Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (5)*

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

	x	y	z	$U_{\rm eq}$
Si3	0.05596 (9)	0.61716 (9)	0.24884 (9)	0.0525 (3)
Si3′	0.41369 (11)	0.06744 (9)	0.22692 (13)	0.0660 (4)
O1	0.3110 (2)	0.7365 (2)	0.2643 (2)	0.0618 (8)
O2	0.1502 (2)	0.7739 (3)	0.1150 (3)	0.0669 (9)
O3	0.2256 (2)	0.7029 (2)	0.4337 (2)	0.0472 (6)
O1′	0.5460 (2)	0.2677 (3)	0.4319 (2)	0.0701 (9)
O2′	0.3938 (3)	0.2080 (2)	0.4454 (3)	0.0691 (9)
O3′	0.4678 (2)	0.2527 (2)	0.1849 (2)	0.0498 (6)
C1	0.2130 (3)	0.7471 (2)	0.2284 (3)	0.0418 (7)
C2	0.1497 (3)	0.7287 (2)	0.3107 (3)	0.0381 (7)
C4	−0.0691 (4)	0.6505 (6)	0.1109 (4)	0.104 (2)
C5	0.1371 (5)	0.5233 (4)	0.2050 (6)	0.094 (2)
C6	0.0160 (4)	0.5752 (4)	0.3779 (4)	0.0758 (14)
C7	0.0863 (4)	0.8198 (3)	0.3192 (4)	0.0698 (13)
C1′	0.4572 (3)	0.2323 (3)	0.3840 (3)	0.0453 (8)
C2′	0.4051 (3)	0.2055 (2)	0.2479 (3)	0.0391 (7)
C4′	0.3711 (10)	0.0454 (5)	0.0572 (6)	0.172 (5)
C5′	0.3218 (6)	0.0020 (4)	0.2895 (7)	0.121 (3)
C6′	0.5583 (6)	0.0315 (5)	0.3102 (8)	0.146 (4)
C7′	0.2886 (3)	0.2453 (4)	0.1883 (4)	0.0655 (11)

Table 4. *Intermolecular hydrogen-bond geometry (Å, °) for (5)*

	O···O	O—H	H···O	O—H···O
$O3^{ii}$—$H3^{ii}$···$O1^{ii}$	2.640 (3)	0.810	2.213	113.3
$O3^{iii}$—$H3^{iii}$···O1′	2.834 (4)	0.810	2.117	147.6
$O2^{ii}$—$H2^{ii}$···$O3^{iii}$	2.655 (4)	0.803	1.902	155.7
O3′—H3′···O1′	2.647 (4)	0.830	2.309	105.0
O3′—H3′···$O1^{ii}$	2.747 (4)	0.830	2.009	147.9
$O2'^{i}$—$H2'^{i}$···O3′	2.616 (4)	0.939	1.699	164.4

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $1 - x, 1 - y, 1 - z$.

Methyl group H atoms were refined using a riding model. Hydroxy H atoms were refined with a riding model for (3), allowing torsional freedom, and were freely refined along with the solvent water unique H atom for (5). For riding H atoms, $U(H) = 1.5U_{\rm eq}(X)$, where X is the parent bonded atom. The relatively high residuals are due to poor quality for both compounds and to probable unresolved disorder and crystal decomposition for (5).

For both compounds, data collection: *P3/PC Diffractometer Program* (Siemens, 1989); cell refinement: *P3/PC Diffractometer Program*; data reduction: *SHELXTL* (Sheldrick, 1994); program(s) used to solve structures: *SHELXTL*; program(s) used to refine structures: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1021). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Phenyl-4-morpholinecarbothioamide and *N*-(2-Tolyl)-4-morpholinecarbothioamide

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Abstract

The structures of two unsymmetrically substituted thiourea derivatives, $C_{11}H_{14}N_2OS$, (1), and $C_{12}H_{16}N_2OS$, (2), are reported. In (1), the two molecules present in the asymmetric unit show differences in the orientations of the phenyl and cyclohexyl rings. In both of the structures, N—H···S hydrogen bonds link the molecules into chains that run parallel to a crystallographic axis.

Comment

Unsymmetrically substituted thioureas are found to exhibit agrochemical properties (Sarkis & Faisal, 1985; Ramadas, Srinivasan & Janarthanan, 1993). As a part of our investigations on substituted thiourea derivatives, we have determined the crystal structures of two more unsymmetrically substituted thiourea derivatives, (1) and (2).

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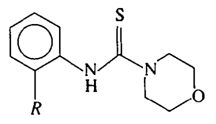

R = H for (1)
R = Me for (2)

In (1) and (2), the C1=S1 distances (Table 2) are 0.01–0.02 Å longer than the mean value (1.681 Å; Allen *et al.*, 1987) for thioureas. The N1—C2 bond lengths of 1.406 (7)–1.418 (4) Å are also longer than the usual C(ar)—N distance of 1.353 Å. In (1), the phenyl and morpholine rings are oriented in opposite directions with different angles to the thiourea moiety in the two molecules. For (1)-*A*, the dihedral angle between the plane of the thiourea moiety and that of the phenyl ring is 53.5 (2)°, that between the thiourea plane and the best plane through the morpholine ring is 21.0 (2)° and that between the phenyl and morpholine ring planes is 61.1 (2)°. In molecule (1)-*B* these angles are 116.7 (2), 36.8 (2) and 90.5 (2)°, respectively. The corresponding angles in (2) are 70.5 (1), 16.6 (1) and 76.8 (1)° and the molecule has nearly the same conformational features as (1)-*A*. The phenyl rings of (1)-*A* and (1)-*B* lie parallel to one another [3.9 (2)°]. The morpholine rings adopt chair conformations in both (1) and (2).

The two molecules in (1) are connected by an N—H···S hydrogen bond between N1*B* and S1*A* [N1*B*···S1*A* 3.467 (5) Å, N1*B*—H1*B*···S1*A* 158 (4)°]. An additional N—H···S hydrogen bond between N1*A* and S1*B* links the molecules into chains along **b** [N1*A*···S1*B*$^{\rm i}$ 3.447 (6) Å, N1*A*—H1*A*···S1*B* 151 (4)°]. Three C—H···O hydrogen bonds are also observed between molecules (1)-*A* and (1)-*B*: C7*A*···O1*B*$^{\rm ii}$ 3.327 (8) Å, C7*A*—H7*A*···O1*B*$^{\rm ii}$ 135 (5)°; C8*A*···O1*B*$^{\rm iii}$ 3.106 (8) Å, C8*A*—H8*A*···O1*B*$^{\rm iii}$ 119 (5)°; C8*B*···O1*A*$^{\rm iv}$ 3.232 (8) Å, C8*B*—H8*D*···O1*A*$^{\rm iv}$ 114 (6)° [symmetry codes: (i) $x, 1+y, z$; (ii) $x, y, z-1$; (iii) $\frac{1}{2}-x, y, z-\frac{1}{2}$; (iv) $\frac{1}{2}-x, y-1, \frac{1}{2}+z$]. Fig. 2 shows the packing of molecules of (2). N—H···S hydrogen bonds form linear chains along the *c* axis: N1···S1$^{\rm v}$ 3.442 (3) Å, N1—H1···S1$^{\rm v}$ 155 (3)° [symmetry code: (v) $x, -y-\frac{1}{2}, z+\frac{1}{2}$]. There is one short C—H···O contact: C9—O1$^{\rm vi}$ 3.087 (5) Å, C9—H9B···O1$^{\rm vi}$ 109 (2)° [symmetry code: (vi) $\frac{1}{2}-x, y, z-\frac{1}{2}$].

In compound (1), the C—H···O hydrogen bonds probably influence the packing of the two molecules in the asymmetric unit. The lone N—H group present in the molecules of (1) and (2) is involved in N—H···S hydrogen bonding to neighbouring molecules forming chains that run parallel to a crystallographic axis. Our structural studies on substituted thiourea derivatives reveal that the formation of one-dimensional chains is associated with an axial length of *ca* 8 Å (two molecules as repeat unit) along which the chains run: 7.912Å in 1-dimethyl-3-tolylthiourea (Ramnathan *et al.*, 1996), 8.488Å in 1-diethyl-3-tolylthiourea (Ramnathan *et al.*, 1996), 8.482Å in 1-cyclohexyl-3-tolylthiourea (Ramnathan *et al.*, 1995*a*), 7.951 Å in diphenylthiourea (Ramnathan *et al.*, 1995*b*), 8.094 (2) and 7.829 (1) Å in the title compounds (1) and (2), respectively.

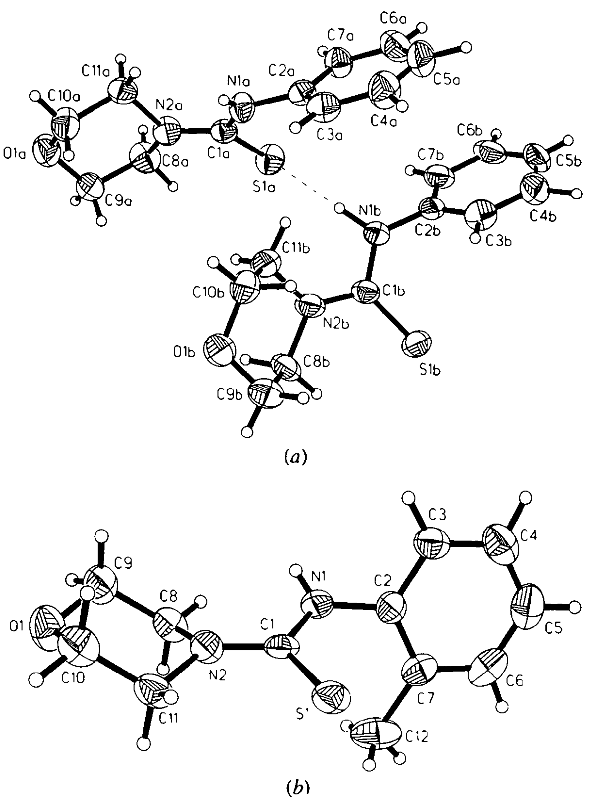

Fig. 1. Displacement ellipsoid (50% probability) plots of (*a*) (1) and (*b*) (2) with the atom-numbering schemes. H atoms are shown as spheres of arbitrary radii.

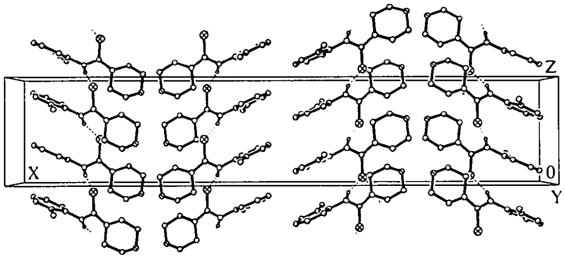

Fig. 2. Packing diagram of the molecules in (2).

Experimental

Suitable single crystals were obtained by slow evaporation of ethanol solutions of the title compounds.

Compound (1)

Crystal data

$C_{11}H_{14}N_2OS$	Mo *Kα* radiation
$M_r = 222.3$	$\lambda = 0.71073$ Å

Orthorhombic
$Pca2_1$
$a = 27.858\ (5)$ Å
$b = 8.094\ (2)$ Å
$c = 9.951\ (2)$ Å
$V = 2243.8\ (8)$ Å^3
$Z = 8$
$D_x = 1.316$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 8–25°$
$\mu = 0.263$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.52 × 0.30 × 0.22 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2895 measured reflections
2432 independent reflections
1869 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.037$

$\theta_{\rm max} = 27.5°$
$h = -1 \rightarrow 36$
$k = -1 \rightarrow 9$
$l = -1 \rightarrow 12$
3 standard reflections monitored every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0470$
$wR(F^2) = 0.1401$
$S = 1.065$
2431 reflections
383 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.086P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.32$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.36$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = 0.22 (15)

Compound (2)

Crystal data

$C_{12}H_{16}N_2OS$
$M_r = 236.3$
Orthorhombic
Pccn
$a = 40.201\ (5)$ Å
$b = 7.590\ (1)$ Å
$c = 7.829\ (1)$ Å
$V = 2388.8\ (5)$ Å^3
$Z = 8$
$D_x = 1.314$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–25°$
$\mu = 0.252$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.60 × 0.28 × 0.08 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2873 measured reflections
2096 independent reflections
1317 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.058$

$\theta_{\rm max} = 25°$
$h = -1 \rightarrow 47$
$k = -1 \rightarrow 9$
$l = -1 \rightarrow 9$
3 standard reflections monitored every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0507$
$wR(F^2) = 0.305$
$S = 0.966$
2079 reflections
209 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1049P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for (1) and (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*	$U_{\rm eq}$
Compound (1)-*A*				
S1*A*	0.30130 (5)	0.5725 (2)	0.1773 (2)	0.0483 (4)
O1*A*	0.21187 (14)	1.0912 (5)	0.3805 (5)	0.056 (1)
N1*A*	0.3607 (2)	0.8223 (6)	0.2333 (6)	0.039 (1)
N2*A*	0.2805 (2)	0.8784 (5)	0.2582 (5)	0.043 (1)
C1*A*	0.3149 (2)	0.7690 (6)	0.2242 (5)	0.034 (1)
C2*A*	0.4028 (2)	0.7442 (6)	0.1848 (6)	0.035 (1)
C3*A*	0.4433 (2)	0.7427 (7)	0.2666 (6)	0.046 (1)
C4*A*	0.4852 (2)	0.6761 (9)	0.2207 (8)	0.057 (2)
C5*A*	0.4879 (2)	0.6087 (8)	0.0917 (9)	0.060 (2)
C6*A*	0.4482 (2)	0.6118 (8)	0.0120 (8)	0.055 (2)
C7*A*	0.4055 (2)	0.6789 (7)	0.0562 (6)	0.044 (1)
C8*A*	0.2295 (2)	0.8331 (8)	0.2629 (8)	0.053 (2)
C9*A*	0.2066 (2)	0.9170 (8)	0.3818 (9)	0.056 (2)
C10*A*	0.2618 (2)	1.1338 (8)	0.3776 (8)	0.051 (2)
C11*A*	0.2868 (2)	1.0591 (7)	0.2586 (7)	0.042 (1)
Compound (1)-*B*				
S1*B*	0.38993 (6)	0.0771 (2)	0.49850 (15)	0.0463 (4)
O1*B*	0.3587 (2)	0.5988 (5)	0.7555 (4)	0.053 (1)
N1*B*	0.3888 (2)	0.3276 (6)	0.3247 (5)	0.045 (1)
N2*B*	0.3569 (2)	0.3830 (5)	0.5327 (5)	0.046 (1)
C1*B*	0.3784 (2)	0.2737 (7)	0.4511 (6)	0.039 (1)
C2*B*	0.4173 (2)	0.2449 (6)	0.2293 (6)	0.038 (1)
C3*B*	0.4647 (2)	0.1989 (8)	0.2589 (8)	0.054 (2)
C4*B*	0.4921 (3)	0.1201 (9)	0.1625 (10)	0.065 (2)
C5*B*	0.4732 (3)	0.0883 (8)	0.0375 (9)	0.072 (3)
C6*B*	0.4271 (3)	0.1343 (8)	0.0067 (7)	0.059 (2)
C7*B*	0.3993 (2)	0.2120 (8)	0.1018 (6)	0.048 (1)
C8*B*	0.3342 (2)	0.3380 (8)	0.6603 (6)	0.048 (1)
C9*B*	0.3592 (3)	0.4227 (9)	0.7748 (7)	0.057 (2)
C10*B*	0.3812 (3)	0.6413 (8)	0.6321 (7)	0.053 (2)
C11*B*	0.3567 (3)	0.5625 (7)	0.5129 (7)	0.052 (2)
Compound (2)				
S1	0.35386 (2)	0.00778 (10)	0.07536 (10)	0.0417 (3)
O1	0.27435 (7)	0.0169 (3)	0.6141 (3)	0.0534 (7)
N1	0.38057 (7)	−0.1469 (4)	0.3473 (4)	0.0382 (7)
N2	0.33011 (7)	−0.0145 (3)	0.3932 (3)	0.0360 (7)
C1	0.35479 (8)	−0.0522 (4)	0.2834 (4)	0.0327 (8)
C2	0.41193 (8)	−0.1701 (4)	0.2674 (4)	0.0360 (8)
C3	0.42276 (9)	−0.3411 (5)	0.2336 (5)	0.0428 (9)
C4	0.45356 (11)	−0.3693 (6)	0.1637 (5)	0.054 (1)
C5	0.47382 (11)	−0.2302 (7)	0.1284 (5)	0.060 (1)
C6	0.46352 (10)	−0.0612 (7)	0.1638 (5)	0.057 (1)
C7	0.43307 (9)	−0.0267 (4)	0.2377 (4)	0.0425 (9)
C8	0.30004 (10)	0.0829 (5)	0.3426 (5)	0.0444 (9)
C9	0.27058 (11)	0.0121 (6)	0.4338 (5)	0.0486 (9)
C10	0.30247 (10)	−0.0863 (6)	0.6610 (5)	0.0488 (9)
C11	0.33374 (10)	−0.0192 (5)	0.5804 (4)	0.0397 (8)
C12	0.4234 (2)	0.1579 (6)	0.2887 (8)	0.068 (1)

Table 2. *Comparison of bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

For (1)-*A* and (2) the signs of the torsion angles obtained from the coordinates in Table 1 have been reversed so that molecules of similar configuration can be compared.

	(1)-*A*	(1)-*B*	(2)
S1—C1	1.701 (5)	1.691 (6)	1.691 (3)
O1—C9	1.417 (7)	1.438 (9)	1.420 (4)
O1—C10	1.433 (8)	1.420 (8)	1.423 (4)

N1—C1	1.350 (7)	1.362 (7)	1.357 (4)
N1—C2	1.417 (7)	1.406 (7)	1.418 (4)
N2—C1	1.348 (7)	1.342 (7)	1.344 (4)
N2—C8	1.468 (7)	1.464 (7)	1.471 (4)
N2—C11	1.473 (7)	1.467 (7)	1.473 (4)
C8—C9	1.506 (10)	1.501 (10)	1.484 (6)
C10—C11	1.501 (10)	1.510 (10)	1.496 (5)
C7—C12			1.508 (6)
C10—O1—C9	109.8 (5)	110.6 (5)	109.1 (3)
C1—N1—C2	128.1 (5)	126.2 (5)	125.6 (3)
C1—N2—C8	122.1 (5)	123.6 (4)	122.8 (3)
C1—N2—C11	124.7 (4)	125.0 (5)	123.9 (3)
C11—N2—C8	111.2 (5)	111.2 (5)	111.2 (3)
N2—C1—N1	116.5 (4)	116.3 (5)	116.2 (3)
N2—C1—S1	121.6 (4)	122.4 (4)	122.9 (2)
N1—C1—S1	121.9 (4)	121.2 (4)	121.0 (3)
C3—C2—C7	119.6 (5)	118.6 (6)	120.2 (3)
C7—C2—N1	121.8 (5)	120.3 (5)	121.1 (3)
C3—C2—N1	118.4 (5)	121.0 (6)	118.5 (3)
C6—C7—C2	119.3 (6)	120.9 (7)	117.4 (4)
N2—C8—C9	108.8 (5)	110.2 (5)	110.1 (3)
O1—C9—C8	113.4 (6)	110.2 (6)	112.6 (3)
O1—C10—C11	111.7 (6)	112.2 (5)	111.8 (3)
N2—C11—C10	110.3 (5)	108.1 (6)	110.2 (3)
C2—C7—C12			121.4 (4)
C6—C7—C12			121.1 (4)
C2—N1—C1—N2	169.2 (5)	170.0 (5)	166.0 (3)
C2—N1—C1—S1	−12.7 (8)	−12.8 (8)	−15.8 (4)
C1—N1—C2—C3	137.3 (6)	−56.2 (8)	122.5 (4)
C1—N1—C2—C7	−46.5 (8)	125.8 (6)	−63.6 (4)
C8—N2—C1—N1	175.2 (6)	166.7 (5)	178.3 (3)
C8—N2—C1—S1	−2.9 (8)	−10.4 (8)	0.1 (4)
C11—N2—C1—N1	−22.0 (9)	−19.1 (9)	19.6 (6)
C11—N2—C1—S1	159.9 (5)	163.7 (5)	162.2 (3)
C1—N2—C8—C9	−141.0 (6)	117.6 (6)	−143.4 (3)
C1—N2—C11—C10	140.5 (6)	−118.7 (7)	143.4 (3)

Both of the structures were solved by direct methods and refined by the full-matrix least-squares technique. All H atoms in both structures were located from difference maps and refined isotropically. The *PARST* (Nardelli, 1983) program was used for geometric calculations.

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990*a*); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*); software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1993); geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1225). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hydrogen Bonding *versus* van der Waals Repulsion in 1,3,5-Trideoxy-1,3,5-tris(trimethylammonio)-*cis*-inositol Triiodide Sesquihydrate, $C_{15}H_{36}N_3O_3^{3+}.3I^-.1.5H_2O$

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Abstract

The crystal structure of the trimethylammonio-*cis*-inositol molecule displays one bifurcated intramolecular hydrogen bond of the form O—H···O, a strong intermolecular O—H···O*W* contact and possible intermolecular C—H···O and O—H···I interactions. C—H···I interactions may also be considered. As a result of the three bulky $N(CH_3)_3$ groups and the OH groups, increased puckering of the cyclohexane ring is observed compared with an undistorted ideal cyclohexane chair conformation.

Comment

This paper reports the structure of a cyclohexane chair substituted by bulky $N(CH_3)_3$ groups and OH groups. Intra- and intermolecular hydrogen bonds are competitors in this structure. Intramolecular three-center bonds, intermolecular OH···O*W*, CH···O*W*, OH···I and possible CH···I interactions are discussed. Fundamental work is summarized by Jeffrey & Saenger (1994). Additional information on OH···O and CH···O interactions is given by Steiner & Saenger (1993, 1994) and by

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