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Acta Cryst. (1996). C**52**, 2844–2847

Three 1,4-*trans*-Substituted Cyclohexanes

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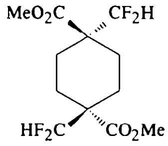
(*Received 1 May 1995; accepted 16 July 1996*)

Abstract

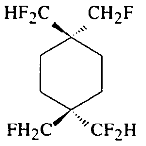
The crystal structure determinations of *trans*-dimethyl 1,4-bis(difluoromethyl)-1,4-cyclohexanedicarboxylate, $C_{12}H_{16}F_4O_4$, *trans*-1,4-bis(difluoromethyl)-1,4-bis(fluoromethyl)cyclohexane, $C_{10}H_{14}F_6$, and *trans*-1,4-bis(fluoromethyl)-1,4-bis(trifluoromethyl)cyclohexane, $C_{10}H_{12}F_8$, confirmed the *trans* assignment for each of the 1,4-disubstituted cyclohexanes.

Comment

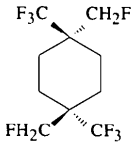
Stereoselective reduction of tetramethyl 1,1,4,4-cyclohexanetetracarboxylate with diisobutyl aluminium hydride yields *trans*-1,4-dialdehyde as the major product (90:10, 63% yield). Fluorination and additional elaboration provides a convenient synthesis of 1,1,4,4-tetrasubstituted cyclohexanes containing trifluoromethyl, difluoromethyl and fluoromethyl groups (Davis, Swenson & Burton, 1993). These compounds should have further application as intermediates of other selectively fluorinated compounds. The crystal structures of three of these compounds [*trans*-dimethyl 1,4-bis(difluoromethyl)-1,4-cyclohexanedicarboxylate, (I), *trans*-1,4-bis(difluoromethyl)-1,4-bis(fluoromethyl)cyclohexane, (II), and *trans*-1,4-bis(trifluoromethyl)-1,4-bis(fluoromethyl)cyclohexane, (III)] were determined in order to confirm the conformations.



(I)

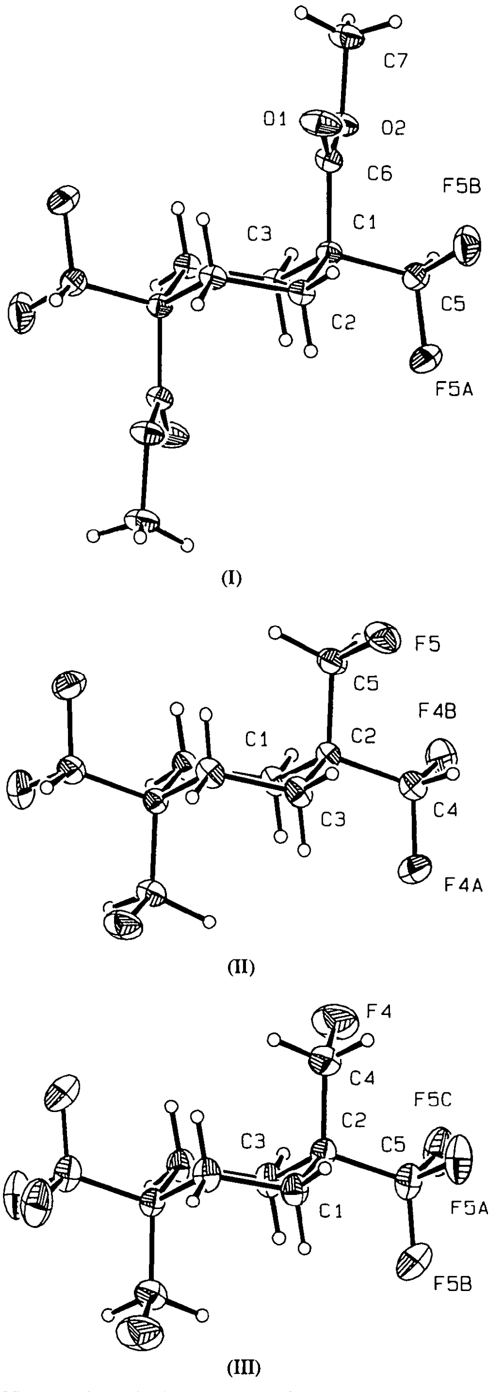


(II)



(III)

The cyclohexane rings of (I) and (III) are centered on an inversion center. A twofold axis passes through the centers of the C1—C1^i and C3—C3^i bonds in (II) [(i) = 1 − *x*, *y*, 1/2 − *z*)]. The cyclohexane ring of each compound adopts a chair conformation. The 1,4-substituents are arranged in a *trans* conformation with the more highly fluorinated substituent occupying the equatorial position in each case. Fig. 2 shows the packing in the crystals of the title compounds.



(III)

Fig. 1. Views and numbering schemes of compounds (I), (II) and (III). Thermal displacement ellipsoids are drawn at the 35% probability level. H atoms are drawn with equal arbitrary radius.

Acta Crystallographica Section C
ISSN 0108-2701 © 1996

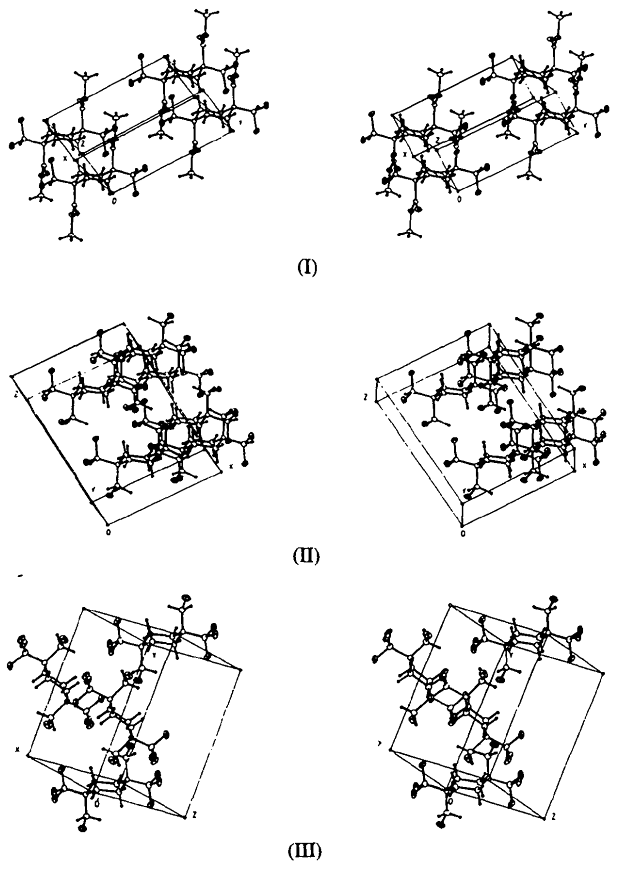


Fig. 2. Stereoviews of the unit cells for compounds (I), (II) and (III).

Experimental

Details of the preparation and crystallization are reported elsewhere (Davis, Swenson & Burton, 1993).

Compound (I)

Crystal data

$C_{12}H_{16}F_4O_4$
$M_r = 300.25$
Triclinic
$P\bar{1}$
$a = 6.222$ (2) Å
$b = 9.778$ (2) Å
$c = 5.966$ (2) Å
$\alpha = 102.83$ (2)°
$\beta = 108.44$ (2)°
$\gamma = 96.57$ (2)°
$V = 329.0$ (4) Å^3
$Z = 1$
$D_x = 1.52$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11$–$19°$
$\mu = 0.140$ mm^{-1}
$T = 291$ K
Prismatic
0.25 × 0.22 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical, ψ scans (*MolEN*; Fair, 1990)
$T_{\min} = 0.960$, $T_{\max} = 0.998$
2898 measured reflections
1893 independent reflections

1652 observed reflections [$I > 2.0\sigma(I)$]
$R_{\rm int} = 0.017$
$\theta_{\max} = 30°$
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -8 \rightarrow 4$
4 standard reflections
frequency: 240 min
intensity decay: 0.01%

Refinement

Refinement on F
$R = 0.039$
$wR = 0.046$
$S = 1.052$
1652 reflections
123 parameters
All H-atom parameters refined
$w = 1/[\sigma(F^2) + (0.015F)^2 + 0.5]$ (Killean & Lawrence, 1969)

$(\Delta/\sigma)_{\max} = 0.071$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
F5*A*	0.0636 (1)	0.1402 (1)	0.7146 (2)	0.0553 (3)
F5*B*	0.3266 (2)	0.33540 (9)	0.8391 (2)	0.0631 (3)
O1	0.8147 (2)	0.2924 (1)	1.1828 (2)	0.0531 (3)
O2	0.5815 (1)	0.3077 (1)	1.4027 (2)	0.0402 (2)
C1	0.4291 (2)	0.1424 (1)	1.0076 (2)	0.0281 (2)
C2	0.5034 (2)	0.0645 (1)	0.7994 (2)	0.0303 (2)
C3	0.3294 (2)	0.0321 (1)	1.1168 (2)	0.0296 (2)
C5	0.2414 (2)	0.2258 (1)	0.9148 (2)	0.0385 (3)
C6	0.6335 (2)	0.2545 (1)	1.2041 (2)	0.0315 (3)
C7	0.7561 (3)	0.4201 (2)	1.5979 (3)	0.0481 (4)

Table 2. *Selected geometric parameters (Å, °) for (I)*

F5*A*—C5	1.363 (2)	C1—C2	1.537 (2)
F5*B*—C5	1.368 (2)	C1—C3	1.545 (2)
O1—C6	1.200 (1)	C1—C5	1.531 (2)
O2—C6	1.337 (1)	C1—C6	1.537 (2)
O2—C7	1.450 (2)	C2—C3$^{\rm i}$	1.527 (2)
C6—O2—C7	116.5 (1)	C1—C3—C2	112.46 (9)
C2 C1 C3	109.64 (9)	F5*A*—C5—F5*B*	106.0 (1)
C2—C1—C5	111.6 (1)	F5*A*—C5—C1	110.8 (1)
C2—C1—C6	110.35 (9)	F5*B*—C5—C1	110.6 (1)
C3—C1—C5	107.73 (9)	O1—C6—O2	123.9 (1)
C3—C1—C6	111.81 (9)	O1—C6—C1	125.2 (1)
C5—C1—C6	105.7 (1)	O2—C6—C1	110.87 (9)
C1—C2—C3$^{\rm i}$	112.03 (9)		

Symmetry code: (i) $1 - x, -y, 2 - z$.

Compound (II)

Crystal data

$C_{10}H_{14}F_6$
$M_r = 248.21$
Monoclinic
$C2/c$
$a = 8.807$ (3) Å
$b = 11.447$ (3) Å
$c = 10.644$ (3) Å
$\beta = 98.54$ (3)°
$V = 1061.1$ (9) Å^3
$Z = 4$
$D_x = 1.55$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–$16°$
$\mu = 0.157$ mm^{-1}
$T = 291$ K
Tabular
0.44 × 0.33 × 0.29 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical, ψ scans (*MolEN*; Fair, 1990)
$T_{\min} = 0.957$, $T_{\max} = 0.998$
5474 measured reflections
2323 independent reflections
1070 observed reflections [$I > 2.0\sigma(I)$]
$R_{\text{int}} = 0.031$
$\theta_{\max} = 35°$
$h = -14 \rightarrow 6$
$k = -16 \rightarrow 18$
$l = -17 \rightarrow 17$
4 standard reflections
frequency: 240 min
intensity decay: 0.28% [linear correction applied (*MolEN*)]

Refinement

Refinement on F
$R = 0.038$
$wR = 0.049$
$S = 1.088$
1070 reflections
101 parameters
All H-atom parameters refined
$w = 1/[\sigma(F^2) + (0.02F)^2 + 0.1]$ (Killean & Lawrence, 1969)
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.03$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
F4*A*	0.6985 (1)	0.3745 (1)	0.00942 (9)	0.0556 (3)
F4*B*	0.8645 (1)	0.4724 (1)	0.1401 (1)	0.0615 (3)
F5	0.8559 (1)	0.2745 (1)	0.3753 (1)	0.0604 (3)
C1	0.5669 (2)	0.4845 (1)	0.2110 (1)	0.0356 (3)
C2	0.6659 (2)	0.3736 (1)	0.2281 (1)	0.0319 (3)
C3	0.5644 (2)	0.2636 (1)	0.2085 (2)	0.0386 (4)
C4	0.7768 (2)	0.3728 (2)	0.1312 (2)	0.0423 (4)
C5	0.7627 (2)	0.3749 (2)	0.3595 (2)	0.0415 (4)

Table 4. *Selected geometric parameters (Å, °) for (II)*

F4*A*—C4	1.375 (2)	C2—C3	1.541 (2)
F4*B*—C4	1.372 (2)	C2—C4	1.523 (2)
F5—C5	1.407 (2)	C2—C5	1.526 (2)
C1—C1^{i}	1.538 (3)	C3—C3^{i}	1.538 (4)
C1—C2	1.536 (2)		
C1^{i}—C1—C2	113.7 (1)	C4—C2—C5	107.1 (1)
C1—C2—C3	110.6 (1)	C2—C3—C3^{i}	112.6 (1)
C1—C2—C4	109.6 (1)	F4*A*—C4—F4*B*	104.5 (1)
C1—C2—C5	109.3 (1)	F4*A*—C4—C2	110.8 (1)
C3—C2—C4	108.7 (1)	F4*B*—C4—C2	111.0 (1)
C3—C2—C5	111.5 (1)	F5—C5—C2	109.8 (1)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Compound (III)

Crystal data

$C_{10}H_{12}F_8$
$M_r = 284.19$
Monoclinic
$P2_1/n$
$a = 6.808$ (1) Å
$b = 10.747$ (2) Å
$c = 7.929$ (1) Å
$\beta = 102.40$ (1)°
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11–15°$
$\mu = 0.181$ mm^{-1}
$T = 291$ K
Tabular
$V = 566.6$ (3) Å^3
$Z = 2$
$D_x = 1.67$ Mg m^{-3}
0.29 × 0.23 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5691 measured reflections
1634 independent reflections
1074 observed reflections [$I > 2.0\sigma(I)$]
$R_{\text{int}} = 0.023$
$\theta_{\max} = 30°$
$h = -9 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -11 \rightarrow 11$
4 standard reflections
frequency: 240 min
intensity decay: 1.60%

Refinement

Refinement on F
$R = 0.034$
$wR = 0.044$
$S = 1.008$
1074 reflections
106 parameters
All H-atom parameters refined
$w = 1/[\sigma(F^2) + (0.02F)^2 + 0.1]$ (Killean & Lawrence, 1969)
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.05$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (III)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
F4	0.5963 (2)	0.2848 (1)	0.6547 (2)	0.0858 (4)
F5*A*	0.1316 (1)	0.1619 (1)	0.7138 (1)	0.0753 (3)
F5*B*	0.2686 (2)	−0.0056 (1)	0.8266 (1)	0.0816 (3)
F5*C*	0.4153 (2)	0.1686 (1)	0.8945 (1)	0.0769 (3)
C1	0.2802 (2)	0.0143 (1)	0.4687 (2)	0.0420 (3)
C2	0.4168 (2)	0.0897 (1)	0.6134 (2)	0.0370 (3)
C3	0.6172 (2)	0.0213 (1)	0.6775 (2)	0.0428 (3)
C4	0.4466 (2)	0.2195 (1)	0.5436 (2)	0.0529 (4)
C5	0.3094 (2)	0.1035 (2)	0.7620 (2)	0.0520 (4)

Table 6. *Selected geometric parameters (Å, °) for (III)*

F4—C4	1.386 (2)	C1—C3^{i}	1.525 (2)
F5*A*—C5	1.344 (2)	C2—C3	1.537 (2)
F5*B*—C5	1.333 (2)	C2—C4	1.530 (2)
F5*C*—C5	1.337 (2)	C2—C5	1.522 (2)
C1—C2	1.543 (2)		
C2—C1—C3^{i}	113.3 (1)	F4—C4—C2	111.6 (1)
C1—C2—C3	110.2 (1)	F5*A*—C5—F5*B*	105.7 (1)
C1—C2—C4	108.4 (1)	F5*A*—C5—C2	112.2 (1)
C1—C2—C5	108.3 (1)	F5*B*—C5—C2	112.7 (1)
C3—C2—C4	112.2 (1)	F5*C*—C5—F5*A*	106.3 (1)
C3—C2—C5	109.3 (1)	F5*C*—C5—F5*B*	106.3 (1)
C4—C2—C5	108.3 (1)	F5*C*—C5—C2	113.1 (1)
C1—C3—C2	112.8 (1)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974). The H-atom coordinates and displacement parameters were refined for each compound. The C—H bond ranges and U values were 0.95 (2)–0.98 (2) Å and 0.035 (4)–0.077 (4) Å^2, respectively, for (I), 0.93 (2)–1.08 (2) Å and 0.031 (4)–0.050 (5) Å^2, respectively, for (II), and 0.94 (2)–0.99 (2) Å and 0.050–0.066 (5) Å^2, respectively, for (III).

For all compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *MULTAN* (Main *et al.*, 1980); program(s) used to refine structures: *MolEN*; molecular graphics: *ORTEP* (Johnson, 1976); software used to prepare material for publication: *MolEN*.

DJB thanks the National Science Foundation for support of this work

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2847–2849

α,α'-Bis(2-pyridylseleno)-*p*-xylene

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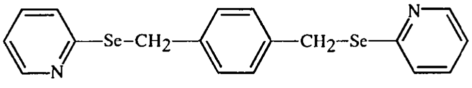
(*Received 15 May 1996; accepted 6 June 1996*)

Abstract

The title compound, $C_{18}H_{16}N_2Se_2$, crystallizes with two half molecules per asymmetric unit. Each independent molecule displays crystallographic inversion symmetry. The pyridyl rings are oriented such that the N atoms are *endo* to the methylene groups. The —Se—CH_2— bridges display extended conformations.

Comment

The title compound, (I), crystallizes with two half molecules per asymmetric unit. The second halves are generated by inversion symmetry at the centres of the rings C31–C36 at (1/2,0,0) and C41–C46 at (0,0,0) (Fig. 1). Where appropriate, values for the second molecule are given in square brackets in the discussion below.



(I)

The relative orientation of the Se- and N-atom pairs across the central phenyl ring is *trans* as a result of the inversion symmetry. The orientation of the N atoms can be regarded as *endo* (or *cis*) relative to the neighbouring methylene groups, as indicated by the torsion angle C1—Se1—C11—N1 = 9.2 (3)° [C2—Se2—C21—N2 = −4.2 (3)°]. The bridges between rings display extended conformations, C31—C1—Se1—C11 = 164.7 (3)° [C41—C2—Se2—C21 = −164.6 (3)°].

The interplanar angle between the phenyl (C31–C36) and the adjacent pyridine (C11–N1) rings is 66.7 (2)° [63.3 (3)° (phenyl C41–C46 and pyridine C21–N2)].

The C—C—Se angles at the methylene C atoms are tetrahedral at 109.2 (2)°. The angles at the Se atoms, 98.6 (3)° [99.3 (2)°], are in agreement with reported values for comparable compounds (with a methylene group and an aromatic ring attached to the Se atom) ranging from 100.5 (3) to 101.6 (2)° (Brussani, Ley, Wright & Williams, 1986; Dupont, Dideberg & Jacquemin, 1990; Iwaoka & Tomoda, 1994). The same is true for the Se—CH_2 and the Se—C(aryl) bond lengths with values of 1.957 (3) Å [1.959 (4) Å] and 1.909 (4) Å [1.909 (4) Å], respectively, compared with the corresponding values for the above-cited compounds ranging from 1.940 (3) to 1.966 (8) Å (Se—CH_2) and from 1.913 (4) to 1.924 (8) Å (Brussani *et al.*, 1986; Dupont *et al.*, 1990; Iwaoka & Tomoda, 1994).

The molecules pack in 'herringbone' layers in the planes 202 (Fig. 2). The interplanar angles between the two independent phenyl and pyridine rings are 23.1 (6) and 4.0 (2)°, respectively.

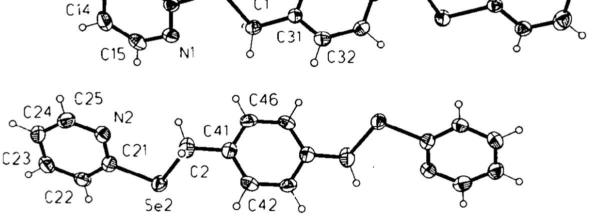


Fig. 1. The two independent molecules of the title compound in the crystal. Ellipsoids correspond to the 50% probability level. H-atom radii are arbitrary. Only the asymmetric unit is numbered.