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Structure of 5′-Acetyl-3′-(4-bromophenyl)spiro[4*H*-1-benzopyran-4,2′-(3′*H*)-[1,3,4]thiadiazole]

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Abstract. $C_{18}H_{13}BrN_2O_2S$, $M_r = 401{\cdot}3$, monoclinic, $P2_1/a$, $a = 11{\cdot}941\ (2)$, $b = 10{\cdot}038\ (2)$, $c = 14{\cdot}340\ (2)$ Å, $\beta = 101{\cdot}12\ (1)°$, $V = 1686{\cdot}6\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}580$ Mg m^{-3}, $F(000) = 808$, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 1{\cdot}543$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}040$ for 2515 observed reflections. The cycloaddition reaction of the conjugated thiocarbonyl compound 4*H*-1-benzopyran-4-thione with *C*-acetyl-*N*-(*p*-bromophenyl)nitrilimine was reported to occur at the C=C bond leaving the C=S function intact [Baruah, Prajapati & Sandhu (1988). *Tetrahedron*, **44**, 6137–6142; (1989). *Tetrahedron*, **45**, 1231]. Investigation of the molecular structure of the product by X-ray diffraction techniques has shown that it is the C=S bond which is involved in the cycloaddition process. The molecule shows no unusual bond lengths or angles.

Experimental. Yellow crystal, dimensions 0·30 × 0·35 × 0·50 mm, Nicolet *R3m/V* diffractometer with graphite monochromator. Cell dimensions from setting angles of 44 reflections with 2θ values 36–44°. Total of 6268 reflections measured to $2\theta = 50°$ using 2θ–θ scans in the range $0 \le h \le 14$, $-13 \le k \le 11$, $-17 \le l \le 16$. Three reference reflections monitored periodically showed no significant change. Correction was made for absorption using a semi-empirical method, transmission 0·26–0·31. Structure solution, refinement and graphics using *SHELXTL-Plus* (Sheldrick, 1988). Structure determined using direct methods, refined by full-matrix least squares on *F* with anisotropic thermal parameters for C, O, N, S and Br, H atoms included in the model in their calculated positions and allowed to ride on their parent atoms with fixed isotropic thermal param-

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
Br(1)	3121 (1)	203 (1)	79 (1)	62 (1)
S(1)	1792 (1)	−6125 (1)	−4113 (1)	51 (1)
N(1)	1962 (2)	−4216 (3)	−2848 (2)	49 (1)
N(2)	855 (2)	−4585 (3)	−3045 (2)	45 (1)
O(1)	5120 (2)	−4871 (3)	−3636 (2)	66 (1)
O(2)	−674 (2)	−6969 (2)	−4535 (2)	62 (1)
C(1)	3523 (3)	−6354 (4)	−1960 (3)	59 (1)
C(2)	4394 (4)	−7044 (4)	−1382 (3)	74 (2)
C(3)	5476 (4)	−7037 (4)	−1596 (3)	74 (2)
C(4)	5700 (3)	−6311 (4)	−2348 (3)	65 (1)
C(5)	4801 (3)	−5594 (3)	−2916 (2)	49 (1)
C(6)	3713 (3)	−5617 (3)	−2739 (2)	42 (1)
C(7)	4314 (3)	−4009 (3)	−4114 (2)	56 (1)
C(8)	3243 (3)	−3961 (3)	−4008 (2)	50 (1)
C(9)	2768 (3)	−4894 (3)	−3383 (2)	42 (1)
C(10)	635 (3)	−5521 (3)	−3658 (2)	42 (1)
C(11)	−515 (3)	−6086 (3)	−3943 (2)	47 (1)
C(12)	−1438 (3)	−5532 (4)	−3490 (3)	61 (1)
C(13)	2260 (2)	−3162 (3)	−2195 (2)	39 (1)
C(14)	1402 (3)	−2504 (3)	−1851 (2)	47 (1)
C(15)	1661 (3)	−1517 (3)	−1179 (2)	51 (1)
C(16)	2774 (3)	−1156 (3)	−856 (2)	43 (1)
C(17)	3630 (3)	−1780 (4)	−1202 (3)	57 (1)
C(18)	3379 (3)	−2789 (4)	−1858 (3)	58 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

Br(1)—C(16)	1·901 (3)	S(1)—C(9)	1·874 (3)
S(1)—C(10)	1·746 (3)	N(1)—N(2)	1·349 (3)
N(1)—C(9)	1·505 (4)	N(1)—C(13)	1·412 (4)
N(2)—C(10)	1·279 (4)	O(1)—C(5)	1·374 (5)
O(1)—C(7)	1·374 (4)	O(2)—C(11)	1·217 (4)
C(1)—C(2)	1·384 (5)	C(1)—C(6)	1·394 (5)
C(2)—C(3)	1·385 (7)	C(3)—C(4)	1·370 (6)
C(4)—C(5)	1·412 (5)	C(5)—C(6)	1·371 (5)
C(6)—C(9)	1·500 (4)	C(7)—C(8)	1·318 (5)
C(8)—C(9)	1·482 (5)	C(10)—C(11)	1·469 (4)
C(11)—C(12)	1·491 (5)	C(13)—C(14)	1·387 (5)
C(13)—C(18)	1·382 (4)	C(14)—C(15)	1·375 (5)
C(15)—C(16)	1·369 (4)	C(16)—C(17)	1·371 (5)
C(17)—C(18)	1·376 (5)		

C(9)—S(1)—C(10)	90·8 (1)	N(2)—N(1)—C(9)	117·8 (2)
N(2)—N(1)—C(13)	117·1 (3)	C(9)—N(1)—C(13)	124·9 (2)
N(1)—N(2)—C(10)	114·2 (3)	C(5)—O(1)—C(7)	116·0 (3)
C(2)—C(1)—C(6)	121·7 (4)	C(1)—C(2)—C(3)	119·4 (4)
C(2)—C(3)—C(4)	120·5 (4)	C(3)—C(4)—C(5)	118·9 (4)
O(1)—C(5)—C(4)	114·4 (3)	O(1)—C(5)—C(6)	123·9 (3)
C(4)—C(5)—C(6)	121·7 (3)	C(1)—C(6)—C(5)	117·7 (3)
C(1)—C(6)—C(9)	122·0 (3)	C(5)—C(6)—C(9)	120·3 (3)
O(1)—C(7)—C(8)	124·5 (3)	C(7)—C(8)—C(9)	122·6 (3)
S(1)—C(9)—N(1)	101·1 (2)	S(1)—C(9)—C(6)	109·7 (2)
N(1)—C(9)—C(6)	112·6 (3)	S(1)—C(9)—C(8)	110·4 (2)
N(1)—C(9)—C(8)	112·3 (2)	C(6)—C(9)—C(8)	110·4 (3)
S(1)—C(10)—N(2)	116·0 (2)	S(1)—C(10)—C(11)	121·8 (2)
N(2)—C(10)—C(11)	122·1 (3)	O(2)—C(11)—C(10)	119·1 (3)
O(2)—C(11)—C(12)	123·2 (3)	C(10)—C(11)—C(12)	117·7 (3)
N(1)—C(13)—C(14)	118·9 (3)	N(1)—C(13)—C(18)	122·5 (3)
C(14)—C(13)—C(18)	118·5 (3)	C(13)—C(14)—C(15)	120·7 (3)
C(14)—C(15)—C(16)	120·0 (3)	Br(1)—C(16)—C(15)	119·6 (3)
Br(1)—C(16)—C(17)	120·4 (2)	C(15)—C(16)—C(17)	120·0 (3)
C(16)—C(17)—C(18)	120·0 (3)	C(13)—C(18)—C(17)	120·5 (3)

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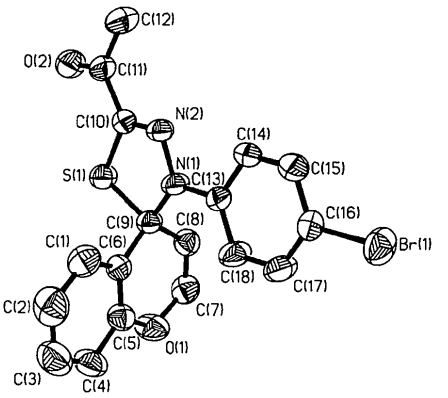

Fig. 1. Molecular structure and numbering scheme for the title compound.

eters. 2983 unique reflections, $R_{int} = 0{\cdot}025$, 2515 with $F > 4\sigma(F)$. At convergence $R = 0{\cdot}040$, $wR = 0{\cdot}049$ (on all data $R = 0{\cdot}049$, $wR = 0{\cdot}050$), $S = 1{\cdot}73$ for 217 parameters, $w = [\sigma^2(F) + 0{\cdot}0004F^2]^{-1}$, $\Delta/\sigma < 0{\cdot}001$, data/parameter ratio 11·6:1, $(\Delta\rho)_{max} = 0{\cdot}26$, $(\Delta\rho)_{min} = -0{\cdot}58$ e Å^{-3}. Scattering factors for all atoms from *SHELXTL-Plus* (Sheldrick, 1988).

Atomic parameters are given in Table 1,* bond distances and angles in Table 2, and Fig. 1 shows the molecule together with the atomic numbering scheme used.

Related literature. The cycloaddition reaction of chromone and diphenylnitrilimine resulted in the formation of the corresponding oxadiazole (Shawali, Eltawil & Albar, 1984).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52648 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9-Acridineethanol

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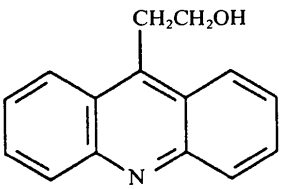
Abstract. $C_{15}H_{13}NO$, $M_r = 223{\cdot}28$, triclinic, $P\bar{1}$, $a = 7{\cdot}537$ (4), $b = 9{\cdot}283$ (5), $c = 16{\cdot}408$ (4) Å, $\alpha = 87{\cdot}50$ (3), $\beta = 83{\cdot}44$ (3), $\gamma = 79{\cdot}04$ (3)°, $V = 1119$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}325$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}78$ cm^{-1}, $F(000) = 472$, $T = 293$ K, $R = 0{\cdot}039$ for 1697 observed reflections. The two molecules per asymmetric unit are in virtually the same conformation. The acridine ring has the expected structure, but the side chain is in the *gauche* conformation. The acridine rings are stacked in pairs held together by hydrogen bonds involving the alcohol functionality and the nitrogen of the acridine ring.

Experimental. Crystals of the synthetic compound were prepared by reacting 9-methylacridine with formaldehyde following published procedures (Eisleb, 1936). Crystal: 0·59 × 0·15 × 0·14 mm, amber needle from ethyl acetate. Data collected using ω–2θ scan techniques; scan rate varied from 2 to 20° min^{-1} (in ω); 2915 intensities recorded on an



Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $2\theta_{max} = 45°$, $-7 \geq h \geq 8$, $-9 \geq k \geq 9$, $0 \geq l \geq 17$. Three check reflections measured every 97 reflections, 2% intensity

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