Purdue University) for his assistance in preparing this paper.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1107). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. J. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.
Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Etter, M. C. & Baures, P. W. (1988). *J. Am. Chem. Soc.* **110**, 639–640.
Etter, M. C. & Huang, K. S. (1992). *Chem. Mater.* **4**, 824–827.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Etter, M. C. & Reutzel, S. M. (1991). *J. Am. Chem. Soc.* **113**, 2586–2598.
Etter, M. C., Urbanczyk-Lipkowska, Z., Zia-Ebrahimi, M. & Panunto, T. W. (1990). *J. Am. Chem. Soc.* **112**, 8415–8426.
Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
Katrusiak, A. (1990). *Acta Cryst.* B**46**, 246–256.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Panunto, T. W., Urbanczyk-Lipkowska, Z., Johnson, R. & Etter, M. C. (1987). *J. Am. Chem. Soc.* **109**, 7786–7797.

Acta Cryst. (1995). C**51**, 1663–1665

2,3,5,6-Tetrakis(trifluoromethyl)terephthaloyl Difluoride

Norman C. Baenziger and Donald J. Burton

*Department of Chemistry, University of Iowa,
Iowa City, Iowa 52242, USA*

Vito Tortelli

Ausimont, Via S. Pietro 50, 20021 Bollate, Milan, Italy

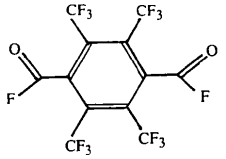
(*Received* 27 *July* 1993; *accepted* 13 *February* 1995)

Abstract

2,3,5,6-Tetrakis(trifluoromethyl)terephthaloyl difluoride, $C_{12}F_{14}O_2$, crystallizes in space group *Fddd*, which requires the molecule to have 222 symmetry. This is achieved by disordering the F, O and C atoms of the acyl halide group. The bulky CF_3 groups cause a slight distortion from planarity of the six-membered ring and they are forced out of coplanarity with the ring. The plane of the —(C═O)—F group is rotated 62° from the plane of the phenyl ring.

Comment

The title compound, (I), was obtained while carrying out the exploratory oxidative coupling of *cis*–$F_2C{=}CFC(CF_3){=}C(CF_3)Cu$. The title compound exhibits two peaks in the ^{19}F NMR spectrum ($CFCl_3$, acetone-d_6) at +55.4 p.p.m. (*s*) [—C(═O)—F] and −54.0 p.p.m. (*s*) (CF_3) in a ratio of 1:6. The acyl halide group appears at 1858 cm^{-1} in the IR spectrum and HRMS analysis shows a molecular ion at 441.9687 corresponding to $C_{12}F_{14}O_2$.



(I)

The bond distances and angles in the title molecule have values close to those expected for this type of compound. The observed deviations from idealized bond

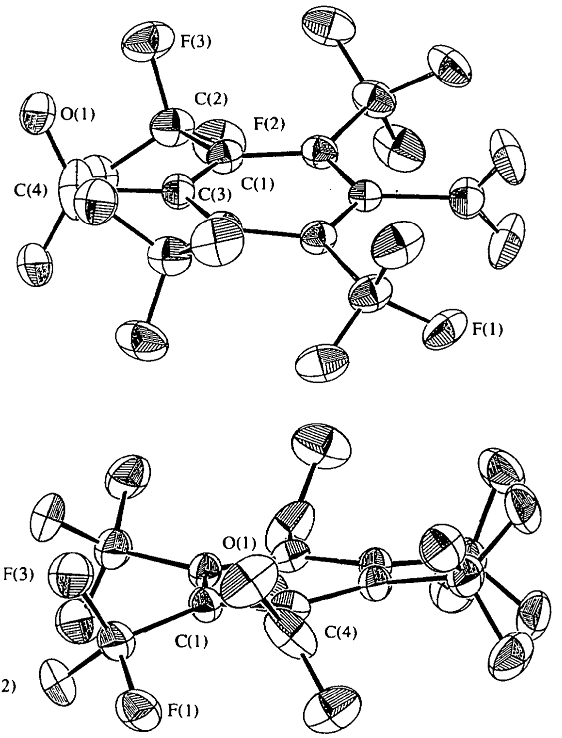


Fig. 1. Two views of the title compound. The upper view shows the —C—C(═O)—F plane lying horizontal, while the lower view is almost end-on along the —C—C(═O)—F plane showing the orientation of these groups relative to the plane of the phenyl ring. Displacement ellipsoids are plotted at the 25% probability level.

distance and angle values appear to follow logically from the crowding of the bulky CF_3 groups, *i.e.* the slight elongation of the C(1)—C(1^{i}) bond [1.406 (6) Å], the enlarged C(2)—C(1)—C(1^{i}) angle [121.7 (2)°; symmetry code: (i) x, $\frac{3}{4}-y$, $\frac{3}{4}-z$], and the buckling of the ring and the associated CF_3 groups [C(1) ±0.06 and C(2) ±0.27 Å from the plane of the ring]. The acyl halide group is rotated so that the dihedral angle between the normals of the phenyl ring plane and the —C(=O)—F plane is 61.8 (8)°. The range of C—F distances in the CF_3 group appears to be typical. The longest C—F distances are to those F atoms which lie closest to the plane of the ring and the shortest distance is associated with the '*exo*' F atom. This correlation of C—F distance with orientation of the CF_3 group relative to the ring appears to be accidental, as it does not appear in tetrakis(trifluoromethyl)cyclopentadienones (Burk, Calabrese, Davidson, Harlow & Roe, 1991).

Experimental

Crystals of the title compound were isolated from a silica gel chromatographic fraction.

Crystal data

$C_{12}F_{14}O_2$
$M_r = 441.97$
Orthorhombic
Fddd
$a = 9.5781\ (8)$ Å
$b = 16.3763\ (18)$ Å
$c = 17.4396\ (14)$ Å
$V = 2735.5\ (7)$ Å^3
$Z = 8$
$D_x = 2.146$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 50$–$66°$
$\mu = 2.62$ mm^{-1}
$T = 295$ K
Tabular
0.50 × 0.32 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
Profile data from θ–2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.967$, $T_{max} = 0.999$
3649 measured reflections
657 independent reflections
537 observed reflections [$I > 3\sigma$]

$R_{int} = 0.026$
$\theta_{max} = 70°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 0$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 200 reflections
frequency: 60 min
intensity decay: <1.2%

Refinement

Refinement on F
$R = 0.051$
$wR = 0.087$
$S = 1.032$
532 reflections
66 parameters

$\Delta\rho_{max} = 0.07$ e Å^{-3}
$\Delta\rho_{min} = -0.06$ e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: 0.00001

$w = 1/[\sigma^2(F) + (0.06F)^2 + 1.0]$
$(\Delta/\sigma)_{max} = 0.12$

Atomic scattering factors from Cromer & Waber (1974)

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

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

	x	y	z	U_{eq}
F(1)	0.6424 (2)	0.2164 (1)	0.3178 (1)	0.0837 (5)
F(2)	0.5223 (2)	0.3265 (2)	0.3180 (1)	0.0890 (6)
F(3)	0.5551 (2)	0.2605 (2)	0.4233 (1)	0.0960 (6)
O(1)†	0.8208 (3)	0.1621 (1)	0.4307 (2)	0.0968 (8)
C(1)	0.7490 (3)	0.3322 (2)	0.3718 (2)	0.0509 (6)
C(2)	0.6140 (3)	0.2840 (2)	0.3592 (2)	0.0694 (8)
C(3)	7/8	0.2905 (2)	3/8	0.0504 (8)
C(4)‡	7/8	0.1986 (3)	3/8	0.0912 (13)

† Disordered O/F site of the acyl halide moiety.
‡ Positionally disordered.

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

C(1)—C(2)	1.531 (4)	C(2)—F(2)	1.331 (5)
C(1)—C(3)	1.388 (3)	C(2)—F(3)	1.309 (4)
C(1)—C(1^{i})	1.406 (6)	C(3)—C(4)	1.506 (7)
C(2)—F(1)	1.348 (5)	C(4)—O(1)	1.252 (4)
C(3)—C(1)—C(1^{i})	119.1 (2)	F(2)—C(2)—F(3)	109.2 (3)
C(3)—C(1)—C(2)	119.1 (3)	C(1)—C(3)—C(4)	119.4 (2)
C(2)—C(1)—C(1^{i})	121.7 (2)	C(1)—C(3)—C(1^{ii})	121.1 (3)
C(1)—C(2)—F(1)	109.3 (3)	C(1^{ii})—C(3)—C(4)	119.4 (2)
C(1)—C(2)—F(2)	111.3 (3)	C(3)—C(4)—O(1)	118.5 (3)
C(1)—C(2)—F(3)	113.2 (3)	C(3)—C(4)—O(1^{ii})	118.5 (3)
F(1)—C(2)—F(2)	105.9 (3)	O(1)—C(4)—O(1^{ii})	123.0 (5)
F(1)—C(2)—F(3)	107.6 (3)		

Symmetry codes: (i) $x, \frac{3}{4}-y, \frac{3}{4}-z$; (ii) $\frac{7}{4}-x, y, \frac{3}{4}-z$.

Data collection used a scan range of $(0.7 + 0.14\tan\theta)°$, a scan speed of 0.7 to 5.5° min^{-1}, a background 25% of the scan range, both below and above, and a counting time reflection/background ratio of 2:1. The structure was determined by direct methods. All atoms were refined with anisotropic displacement parameters. The O and F atoms of the acyl halide group were assumed to be disordered and were refined as a single O atom. The appearance of the displacement ellipsoids for the disordered O/F atoms and the acyl halide C atom indicates that the O and F atoms have typical displacement parameters for their site in the molecule and that they are site disordered. The much greater displacement parameter of the C atom in the plane of the acyl halide group indicates that it is modelling a random placement of the C atom, closer to O and further from F, and it is thus positionally disordered. No attempt was made to model the disorder other than by the displacement ellipsoid.

Data collection, cell refinement and data reduction were carried out using *CAD-4 Software* (Enraf–Nonius, 1989). All other calculations were performed using the *MolEN* package (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: CR1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Burk, M. J., Calabrese, J. C., Davidson, R., Harlow, R. L. & Roe, D. C. (1991). *J. Am. Chem. Soc.* **113**, 2209–2222.
Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A, 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Enraf–Nonius (1989). *CAD-4 Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1995). C**51**, 1665–1667

1,2,3,4,5,8-Hexakis(trimethylsilyl)-1,2,3,4,5,8-hexahydronaphthalene: the First Example of Double-*peri* Trimethylsilyl Substitution

Jean Michel Leger

Laboratoire de Chimie Analytique, UFR des Sciences Pharmaceutiques, 3 Place de la Victoire, 33076 Bordeaux CEDEX, France

Micheline Grignon-Dubois

Laboratoire de Chimie Organique et Organométallique, CNRS URA 35, Université Bordeaux I, 351 Cours de le Libération, 33405 Talence CEDEX, France

Michel Laguerre

Laboratoire de Chimie Analytique, UFR des Sciences Pharmaceutiques, 3 Place de la Victoire, 33076 Bordeaux CEDEX, France

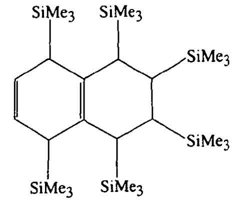
(*Received* 3 *January* 1995; *accepted* 17 *February* 1995)

Abstract

The crystal structure determination of the title compound, $C_{28}H_{62}Si_6$, is the first reporting double-*peri* trimethylsilyl substitution. Four trimethyl groups are in axial positions, the remaining two groups are equatorial.

Comment

Polysilylation of naphthalene has been the subject of numerous works (Dunoguès, Calas, Biran & Duffaut, 1970; Birkofer & Ramadan, 1971; Yang, Liu & Liu, 1978; Laguerre, Dunoguès & Calas, 1981). In particular, it has been claimed (Dunoguès, Calas, Biran & Duffaut, 1970) that derivatives with two silicon groups in *peri* positions (*i.e.* 1,8 substitution of naphthalene) might be difficult to obtain due to exaggerated steric hindrance. On reinvestigation, we showed that some of the previous interpretations were wrong (Grignon-Dubois, Laguerre & Saux, 1995; Grignon-Dubois & Laguerre, 1995). This is well demonstrated by the isolation of 1,2,3,4,5,8-hexakis(trimethylsilyl)-1,2,3,4,5,8-hexahydronaphthalene, (I).



(I)

The geometry of the molecule is shown in Fig. 1. From a close examination of the structure, it appears that this molecule is less sterically hindered than expected. The distances between the *peri* Si atoms are essentially the same as the distance between two adjacent Si atoms (mean 4.34 Å for the five Si···Si distances). The C—Si bond lengths have standard values only for the Si atoms at the naphthalene positions 2 and 3 [C3—Si12 and C4—Si13 1.885 (4) and 1.889 (4) Å, respectively], but for the four Si atoms in the *peri* positions 1,8 and 4,5, the bond lengths are significantly longer than the standard value (mean of C2—Si11, C5—Si14, C7—Si15 and C10—Si16 1.931 Å). This behaviour may contribute to a noticeable lowering of the total steric hindrance. The three (Si)C—C(Si) bonds in the tetrasilylated ring (C2—C3, C3—C4, C4—C5) are longer than usual (1.560–1.574 Å), as found previously

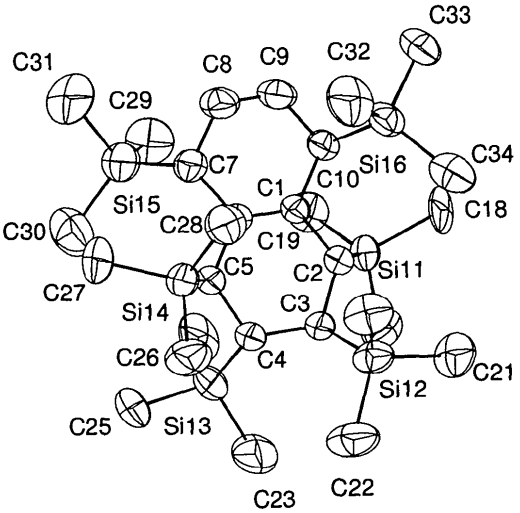


Fig. 1. Perspective view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.