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2′,3′-Diphenyl-4′-*p*-tolylspiro[isothiachroman-3,5′-isoxazolidin]-4(2*H*)-one

Brahim Bennani,[a] Bouchra Filalibaba,[a] Ahmed El-Fazazi,[a] Ghali Al Houari,[a] Najib Bilit,[a] Abdelali Kerbal,[a] Brahim El-Bali[a] and Michael Bolte[b]*

[a]Laboratoire de Chimie Organique, Faculté des Sciences, Dhar El Mahraz, Université Sidi Mohammed Ben Abdelah, 1796 Atlas, Fès, Morocco, and [b]Institut für Organische Chemie, J.-W.-Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.099
Data-to-parameter ratio = 13.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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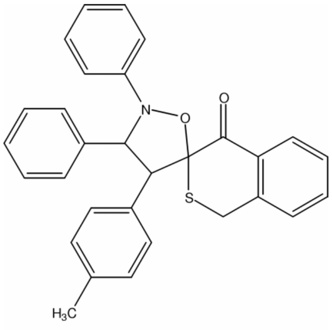
The title compound, $C_{30}H_{25}NO_2S$, contains an isoxazolidine ring in a twist conformation with the spiro-C [0.404 (2) Å] and the adjacent C atom [0.266 (2) Å] deviating from the plane of the remaining three atoms. The thiapyran ring adopts a sofa conformation, with the five C atoms in a common plane (r.m.s.d. = 0.033 Å) and the S atom deviating by 0.984 (2) Å from this plane.

Comment

The dipolar 1,3-cycloaddition reaction between alkenes and nitrones is an important method for producing isoxazolidines, which can be easily converted to other compounds, as reported in the literature (Gothelf & Jorgensen, 1994; Broggini & Zecchi, 1997; Coutouli-Argyropoulou *et al.*, 1997). The present work is a contribution to these investigations, focusing on the dipole–dipolarophile approach in dipolar 1,3-cycloaddition reactions. The case in which the dipolarophilic site is inserted into different cyclic systems (Tshiamala *et al.*, 1988; Kerbal *et al.*, 1989; Filali Baba *et al.*, 2000) or non-cyclic systems (Kerbal *et al.*, 1988, 1991) has already been studied. After having explored the reaction between diphenylnitrilimines (DANI) and 3-arylidenisothiochroman-4-ones (Badri *et al.*, 1999), we investigated the effects of the former on nitrones. In this case, the reaction is regiospecific and leads to spiro products. The synthesis of 2-arylideneisothiochroman-4-ones by condensation of *para*-substituted benzaldehyde with isothiochroman-4-one in an acidic medium has been accomplished according to reported procedures (Kerbal *et al.*, 1990; Riahi *et al.*, 1998) (see Scheme). Since NMR spectroscopy did not provide sufficient information about the nature of the reaction product, we have carried out the X-ray structure analysis of the title compound, (I).



(I)

The ioxazolidine ring shows a twist conformation with C1 [0.404 (2) Å] and C5 [0.266 (2) Å] deviating from the plane of the remaining three atoms. The thiapyran ring adopts a sofa

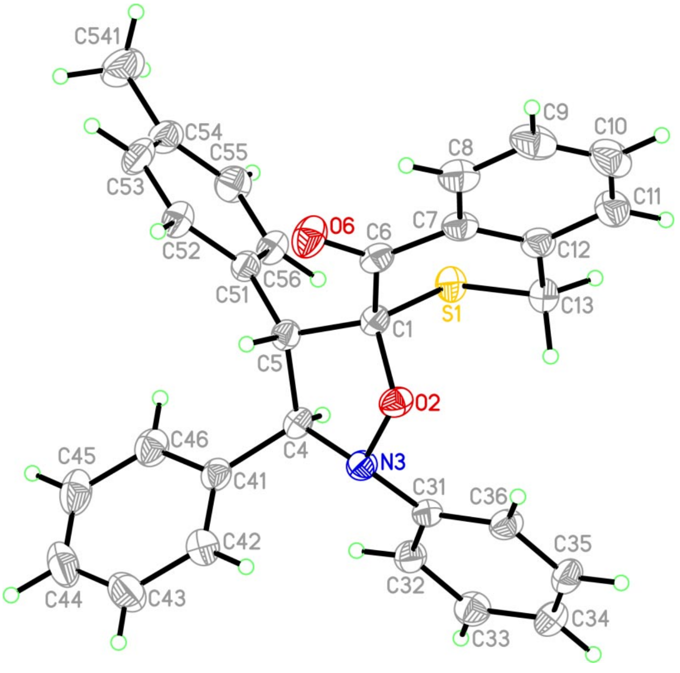


Figure 1
A perspective view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

conformation, with the five C atoms in a common plane (r.m.s.d. = 0.033 Å) and the S atom deviating by 0.984 (2) Å from this plane.

Experimental

The title compound was prepared according to Badri *et al.* (1999). Crystals were grown from an ethanol solution.

Crystal data

$C_{30}H_{25}NO_2S$
$M_r = 463.57$
Triclinic, $P\bar{1}$
$a = 9.6687$ (2) Å
$b = 11.2722$ (3) Å
$c = 11.5579$ (3) Å
$\alpha = 78.850$ (2)°
$\beta = 73.486$ (2)°
$\gamma = 75.860$ (2)°
$V = 1160.66$ (5) Å^3
$Z = 2$
$D_x = 1.326$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6557 reflections
$\theta = 1$–25°
$\mu = 0.17$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.41 × 0.36 × 0.18 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.934$, $T_{\max} = 0.970$
22101 measured reflections
4250 independent reflections
3575 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 25.4°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$
227 standard reflections
frequency: 1200 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.099$
$S = 1.05$
4250 reflections
308 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.6774P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C13	1.8121 (19)	O2—N3	1.4609 (18)
S1—C1	1.8280 (18)	N3—C4	1.476 (2)
C1—O2	1.431 (2)	C4—C5	1.547 (2)
C1—C5	1.529 (2)		
C13—S1—C1	95.14 (9)	C31—N3—C4	118.55 (14)
C1—O2—N3	106.71 (12)	O2—N3—C4	108.29 (12)
C31—N3—O2	111.65 (13)		

All H atoms were located by difference Fourier syntheses and refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm C_{methyl}})$], using a riding model with tertiary C—H = 1.00 Å, secondary C—H = 0.99 Å, methyl C—H = 0.98 Å and aromatic C—H = 0.95 Å. The methyl group was allowed to rotate about its local threefold axes.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1991).

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