

only intramolecular interaction is that between $N(16)^+$ and $OCIO_3^-$ [$N(16)^+ \cdots O(1Cl) = 3.026$ (4) Å].

In the crystal structure of (+)-lupanine perchlorate, which is much the same as sparteine perchlorate, no intermolecular contacts (apart from normal van der Waals contacts) are observed (Fig. 2).

We thank Professor M. Wiewiórowski for suggesting this structure determination. The study was supported by the Polish Academy of Sciences (MRI-9).

References

BOROWIAK, T., BOKIY, N. G. & STRUCHKOV, YU. T. (1980). In preparation.

DOUCERAIN, H., CHIARONI, A. & RICHE, C. (1976). *Acta Cryst.* **B32**, 3213–3215.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

MALUSZYŃSKA, H., HOSER, A. & KALUSKI, Z. (1979). *Acta Cryst.* **B35**, 970–973.

PERKOWSKA, A., PIECZONKA, G. & WIEWIÓROWSKI, M. (1980). *Bull. Acad. Pol. Sci. Sér. Sci. Chim.* **9**. In the press.

SKRZYPCZAK-JANKUN, E. & KALUSKI, Z. (1978). *Acta Cryst.* **B34**, 2651–2653.

Syntax (1976). The Syntax *XTL/E-XTL* structure determination system. Syntax Analytical Instruments, Inc., 10040 Bubb Road, Cupertino, California 95014, USA.

WIEWIÓROWSKI, M., EDWARDS, O. E. & BRATEK-WIEWIÓROWSKA, M. D. (1967). *Can. J. Chem.* **45**, 1447–1457.

Acta Cryst. (1980). **B36**, 1520–1522

4,5,9,10,11,12-Hexafluoropentacyclo[6.4.0.0^{3,6}.0^{4,12}.0^{5,9}]dodec-10-ene*

BY LJUBO GOLJIĆ AND IVAN LEBAN

Chemistry Department, University Edvard Kardelj, 61000 Ljubljana, Yugoslavia

(Received 9 January 1980; accepted 18 February 1980)

Abstract. $C_{12}H_8F_6$, $M_r = 266.2$, triclinic, $P\bar{1}$, $a = 6.625$ (3), $b = 7.176$ (4), $c = 11.427$ (6) Å, $\alpha = 79.43$ (4), $\beta = 78.67$ (4), $\gamma = 67.56$ (4)°, $V = 488.8$ Å³, $D_m = 1.85$ (5) (floatation), $D_x = 1.808$ Mg m⁻³, $\mu(\text{Mo } K\alpha) = 0.202$ mm⁻¹, $Z = 2$, at 293 (1) K; final $R = 0.048$ for 1700 observed reflexions [$I > 2.5\sigma(I)$]. There is considerable strain in the molecule resulting in two rather long C–C distances of 1.567 (4) and 1.581 (3) Å.

Introduction. The title compound was prepared by cycloaddition between hexafluorobenzene and 1,4-cyclohexadiene by Drs M. Zupan and B. Šket, Chemistry Department, University E. Kardelj, Ljubljana, Yugoslavia. An inspection of Weissenberg photographs and the mean value of $|E^2 - 1| = 0.977$ indicated the space group $P\bar{1}$. The cell parameters and their e.s.d.'s were obtained by least squares from 2θ values of 45 centered reflexions (Mo $K\alpha_1$, $\lambda = 0.70926$ Å) at 293 (1) K. A prismatic single crystal $0.4 \times 0.4 \times 0.5$ mm, sealed in a Lindemann-glass capillary, was used for data collection on an automated Enraf-Nonius CAD-4 diffractometer with graphite-mono-

chromatized Mo $K\alpha$ radiation. A summary of the data collection and reduction is given in Table 1. The data were corrected for variation in reference reflexions and Lorentz-polarization effects, but not for absorption. Multisolution \sum_2 sign expansion (Sheldrick, 1976) located all non-hydrogen atoms. Isotropic full-matrix

Table 1. *Data-collection summary*

Temperature (K)	293 (1)
Diffractometer	CAD-4, automated, four-circle
Radiation	Mo $K\alpha$ (graphite monochromator) ($\lambda = 0.71069$ Å)
Scan method	$\omega-2\theta$
2θ scan width (°)	$0.8 + 0.2 \tan \theta$
Scan rate (deg min ⁻¹)	Minimum 1.8; maximum 20.1
Background	$\frac{1}{4}$ of the scan time at each of the scan limits
$2\theta_{\max}$ (°)	54
Maximum scan time (s)	40
Aperture (mm)	$2.5 + 0.9 \tan \theta$
Reference reflexions	115, 214, 332
Intensity decrease (%)	12
Measured reflexions	3834 ($\pm h, \pm k, \pm l$)
Averaged reflexions	2035
Mean discrepancy on I (%)	2.0
Observed reflexions	1700 [$I > 2.5\sigma(I)$]
Unobserved reflexions	335
$\sigma(I)$ base	Counting statistics
μ (mm ⁻¹)	0.202 for Mo $K\alpha$

* It should be noted that the numbering scheme used for naming the title compound differs from the crystallographic numbering.

Table 2. Final fractional coordinates ($\times 10^4$; for H $\times 10^3$) with U_{eq} ($\times 10^4$)

	x	y	z	U_{eq} (\AA^2)
F(1)	4169 (3)	6070 (2)	7017 (1)	449 (9)
F(5)	1847 (3)	5304 (2)	9180 (1)	506 (10)
F(6)	5546 (3)	2691 (3)	9931 (2)	643 (12)
F(7)	8047 (3)	-638 (3)	8558 (2)	538 (11)
F(8)	6425 (3)	-858 (2)	6678 (1)	434 (9)
F(12)	6520 (2)	2897 (2)	5712 (1)	402 (9)
C(1)	3308 (4)	4554 (3)	7168 (2)	303 (12)
C(2)	1229 (4)	4911 (4)	6606 (2)	339 (13)
C(3)	-338 (4)	4338 (4)	7656 (3)	390 (15)
C(4)	1333 (4)	2627 (4)	8382 (2)	347 (13)
C(5)	2891 (4)	3719 (4)	8497 (2)	317 (12)
C(6)	4984 (5)	2328 (4)	8961 (2)	383 (13)
C(7)	6127 (4)	809 (4)	8334 (2)	369 (13)
C(8)	5095 (4)	766 (3)	7291 (2)	296 (12)
C(9)	2712 (4)	785 (4)	7632 (2)	336 (12)
C(10)	1999 (5)	1204 (4)	6375 (2)	404 (14)
C(11)	2596 (4)	3094 (4)	5858 (2)	339 (13)
C(12)	4645 (4)	2767 (4)	6435 (2)	288 (12)
H(2)	26	633	614	
H(3A)	-144	380	735	
H(3B)	-129	560	816	
H(4)	58	202	921	
H(9)	252	-56	816	
H(10A)	25	153	643	
H(10B)	291	-5	585	
H(11)	255	331	490	

coordinates with $U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$ are given in Table 2.* All calculations were carried out on the CDC Cyber 172 computer at RRC Ljubljana with *SHELX* 76 (Sheldrick, 1976).

Discussion. The proposed structural formula has been confirmed in this study. Fig. 1 illustrates the configuration of the molecule. The molecule possesses a mirror plane, which is not a crystallographic mirror plane. The average and the largest deviations from the mirror plane taken through the mid-points of the distances C(1)–C(12), C(2)–C(11), C(3)–C(10), C(4)–C(9), C(5)–C(8), C(6)–C(7), F(1)–F(12), F(5)–F(8), F(6)–F(7) are 0.001 and 0.003 Å. The conformation of the molecule is best described with a series of planes through the atom groups given in Table 3. Important

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35134 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

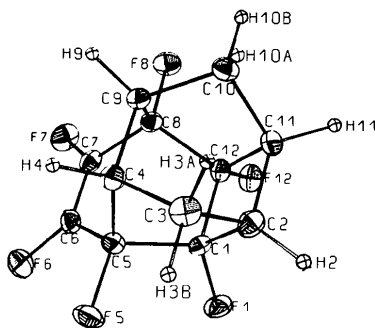


Fig. 1. A view of the molecule. The atoms are represented by thermal ellipsoids drawn at the 50% probability level (Johnson, 1965).

least-squares refinement proceeded to $R = 0.128$, and anisotropic refinement to $R = 0.060$. A subsequent difference synthesis showed all H atoms, which were inserted in calculated positions and refined subject to the constraint that the C–H vectors were constant in magnitude (1.08 Å) and direction but not position (riding model). A common isotropic temperature factor B for H atoms refined to 3.8 (2) Å². The final $R = 0.048$, with $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.058$; the weighting scheme was $w = 4.3 / [\sigma^2(F_o) + 0.0004 F_o^2]$. Analysis of the resulting difference synthesis revealed no peak $> 0.3 \text{ e \AA}^{-3}$. In the final cycle, the average and the maximum shift/error for the atomic parameters were 0.08 and 0.29 for U_{11} of C(1). Final atomic

Table 3. Least-squares planes and interplanar angles

Plane	Plane-defining atoms	Largest deviation (e.s.d. < 0.004 Å)
(I)	F(6), C(6), C(7), F(7)	0.0006 Å
(II)	C(5), C(6), C(7), C(8)	0.0029
(III)	C(5), C(4), C(9), C(8)	0.0011
(IV)	C(5), C(1), C(12), C(8)	0.0005
(V)	F(1), C(1), C(12), F(12)	0.0022
(VI)	C(1), C(2), C(11), C(12)	0.0008
(VII)	C(2), C(3), C(11), C(10)	0.0008
(VIII)	C(3), C(4), C(9), C(10)	0.0009
(IX)	C(4), C(5), F(5)	0
(X)	C(8), C(9), F(8)	0

Interplanar angles (e.s.d.'s 0.4–0.9°)

(I), (II)	0.3°	(III), (VIII)	109.1°
(II), (III)	127.8	(IV), (V)	128.0
(II), (IV)	125.6	(IV), (VI)	108.2
(III), (IX)	2.3	(V), (VI)	123.8
(III), (X)	2.7	(VI), (VII)	105.0
(III), (IV)	106.6	(VII), (VIII)	111.0

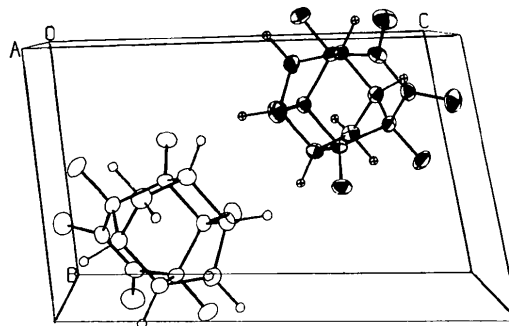


Fig. 2. A view of the unit cell along [100].

Table 4. Bond distances (Å) and angles (°)

C(1)—F(1)	1.380 (3)	C(12)—F(12)	1.374 (3)
C(5)—F(5)	1.377 (3)	C(8)—F(8)	1.378 (3)
C(6)—F(6)	1.334 (3)	C(7)—F(7)	1.335 (3)
C(1)—C(2)	1.547 (4)	C(12)—C(11)	1.544 (3)
C(2)—C(3)	1.531 (4)	C(11)—C(10)	1.539 (4)
C(3)—C(4)	1.539 (4)	C(10)—C(9)	1.543 (3)
C(4)—C(5)	1.551 (4)	C(9)—C(8)	1.545 (4)
C(1)—C(5)	1.539 (3)	C(12)—C(8)	1.543 (3)
C(5)—C(6)	1.496 (4)	C(8)—C(7)	1.497 (4)
C(1)—C(12)	1.538 (3)	C(4)—C(9)	1.581 (3)
C(2)—C(11)	1.567 (4)	C(6)—C(7)	1.310 (4)
F(1)—C(1)—C(2)	120.2 (2)	F(12)—C(12)—C(11)	119.5 (2)
F(1)—C(1)—C(5)	112.6 (2)	F(12)—C(12)—C(8)	112.7 (2)
F(1)—C(1)—C(12)	115.2 (2)	F(12)—C(12)—C(1)	116.0 (2)
C(2)—C(1)—C(5)	107.0 (2)	C(11)—C(12)—C(8)	107.0 (2)
C(2)—C(1)—C(12)	90.6 (2)	C(11)—C(12)—C(1)	90.5 (2)
C(5)—C(1)—C(12)	109.0 (2)	C(8)—C(12)—C(1)	108.8 (2)
C(1)—C(2)—C(3)	104.2 (2)	C(12)—C(11)—C(10)	104.2 (2)
C(1)—C(2)—C(11)	89.3 (2)	C(12)—C(11)—C(2)	89.6 (2)
C(3)—C(2)—C(11)	111.8 (2)	C(10)—C(11)—C(2)	111.3 (2)
C(2)—C(3)—C(4)	100.3 (2)	C(11)—C(10)—C(9)	100.3 (2)
C(3)—C(4)—C(5)	100.2 (2)	C(10)—C(9)—C(8)	100.4 (2)
C(3)—C(4)—C(9)	111.5 (2)	C(10)—C(9)—C(4)	110.9 (2)
C(5)—C(4)—C(9)	108.0 (2)	C(8)—C(9)—C(4)	108.0 (2)
F(5)—C(5)—C(1)	109.7 (2)	F(8)—C(8)—C(12)	110.3 (2)
F(5)—C(5)—C(4)	112.0 (2)	F(8)—C(8)—C(9)	111.3 (2)
F(5)—C(5)—C(6)	110.1 (2)	F(8)—C(8)—C(7)	110.1 (2)
C(1)—C(5)—C(4)	98.9 (2)	C(12)—C(8)—C(9)	99.1 (2)
C(1)—C(5)—C(6)	111.9 (2)	C(12)—C(8)—C(7)	111.5 (2)
C(4)—C(5)—C(6)	113.8 (2)	C(9)—C(8)—C(7)	114.0 (2)
F(6)—C(6)—C(5)	120.2 (2)	F(7)—C(7)—C(8)	119.8 (2)
F(6)—C(6)—C(7)	125.7 (3)	F(7)—C(7)—C(6)	125.9 (3)
C(5)—C(6)—C(7)	114.1 (2)	C(8)—C(7)—C(6)	114.3 (2)

bond lengths and angles are presented in Table 4. The C(6)—C(7) distance of 1.310 (4) Å has double-bond character, whereas the C—F distances [from 1.334 (3) to 1.380 (3) Å] are within the normal range and are close to those found in similar fluoro-organic compounds (Golič & Leban, 1978*a,b*). There is considerable strain in this cage-like molecule and two of the C—C bonds, C(2)—C(11) [1.567 (4) Å] and C(4)—C(9) [1.581 (3) Å], are somewhat longer than a single C—C bond.

A view of the unit cell along [100] is given in Fig. 2. No significant intermolecular contacts were observed.

We thank Drs M. Zupan and B. Šket for providing the sample and the Research Community of Slovenia for financial support.

References

- GOLIČ, L. & LEBAN, I. (1978*a*). *Cryst. Struct. Commun.* **7**, 47–52.
 GOLIČ, L. & LEBAN, I. (1978*b*). *Cryst. Struct. Commun.* **7**, 53–57.
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
 SHELDRIK, G. M. (1976). *SHELX 76*. Program for crystal structure determination. Univ. of Cambridge, England.