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Structure of a Twisted Styrylbenzothiazole, 2-(α-Acetoxy-2-chloro-5-nitrostyryl)-benzothiazole

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Abstract. α-(1,3-Benzothiazol-2-yl)-2-chloro-5-nitro-styryl α-acetate (ac-nsb), $C_{17}H_{11}ClN_2O_4S$, $M_r = 374.81$, triclinic, $P\bar{1}$, $a = 9.762$ (4), $b = 10.689$ (5), $c = 8.387$ (3) Å, $\alpha = 96.97$ (4), $\beta = 102.26$ (3), $\gamma = 76.74$ (4)°, $V = 830$ (1) Å^3, $Z = 2$, $D_x = 1.500$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 3.72$ cm^{-1}, $F(000) = 384$, $T = 298$ K, final $R = 0.047$ for 2463 unique observed reflections. The molecule is distinctly non-planar, with the benzothiazole and phenyl rings rotated with respect to the olefin to give a dihedral angle of 51.1 (1)° between the planes of those rings.

Experimental. The benzothiazole derivative ac-nsb (I) ($X = S$) was prepared as part of a study of substitution in the azole ring ($X = O$, S, Se and NR, $R = C_2H_5$). It was synthesized by refluxing 2-hydroxymethylbenzothiazole and 2-chloro-5-nitro-benzaldehyde in acetic anhydride. The solid product, m.p. 428–430 K, formed on vacuum evaporation. Crystals were obtained by slow evaporation from hexane. Analysis for $C_{17}H_{11}ClN_2O_4S$: calculated C 55.48, H 2.96, N 7.47%; found C 54.43, H 2.97, N 7.47%.
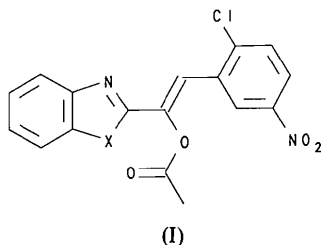
Light-brown plate (D_m not determined), 0.30 × 0.25 × 0.20 mm, used for data collection on an Enraf–Nonius CAD-4 diffractometer with monochromated Mo $K\alpha$ radiation, ω–2θ scan, scan width $(0.9 + 0.34\tan\theta)°$, $2\theta_{max} = 53°$. Lattice parameters were determined from least-squares fit of 24 reflections in the range $10 < \theta < 15°$. No systematic absences; $-h$, $\pm k$, $\pm l$, $-12 \le h \le 0$, $-13 \le k \le 13$, $-10 \le l \le 10$. Three standard reflections ($\bar{4}14$, 422 and $2\bar{4}1$) showed total intensity loss of 1.1% during 51.1 h of exposure. $R_{int} = 0.02$. Total number of reflections measured was 5094, 2463 of these being unique with $F^2 > 3\sigma^2(F_o)$, $\sigma(F_o) = [\sigma^2(I_{raw}) + (0.04F_o^2)^2]^{1/2}/2F_o$. The structure was solved with the Enraf–Nonius *Sructure Determination Package* (Frenz, 1986), heavy-atom and difference Fourier methods. Isotropic then anisotropic refinement was performed on F of all non-H atoms, H atoms were placed at calculated positions using a riding model [C—H 0.95 Å, U(H) 1.3U(C)]. 227 parameters were refined. $R = 0.047$, $wR = 0.060$, $w = 1/[\sigma^2(F_o)]$, $S = 1.501$; difference syntheses showed no densities above 0.37 e Å^{-3} (minimum -0.39 e Å^{-3}); maximum $\Delta/\sigma = 0.01$. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B, 2.3.1), and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No absorption correction was applied. An extinction parameter which refined to 1.10 (1) × 10^{-5} was applied and the calculated structure factor became $F_c = 1/(1 + xI_c)$ (maximum correction 55 for the 012 reflection).

(I)

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* ($\mathring{A}^2$)

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Cl1	0.19749 (8)	1.03661 (8)	0.7570 (1)	5.71 (2)
S1	0.16563 (7)	0.44966 (6)	0.89707 (8)	3.48 (1)
O1	−0.0628 (2)	0.6719 (2)	0.7878 (2)	3.29 (4)
O2	0.0108 (2)	0.6539 (2)	0.5487 (2)	4.70 (4)
O3	−0.5051 (3)	1.1484 (3)	0.6192 (4)	8.65 (8)
O4	−0.4589 (3)	1.0034 (3)	0.7877 (4)	8.91 (8)
N1	0.2766 (2)	0.6346 (2)	1.0657 (3)	3.44 (5)
N2	−0.4220 (3)	1.0697 (3)	0.7041 (4)	5.65 (7)
C1	0.1706 (2)	0.6117 (2)	0.9531 (3)	3.14 (5)
C2	0.3233 (2)	0.4078 (2)	1.0372 (3)	3.23 (5)
C3	0.4027 (3)	0.2866 (3)	1.0758 (3)	4.14 (6)
C4	0.5247 (3)	0.2793 (3)	1.1951 (4)	4.73 (7)
C5	0.5669 (3)	0.3901 (3)	1.2743 (4)	4.63 (7)
C6	0.4892 (3)	0.5104 (3)	1.2374 (3)	4.11 (6)
C7	0.3652 (3)	0.5205 (2)	1.1163 (3)	3.25 (5)
C8	0.0618 (2)	0.7126 (2)	0.8701 (3)	3.13 (5)
C9	0.0770 (3)	0.8330 (3)	0.8728 (3)	3.52 (6)
C10	−0.0272 (3)	0.9404 (2)	0.7948 (3)	3.31 (5)
C11	0.0167 (3)	1.0391 (3)	0.7371 (3)	3.88 (6)
C12	−0.0794 (3)	1.1407 (3)	0.6626 (4)	4.66 (7)
C13	−0.2238 (3)	1.1496 (3)	0.6466 (4)	4.55 (7)
C14	−0.2683 (3)	1.0558 (3)	0.7085 (3)	3.89 (6)
C15	−0.1746 (3)	0.9526 (3)	0.7809 (3)	3.65 (6)
C16	−0.0803 (3)	0.6493 (2)	0.6209 (3)	3.28 (5)
C17	−0.2230 (3)	0.6182 (3)	0.5535 (4)	5.11 (8)

Table 2. *Selected bond distances* ($\mathring{A}$) *and angles* (°)

Cl1—C11	1.732 (3)	C1—C8	1.455 (3)
S1—C1	1.748 (3)	C9—C10	1.464 (3)
S1—C2	1.731 (2)	C8—C9	1.326 (4)
O1—C8	1.400 (3)	N2—C14	1.466 (4)
O1—C16	1.372 (3)	N—O (av.)	1.210 (4)
O2—C16	1.189 (4)	N1—C1	1.291 (3)
C16—C17	1.485 (4)	N1—C7	1.380 (3)
C—C (av. benzo)	1.39 (3)	C—C (av. phenyl)	1.381 (9)
C1—S1—C2	88.7 (1)	O1—C8—C1	113.9 (2)
C1—N1—C7	110.2 (2)	O1—C8—C9	122.4 (2)
S1—C1—N1	116.5 (2)	O1—C16—C17	109.8 (2)
S1—C1—C8	120.1 (2)	O2—C16—C17	128.2 (2)
N1—C1—C8	123.3 (2)	O3—N2—O4	123.5 (3)
S1—C2—C7	109.2 (2)	O1—C16—O2	122.0 (2)
N1—C7—C2	115.4 (2)	C14—N2—O (av.)	118.2 (3)
C1—C8—C9	123.7 (2)	C—C—C (av. phenyl)	120.0 (6)
C8—C9—C10	127.2 (2)	C—C—C (av. benzo)	120.0 (5)

The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, selected bond lengths and angles in Table 2.* An *ORTEP*II plot (Johnson, 1976) of the complex with the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell & Clegg, 1976) in Fig. 2.

Related literature. Derivatives of 2-styrylbenzazoles have been studied owing to their importance as

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* Lists of structure factors, anisotropic thermal factors, H-atom parameters, bond distances and angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55024 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0564]

precursors to the biologically active benzazolo[3,2-*a*]-quinolinium salts (Cox, Jackson, Vargas, Báez, Colón, González & de León, 1982). Derivatives include S, O, Se and N analogues. Moreover, two platinum(II) complexes of the type [NEt$_4$][Pt(L)Br$_3$], L = 2-(2-hydroxystyryl)benzothiazole and 2-(2-chloro-5-chloro-5-nitrostyryl)benzothiazole, respectively, show good cytotoxic activity against CHO-K cells *in vitro* (Muir, Cádiz & Báez, 1988). The most notable feature of the present structure is the 51.1° dihedral angle between the planes of the benzothiazole and the phenyl rings. Other benzoheterazoles have nearly planar geometry with 6.8° for 2-(2-chloro-5-nitrostyryl)benzoxazole (nsbo) (Muir, Cox, Bernard & Muir, 1992*a*), and 4.8° for 2-(2-chloro-5-nitrostyryl)benzoselenazole (nsbse) (Muir, Cox, Bernard & Muir, 1992*b*). The acetoxy substituent on C$_8$ in ac-nsb is probably too bulky to permit the rings to be coplanar, as shown by molecular modeling. The observed twisting permits stacking of pairs of benzothiazole rings (oriented in alternate directions) with a distance of 3.42 (1) Å between the planes. This is similar to the 3.53 (1) Å distance between the phenyl rings in the [Pt(nsb)Br$_3$]$^-$ complex [nsb = 2-(2-chloro-5-nitrostyryl)benzothiazole] (Muir, Gómez, Muir, Cox & Cádiz, 1987). The dihedral angle in nsb in that complex is 60.1 (4)°. The average C—N, C—S and C—C distances and bond angles in (I) are similar to those found in the other compounds.
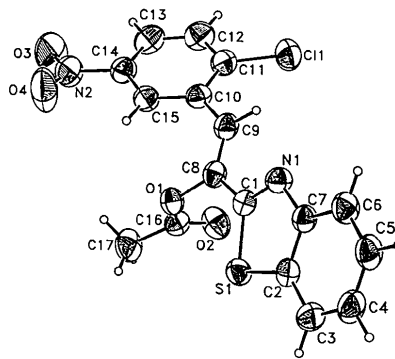

Fig. 1. Labeling of atoms in the title compound (50% thermal ellipsoids).
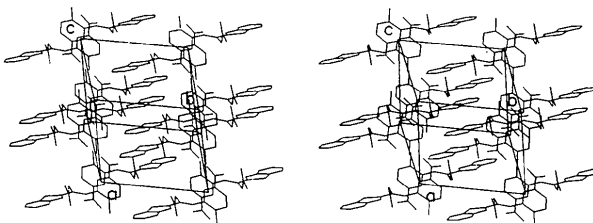

Fig. 2. Packing diagram of the title compound in the unit cell.

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Structure of 3-Nitrato-1-nitroazetidine

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Abstract. $C_3H_5N_3O_5$, $M_r = 163.1$, tetragonal, $P4_1$, $a = 10.646\,(1)$, $c = 5.752\,(2)$ Å, $V = 651.9\,(2)$ Å^3, $Z = 4$, $D_x = 1.662$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.15$ mm^{-1}, $F(000) = 336$, $T = 200$ K, final $R = 0.031$, $wR = 0.030$ for 415 independent observed reflections. The four-membered ring is significantly puckered, with an angle between the C—C—C and C—N—C planes of 12.7°. The amino atom N1 is pyramidal as seen by the 'out-of-plane-bend' angle between the N—N bond and the C—N—C plane, which is 39.5°. The nitro group itself shows no twist relative to the azetidine ring (average value of the four torsion angles about the N1—N1a bond is 0.5°).

Experimental. A clear colorless prism, $0.15 \times 0.15 \times 0.35$ mm, data crystal was provided by Dr Kurt Baum of Fluorochem, Inc., Azusa, CA. An automated Siemens $R3m/V$ diffractometer with incident-beam monochromator was used for data collection. 24 centered reflections within $13 \le 2\theta \le 35°$ were used for determining lattice parameters. $(\sin\theta/\lambda)_{max} = 0.54$ Å^{-1}, range of hkl: $0 \le h \le 11$, $0 \le k \le 11$, $-6 \le l \le 4$. Standards 500, 050, 004, monitored every 97 reflections, showed random variation of 3.0% over data collection: $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.1]$ to $[2\theta(K\alpha_2) + 1.1]°$, ω-scan rate a function of count rate (minimum 8, maximum 15.0° min^{-1}). 544 reflections were measured, of which 482 were unique

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

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N1	5029 (3)	1861 (3)	4851	24 (1)
N1a	4262 (3)	2477 (4)	6353 (10)	28 (1)
O1a	4073 (3)	1982 (3)	8262 (9)	30 (1)
O1b	3785 (3)	3467 (3)	5657 (9)	38 (1)
C2	5802 (4)	2568 (5)	3140 (11)	26 (2)
C3	6853 (4)	1611 (4)	3617 (11)	23 (2)
O3a	6922 (3)	813 (3)	1585 (9)	27 (1)
N3a	7812 (4)	−169 (4)	1873 (12)	30 (2)
O3b	7925 (3)	−787 (3)	131 (9)	44 (1)
O3c	8305 (3)	−275 (3)	3741 (9)	36 (1)
C4	6093 (5)	1055 (5)	5641 (11)	26 (2)

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

N1—N1a	1.358 (5)	N1—C2	1.488 (6)
N1—C4	1.491 (6)	N1a—O1a	1.234 (7)
N1a—O1b	1.237 (5)	C2—C3	1.538 (7)
C3—O3a	1.447 (7)	C3—C4	1.537 (8)
O3a—N3a	1.420 (5)	N3a—O3b	1.205 (8)
N3a—O3c	1.202 (8)		

N1a—N1—C2	120.6 (4)	N1a—N1—C4	122.7 (3)
C2—N1—C4	94.2 (3)	N1—N1a—O1a	117.2 (4)
N1—N1a—O1b	116.9 (5)	O1a—N1a—O1b	125.8 (5)
N1—C2—C3	87.1 (4)	C2—C3—O3a	106.4 (4)
C2—C3—C4	90.4 (4)	O3a—C3—C4	114.3 (4)
C3—O3a—N3a	111.8 (4)	O3a—N3a—O3b	111.9 (5)
O3a—N3a—O3c	117.7 (5)	O3b—N3a—O3c	130.4 (4)
N1—C4—C3	87.0 (4)		