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A Bromocyclohexenetriol

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Abstract. (1β,2α,3α,4β)-3-Bromocyclohex-5-ene-1,2,4-triol, $C_6H_9BrO_3$, $M_r = 209{\cdot}05$, monoclinic, $P2_1/n$, $a = 7{\cdot}592$ (1), $b = 8{\cdot}781$ (2), $c = 11{\cdot}504$ (2) Å, $\beta = 107{\cdot}02$ (1)°, $V = 733{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}893$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 54{\cdot}14$ cm^{-1}, $F(000) = 416$, $T = 295$ K, final $R = 0{\cdot}054$ for 758 unique observed reflections. The structure determination was undertaken so as to establish the stereochemistry of the compound. Important bond lengths are as follows: C—Br = 1·964 (9); C=C = 1·325 (13); C—C, within the cyclohexene ring, range from 1·499 (14) to 1·534 (12); C—OH are 1·435 (11), 1·441 (10), and 1·463 (11) Å. The C—C—C ring angle, at the point of attachment of the Br atom, is 114·4 (8)° and those involving the C=C bond are 123·9 (8) and 124·0 (9)°.

Experimental. Crystal dimensions 0·3 × 0·3 × 0·4 mm. Space group $P2_1/n$ [non-standard setting of $P2_1/c$ (No. 14)], Enraf–Nonius CAD-4*F*-11 diffractometer, monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, variable scan-width where $\Delta\omega = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, scan rate varied from 4 to 20° min^{-1} in ω, scan extended 25% on either side for background measurement, $3 < 2\theta < 45°$, lattice parameters from 25 reflections with $2\theta > 30°$, semi-empirical absorption correction, three intensity standards showed no decay. Br position from Patterson synthesis, other non-H atoms from difference-Fourier synthesis. 964 reflections measured (h 0→7, k 0→9, l 0→12), 758 with $F_o > 4\sigma(F_o)$ used in structure refinement, full-matrix least squares on F (91 variables) using *SHELX*76 (Sheldrick, 1978). All atoms refined anisotropically, H atoms ignored. Scattering factors and anomalous-dispersion corrections from refinement program. Final $R = 0{\cdot}054$, $wR = 0{\cdot}073$. Weights given by $w = 1{\cdot}5468/[\sigma^2(F) + 0{\cdot}000400F^2]$. In final cycle maximum LS shift/e.s.d. 0·001. Final difference-Fourier map maximum and minimum peaks 0·75 and −0·99 e Å^{-3} respectively. Further details of data collection, reduction, and refinement procedures given in Silverman, Dewan, Giandomenico & Lippard (1980). Table 1* gives atomic positional parameters and Table 2 bond lengths and angles. Fig. 1 shows the molecular geometry and atom-labeling scheme.

Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å^2)

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}
Br	0·22739 (13)	0·03832 (15)	−0·05161 (9)	0·0453
O(1)	−0·4103 (8)	0·0140 (7)	−0·2794 (7)	0·038
O(2)	−0·0007 (7)	0·2301 (7)	−0·2891 (6)	0·035
O(3)	0·1338 (8)	−0·2505 (7)	−0·2332 (7)	0·039
C(1)	−0·2773 (12)	0·0776 (11)	−0·3361 (9)	0·036
C(2)	−0·1037 (11)	0·1266 (10)	−0·2368 (8)	0·027
C(3)	0·0116 (11)	−0·0155 (10)	−0·1878 (8)	0·027
C(4)	0·0748 (12)	−0·1024 (11)	−0·2821 (8)	0·030
C(5)	−0·0855 (13)	−0·1204 (12)	−0·3965 (9)	0·037
C(6)	−0·2409 (11)	−0·0419 (11)	−0·4192 (9)	0·033

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

C(3)–Br	1·964 (9)	C(1)–O(1)	1·463 (11)
C(3)–C(2)	1·534 (12)	C(1)–C(6)	1·499 (14)
C(3)–C(4)	1·515 (12)	C(6)–C(5)	1·325 (13)
C(2)–O(2)	1·441 (10)	C(5)–C(4)	1·516 (13)
C(2)–C(1)	1·531 (13)	C(4)–O(3)	1·435 (11)
Br–C(3)–C(2)	110·5 (6)	O(1)–C(1)–C(6)	107·3 (7)
Br–C(3)–C(4)	109·1 (6)	C(2)–C(1)–C(6)	113·2 (7)
C(2)–C(3)–C(4)	114·4 (8)	C(1)–C(6)–C(5)	123·9 (8)
O(2)–C(2)–C(3)	110·2 (6)	C(6)–C(5)–C(4)	124·0 (9)
O(2)–C(2)–C(1)	108·7 (7)	O(3)–C(4)–C(5)	108·9 (8)
C(3)–C(2)–C(1)	108·6 (7)	O(3)–C(4)–C(3)	107·7 (7)
O(1)–C(1)–C(2)	109·3 (8)	C(3)–C(4)–C(5)	109·4 (7)

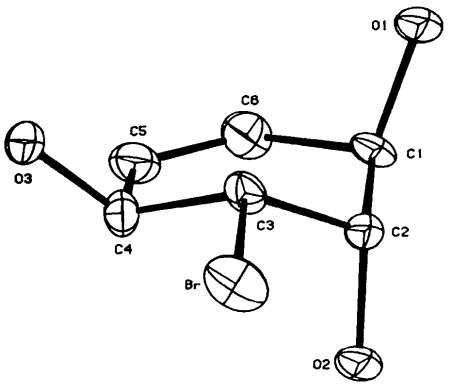

Fig. 1. Diagram of the molecule showing the atom-labeling scheme and 40% probability thermal ellipsoids.

* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42663 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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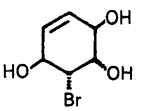
Related literature. The structure determination has established the stereochemistry of the compound (Aleksejczyk, Berchtold & Braun, 1985) to be that displayed in Fig. 1 and represented by the structural formula:



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The Structure of the Norbornadiene* Adduct of 5-Phenyl-1,3,2,4,6-dithiatriazine

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Abstract. 10-Phenyl-1,8-dithia-9,11,12-triazatetracyclo-[6.3.1.1^{3,6}.0^{2,7}]trideca-1(11),4,9,8(12)-tetraene, $C_{14}H_{13}N_3S_2$, $M_r = 287{\cdot}4$, monoclinic, $P2_1$, $a = 5{\cdot}789$ (2), $b = 10{\cdot}064$ (1), $c = 11{\cdot}109$ (2) Å, $\beta = 94{\cdot}78$ (2)°, $V = 644{\cdot}9$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}48$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 3{\cdot}84$ cm^{-1}, $F(000) = 300$, $T = 293$ K, $R = 0{\cdot}037$ for 1203 unique observed reflections. The norbornadiene bonds to the sulfur atoms in the *exo*-β orientation and the nitrogen atom between the sulfur atoms is displaced 0·802 (4) Å from the SNCNS plane. The C—N bond lengths are equal [1·335 (5) Å] and the S—N bond lengths in a very narrow range of values [1·637 (4)–1·652 (4) Å].

Experimental. Compound prepared by the reaction of norbornadiene with 1,3-dichloro-5-phenyl-1,3,2,4,6-dithiatriazine. Crystals obtained from acetonitrile solutions. White platelet data crystal 0·48 × 0·04 × 0·40 mm mounted on glass fiber. Intensities measured with Enraf–Nonius CAD-4 diffractometer, variable-speed ω–2θ scans. Unit cell from least squares of angle data for 18 reflections with $20 < 2\theta < 30°$. Analytical absorption correction based on crystal shape varied from 0·89 to 1·00. Data collected to $\sin\theta/\lambda$ of 0·70 Å^{-1}, $-8 \le h \le 8$, $0 \le k \le 14$, $0 \le l \le 15$. Three standard reflections (060, 224, $\bar{2}14$) varied ±1·9% over 18·6 h of data collection; anisotropic drift correction applied. 2061 reflections measured, 1972 unique ($R_{int} = 0{\cdot}03$), 769 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms refined with isotropic temperature factors, all other atoms refined anisotropically for 224 variables. $R = 0{\cdot}037$, $wR = 0{\cdot}043$, $S = 1{\cdot}09$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0025I^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0{\cdot}01$, $\Delta\rho_{max} = 0{\cdot}21$ (5) and $\Delta\rho_{min} = -0{\cdot}24$ (5) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.† Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

* IUPAC name: 8,9,10-trinorbornadiene.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42630 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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