$C1^{\alpha}$—C1′	1.525 (5)	$C2^{\alpha}$—$C2^{\beta}$	1.525 (5)
$C1^{\beta}$—$C1^{\gamma}$	1.493 (7)	$C2^{\alpha}$—C2′	1.513 (4)
$C1^{\gamma}$—$C1^{\delta}$	1.507 (7)	$C2^{\beta}$—$C2^{\gamma}$	1.497 (5)
$C2^{\gamma}$—$C2^{\delta1}$	1.548 (7)	$C3^{\alpha}$—$C3^{\beta}$	1.551 (5)
$C2^{\gamma}$—$C2^{\delta2}$	1.526 (7)	$C3^{\alpha}$—C3′	1.535 (5)
C2′—O2	1.234 (4)	$C3^{\beta}$—$C3^{\gamma}$	1.519 (6)
C2′—N3	1.330 (4)	$C3^{\gamma}$—$C3^{\delta}$	1.528 (6)
N3—$C3^{\alpha}$	1.453 (4)	C3′—N4	1.325 (4)
N3—$C3^{\delta}$	1.470 (4)	C3′—O3	1.216 (4)
$C1^{\alpha}$—N_1—$C1^{\delta}$	108.3 (6)	N2—$C2^{\alpha}$—$C2^{\beta}$	110.8 (5)
N_1—$C1^{\alpha}$—$C1^{\beta}$	106.2 (5)	N2—$C2^{\alpha}$—C2′	109.1 (4)
N_1—$C1^{\alpha}$—C1′	111.9 (5)	$C2^{\beta}$—$C2^{\alpha}$—C2′	108.3 (5)
$C1^{\beta}$—$C1^{\alpha}$—C1′	110.9 (6)	$C2^{\alpha}$—$C2^{\beta}$—$C2^{\gamma}$	116.2 (5)
$C1^{\alpha}$—$C1^{\beta}$—$C1^{\gamma}$	102.8 (6)	$C2^{\beta}$—$C2^{\gamma}$—$C2^{\delta1}$	109.0 (6)
$C1^{\beta}$—$C1^{\gamma}$—$C1^{\delta}$	104.2 (7)	$C2^{\beta}$—$C2^{\gamma}$—$C2^{\delta2}$	111.4 (6)
N_1—$C1^{\delta}$—$C1^{\gamma}$	105.5 (7)	$C2^{\delta1}$—$C2^{\gamma}$—$C2^{\delta2}$	110.5 (7)
$C1^{\alpha}$—C1′—O1	120.4 (6)	$C2^{\alpha}$—C2′—O2	120.5 (5)
$C1^{\alpha}$—C1′—N2	115.9 (6)	$C2^{\alpha}$—C2′—N3	119.0 (5)
O1—C1′—N2	123.7 (6)	O2—C2′—N3	120.5 (5)
C1′—N2—$C2^{\alpha}$	123.1 (5)	C2′—N3—$C3^{\alpha}$	120.5 (5)
C2′—N3—$C3^{\delta}$	126.6 (5)	$C3^{\beta}$—$C3^{\gamma}$—$C3^{\delta}$	103.6 (5)
$C3^{\alpha}$—N3—$C3^{\delta}$	112.9 (4)	N3—$C3^{\delta}$—$C3^{\gamma}$	103.6 (5)
N3—$C3^{\alpha}$—$C3^{\beta}$	102.5 (4)	$C3^{\alpha}$—C3′—N4	115.7 (5)
N3—$C3^{\alpha}$—C3′	110.1 (4)	$C3^{\alpha}$—C3′—O3	120.5 (5)
$C3^{\beta}$—$C3^{\alpha}$—C3′	112.0 (5)	N4—C3′—O3	123.9 (5)
$C3^{\alpha}$—$C3^{\beta}$—$C3^{\gamma}$	103.3 (5)		

Lists of structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71453 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VJ1000]

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Acta Cryst. (1994). C**50**, 252–254

2-[(5-Phenyl-2,3-dihydro-6*H*-1,3,4-thiadiazin-2-ylidene)amino]-3-pyridinol

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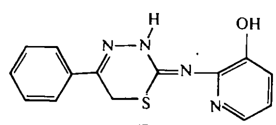
(*Received* 15 *April* 1993; *accepted* 14 *June* 1993)

Abstract

The molecules of $C_{14}H_{12}N_4OS$ are dimerized through N—$H_{endo}\cdots N_{exo}$ hydrogen bonds [N···N 2.971 (3) Å] and linked in chains along the *a* axis by a short O—H···N_{py} hydrogen bond [O···N 2.681 (2) Å]. The thiadiazine ring is in a screw-boat conformation.

Comment

Treatment of functionalized 2-(2-oxopropyl-2-oxo-2-phenylethylthio)oxazole[4,5-*b*]pyridines with N-containing nucleophiles leads to the formation of substituted thiadiazines (Tosheva & Kalcheva, 1993). Some of these compounds have shown pronounced bacteriostatic activity and low toxicity (Kalcheva & Tosheva, 1990). The synthetic route for analogous 2*H*-imidazo[2,1-*b*]thiadiazines was given by Sasaki, Ito & Shimizu (1982).



(I)

Ring puckering parameters for the thiadiazine ring ${}^6S_1$ (Boeyens, 1978) are q_2 = 0.576, q_3 = −0.231 Å, φ = 146.75°; Q = 0.620 (2) Å, θ = 111.8 (2)° (Cremer & Pople, 1975; Evans & Boeyens, 1989). The large φ value indicates that the direction of the ring distortion is towards an inverted screw-boat conformation. The S—C1 1.751 (2) and S—C2 1.815 (2) Å bond lengths correspond to the typical S—C_{sp^2} and S—C_{sp^3} single bonds [1.751 (17), 1.819 (19) Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987]. The endocyclic C1—N2 distance [1.363 (3) Å] is longer than the exocyclic C1—N1 distance [1.294 (3) Å]. The C1—N1 and the endocyclic N3—C3 [1.286 (3) Å] bonds have values close to that of a double N=C_{sp^2} bond [1.329 (14) Å, Allen *et al.*, 1987]. The N2—N3 bond [1.382 (3) Å] is slightly longer than the average single N(1)—N(2) bond [1.366 (19) Å, Allen

ISSN 0108-2701

et al., 1987]. The large C1—N2—N3 angle [127.8 (2)°] at the sp^2-hybridized N atom results from the angular strain introduced by the S atom. The phenyl and hydroxypyridyl rings are twisted out of the best plane through the C1—N2—N3—C3 fragment [maximum deviation 0.125 (2) Å] by angles of 35.9 (1) and 52.1 (1)°, respectively.

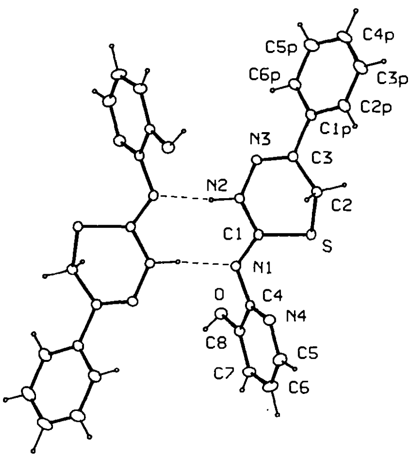


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the dimer with the atom-numbering scheme. Thermal ellipsoids are drawn at the 20% probabily level; H atoms are represented by spheres of arbitrary size.

Experimental

Crystal data

$C_{14}H_{12}N_4OS$
M_r = 284.34
Monoclinic
$P2_1/a$
a = 11.679 (2) Å
b = 8.301 (1) Å
c = 13.554 (1) Å
β = 91.97 (2)°
V = 1313.2 (5) Å^3
Z = 4
D_x = 1.438 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 22 reflections
θ = 20.46–21.58°
μ = 0.23 mm^{-1}
T = 292 K
Plate
0.27 × 0.25 × 0.10 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
Continuous profiles scans
Absorption correction: empirical
T_{min} = 0.980, T_{max} = 0.999
5191 measured reflections
2577 independent reflections
1634 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.028
θ_{max} = 26°
$h = 0 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 16$
3 standard reflections frequency: 120 min intensity variation: 1.9%

Refinement

Refinement on F
R = 0.034
wR = 0.030
S = 1.480
1634 reflections
181 parameters
H atoms refined as riding
$w = 1/[\sigma^2(F) + (0.001F)^2]$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.291 e Å^{-3}
$\Delta\rho_{min}$ = −0.210 e Å^{-3}
Atomic scattering factors as coded in *SDP*/*PDP* (Enraf-Nonius, 1985)

The hydroxyl H atom was localized from a $\Delta\rho$ map, the other H-atom positions were calculated. Data collection: CAD-4 (Enraf-Nonius, 1988). Data reduction: *SDP*/*PDP* (Enraf-Nonius, 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP*/*PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Macíček, 1992; unpublished).

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

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

	x	y	z	U_{eq}
S	0.36122 (5)	0.07458 (8)	0.01553 (5)	0.0285 (1)
O	0.6803 (1)	0.2665 (2)	−0.1377 (1)	0.0330 (5)
N1	0.4711 (2)	0.3220 (2)	−0.0720 (1)	0.0256 (5)
N2	0.3883 (2)	0.3798 (2)	0.0726 (1)	0.0283 (5)
N3	0.3484 (2)	0.3484 (3)	0.1654 (1)	0.0279 (5)
N4	0.3882 (2)	0.1877 (3)	−0.2110 (1)	0.0305 (5)
C1	0.4102 (2)	0.2719 (3)	−0.0002 (2)	0.0240 (6)
C2	0.2397 (2)	0.1284 (3)	0.0887 (2)	0.0297 (6)
C3	0.2791 (2)	0.2293 (3)	0.1745 (2)	0.0268 (6)
C4	0.4813 (2)	0.2357 (3)	−0.1597 (2)	0.0231 (6)
C5	0.4004 (2)	0.1161 (4)	−0.2989 (2)	0.0410 (8)
C6	0.5051 (2)	0.0956 (4)	−0.3402 (2)	0.0443 (8)
C7	0.6025 (2)	0.1490 (3)	−0.2890 (2)	0.0354 (7)
C8	0.5923 (2)	0.2177 (3)	−0.1976 (2)	0.0255 (6)
C1p	0.2362 (2)	0.1978 (3)	0.2740 (2)	0.0297 (6)
C2p	0.1365 (2)	0.1082 (4)	0.2869 (2)	0.0410 (7)
C3p	0.0986 (2)	0.0776 (4)	0.3801 (2)	0.0532 (9)
C4p	0.1572 (3)	0.1378 (4)	0.4621 (2)	0.061 (1)
C5p	0.2545 (3)	0.2278 (5)	0.4508 (2)	0.065 (1)
C6p	0.2945 (3)	0.2567 (4)	0.3573 (2)	0.0492 (9)

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

S—C1	1.751 (2)	C3—C1p	1.478 (4)
S—C2	1.815 (2)	C4—C8	1.419 (3)
O—C8	1.349 (3)	C5—C6	1.373 (4)
N1—C1	1.294 (3)	C6—C7	1.385 (4)
N1—C4	1.395 (3)	C7—C8	1.373 (4)
N2—N3	1.382 (3)	C1p—C2p	1.398 (4)
N2—C1	1.363 (3)	C1p—C6p	1.388 (4)
N3—C3	1.286 (3)	C2p—C3p	1.377 (4)
N4—C4	1.332 (3)	C3p—C4p	1.379 (4)
N4—C5	1.343 (3)	C4p—C5p	1.373 (5)
C2—C3	1.492 (3)	C5p—C6p	1.386 (4)
C1—S—C2	95.8 (1)	N4—C5—C6	122.7 (2)
C1—N1—C4	122.7 (2)	C5—C6—C7	118.9 (3)
N3—N2—C1	127.8 (2)	C6—C7—C8	119.4 (2)
N2—N3—C3	117.7 (2)	O—C8—C4	115.9 (2)
C4—N4—C5	119.1 (2)	O—C8—C7	125.4 (2)
S—C1—N1	125.7 (2)	C4—C8—C7	118.6 (2)
S—C1—N2	117.1 (2)	C3—C1p—C2p	121.2 (2)
N1—C1—N2	117.1 (2)	C3—C1p—C6p	120.5 (2)
S—C2—C3	109.7 (2)	C2p—C1p—C6p	118.3 (2)
N3—C3—C2	122.4 (2)	C1p—C2p—C3p	120.6 (3)
N3—C3—C1p	117.2 (2)	C2p—C3p—C4p	120.4 (3)
C2—C3—C1p	120.4 (2)	C3p—C4p—C5p	119.8 (3)
N1—C4—N4	120.4 (2)	C4p—C5p—C6p	120.2 (3)
N1—C4—C8	117.9 (2)	C1p—C6p—C5p	120.7 (3)
N4—C4—C8	121.3 (2)		
C2—S—C1—N2	29.2 (2)	C1—N2—N3—C3	−33.0 (3)
C1—S—C2—C3	−53.4 (2)	N3—N2—C1—S	11.4 (3)
C4—N1—C1—S	17.1 (3)	N2—N3—C3—C2	−1.3 (3)
C1—N1—C4—N4	52.9 (3)	S—C2—C3—N3	46.2 (3)

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]

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Acta Cryst. (1994). **C50**, 254–255

4,4′-Difluorobenzophenone

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(*Received* 22 *March* 1993; *accepted* 9 *July* 1993)

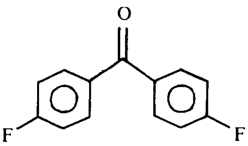
Abstract

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,



(I)

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.

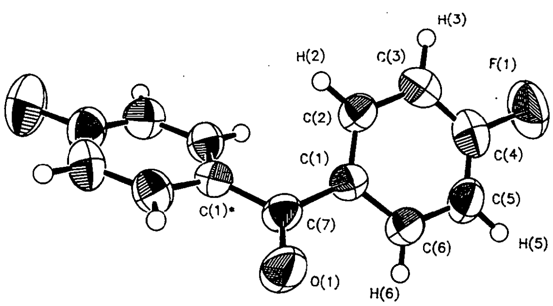


Fig. 1. *ORTEP* (Johnson, 1965) view of the title molecule with ellipsoids at the 50% probability level.

Experimental

Crystal data

$C_{13}H_8F_2O$	Mo $K\alpha$ radiation
M_r = 218.1	λ = 0.71069 Å
Monoclinic	Cell parameters from 18 reflections
$C2/c$	θ = 7-33°
a = 23.184 (7) Å	μ = 0.11 mm^{-1}
b = 6.17 (1) Å	T = 295 K
c = 7.409 (9) Å	Tabular
β = 79.868 (9)°	