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erythro-1-(Benzothiazol-2-yl)-1,2-dibromo-2-(2-chloro-5-nitrophenyl)ethane†

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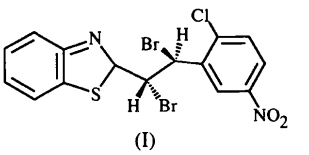
Abstract

The title compound, $C_{15}H_9Br_2ClN_2O_2S$, was synthesized by electrophilic addition of bromine to 2-(2-chloro-5-nitrostyryl)benzothiazole. The molecule consists of benzothiazole and 2-chloro-5-nitrophenyl rings linked by a 1,2-dibromoethane moiety. The dihedral angle between the benzothiazole and phenyl rings is 8.2 (9)°. The benzothiazole ring is planar with a mean deviation of 0.168 (7) Å. The Br1—C8 and Br2—C9 bond distances are 1.972 (5) and 2.007 (6) Å, respectively.

Comment

We have been interested in utilizing 2-styrylbenzazols and their oxo and thio derivatives as potential ligands in the synthesis of new platinum-based anticancer agents (Cox *et al.*, 1982; Gòmez *et al.*, 1988; Muir *et al.*, 1987, 1988, 1992*a,b,c,d*, 1993; Muir, Cox, Rivera *et al.*, 1992). As part of our continuing work on the benzothiazole series, we studied the electrophilic addition of bromine to 2-(2-chloro-5-nitrostyryl)benzothiazole. Such a reaction produced the corresponding dibromide in excellent yield. The proton–proton coupling constant (J = 11.1 Hz) in the NMR spectrum indicates that the compound is an *erythro* isomer. In order to confirm the identity of this compound, (I), a single-crystal X-ray structure analysis was carried out.

† Alternative name: 2-[*erythro*-1,2-dibromo-2-(2-chloro-5-nitrophenyl)-ethyl]-1,3-benzothiazole.



(I)

An *ORTEP*II (Johnson, 1976) representation of the title compound is presented in Fig. 1. The molecular structure of this compound features a benzothiazole and a 2-chloro-5-nitrophenyl ring linked by a 1,2-dibromoethane moiety that is in an *anti* conformation. The dihedral angle between the two aromatic rings is 8.2 (9)°. The C—N and S—C bond distances in the benzothiazole ring are similar to those found in other similar structures (Alegría *et al.*, 1993). In summary, the X-ray structure of the title compound confirms that the electrophilic addition of bromine to (*E*)-2-(2-chloro-5-nitrostyryl)benzothiazole proceeds by a stereoselective *anti* process to produce the racemic *erythro*-dibromide. Furthermore, the conformation observed in the solid state is also the most stable conformation in solution as evidenced by the proton–proton coupling constant.

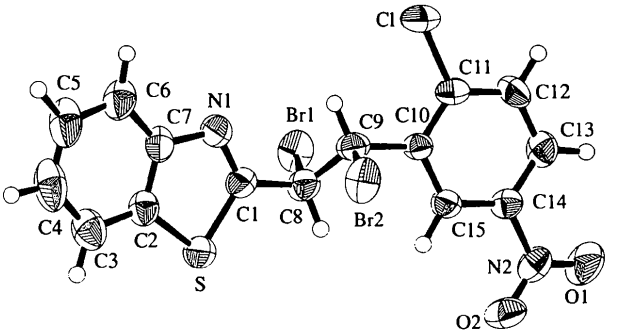


Fig. 1. An *ORTEP*II (Johnson, 1976) representation of the title compound showing 50% probability displacement ellipsoids.

Experimental

A solution of (*E*)-2-(2-chloro-5-nitrostyryl)benzothiazole (8.0 g, 25.0 mmol) in 200 ml of a benzene–dioxane (3:1) mixture was allowed to react with bromine (8.0 g, 50.0 mmol) to afford 9.5 g (79% yield) of the title compound as a pale yellow solid (m.p. 411–413 K). Single crystals suitable for X-ray diffraction studies were obtained from acetonitrile by slow evaporation of the solvent.

Crystal data

$C_{15}H_9Br_2ClN_2O_2S$
M_r = 476.57
Monoclinic
$P2_1/c$
a = 10.557 (4) Å
b = 14.518 (5) Å
c = 11.094 (3) Å
β = 104.51 (2)°
V = 1646.1 (9) Å^3
Z = 4
D_x = 1.923 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 12–13°
μ = 5.237 mm^{-1}
T = 295 (1) K
Prism
0.66 × 0.54 × 0.51 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer	2300 reflections with $I > 2.5\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.028$
Absorption correction: empirical ψ scan (North, Phillips & Mathews, 1968)	$\theta_{max} = 24.97°$
	$h = 0 \rightarrow 12$
	$k = 0 \rightarrow 17$
$T_{min} = 0.046$, $T_{max} = 0.069$	$l = -13 \rightarrow 12$
3202 measured reflections	3 standard reflections
3187 independent reflections	every 200 reflections
	intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.0034$
$R = 0.053$	$\Delta\rho_{max} = 1.41$ e Å^{-3}
$wR = 0.061$	$\Delta\rho_{min} = -0.69$ e Å^{-3}
$S = 1.905$	Extinction correction: none
2300 reflections	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
208 parameters	
H atoms not refined	
Weighting scheme based on measured s.u.'s; $w = 1/[\sigma_c^2(F_o) + (p^2/4F_o^2)]$, with $p = 0.03$	

Table 1. *Selected geometric parameters (Å, °)*

Br(1)—C(8)	1.972 (5)	N(1)—C(1)	1.306 (7)
Br(2)—C(9)	2.007 (6)	N(1)—C(7)	1.385 (7)
Cl—C(11)	1.736 (5)	C(1)—C(8)	1.492 (7)
S—C(1)	1.729 (5)	C(8)—C(9)	1.476 (8)
S—C(2)	1.711 (5)	C(9)—C(10)	1.504 (7)
C(1)—S—C(2)	89.5 (3)	Br(1)—C(8)—C(1)	110.4 (4)
C(1)—N(1)—C(7)	109.9 (4)	Br(1)—C(8)—C(9)	106.2 (4)
S—C(1)—N(1)	116.2 (4)	C(1)—C(8)—C(9)	113.2 (5)
S—C(1)—C(8)	120.4 (4)	Br(2)—C(9)—C(8)	107.0 (4)
N(1)—C(1)—C(8)	123.3 (5)	Br(2)—C(9)—C(10)	106.7 (4)
S—C(2)—C(3)	129.2 (5)	C(8)—C(9)—C(10)	117.0 (5)
S—C(2)—C(7)	109.4 (4)	C(9)—C(10)—C(11)	122.2 (5)
N(1)—C(7)—C(2)	114.9 (5)	C(9)—C(10)—C(15)	120.5 (4)
N(1)—C(7)—C(6)	125.2 (5)		

The largest peak in the final difference Fourier map (1.41 e Å^{-3}) is 0.39 Å from the Br(2) atom.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1024). Services for accessing these data are described at the back of the journal.

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N-(*o*-Tolyl)-2-bromo-4,5-dimethoxybenzamide†

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Abstract

The molecular structure of the title compound, $C_{16}H_{16}BrNO_3$, consists of 2-bromo-4,5-dimethoxybenzene and *o*-methylphenyl rings linked by an amide group. The two methoxy groups are almost coplanar with the phenyl ring.

Comment

A number of antipsychotic agents of the *o*-methoxybenzamide type show interesting selectivity for dopamine–D2 receptors (Hogberg, 1991). Benzamides with an *N*-(*cis*-2-phenylcycloheptyl) or an *N*-ethyl-2-(pyrrolidine)methyl side chain and a series of

† Alternative name: 2-bromo-4,5-dimethoxy-2′-methylbenzanilide.