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THE STRUCTURE OF A TRICLINIC FORM OF OCTAFLUOROSELENANTHRENE, $(C_6F_4)_2Se_2$

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Summary

Octafluoroselenanthrene exists in two different crystal forms, monoclinic and triclinic. It is shown here that the molecular structure in the triclinic modification is very similar to that found previously in monoclinic crystals.

Introduction

Rainville, Zingaro and Meyers have studied the structures of octafluorothianthrene [1], octafluoroselenanthrene [2] and octafluorotelluranthrene [3], the crystals being triclinic, monoclinic and monoclinic, respectively. It seemed to us that there should be a distinct possibility of $Se_2(C_6H_4)_2$ adopting a triclinic crystal form as well, since the molecular parameters are part way between $S_2(C_6F_4)_2$ and $Te_2(C_6F_4)_2$ particularly the angle of fold about the two hetero atoms (126, 123 and 118° for S, Se and Te). Unfortunately the authors do not state the solvent from which their crystal of $Se_2(C_6F_4)_2$ was obtained, but when crystallized from 40–60°C petroleum ether octafluoroselenanthrene does, indeed, give a triclinic crystal form. The molecule in this new modification has very similar parameters to those described in ref. 2.

Experimental

Octafluoroselenanthrene was prepared by heating together selenium powder and 1,2-diiodotetrafluorobenzene in a sealed, evacuated tube at 320°C for 3 days. It was isolated as pale yellow crystals by extraction of the reaction mixture with diethyl ether; m.p. 117–120°C. The crystal chosen for the X-ray study ($0.23 \times 0.08 \times 0.77$ mm) was grown from 40–60°C petroleum ether and mounted in a capillary tube. Data were collected on a Stöe-Stadi-2 diffractometer allowing the measurement of 1896 reflections of which 1534 had $I > 3\sigma(I)$. The intensities were corrected for Lp and absorption effects (t_{min} 0.13, t_{max} 0.51). The structure was solved by direct

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methods [EEES; 4] followed by Fourier maps and full-matrix, least-squares refinement. Geometry calculations used the XRAY72 system of programs [5] as implemented at the University of Manchester Regional Computer Centre.

Crystal data. $C_{12}F_8Se_2$, $M_r = 454.0$, triclinic $P\bar{1}$, a 8.641(2), b 11.139(3), c 6.566(2) Å, α 98.30 (05), β 95.12 (05), γ 88.93° (05), U 622.87 Å^3, $Z = 2$, D_x 2.422 Mg m^{-3}, Cu-K_α, λ 1.5418 Å, μ 93.05 cm^{-1}, $F(000) = 424$, T 293 K, $R = 0.097$ for 1534 observed reflections with $I > 3\sigma(I)$.

Discussion

The bond lengths and angles for octafluoroselenanthrene are shown in Table 1, the numbering scheme being as in Fig. 1. The atomic coordinates and equivalent isotropic parameters are listed in Table 2. Figure 2 shows that the molecule is relatively undistorted with the atoms in the individual C_6F_4 rings being virtually in the same plane. The unit cell contents are depicted in Fig. 3. The V-shaped molecule is hinged about the two selenium atoms with the angle between the two benzene

TABLE 1

BOND LENGTHS (Å) AND ANGLES (°)

C(1)–C(2)	1.39(2)	C(7)–Se(1)	1.90(1)
C(1)–C(6)	1.44(2)	C(7)–C(8)	1.36(2)
C(1)–Se(1)	1.87(1)	C(8)–C(9)	1.39(2)
C(2)–C(3)	1.35(2)	C(8)–F(8)	1.33(2)
C(2)–F(2)	1.34(1)	C(9)–C(10)	1.38(2)
C(3)–C(4)	1.44(2)	C(9)–F(9)	1.33(2)
C(3)–F(3)	1.31(2)	C(10)–C(11)	1.32(2)
C(4)–C(5)	1.41(2)	C(10)–F(10)	1.32(1)
C(4)–F(4)	1.32(1)	C(11)–C(12)	1.44(2)
C(5)–C(6)	1.35(2)	C(11)–F(11)	1.37(1)
C(5)–F(5)	1.34(1)	C(12)–Se(2)	1.87(1)
C(6)–Se(2)	1.89(1)	C(12)–C(7)	1.42(2)
C(1)–Se(1)–C(7)	97.0(4)	C(6)–Se(2)–C(12)	96.2(5)
Se(1)–C(1)–C(2)	121.2(8)	Se(1)–C(7)–C(8)	119.7(8)
Se(1)–C(1)–C(6)	120.9(8)	Se(1)–C(7)–C(12)	119.7(8)
C(2)–C(1)–C(6)	117.8(10)	C(8)–C(7)–C(12)	120.6(10)
C(1)–C(2)–C(3)	124.0(12)	C(7)–C(8)–C(9)	121.2(13)
C(1)–C(2)–F(2)	118.4(11)	C(7)–C(8)–F(8)	120.8(11)
C(3)–C(2)–F(2)	117.6(12)	C(9)–C(8)–F(8)	117.8(13)
C(2)–C(3)–C(4)	118.3(12)	C(8)–C(9)–C(10)	120.1(13)
C(2)–C(3)–F(3)	125.0(12)	C(8)–C(9)–F(9)	120.6(14)
C(4)–C(3)–F(3)	116.6(10)	C(10)–C(9)–F(9)	119.3(12)
C(3)–C(4)–C(5)	118.0(10)	C(9)–C(10)–C(11)	118.7(11)
C(3)–C(4)–F(4)	118.1(11)	C(9)–C(10)–F(10)	119.5(13)
C(5)–C(4)–F(4)	123.9(11)	C(11)–C(10)–F(10)	121.8(14)
C(4)–C(5)–C(6)	122.7(11)	C(10)–C(11)–C(12)	124.5(12)
C(4)–C(5)–F(5)	115.0(11)	C(10)–C(11)–F(11)	118.7(11)
C(6)–C(5)–F(5)	121.95(12)	C(12)–C(11)–F(11)	116.75(11)
C(5)–C(6)–C(1)	119.0(10)	C(11)–C(12)–C(7)	114.8(10)
C(5)–C(6)–Se(2)	119.5(9)	C(11)–C(12)–Se(2)	121.3(9)
C(1)–C(6)–Se(2)	121.6(8)	C(7)–C(12)–Se(2)	123.6(8)

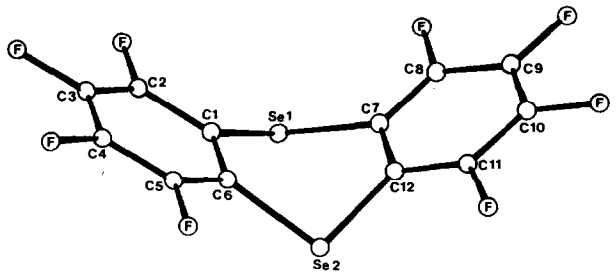

Fig. 1. ORTEP drawing of the octafluoroselenanthrene molecule and atom numbering.

TABLE 2

ATOMIC COORDINATES ($\times 10^4$) AND EQUIVALENT ISOTROPIC THERMAL PARAMETERS $B_{eq} = \frac{1}{3}\Sigma_i B_{ii}$

Atom	x	y	z	B_{eq} (Å^2)
Se(1)	424(1)	3872(1)	7163(2)	3.5
Se(2)	−357(2)	2604(2)	2150(2)	3.9
C(1)	−1627(14)	3599(8)	6028(17)	3.3
C(2)	−2877(15)	3944(9)	7183(20)	3.4
C(3)	−4391(14)	3830(10)	6423(22)	4.2
C(4)	−4719(13)	3333(10)	4284(21)	4.2
C(5)	−3457(16)	2929(10)	3125(20)	3.8
C(6)	−1962(12)	3078(8)	3894(19)	3.4
C(7)	1231(12)	2319(9)	6156(18)	3.5
C(8)	2195(16)	1724(11)	7430(21)	4.2
C(9)	2912(16)	637(12)	6701(28)	5.9
C(10)	2600(17)	115(9)	4665(26)	5.1
C(11)	1642(18)	685(10)	3430(20)	4.3
C(12)	869(13)	1813(9)	4063(19)	3.9
F(2)	−2581(10)	4443(6)	9156(12)	4.7
F(3)	−5577(9)	4155(7)	7480(14)	5.8
F(4)	−6186(9)	3237(8)	3545(15)	5.7
F(5)	−3848(9)	2489(6)	1141(12)	4.6
F(8)	2563(10)	2206(8)	9385(12)	5.6
F(9)	3910(11)	96(6)	7937(16)	7.9
F(10)	3303(12)	−909(6)	3986(16)	7.1
F(11)	1314(11)	160(6)	1422(13)	6.1

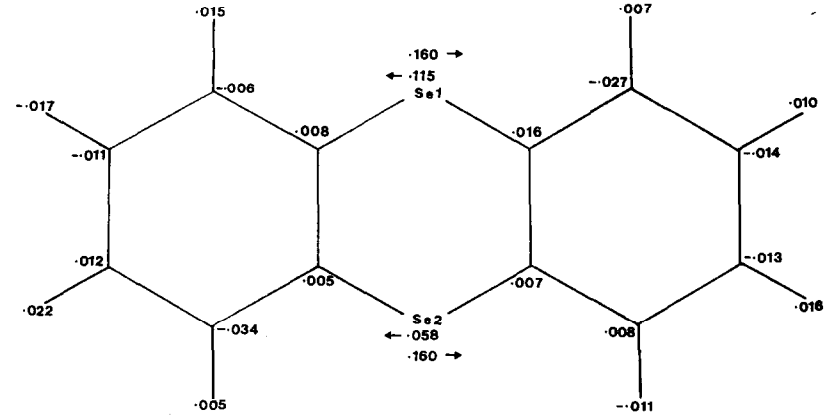

Fig. 2. Deviations from planarity (in Å) for the members of each C_6F_4 ring.

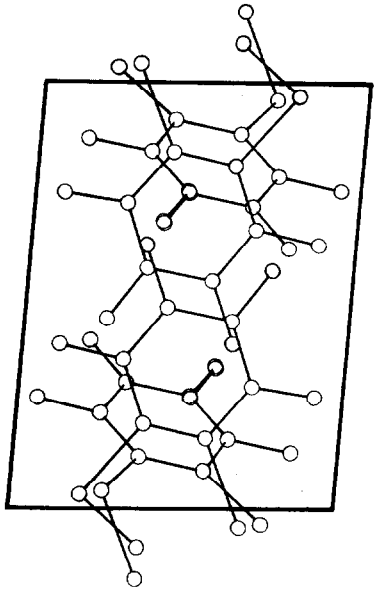

Fig. 3. Contents of the unit cell projected down the c axis.

rings being 130.2°; the angle between the [Se(1), C(1), C(6) Se(2)] and [Se(1), C(7), C(12), Se(2)] planes is 122.2°. The two selenium atoms lie 0.79 Å (Se(1)) and 0.76 Å (Se(2)) above the plane through C(1), C(6), C(7) and C(12). There were no abnormal intermolecular contacts. All the molecular parameters of triclinic octafluoroselenanthrene are very similar to those found in the monoclinic form. The fact that the densities of the two crystalline phases are essentially identical shows there is little to choose between the two methods of packing the molecules in the two unit cells.

Lists of structure factors and anisotropic thermal parameters may be obtained from D.S.B.

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