All non-H atoms in the structure were found by direct methods. All H atoms were located by subsequent isotropic refinement and difference electron-density synthesis. Final full-matrix least-squares refinement of the coordinates and anisotropic displacement parameters for the non-H atoms, and of the coordinates and isotropic temperature factors for the H atoms, reduced R to 0.0434.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*92 (Vicković, 1994) and *PLUTON* (Spek, 1993). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *CSU* (Vicković, 1988).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,4-Bis(1,3-benzothiazol-2-yl)furan

DRAGINJA MRVOŠ-SERMEK,[a]* VESNA TRALIĆ-KULENOVIĆ,[b] ANTE NAGL[b] AND LELJA FIŠER-JAKIĆ[b]

[a]*Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, 10000 Zagreb, Croatia, and* [b]*Faculty of Textile Technology, University of Zagreb, Pierottijeva 6, 10000 Zagreb, Croatia.*
E-mail: mrvos@olimp.irb.hr

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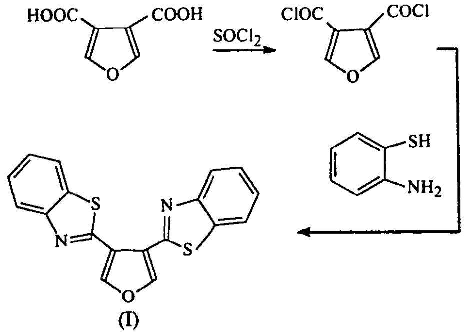
Abstract

The title compound, $C_{18}H_{10}N_2OS_2$, was prepared and characterized on the basis of its ^1H NMR and UV spectra, and X-ray diffraction analysis. The five-membered heterocyclic rings are flat and coplanar with their fused benzene rings. The furan ring is planar, with the 2-benzothiazolyl groups rotated out of plane by 6.3 (1) and 32.7 (1)°. The S atoms of the 2-benzothiazolyl groups are positioned *anti* with respect to one another.

Comment

In the course of our previous studies, we have paid attention on the synthesis and spectroscopic properties of some aryl- and heteroaryl-substituted benzothiazoles (Fišer-Jakić, Karaman & Jakopčić, 1980; Fišer-Jakić & Jakopčić, 1981; Tralić-Kulenović, Fišer-Jakić & Lazarević, 1993, 1994; Racané, Tralić-Kulenović, Karminski-Zamola & Fišer-Jakić, 1995). Similar substances are known and used as plant protectors and pharmaceuticals (Papenfuhs, 1987), growth-regulating substances (Chulák, Sutoris, Gáplovský & Sekerka, 1990), pesticides (Lácová, Chovancová, Hýblová & Varkonda, 1991), intermediates for dyes (Barni, Savarino, Larovere & Viscardi, 1986) and optical brighteners (Allen, 1971). Continuing the earlier investigations of furylbenzothiazoles, the title compound, 3,4-bis(1,3-benzothiazol-2-yl)furan, (I), was prepared.

The bond distances in both five-membered heterocyclic rings are consistent with those usually found



(I)

(Potenza & Mastropaolo, 1974; Rudd & Barany, 1984). Each of the phenyl rings and their fused five-membered heterocyclic rings are coplanar. The S1—C7 and S2—C14 distances [1.728 (4) and 1.717 (4) Å, respectively] are less than the S1—C6 and S2—C13 distances [1.745 (4) and 1.759 (4) Å, respectively] owing to the fact that the C6 and C13 atoms are sp^2 hybridized, whereas atoms C7 and C14 are part of the aromatic ring (Yeap, Fun, Teo & Teoh, 1991). The S1 and S2 atoms of the 2-benzothiazolyl groups are positioned *anti* with respect to one another. The S1···N2 distance [2.928 (4) Å] is shorter than the sum of the van der Waals radii of S and N (3.35 Å), indicating some interaction between these atoms (Hori *et al.*, 1987). The molecular structure of (I) is therefore not planar. The dihedral angle between the planes of the 2-benzothiazolyl groups is 35.3 (1)°. The twisting is more pronounced about the axis through the C4 and C13 atoms than about the axis through the C3 and C6 atoms [C2—C3—C6—N1 −5.2 (7), C4—C3—C6—S1 −6.9 (7), C3—C4—C13—N2 33.6 (7) and C5—C4—C13—S2 31.7 (6)°].

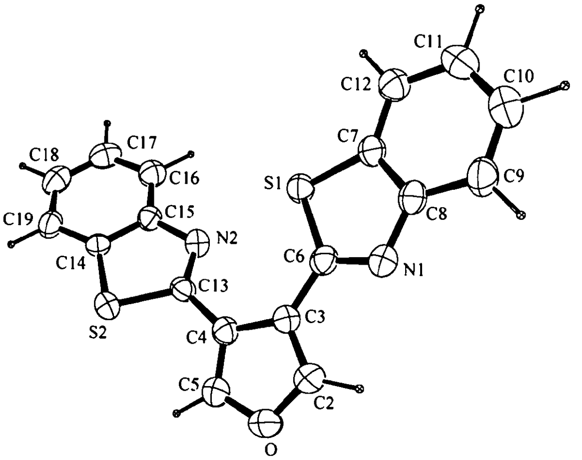


Fig. 1. *ORTEP*92 (Vicković, 1994) view of the title molecule with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared by heating a mixture of 3,4-furandicarboxylic acid (5.3 g, 40 mmol) with an excess of thionyl chloride (24 g, 34 mmol). After evaporation of unreacted $SOCl_2$, residual dichloride of 3,4-furandicarboxylic acid (7.6 g, 40 mmol) was dissolved in benzene, *o*-aminobenzenethiol (10 g, 80 mmol) added and the resulting mixture refluxed for 1 h. After cooling, the product was isolated by filtration in 60% yield. Recrystallization from a dimethylformamide/water solution afforded the pure compound (m.p. 359–361 K). ^1H NMR (DMSO, Jeol FX-100): δ 7.46–7.60 (4H, *m*), 8.08–8.21 (4H, *m*), 8.69 p.p.m. (2H, *s*). UV/Vis (EtOH, Hitachi Perkin-Elmer 124 spectrophotometer): λ_{max} 283 nm. Single crystals of X-ray diffraction quality were obtained from acetone.

Crystal data

$C_{18}H_{10}N_2OS_2$
$M_r = 334.42$
Monoclinic
$P2_1/n$
$a = 20.753$ (7) Å
$b = 4.017$ (1) Å
$c = 17.476$ (3) Å
$\beta = 93.25$ (2)°
$V = 1454.5$ (6) $Å^3$
$Z = 4$
$D_x = 1.527$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71070$ Å
Cell parameters from 20 reflections
$\theta = 3.94–12.64°$
$\mu = 0.371$ mm^{-1}
$T = 293$ (2) K
Needle
0.80 × 0.20 × 0.15 mm
Pale yellow

Data collection

Philips PW1100 diffractometer updated by Stoe
$\theta/2\theta$ scans
Absorption correction: none
1645 measured reflections
1645 independent reflections
1622 observed reflections $[I > 2\sigma(I)]$

$\theta_{max} = 29.90°$
$h = -28 \rightarrow 27$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 24$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.0440$
$wR(F^2) = 0.1088$
$S = 1.036$
1645 reflections
206 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.1898P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.021$
$\Delta\rho_{max} = 0.270$ e $Å^{-3}$
$\Delta\rho_{min} = -0.215$ e $Å^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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}
S1	0.31014 (5)	−0.0063 (3)	0.60064 (6)	0.0404 (3)
S2	0.21375 (5)	0.1360 (3)	0.33218 (6)	0.0422 (3)
N1	0.42147 (15)	−0.2846 (9)	0.5858 (2)	0.0434 (9)
N2	0.24741 (15)	0.3409 (9)	0.4692 (2)	0.0383 (8)
O	0.42605 (14)	−0.0697 (10)	0.3523 (2)	0.0617 (10)
C2	0.4331 (2)	−0.1448 (14)	0.4282 (2)	0.0527 (12)
C3	0.3795 (2)	−0.0664 (11)	0.4653 (2)	0.0383 (10)
C4	0.3347 (2)	0.0665 (11)	0.4062 (2)	0.0399 (11)
C5	0.3659 (2)	0.0565 (12)	0.3407 (2)	0.0487 (12)
C6	0.3748 (2)	−0.1272 (11)	0.5474 (2)	0.0380 (10)
C7	0.3483 (2)	−0.1836 (10)	0.6811 (2)	0.0356 (9)
C8	0.4074 (2)	−0.3198 (11)	0.6620 (2)	0.0379 (10)
C9	0.4468 (2)	−0.4742 (12)	0.7183 (2)	0.0499 (12)
C10	0.4258 (2)	−0.4967 (12)	0.7907 (2)	0.0504 (11)
C11	0.3679 (2)	−0.3591 (8)	0.8101 (2)	0.0489 (11)
C12	0.3280 (2)	−0.2027 (8)	0.7556 (2)	0.0440 (11)
C13	0.2690 (2)	0.1888 (11)	0.4114 (2)	0.0355 (9)
C14	0.1566 (2)	0.3379 (11)	0.3823 (2)	0.0361 (9)
C15	0.1830 (2)	0.4303 (10)	0.4549 (2)	0.0361 (10)
C16	0.1465 (2)	0.6004 (11)	0.5057 (2)	0.0457 (11)
C17	0.0831 (2)	0.6754 (13)	0.4835 (3)	0.0535 (12)
C18	0.0570 (2)	0.5828 (12)	0.4123 (3)	0.0527 (13)
C19	0.0925 (2)	0.4152 (12)	0.3608 (2)	0.0488 (12)

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

S1—C7	1.728 (4)	N1—C6	1.310 (5)
S1—C6	1.745 (4)	N1—C8	1.386 (5)
S2—C14	1.717 (4)	N2—C13	1.284 (5)
S2—C13	1.759 (4)	N2—C15	1.393 (5)
C7—S1—C6	89.3 (2)	C6—N1—C8	110.6 (3)
C14—S2—C13	88.8 (2)	C13—N2—C15	110.8 (3)

H atoms were added at calculated positions and refined with a common isotropic displacement parameter using a riding model.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*92 (Vicković, 1994). Geometric calculations: *CSU* (Vicković, 1988). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: SK1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(2-Chlorophenyl)-1-(2-naphthyl)-2-propen-1-one†

D. Kumaran,[a] S. Eswaramoorthy,[a] M. N. Ponnuswamy,[a]* K. S. Raju[a] and S. Nanjundan[b]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras 600 025, India, and* [b]*Department of Chemistry, Anna University, Madras 600 025, India*

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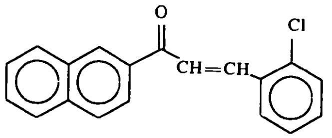
Abstract

The carbonyl group in the α,β-unsaturated ketone system of the title compound, $C_{19}H_{13}ClO$, is in an *s-cis* conformation. The naphthyl ring is twisted by 27.68 (5)° with respect to the phenyl ring. The naphthoyl group is *trans* to the chlorophenyl group.

Comment

Chalcones (α,β-unsaturated ketones) derived from acetonaphthones are potential germicides, fungicides, bactericides and carcinogenics (Eaton & Davis, 1950; Misra, Kushwaha & Lal, 1970; Misra, Tewari & Nath, 1971; Geiger & Conn, 1945). The effectiveness of these compounds is associated with the tendency of substituents to withdraw electrons from the double bond, since saturation of the double bond by hydrogenation makes the compounds inactive. The substitution in benzene by a halogen enhances the activity (Kamoda, 1954). Even though greater anticancer properties have been found in many unsaturated ketones, the compounds with halogen atoms have higher cytotoxicity to leukemic cells. The structure of 3-(2-chlorophenyl)-1-(2-naphthyl)-2-propen-1-one, (I), is presented here.



(I)

The title molecule is found to have an *s-cis* conformation of the α,β-unsaturated ketone system as evidenced by the torsion angle O20—C11—C12—C13 of −21.4 (3)° (Fig. 1). The torsion angle C2—C11—C12—C13 of 153.6 (2)° indicates that the ketone system is not planar.

The C═O bond distance of 1.222 (3) Å is in agreement with the values reported in the literature (Carpy,

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† DCB contribution No. 869.