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2-*tert*-Butylthio-4-methylbicyclo[2.1.1]hexane-1,2-dicarbonitrile

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Abstract. $C_{13}H_{18}N_2S$, $M_r = 234{\cdot}36$, orthorhombic, $Pna2_1$, $a = 20{\cdot}341\ (2)$, $b = 6{\cdot}594\ (1)$, $c = 10{\cdot}579\ (1)$ Å, $V = 1418{\cdot}9\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}10$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}05$ cm^{-1}, $F(000) = 504$, $T = 291$ K, $R = 0{\cdot}040$ for 891 observed reflections. The two five-membered rings have envelope conformations with C1—C2—C3—C4 planar (max. deviation = 0·003 Å) and C5, C6 at 1·06 Å on each side of this plane. The mean torsion angle about the ring bonds of the four-membered ring is 34·5 (5)°. All ring bond lengths are slightly elongated but no significant lengthening is observed for the *cd*-substituted C1—C2 bond {*cd* substitution means the simultaneous substitution on the same C atom by an electron acceptor group (*c*) and an electron donor (*d*) [Viehe, Janousek, Mérényi & Stella (1985). *Acc. Chem. Res.* **18**, 148–154]}.

Experimental. The compound was prepared by Dr V. Gallez (De Meijere, Wenck, Seyed-Madhavi, Viehe, Gallez & Erden, 1986) and recrystallized by evaporation from ethanol. The crystal structure analysis was undertaken to determine which isomer was obtained. Parallelepiped crystal with dimensions 0·15 × 0·15 × 0·5 mm. Lattice parameters were refined using 17 reflections in the range $5° \le 2\theta \le 30°$. Huber four-circle diffractometer, graphite monochromated Mo $K\alpha$ radiation. 1323 independent reflections with $\sin\theta/\lambda \le 0{\cdot}595$ Å^{-1}; $0 \le h \le 24$, $0 \le k \le 7$, $0 \le l \le 12$; 1323 with $I \ge 2{\cdot}5\sigma(I)$. Standard reflection (113) checked every 50 reflections: no significant deviation. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Nine H atoms were located from difference Fourier synthesis; those of the *tert*-butyl group were calculated with C—H distances of 1·08 Å. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0{\cdot}00285F^2)$, $R = 0{\cdot}040$, wR

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
C1	0·4484 (2)	0·3712 (7)	0·3116 (8)	3·71 (9)
C2	0·4018 (2)	0·3000 (8)	0·4200 (8)	3·98 (9)
C3	0·4431 (3)	0·1125 (10)	0·4652 (9)	4·45 (10)
C4	0·5012 (3)	0·1275 (8)	0·3731 (9)	4·52 (10)
C5	0·4656 (3)	0·1684 (9)	0·2454	4·52 (10)
C6	0·5166 (3)	0·3583 (9)	0·3755 (8)	4·43 (10)
C7	0·4283 (3)	0·5509 (9)	0·2414 (9)	4·49 (10)
N8	0·4156 (3)	0·6913 (8)	0·1827 (9)	6·55 (12)
C9	0·3971 (2)	0·4627 (9)	0·5158 (8)	4·53 (10)
N10	0·3963 (3)	0·5889 (8)	0·5872 (8)	6·24 (11)
S11	0·3215 (1)	0·2451 (2)	0·3500 (7)	4·96 (2)
C12	0·5542 (4)	−0·0345 (14)	0·3852 (11)	6·57 (16)
C13	0·2617 (2)	0·2003 (11)	0·4784 (9)	5·80 (12)
C14	0·2075 (4)	0·0884 (14)	0·4137 (12)	9·04 (19)
C15	0·2894 (4)	0·0881 (15)	0·5884 (10)	9·24 (20)
C16	0·2346 (4)	0·4045 (12)	0·5249 (10)	8·27 (18)

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

C1—C5	1·550 (8)	C4—C5	1·556 (9)
C2—C1	1·560 (7)	C6—C1	1·545 (7)
C7—C1	1·457 (8)	C3—C2	1·570 (8)
C9—C2	1·479 (8)	S11—C2	1·828 (5)
C4—C3	1·534 (8)	C6—C4	1·554 (8)
C12—C4	1·523 (8)	N8—C7	1·144 (7)
N10—C9	1·124 (7)	C13—S11	1·849 (6)
C14—C13	1·492 (10)	C15—C13	1·490 (10)
C16—C13	1·535 (10)		
C4—C5—C1	82·1 (4)	C2—C1—C5	102·1 (4)
C6—C1—C5	87·0 (4)	C6—C1—C2	102·0 (4)
C7—C1—C5	122·3 (4)	C7—C1—C2	116·7 (4)
C7—C1—C6	121·3 (4)	C3—C2—C1	97·8 (4)
C9—C2—C1	108·9 (4)	C9—C2—C3	113·4 (4)
S11—C2—C1	107·8 (3)	S11—C2—C3	116·5 (4)
S11—C2—C9	111·3 (3)	C4—C3—C2	99·7 (5)
C3—C4—C5	101·8 (4)	C6—C4—C5	86·5 (4)
C6—C4—C3	102·0 (5)	C12—C4—C5	121·6 (6)
C12—C4—C3	116·5 (6)	C12—C4—C6	122·9 (5)
C4—C6—C1	82·3 (4)	N8—C7—C1	176·4 (6)
N10—C9—C2	176·9 (5)	C13—S11—C2	108·8 (3)
C14—C13—S11	103·1 (5)	C15—C13—S11	113·8 (4)
C15—C13—C14	113·1 (7)	C16—C13—S11	109·4 (5)
C16—C13—C14	108·4 (6)	C16—C13—C15	108·7 (7)

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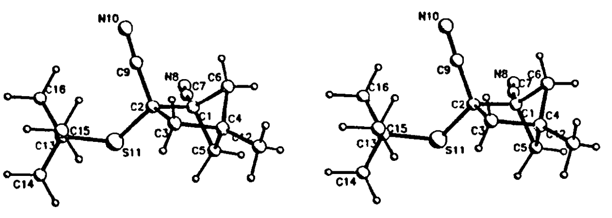

Fig. 1. Stereoscopic view of the molecule (program *PLUTO*; Motherwell & Clegg, 1978).

= 0·046 for 891 observed reflections. Final maximum shift to e.s.d. = 0·28. S = 0·86. Maximum and minimum heights in final difference Fourier synthesis = 0·16 and −0·18 e Å^{-3}. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974).

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 is a stereoscopic view of the molecule.

Related literature. The structure of (1′*R*,2′*R*,4′*R*)-2-(1′-methyl-5′-oxo-2′-bicyclo[2.1.1]hexyl)ethyl 4-bromobenzoate has been reported recently (Rettig & Trotter, 1987).

The geometry of *cd*-substituted ethane and cyclopropane derivatives has been discussed (Parfonry, Declercq, Tinant, Van Meerssche & Schweiss, 1988; Tinant, Wu, Declercq, Van Meerssche, Masamba, De Mesmaeker & Viehe, 1988).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52464 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dimethyl 5-Amino-3-methyl-4-triisopropylsilyl-1,2-benzenedicarboxylate

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Abstract. $C_{20}H_{33}NO_4Si$, M_r = 379·57, monoclinic, $P2_1/c$, a = 17·287 (5), b = 8·895 (2), c = 15·049 (4) Å, β = 109·53 (2)°, V = 2181 (1) Å^3, Z = 4, D_x = 1·16 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·35 cm^{-1}, $F(000)$ = 824, T = 291 K, R = 0·050 for 3013 observed reflections. Severe steric strain in this pentasubstituted benzene derivative leads to: (*a*) rotation of the methoxycarbonyls at C1, C2 out of the benzene plane by 29·5 (3) and 99·7 (3)° respectively. (*b*) Significant lengthening of the C3—C4 and C4—C5 bonds to 1·418 (3) and 1·429 (3) Å and a stretching of the Si—C(ar.) bond to 1·930 (2) compared with an expected value of 1·868 Å [Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem.*