−6086 (10)	5853 (3)	370 (7)	49 ⟨8⟩
C(6)	4969 (10)	6013 (2)	5162 (6)	50 ⟨6⟩
C(2)	2041 (10)	5856 (2)	5408 (7)	86 ⟨8⟩
C(3)	4012 (10)	5455 (3)	6711 (8)	86 ⟨10⟩
C(5)	6905 (10)	5608 (3)	6432 (7)	60 ⟨8⟩
C(7)	1503 (10)	6731 (2)	2414 (6)	52 ⟨6⟩
C(4)	6369 (10)	5331 (3)	7201 (7)	61 ⟨9⟩

Table 2. *Bond lengths* (Å) *and bond angles* (°), *with e.s.d.'s in parentheses*

Se–C(6)	1·917 (7)	C(11)–C(12)	1·503 (12)
Se–C(7)	1·965 (7)	C(10)–C(1)	1·508 (6)
Cl(2)–C(9)	1·787 (8)	C(10)–C(7)	1·559 (11)
Cl(1)–C(8)	1·779 (7)	C(1)–C(6)	1·383 (11)
O(2)–C(11)	1·373 (5)	C(1)–C(2)	1·369 (11)
O(2)–C(10)	1·427 (8)	C(6)–C(5)	1·384 (8)
O(1)–C(11)	1·186 (10)	C(2)–C(3)	1·398 (9)
C(9)–C(10)	1·557 (6)	C(3)–C(4)	1·359 (16)
C(9)–C(8)	1·510 (9)	C(5)–C(4)	1·393 (13)

C(6)–Se–C(7)	89·6 (3)	C(10)–C(1)–C(2)	125·3 (6)
C(11)–O(2)–C(10)	116·3 (4)	C(6)–C(1)–C(2)	120·2 (6)
Cl(2)–C(9)–C(10)	118·0 (4)	Cl(1)–C(8)–C(9)	117·1 (4)
Cl(2)–C(9)–C(8)	116·4 (4)	Cl(1)–C(8)–C(7)	119·8 (5)
C(10)–C(9)–C(8)	88·8 (4)	C(9)–C(8)–C(7)	89·8 (5)
O(2)–C(11)–O(1)	122·7 (6)	Se–C(6)–C(1)	114·0 (5)
O(2)–C(11)–C(12)	109·0 (5)	Se–C(6)–C(5)	124·6 (5)
O(1)–C(11)–C(12)	128·3 (6)	C(1)–C(6)–C(5)	121·4 (6)
O(2)–C(10)–C(9)	108·8 (4)	C(1)–C(2)–C(3)	118·6 (7)
O(2)–C(10)–C(1)	114·2 (4)	C(2)–C(3)–C(4)	121·2 (8)
O(2)–C(10)–C(7)	114·5 (5)	C(6)–C(5)–C(4)	117·9 (7)
C(9)–C(10)–C(1)	115·9 (5)	Se–C(7)–C(10)	105·3 (4)
C(9)–C(10)–C(7)	86·6 (4)	Se–C(7)–C(8)	117·5 (4)
C(1)–C(10)–C(7)	114·0 (5)	C(10)–C(7)–C(8)	88·4 (5)
C(10)–C(1)–C(6)	114·5 (5)	C(3)–C(4)–C(5)	120·7 (8)

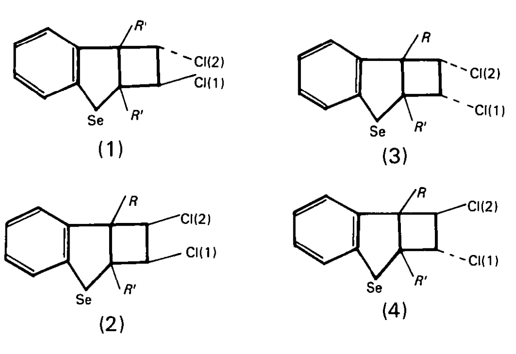

Fig. 1. The four possible diastereoisomers of the compound.

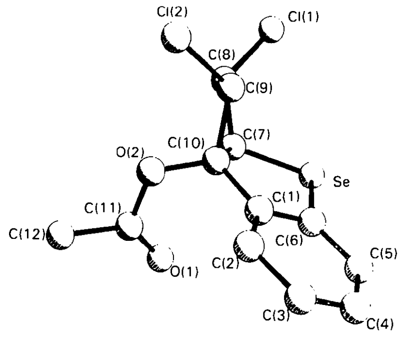

Fig. 2. Perspective view of the molecule.

65·5° with the benzo[*b*]selenole fragment (63·9° for the other isomer). The benzene ring of the benzo[*b*]selenole is perturbed very little, just as in the compound studied by Dideberg *et al.* (1977) ($\langle r \rangle = 1{\cdot}381 \pm 0{\cdot}014$ Å and $\langle \varphi \rangle = 119{\cdot}76 \pm 1{\cdot}4°$).

The angle C–Se–C (89·6°) is larger than that observed in related compounds: 86·7° in dibenzoselenole (Hope, Knobler & McCullough, 1970) and 87·7° in selenole (Brown, Burden & Godfrey, 1968). The Se–C distances are significantly longer in this compound (1·917, 1·965 Å, instead of 1·905, 1·945 Å and 1·899, 1·863 Å). The bond C(10)–C(7) (1·559 Å) has sp^3 character. All these features indicate that there is no participation of the lone pairs of the Se atom in conjugation in the ring.

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