Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2	H2	N1	0.95	2.16	2.971 (3)	143.0 (1)
O	H	N4	0.92	1.76	2.681 (3)	170.7 (1)

2-(2-Oxo-2-phenylethylthio)oxazole[4,5-*b*]pyridine, concentrated acetic acid and hydrazine hydrate reacted at room temperature. After 4 d the mixture was poured into a saturated solution of NaCl and neutralized with 20% NaOH. The precipitate was recrystallized from EtOH.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71513 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1048]

References

Allen, F. N., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1-S19.

Boeyens, J. C. A. (1978). *J. Cryst. Mol. Struct.* **8**, 317-320.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354-1358.

Enraf-Nonius (1985). *Structure Determination Package. SDP/PDP User's Guide.* Version 3.0. Enraf-Nonius, Delft, The Netherlands.

Enraf-Nonius (1988). *CAD-4 Manual.* Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Evans, D. G. & Boeyens, J. C. A. (1989). *Acta Cryst.* **B45**, 581-590.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kalcheva, V. & Tosheva, M. (1990). *Bulg. Appl.* p. 92211.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sasaki, T., Ito, E. & Shimizu, I. (1982). *Heterocycles*, **19**, 2119-2129.

Tosheva, M. & Kalcheva, V. (1993). *Synth. Commun.* In the press.

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4,4′-Difluorobenzophenone

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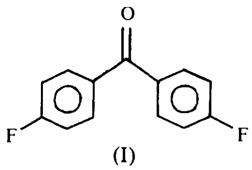
Abstract

The crystal structure of 4,4′-difluorobenzophenone, $C_{13}H_8F_2O$, has been determined and found to be isostructural with the dichloro analogue, though not with the diiodo analogue.

Comment

4,4′-Difluorobenzophenone is a fine chemical product of industrial significance. Crystals were prepared by small-scale recrystallization of the compound from cyclohexane solution.

The title compound (I) is found to be isotructural with the 4,4′-dichloro analogue (Shields & Kennard, 1977; Granger & Coillot, 1985) although the cell volume is slightly smaller, as one would expect from the smaller size of the F atom. The molecule lies on a crystallographic twofold axis along the central carbonyl bond. The phenyl rings are not coplanar; the torsion angle C(2)—C(1)—C(1′)—C(2′) is 47.1 (1)°. This can be compared with values of 48.1 (Shields & Kennard, 1977) and 48.3° (Granger & Coillot, 1985) in 4,4′-dichlorobenzophenone, 48.3° in 4,4′-diiodobenzophenone (van der Velden & Noordik, 1979), 54.5° in 4,4-diaminobenzophenone (van der Velden & Noordik, 1980), and 111.0° in 4,4′-dimethylbenzophenone (Ito *et al.*, 1987; Kojić-Prodić, Bresciani-Pahor & Horvatić, 1990). The 4,4′-diiodo analogue has a different structure, in space group *Ccc*2, reflecting the slightly larger size of iodine and the fine balance of intermolecular forces guiding crystal packing.



(I)

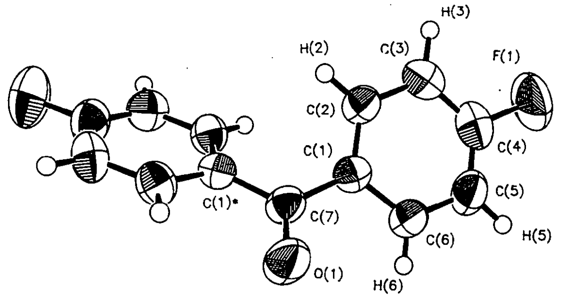


Fig. 1. *ORTEP* (Johnson, 1965) view of the title molecule with ellipsoids at the 50% probability level.

Experimental

Crystal data

$C_{13}H_8F_2O$
$M_r = 218.1$
Monoclinic
$C2/c$
$a = 23.184\ (7)$ Å
$b = 6.17\ (1)$ Å
$c = 7.409\ (9)$ Å
$\beta = 79.868\ (9)°$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 18 reflections
$\theta = 7\text{-}33°$
$\mu = 0.11\ \text{mm}^{-1}$
$T = 295$ K
Tabular

$V = 1043.3$ Å^3
$Z = 4$
$D_x = 1.39$ Mg m^{-3}

0.45 × 0.25 × 0.20 mm
Clear

Data collection

Stoe Stadi-2 diffractometer
ω scans
Absorption correction: none
957 measured reflections
772 independent reflections
557 observed reflections [$|F| > 4\sigma(|F|)$]
$R_{\text{int}} = 0.026$
$\theta_{\max} = 50°$

$h = -28 \rightarrow 28$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 9$
1 standard reflection per layer of k monitored every 25 reflections intensity variation: >2% of I

Refinement

Refinement on F
$R = 0.0465$
$wR = 0.0465$
$S = 0.74$
557 reflections
94 parameters
All H-atom parameters refined

Unit weights applied
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
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)

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

	x	y	z	U_{eq}
C(1)	0.05674 (11)	0.1495 (5)	0.2361 (3)	0.053 (2)
C(2)	0.06132 (13)	0.3522 (6)	0.3090 (4)	0.060 (2)
C(3)	0.11540 (14)	0.4519 (6)	0.3057 (4)	0.070 (2)
C(4)	0.16412 (13)	0.3382 (7)	0.2237 (4)	0.077 (2)
C(5)	0.16191 (13)	0.1389 (7)	0.1492 (5)	0.074 (2)
C(6)	0.10800 (13)	0.0428 (6)	0.1560 (4)	0.062 (2)
C(7)	0	0.0309 (7)	$\frac{1}{4}$	0.055 (2)
F(1)	0.21733 (8)	0.4348 (4)	0.2177 (3)	0.118 (3)
O(1)	0	−0.1663 (6)	$\frac{1}{4}$	0.088 (3)

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

C(1)—C(2)	1.374 (4)	C(1)—C(6)	1.396 (4)
C(2)—C(3)	1.393 (4)	C(1)—C(7)	1.493 (3)
C(3)—C(4)	1.377 (5)	C(7)—O(1)	1.217 (5)
C(4)—C(5)	1.353 (5)	C(4)—F(1)	1.364 (3)
C(5)—C(6)	1.376 (4)		
C(7)—C(1)—C(2)	123.2 (3)	F(1)—C(4)—C(5)	118.9 (3)
C(7)—C(1)—C(6)	118.2 (3)	C(3)—C(4)—C(5)	123.8 (3)
C(2)—C(1)—C(6)	118.5 (3)	C(4)—C(5)—C(6)	118.4 (3)
C(1)—C(2)—C(3)	121.8 (3)	C(5)—C(6)—C(1)	120.8 (3)
C(2)—C(3)—C(4)	116.7 (3)	C(1)—C(7)—O(1)	119.3 (2)
F(1)—C(4)—C(3)	117.3 (4)	C(1)—C(7)—C(1′)	121.4 (2)

The data were collected, by two-circle diffractometer, in layers of k and, therefore, inter-layer scale factors were refined (final values of these ranged between 0.73 and 0.94). Structure solution was by *SHELXS*86 (Sheldrick, 1985) and the refinement used *SHELX*76 (Sheldrick, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71516 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1056]

References

Granger, M. M. & Coillot, M. F. (1985). *Acta Cryst.* **C41**, 542–543.
Ito, Y., Matsuura, T., Tabata, K., Meng Ji-Ben, Fukuyama, K., Sasaki, M. & Okada, S. (1987). *Tetrahedron*, **43**, 1307–1312.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kojić-Prodić, B., Bresciani-Pahor, N. & Horvatić, D. (1990). *Acta Cryst.* **C46**, 430–432.
Shields, K. G. & Kennard, C. H. L. (1977). *J. Chem. Soc. Perkin Trans.* 2, pp. 463–465.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Velden, G. P. M. van der & Noordik, J. H. (1979). *J. Cryst. Mol. Struct.* **10**, 83–92.
Velden, G. P. M. van der & Noordik, J. H. (1980). *J. Cryst. Mol. Struct.* **9**, 283–294.

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Substitution Effects on the Structure of *o*-Aryliminophosphoranes

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Abstract

The iminophosphorane group in (*o*-dimethylaminophenylimino)triphenylphosphorane, $C_{26}H_{25}N_2P$, lies almost in the benzene plane [C6—C1—N11—P12 = −8.3 (3)°] giving rise to an inward distortion of the external angle at C1. The conformation adopted by the dimethylamino group reflects the electronic repulsion between both N-atom lone pairs and places one methyl almost eclipsed by the benzene ring [C3—C2—N31—C33 = −14.5 (3)°]. The optimized geometry (AM1) of *o*-dimethylaminobenzene shows both groups equally twisted (−27.8°) but to a lesser extent than in (*o*-dimethylaminophenylimino)triphenylphosphorane.

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