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2,3-Dichloro-1-benzothiophene

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Abstract. $C_8H_4Cl_2S$, $M_r = 203{\cdot}1$, monoclinic, $P2_1/c$, $a = 3{\cdot}89$ (1), $b = 14{\cdot}97$ (2), $c = 14{\cdot}21$ (4) Å, $\beta = 92{\cdot}0$ (2)°, $V = 827{\cdot}0$ Å^3, $Z = 4$, $D_m = 1{\cdot}63$, $D_x = 1{\cdot}63$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 8{\cdot}54$ mm^{-1}, $F(000) = 408$, $T = 283$ K, $R = 0{\cdot}094$ for 1054 observed visually measured equi-inclination Weissenberg data. The five- and six-membered rings are planar and inclined to one another at 1·04 (13)°. The average C–C bond length in the phenyl ring is 1·386 (6) Å. In the five-membered ring the average C–S bond length is 1·762 (7) Å, the C–S–C angle is 90·0 (5)°; the C–C bond lengths are 1·341 (14) and 1·418 (13) Å. The average C–Cl bond length is 1·727 (13) Å. The Cl atoms are displaced on either side of the five-membered rings with the Cl(1)–C(7) and Cl(2)–C(8) bonds making angles of 0·5 (9) and 1·0 (9)°, respectively, with the ring plane.

Introduction. This paper forms part of an investigation into 1-benzothiophene derivatives. These frequently discolour on exposure to the atmosphere and sublime quite rapidly under X-ray irradiation.

Experimental. D_m measured by flotation using aqueous cadmium *n*-dodecatungstoborate. To keep the material as long as possible the selected crystals together with some additional material were sealed in Lindemann-glass capillaries. White transparent crystals used in data collection, dimensions 0·27 × 0·09 × 0·11 and 0·16 × 0·1 × 0·16 mm for *a*-axis $h = 0$–3 and *c*-axis *hk*0 Weissenberg data, respectively. 1087 reflections measured by visual estimation from multiple-film photographs, Cu $K\alpha$ radiation, $-3 \le h \le 3$, $0 \le k \le 18$, $0 \le l \le 17$. Data merged to give 1054 unique observed reflections; $R_{int} = 0{\cdot}06$. Absorption ignored. Structure solved by Patterson synthesis and refined (on *F*) by blocked-matrix least squares with anisotropic thermal parameters for non-H atoms. H-atom positions, initially obtained from difference synthesis and placed at geometrically reasonable positions, refined with constrained C–H bond distances and isotropic thermal parameters. Four reflections (120, $\bar{1}41$, $\bar{1}53$, 102) omitted from refinement; final $R = 0{\cdot}094$, unit weights used. The high *R* factor is mainly due to the crystal quality and degradation during data collection. $(\Delta/\sigma)_{max}$ in final refinement cycle 0·04 for positional and 0·06 for thermal parameters. Max. and min. heights in final $\Delta\rho$ map +0·7 and −0·5 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs used: *SHELX*76 (Sheldrick, 1976) and local programs supplied by HHS and Drs C. Morgan and M. J. Mottram.

Discussion. Table 1* gives atomic parameters and Table 2 bond lengths and angles. The atomic numbering is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, mean-plane calculations and intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42720 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	*y*	*z*	U_{eq}
Cl(1)	1007 (9)	1115 (2)	287 (2)	74 (2)
Cl(2)	445 (8)	3203 (2)	−546 (2)	67 (2)
S(1)	−1723 (8)	1908 (2)	2039 (2)	61 (2)
C(1)	−2059 (25)	3487 (6)	1237 (7)	50 (6)
C(2)	−2707 (30)	4406 (6)	1144 (6)	63 (7)
C(3)	−4070 (32)	4840 (5)	1891 (9)	72 (8)
C(4)	−4756 (30)	4404 (7)	2732 (7)	69 (7)
C(5)	−4097 (29)	3506 (7)	2828 (6)	61 (7)
C(6)	−2753 (28)	3056 (6)	2082 (7)	54 (6)
C(7)	−478 (28)	2032 (6)	869 (7)	56 (6)
C(8)	−694 (26)	2883 (7)	579 (6)	53 (6)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Cl(1)–C(7)	1·714 (10)	C(5)–C(6)	1·375 (13)
Cl(2)–C(8)	1·740 (9)	C(6)–C(1)	1·398 (13)
C(1)–C(2)	1·403 (13)	C(6)–S(1)	1·765 (10)
C(2)–C(3)	1·368 (15)	S(1)–C(7)	1·758 (10)
C(3)–C(4)	1·396 (15)	C(7)–C(8)	1·341 (14)
C(4)–C(5)	1·374 (14)	C(8)–C(1)	1·418 (13)
Cl(1)–C(7)–S(1)	118·7 (6)	C(7)–S(1)–C(6)	90·0 (5)
Cl(1)–C(7)–C(8)	129·2 (8)	S(1)–C(6)–C(1)	111·7 (7)
C(8)–C(7)–S(1)	112·0 (7)	S(1)–C(6)–C(5)	126·6 (7)
Cl(2)–C(8)–C(7)	121·9 (8)	C(5)–C(6)–C(1)	121·7 (9)
Cl(2)–C(8)–C(1)	122·9 (7)	C(6)–C(5)–C(4)	118·5 (8)
C(1)–C(8)–C(7)	115·1 (9)	C(5)–C(4)–C(3)	120·2 (9)
C(8)–C(1)–C(6)	111·1 (8)	C(4)–C(3)–C(2)	122·2 (8)
C(8)–C(1)–C(2)	129·3 (9)	C(3)–C(2)–C(1)	117·8 (8)
C(2)–C(1)–C(6)	119·7 (9)		

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The individual five- and six-membered rings are planar but the molecule as a whole shows a small deviation from planarity, the rings being inclined at 1·04 (13)° to one another. An angle of 0·6° was determined in 5-bromo-2,3-dimethyl-1-benzothiophene (Hogg & Sutherland, 1974) and in 2-methyl-1-benzothiophene (Sutherland & Rawas, 1985). Cl(1) is displaced by −0·014 (9) Å and Cl(2) by 0·030 (9) Å from the five-membered ring with the Cl(1)—C(7) and Cl(2)—C(8) bonds inclined at −0·5 (9) and 1·0 (9)°, respectively, to the plane of the ring. The C—S—C bond angle of 90·0 (5)° is in good agreement with the values of 91·1 (12)° found in 2,3-dibromo-1-benzothiophene (Sutherland & Ali-Adib, 1986) and 92·4 (7)° in 2-methyl-1-benzothiophene. The C(1)—C(8) bond of 1·418 (13) Å is shorter than the corresponding values of 1·441 (1), 1·485 (17) and 1·446 (12) Å in dibenzothiophene (Schaffrin & Trotter, 1970), 2-methyl-1-benzothiophene and 5-bromo-2,3-dimethyl-1-benzothiophene, respectively. The S—C(7) bond of 1·758 (10) Å is in good agreement with the value of 1·754 (10) Å for the corresponding bond in 5-bromo-2,3-dimethyl-1-benzothiophene. The S—C(6) bond of 1·765 (10) Å is similar to the value of 1·772 (16) Å found in 2-methyl-1-benzothiophene. The C(7)—C(8) bond of 1·341 (14) Å is shorter than the corresponding values of 1·355 (14), 1·369 and 1·382 (16) Å in 5-bromo-2,3-dimethyl-1-benzothiophene, thiophene (Harshbarger & Bauer, 1970) and 2-methyl-1-benzothiophene, respectively.

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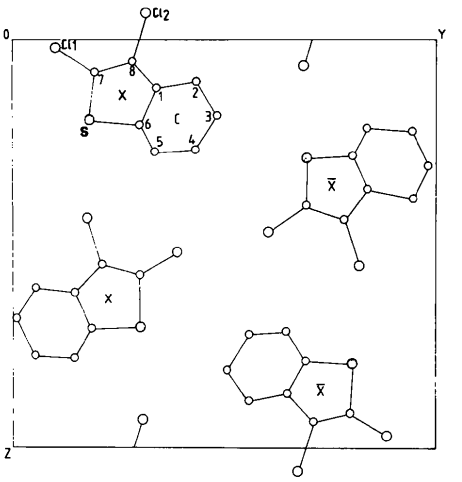

Fig. 1. The arrangement of the molecules in the unit cell viewed along **a**.

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Structure of 2′,3′,5′-Tri-*O*-acetyladenosine

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Abstract. $C_{16}H_{19}N_5O_7$, $M_r = 393{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}46$ (2), $b = 20{\cdot}26$ (3), $c = 8{\cdot}42$ (1) Å, $U = 1955$ Å^3, $Z = 4$, $D_x = 1{\cdot}34$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}68$ cm^{-1}, $F(000) = 824$, $T = 293$ K, $R = 0{\cdot}050$ for 1926 unique reflections (including unobserveds). The molecule is *syn*, with χ[C(4)—N(9)—C(1′)—O(4′)] = 61·1 (7)°, has sugar pucker $^2T_3$, with $P = 171{\cdot}1$ (8)°, and C(4′)—C(5′) conformation *ap* (*gauche–trans*). The structure has

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