$S = 1.54$
2038 reflections
226 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O(1)	0.0710 (1)	0.0464 (2)	0.3216 (4)	0.049 (1)
C(2)	0.0928 (1)	0.0824 (3)	0.1900 (5)	0.044 (1)
C(3)	0.0687 (2)	0.1456 (3)	0.0905 (6)	0.050 (1)
C(4)	0.0901 (2)	0.1838 (3)	−0.0385 (6)	0.049 (1)
C(5)	0.1361 (2)	0.1589 (3)	−0.0713 (5)	0.045 (1)
C(6)	0.1585 (2)	0.1967 (3)	−0.2060 (6)	0.054 (2)
C(7)	0.2022 (2)	0.1720 (4)	−0.2358 (6)	0.062 (2)
C(8)	0.2258 (2)	0.1083 (4)	−0.1379 (6)	0.060 (2)
C(9)	0.2053 (2)	0.0689 (4)	−0.0099 (6)	0.051 (2)
C(10)	0.1596 (1)	0.0938 (3)	0.0241 (5)	0.042 (1)
C(11)	0.1369 (1)	0.0556 (3)	0.1602 (5)	0.041 (1)
N(12)	0.1581 (1)	−0.0184 (3)	0.2483 (5)	0.046 (1)
C(13)	0.1409 (1)	−0.0382 (2)	0.3829 (5)	0.044 (2)
C(14)	0.1570 (2)	−0.1214 (3)	0.4811 (6)	0.048 (2)
C(15)	0.1139 (2)	−0.1762 (3)	0.5264 (6)	0.048 (2)
C(16)	0.1113 (2)	−0.2770 (4)	0.5289 (7)	0.060 (2)
C(17)	0.0724 (2)	−0.3229 (4)	0.5812 (7)	0.071 (2)
C(18)	0.0364 (2)	−0.2699 (5)	0.6299 (8)	0.079 (2)
C(19)	0.0374 (2)	−0.1699 (4)	0.6250 (7)	0.064 (2)
C(20)	0.0759 (2)	−0.1237 (3)	0.5731 (6)	0.051 (2)
N(21)	0.0789 (1)	−0.0216 (3)	0.5717 (5)	0.054 (1)
C(22)	0.1027 (2)	0.0237 (3)	0.4485 (5)	0.045 (1)
C(23)	0.1796 (2)	−0.0801 (4)	0.6291 (6)	0.062 (2)
C(24)	0.1917 (2)	−0.1819 (4)	0.3930 (7)	0.066 (2)
C(25)	0.0461 (2)	0.0378 (4)	0.6558 (6)	0.064 (2)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.385 (5)	O(1)—C(22)	1.463 (6)
C(2)—C(3)	1.411 (6)	C(2)—C(11)	1.375 (4)
C(3)—C(4)	1.373 (7)	C(4)—C(5)	1.426 (8)
C(5)—C(6)	1.423 (7)	C(5)—C(10)	1.398 (6)
C(6)—C(7)	1.356 (8)	C(7)—C(8)	1.399 (8)
C(8)—C(9)	1.361 (8)	C(9)—C(10)	1.420 (7)
C(10)—C(11)	1.439 (6)	C(11)—N(12)	1.416 (6)
N(12)—C(13)	1.283 (6)	C(13)—C(14)	1.501 (6)
C(13)—C(22)	1.522 (6)	C(14)—C(15)	1.529 (8)
C(14)—C(23)	1.536 (7)	C(14)—C(24)	1.520 (8)
C(15)—C(16)	1.399 (7)	C(15)—C(20)	1.393 (8)
C(16)—C(17)	1.385 (8)	C(17)—C(18)	1.355 (9)
C(18)—C(19)	1.387 (9)	C(19)—C(20)	1.376 (8)
C(20)—N(21)	1.418 (6)	N(21)—C(22)	1.409 (6)
N(21)—C(25)	1.458 (7)		
C(2)—O(1)—C(22)	112.3 (3)	O(1)—C(2)—C(11)	119.4 (4)
O(1)—C(2)—C(3)	118.5 (4)	C(3)—C(2)—C(11)	122.2 (4)
C(2)—C(3)—C(4)	119.3 (5)	C(3)—C(4)—C(5)	120.0 (4)
C(4)—C(5)—C(10)	120.9 (4)	C(4)—C(5)—C(6)	120.6 (5)
C(6)—C(5)—C(10)	118.5 (4)	C(5)—C(6)—C(7)	119.9 (5)
C(6)—C(7)—C(8)	121.3 (5)	C(7)—C(8)—C(9)	120.7 (5)
C(8)—C(9)—C(10)	119.1 (5)	C(5)—C(10)—C(9)	120.6 (4)
C(9)—C(10)—C(11)	121.0 (3)	C(5)—C(10)—C(11)	118.4 (3)
C(2)—C(11)—C(10)	119.2 (3)	C(10)—C(11)—N(12)	119.2 (3)
C(2)—C(11)—N(12)	121.0 (3)	C(11)—N(12)—C(13)	117.0 (3)
N(12)—C(13)—C(22)	120.0 (4)	N(12)—C(13)—C(14)	122.5 (4)
C(14)—C(13)—C(22)	117.5 (4)	C(13)—C(14)—C(24)	111.2 (4)
C(13)—C(14)—C(23)	107.9 (4)	C(13)—C(14)—C(15)	105.1 (4)
C(23)—C(14)—C(24)	108.6 (5)	C(15)—C(14)—C(24)	114.1 (4)
C(15)—C(14)—C(23)	109.8 (4)	C(14)—C(15)—C(20)	118.7 (4)
C(14)—C(15)—C(16)	123.0 (5)	C(16)—C(15)—C(20)	118.2 (5)
C(15)—C(16)—C(17)	120.6 (5)	C(16)—C(17)—C(18)	119.8 (6)
C(17)—C(18)—C(19)	121.1 (6)	C(18)—C(19)—C(20)	119.5 (5)
C(15)—C(20)—C(19)	120.8 (4)	C(19)—C(20)—N(21)	121.3 (5)
C(15)—C(20)—N(21)	118.0 (5)	C(20)—N(21)—C(25)	121.2 (4)
C(20)—N(21)—C(22)	118.8 (4)	C(22)—N(21)—C(25)	116.4 (4)
C(13)—C(22)—N(21)	113.0 (4)	O(1)—C(22)—N(21)	109.2 (4)
O(1)—C(22)—C(13)	108.8 (3)		

The structure was solved by direct methods (*MULTAN*80; Main *et al.*, 1980) and refined by block-matrix least squares (*SHELX*76; Sheldrick, 1976). Other programs used: *PARST* (Nardelli, 1983) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: NA1085). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stereochemistry of Rings. XVIII.† 8-*tert*-Butyl-1,4-dithiaspiro[4.5]decane

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(*Received* 20 *December* 1993; *accepted* 26 *May* 1994)

Abstract

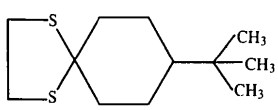
The two mean ring planes of the title compound, $C_{12}H_{22}S_2$, are almost perpendicular [dihedral angle

† Part XVII: Bocelli (1990).

96.9 (3)°] and are not individually planar. The cyclohexane ring has a chair conformation while the conformation of the five-membered ring is intermediate between envelope and twist.

Comment

This paper forms part of a wider study designed to elucidate the conformational variations of the cyclohexane ring in the presence of a spiro connection. Fig. 1, which presents a projection of the title molecule (I), shows that the mean ring planes are almost perpendicular [dihedral angle 96.9 (3)°]. The rings are not individually planar and in terms of the Cremer & Pople (1975) notation the cyclohexane ring has puckering parameters [Q = 0.56 (1), θ = 180.0 (10)°, φ = −26.3 (17)°] which are near to those calculated for a chair conformation, while those of the five-membered ring [Q = 0.51 (1), φ = −99.7 (11)°] are intermediate between those of envelope and twist conformations.



(I)

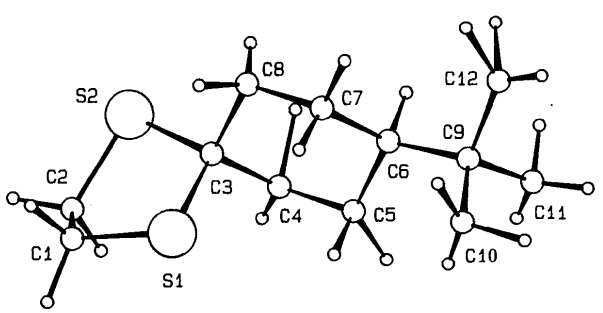


Fig. 1. Projection of the title molecule showing the arbitrary atom-numbering scheme.

Experimental

Crystal data

$C_{12}H_{22}S_2$
M_r = 230.43
Orthorhombic
$Pca2_1$
a = 10.041 (2) Å
b = 17.962 (2) Å
c = 7.259 (3) Å
V = 1309.2 (6) Å^3
Z = 4
D_x = 1.169 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 30 reflections
θ = 10.8–35.8°
μ = 3.324 mm^{-1}
Room temperature
Prism
0.21 × 0.16 × 0.11 mm
Colourless
Crystal source: Grenier-Loustalot (1994)

Data collection

Siemens AED diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1479 measured reflections
1358 independent reflections
797 observed reflections [$I > 3\sigma(I)$]
θ_{max} = 70°
h = 0 → 21
k = 0 → 12
l = 0 → 8
2 standard reflections monitored every 50 reflections
intensity variation: 11%

Refinement

Refinement on F^2
R = 0.066
wR = 0.068
S = 0.731
1479 reflections
214 parameters
w = 1.881/[$\sigma^2(F)$ + 0.039996F^2]
$(\Delta/\sigma)_{max}$ = 0.654
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol IV, Tables 2.2A, 2.2C and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) (*Hamilton*, 1959)

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

	x	y	z	U_{eq}
S1	−0.1225 (2)	0.1643 (1)	0.55790	0.0849 (12)
S2	0.1252 (2)	0.0794 (1)	0.5172 (7)	0.0687 (9)
C1	−0.1014 (12)	0.0865 (8)	0.7062 (20)	0.087 (4)
C2	0.0458 (11)	0.0744 (6)	0.7423 (19)	0.084 (5)
C3	0.0387 (7)	0.1628 (4)	0.4426 (17)	0.0533 (31)
C4	0.1178 (8)	0.2328 (4)	0.4909 (19)	0.0589 (38)
C5	0.0587 (9)	0.3034 (4)	0.4139 (17)	0.0667 (35)
C6	0.0465 (8)	0.3004 (4)	0.2045 (16)	0.0516 (29)
C7	−0.0360 (13)	0.2312 (5)	0.1531 (19)	0.069 (5)
C8	0.0216 (12)	0.1608 (4)	0.2319 (18)	0.0616 (35)
C9	0.0001 (9)	0.3730 (4)	0.1129 (20)	0.0678 (33)
C10	−0.1400 (13)	0.3952 (9)	0.1656 (20)	0.096 (6)
C11	0.0968 (18)	0.4361 (6)	0.1559 (21)	0.097 (6)
C12	0.0033 (18)	0.3633 (7)	−0.0954 (18)	0.095 (5)

Table 2. *Selected geometric parameters* (Å, °)

S1—C1	1.777 (15)	C5—C6	1.526 (17)
S1—C3	1.822 (8)	C6—C7	1.540 (13)
S2—C2	1.820 (14)	C6—C9	1.536 (12)
S2—C3	1.814 (8)	C7—C8	1.504 (14)
C1—C2	1.517 (17)	C9—C10	1.511 (16)
C3—C4	1.528 (11)	C9—C11	1.525 (17)
C3—C8	1.539 (18)	C9—C12	1.522 (20)
C4—C5	1.508 (12)		
C1—S1—C3	99.3 (5)	C5—C6—C9	115.2 (8)
C2—S2—C3	95.7 (5)	C5—C6—C7	108.3 (8)
S1—C1—C2	109.5 (9)	C7—C6—C9	114.7 (8)
S2—C2—C1	105.4 (9)	C6—C7—C8	112.3 (9)
S1—C3—S2	107.5 (4)	C3—C8—C7	113.7 (9)
S2—C3—C8	109.3 (5)	C6—C9—C12	109.1 (8)
S2—C3—C4	111.2 (5)	C6—C9—C11	110.4 (9)
S1—C3—C8	111.0 (5)	C6—C9—C10	113.4 (8)
S1—C3—C4	110.1 (6)	C11—C9—C12	106.0 (9)
C4—C3—C8	107.8 (7)	C10—C9—C12	107.5 (10)
C3—C4—C5	113.7 (7)	C10—C9—C11	110.2 (9)
C4—C5—C6	111.8 (7)		

The structure was solved by direct methods using the *SIR*92 package (Altomare *et al.*, 1994) and refined by full-matrix anisotropic least squares for the non-H atoms. H atoms, in part found in a ΔF map and the rest positioned geometrically, were refined with isotropic temperature factors.

Data collection: Belletti, Cantoni & Pasquinelli (1993). Cell refinement: Belletti *et al.* (1993). Data reduction: Belletti *et al.* (1993). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1987) .

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: PA1102). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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