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9-(1,3-Dithian-2-yl)-10-oxa-1-azatricyclo-[7.4.0.0^{3,8}]trideca-3,5,7-trien-2-one

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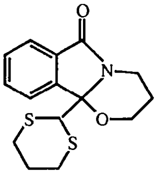
Abstract

The title compound, $C_{15}H_{17}NO_2S_2$, was produced during attempts to synthesize new serotonergic agents based upon the NAN-190 compounds [Dawson, Lawrence, Wilson, Olverman & Kelly (1991). *Br. J. Pharmacol.* **102**, 244P]. While the geometry around the N atom shows some distortion from ideal tetrahedral geometry, the remaining bond lengths and angles are well within the expected limits.

Comment

The title compound (I) was prepared by reacting *N*-(3-bromopropyl)phthalimide with one equivalent of lithiated 1,3-dithiane. The synthesis of *N*-(3-dithianylpropyl)phthalimide was attempted in order to use it as a synthon in the Fischer indole synthesis of a novel serotonergic agent similar to the NAN-190 compounds (Glennon, Naiman, Lyon & Titeler, 1988; Glennon, Naiman, Poerson, Titeler, Lyon & Weisberg, 1988) but with an S atom in the ring system rather than an N atom. Addition of the dithiane anion to the carboxyl group of the phthalimide group occurred preferentially under the conditions used (refluxing in THF), with concomitant intramolecular O-alkylation by the bromopropyl group to give the title compound.



(I)

In the crystal, the geometry of the group of atoms C(7), C(8) and C(11)–C(14) conforms to that of a near perfect benzene ring. The angles at the N atom are somewhat distorted from true tetrahedral values, which is probably due to the strain caused by cyclizing atoms C(2), C(3), C(4) and O(5). The molecules form stacks parallel to the *b* axis with a herringbone pattern typical of aromatic systems.

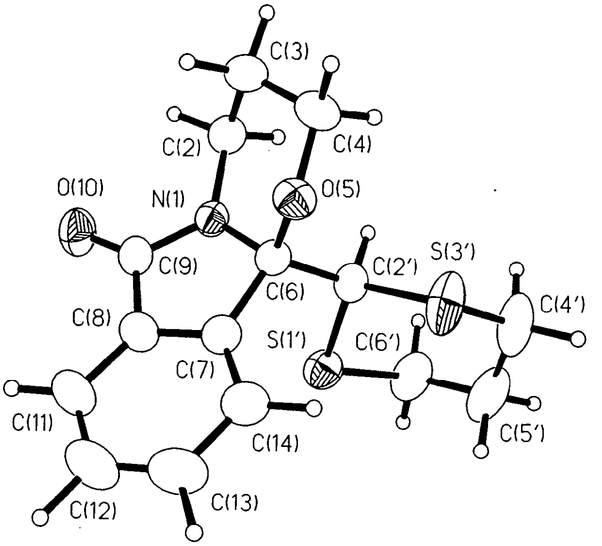


Fig. 1. A general view of the molecule showing the numbering scheme with the H atoms numbered according to the atoms to which they are attached. Displacement ellipsoids of the non-H atoms are drawn at the 50% probability level and H-atom radii are 0.15 Å.

Experimental

Crystal data

$C_{15}H_{17}NO_2S_2$
$M_r = 306.42$
Monoclinic
$P2_1/c$
$a = 12.2411\ (15)$ Å
$b = 8.0563\ (9)$ Å
$c = 15.3544\ (20)$ Å
$\beta = 98.997\ (13)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 15–16°$
$\mu = 0.341\ \text{mm}^{-1}$
$T = 293$ K
Prism

$V = 1495.6$ (2) Å^3
$Z = 4$
$D_x = 1.365$ Mg m^{-3}
0.80 × 0.55 × 0.45 mm
Colourless
Crystal source: grown from ethyl acetate/hexane solution

Data collection

Siemens Stoe AED-2 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{\min} = 0.481$, $T_{\max} = 0.622$
2130 measured reflections
1913 independent reflections
1766 observed reflections $[|F_o| > 4\sigma(F_o)]$
$\theta_{\max} = 22.5°$
$h = -11 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 16$
4 standard reflections
frequency: 120 min
intensity variation: 0.36%

Refinement

Refinement on F
$R = 0.0350$
$wR = 0.0498$
$S = 1.303$
1766 reflections
181 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.000060F^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.257$ e Å^{-3}
$\Delta\rho_{\min} = -0.279$ 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)

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

	x	y	z	$U_{\rm eq}$
N(1)	0.10402 (15)	−0.06385 (24)	0.28621 (12)	0.0340 (11)
C(2)	−0.00601 (19)	−0.0593 (3)	0.31189 (17)	0.0440 (15)
C(3)	−0.00961 (22)	−0.1888 (4)	0.38304 (18)	0.0516 (17)
C(4)	0.08798 (22)	−0.1679 (4)	0.45593 (17)	0.0505 (17)
O(5)	0.19167 (14)	−0.17680 (20)	0.42223 (10)	0.0398 (10)
C(6)	0.20147 (18)	−0.0593 (3)	0.35495 (14)	0.0329 (13)
C(7)	0.29152 (19)	−0.1184 (3)	0.30553 (15)	0.0353 (14)
C(8)	0.24594 (20)	−0.1561 (3)	0.21934 (16)	0.0384 (15)
C(9)	0.12484 (20)	−0.1238 (3)	0.20782 (16)	0.0365 (14)
O(10)	0.05559 (15)	−0.14692 (24)	0.14251 (11)	0.0524 (12)
C(11)	0.30853 (24)	−0.2214 (3)	0.16014 (18)	0.0519 (17)
C(12)	0.4204 (3)	−0.2447 (4)	0.18893 (21)	0.0632 (21)
C(13)	0.46653 (24)	−0.2046 (4)	0.27443 (22)	0.0613 (20)
C(14)	0.40296 (20)	−0.1416 (3)	0.33475 (18)	0.0476 (17)
S(1′)	0.26498 (6)	0.24926 (8)	0.30941 (4)	0.0431 (4)
C(2′)	0.21661 (20)	0.1200 (3)	0.39186 (15)	0.0352 (14)
S(3′)	0.30770 (7)	0.12121 (9)	0.49747 (5)	0.0596 (5)
C(4′)	0.3015 (3)	0.3420 (4)	0.52080 (20)	0.0675 (21)
C(5′)	0.3399 (3)	0.4539 (4)	0.45272 (21)	0.0647 (20)
C(6′)	0.26530 (24)	0.4483 (3)	0.36402 (19)	0.0551 (18)

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

N(1)—C(6)	1.464 (3)	C(8)—C(7)	1.387 (3)
N(1)—C(9)	1.358 (3)	C(9)—C(8)	1.488 (3)
C(2)—N(1)	1.462 (3)	C(9)—O(10)	1.222 (3)
C(4)—O(5)	1.446 (3)	C(2′)—S(1′)	1.809 (2)
C(6)—O(5)	1.420 (3)	C(2′)—S(3′)	1.819 (3)
C(6)—C(2′)	1.552 (3)	S(3′)—C(4′)	1.819 (3)
C(7)—C(6)	1.510 (3)	C(6′)—S(1′)	1.809 (3)
C(2)—N(1)—C(9)	123.83 (20)	C(14)—C(7)—C(6)	129.90 (21)
C(2)—N(1)—C(6)	119.08 (18)	C(9)—C(8)—C(11)	129.28 (23)
C(9)—N(1)—C(6)	113.78 (18)	C(2′)—S(1′)—C(6′)	99.39 (12)
C(4)—O(5)—C(6)	113.96 (18)	S(3′)—C(2′)—S(1′)	113.50 (13)
N(1)—C(6)—O(5)	110.82 (17)	C(2′)—S(3′)—C(4′)	98.18 (13)
C(2)—N(1)—C(6)—C(2′)	−74.78 (24)		
C(9)—N(1)—C(6)—O(5)	−111.48 (21)		
C(9)—N(1)—C(6)—C(7)	3.06 (24)		
C(9)—N(1)—C(6)—C(2′)	124.96 (20)		
N(1)—C(6)—C(2′)—S(3′)	162.71 (15)		
O(5)—C(6)—C(2′)—S(3′)	39.81 (22)		
O(5)—C(6)—C(2′)—S(1′)	164.77 (15)		

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990). Refinement was carried out with *SHELX*76 (Sheldrick, 1976). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985). The atomic numbering scheme is shown in Fig. 1 which was produced by *XP* (Sheldrick, 1989).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: HA1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,5-Bis(methylthio)-7,7′,8,8′-tetracyanoquinodimethane

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Abstract

The title compound 2,2′-[2,5-bis(methylthio)-2,5-cyclohexadiene-1,4-diylidene]bis(propanedinitrile),