Venkatesan, 1988). $C(sp^3)$—$C(sp^3)$ distances in the two piperidine rings are shorter than the $C(sp^2)$—$C(sp^3)$ distances. This feature is found to be common in the fused piperidine ring systems (Chinnakali, Sivakumar & Natarajan, 1990). The benzene and pyrone rings are planar (χ^2 = 5·2 and 4·6) and the dihedral angle between them is 0·8 (1)°, thus making the coumarin moiety exactly planar.

Endocyclic torsion angles characterizing the piperidine-ring conformations are given in Table 2. The two piperidine rings *A* and *B* adopt significantly flattened half-chair conformations [$\Delta C_2(6–7)$ = 3·6 and $\Delta C_2(7–8)$ = 2·9°] (Duax, Weeks & Rohrer, 1976). This flattening results from the fusion of the piperidine rings with the benzene ring of the coumarin moiety. The average values of the absolute magnitudes of the torsion angles are 24·6 and 28·8° respectively.

The packing of the molecules viewed down the *c* axis is shown in Fig. 1. The crystal structure is stabilized by van der Waals interactions.

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Conformation of 1,3,5-Tri-*p*-tolylmethyl-1,3,5-triazacyclohexane

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Abstract. $C_{27}H_{33}N_3$, M_r = 399·62, triclinic, $P\bar{1}$, a = 6·020 (2), b = 15·326 (2), c = 13·009 (1) Å, α = 97·00 (1), β = 98·05 (2), γ = 90·55 (2)°, V = 1179 (1) Å^3, Z = 2, D_x = 1·13 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·07 mm^{-1}, F(000) = 432, T = 293 K, R = 0·047 for 2713 independent observed reflections. The triazacyclohexane ring adopts a chair conformation with equatorial–diaxial orientation of the *p*-tolylmethyl groups. The torsion angles around the N—C bonds in the triazacyclohexane ring are 50·6 (2)–61·1 (2)°, the CH_2—N—CH_2 angles in the ring are 108·0 (2)–109·4(2)°, the N—CH_2—N angles are 111·5 (2)–116·8 (2)°, and the N—C ring bonds are 1·446 (3)–1·477 (3) Å. The axial N—CH_2 exocyclic bonds are bent outwards from ideal tetrahedral positions by *ca* 9° to reduce 1,3-diaxial repulsion.

Introduction. Conformational effects of non-bonding electrons are a feature of heterocyclic chemistry (Riddell, 1980). The heterocyclic nucleus in the 1,3,5-trialkyl-1,3,5-triazacyclohexanes (1) (hexahydro-*s*-triazines) is expected to adopt a chair conformation and four distinct types of substituent orientation can be postulated, *eee*, *eea*, *eaa* and *aaa*, where *e* = equatorial and *a* = axial; all four conformers have axial repulsions involving substituents and/or lone pairs of electrons on the N atoms. Several 1,3,5-trialkyl derivatives have been investigated in solution by NMR spectroscopy and dipole-moment measurements and the results interpreted in terms of the *eee* conformer (Farmer & Hamer, 1968; Gutowsky & Temussi, 1967; Lehn, Riddell, Price & Sutherland, 1967), the *eea* conformer (Bushweller, Lourandos & Brunelle, 1974; Baker, Ferguson,

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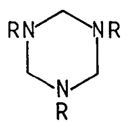
Katritzky, Patel & Rahimi-Rastgoo, 1978), and varying amounts of the *eee*, *eea* and *eaa* conformers (Duke, Jones, Katritzky, Scattergood & Riddell, 1973). X-ray studies of the 1,3,5-tricyclohexyl compound [(1): $R = C_6H_{11}$] established that it adopts the *eea* conformation in the crystal (Zangrando, Poggi, Giumanini & Verardo, 1987; Bouchemma, McCabe & Sim, 1988). The 1,3,5-tribenzyl compound [(1): $R = CH_2C_6H_5$] exhibits a single conformation *eaa* in the solid state below *ca* 250 K but at higher temperatures there is a disordered mixture of *eaa* and *eea* conformations (Sim, 1987).

The 1,3,5-tri-*p*-tolylmethyl compound [(1): $R = CH_2C_6H_4CH_3$] was chosen for an X-ray investigation to provide a comparison with the 1,3,5-tribenzyl derivative [(1): $R = CH_2C_6H_5$].



(1)

Experimental. 1,3,5-Tri-*p*-tolylmethyl-1,3,5-triazacyclohexane [(1): $R = CH_2C_6H_4CH_3$] was obtained by reaction of *p*-methylbenzylamine (1·22 g, 10 mmol) with formalin (38% *w*/*w*, 1 ml) under reflux in ethanol (30 ml) for 2 h (Eckstein, Gluzinski, Plenkiewicz & Urbanski, 1962). On cooling, the product crystallized as needles (1·13 g, 84%), m.p. 414–416 K (lit. m.p. 405–406 K); (found: C 80·80, H 8·45, N 10·54%; $C_{27}H_{33}N_3$ requires C 81·16, H 8·33, N 10·52%); *m*/*z* 133 ($\frac{1}{3}$ M), 105 (base peak), 91 and 77; ν_{max} (KBr) 3015, 2950, 2885, 2845, 2795, 1512, 1345, 1260, 1171, 1151, 988, 902 and 800 cm^{-1}; δ_H($CDCl_3$, 90 MHz) 2·31 (*s*; CH_3), 3·41 (*s*; CH_2), 3·62 (*s*; CH_2), 7·08(*d*, *J* = 9 Hz; Ar) and 7·22 (*d*, *J* = 9Hz; Ar).

Crystal dimensions 0·2 × 0·5 × 0·6 mm. Enraf–Nonius CAD-4 diffractometer, Mo *Kα* radiation, generator settings 50 kV, 20 mA. Cell dimensions from setting angles of 25 reflections with $14 \leq \theta \leq 19°$; 5362 reflections surveyed in the range 1·5–26·5°; h 0→7, k −18→18, l −16→16; scan width $(1{\cdot}1 + 0{\cdot}25\tan\theta)°$; max. scan time 100 s; 481 pairs of equivalent reflections merged, $R_{int} = 0{\cdot}021$; 2713 independent reflections with $I > 2{\cdot}5\sigma(I)$. Two reference reflections monitored periodically showed no significant variation in intensity. Corrections applied for Lorentz–polarization effects, assuming an ideally imperfect monochromator crystal. Structure determined by direct phasing, using *MITHRIL* (Gilmore, 1984). H atoms were located in difference Fourier synthesis. Full-matrix least-squares calculations on *F* with anisotropic thermal parameters for C and N atoms and isotropic for H atoms. Convergence at $R = 0{\cdot}047$, $wR = 0{\cdot}056$, $S = 2{\cdot}41$ for 404 parameters, $\Delta/\sigma < 0{\cdot}5$, $w = 1/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 0·15, min. −0·13 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations on an SEL 32/27 computer with the *GX* (Mallinson & Muir, 1985) system of programs.

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

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

	x	*y*	*z*	U_{eq}
N(1)	0·8336 (3)	0·3421 (1)	1·0299 (1)	0·048
C(2)	0·9455 (4)	0·3552 (2)	1·1374 (2)	0·057
N(3)	0·8815 (3)	0·2948 (1)	1·2071 (1)	0·051
C(4)	0·8939 (4)	0·2056 (1)	1·1579 (2)	0·048
N(5)	0·7584 (3)	0·1912 (1)	1·0541 (1)	0·042
C(6)	0·8474 (4)	0·2507 (1)	0·9876 (2)	0·044
C(7)	0·6055 (4)	0·3743 (1)	1·0142 (2)	0·053
C(8)	0·5310 (4)	0·3929 (1)	0·9038 (2)	0·050
C(9)	0·3125 (5)	0·3748 (2)	0·8570 (2)	0·063
C(10)	0·2395 (5)	0·3950 (2)	0·7575 (2)	0·068
C(11)	0·3818 (5)	0·4341 (2)	0·7007 (2)	0·062
C(12)	0·5993 (5)	0·4528 (2)	0·7484 (2)	0·068
C(13)	0·6741 (4)	0·4328 (2)	0·8480 (2)	0·059
C(14)	0·3010 (7)	0·4561 (2)	0·5928 (2)	0·095
C(15)	0·6666 (4)	0·3117 (2)	1·2458 (2)	0·058
C(16)	0·6601 (4)	0·2802 (2)	1·3509 (2)	0·053
C(17)	0·8469 (5)	0·2853 (2)	1·4259 (2)	0·070
C(18)	0·8360 (6)	0·2593 (2)	1·5223 (2)	0·078
C(19)	0·6398 (5)	0·2279 (2)	1·5494 (2)	0·066
C(20)	0·4550 (6)	0·2224 (2)	1·4732 (3)	0·078
C(21)	0·4624 (5)	0·2490 (2)	1·3768 (2)	0·068
C(22)	0·6321 (11)	0·2033 (3)	1·6579 (3)	0·103
C(23)	0·7682 (5)	0·0996 (1)	1·0086 (2)	0·049
C(24)	0·5999 (4)	0·0749 (1)	0·9122 (2)	0·045
C(25)	0·3946 (4)	0·1146 (2)	0·8971 (2)	0·054
C(26)	0·2389 (5)	0·0889 (2)	0·8105 (2)	0·062
C(27)	0·2813 (5)	0·0219 (2)	0·7353 (2)	0·064
C(28)	0·4871 (6)	−0·0179 (2)	0·7505 (2)	0·068
C(29)	0·6426 (5)	0·0081 (2)	0·8366 (2)	0·059
C(30)	0·1105 (10)	−0·0065 (4)	0·6400 (3)	0·105

Discussion. Atomic coordinates are listed in Table 1 and molecular dimensions in Table 2.* Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular structure.

The molecule adopts the *eaa* chair conformation in the solid state, in common with the 1,3,5-tribenzyl compound below 250 K (Sim, 1987) and several 1,3,5-triaryl-1,3,5-triazacyclohexanes (Giumanini, Verardo, Randaccio, Bresciani-Pahor & Traldi, 1985; Bouchemma, McCabe & Sim, 1989), whereas the 1,3,5-tricyclohexyl derivative adopts the alternative *eea* conformation in the solid state, consistent with the greater steric requirement of the cyclohexyl group (Zangrando, Poggi, Giumanini & Verardo, 1987; Bouchemma, McCabe & Sim, 1988). The

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52487 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

N(1)—C(2)	1·454 (4)	N(1)—C(6)	1·449 (3)
N(1)—C(7)	1·459 (4)	C(2)—N(3)	1·456 (3)
N(3)—C(4)	1·446 (3)	N(3)—C(15)	1·466 (4)
C(4)—N(5)	1·466 (3)	N(5)—C(6)	1·477 (3)
N(5)—C(23)	1·461 (3)	C(7)—C(8)	1·505 (4)
C(8)—C(9)	1·381 (4)	C(8)—C(13)	1·384 (4)
C(9)—C(10)	1·380 (4)	C(10)—C(11)	1·384 (4)
C(11)—C(12)	1·380 (5)	C(11)—C(14)	1·499 (4)
C(12)—C(13)	1·382 (4)	C(15)—C(16)	1·511 (4)
C(16)—C(17)	1·377 (4)	C(16)—C(21)	1·378 (5)
C(17)—C(18)	1·371 (5)	C(18)—C(19)	1·378 (5)
C(19)—C(20)	1·377 (5)	C(19)—C(22)	1·512 (5)
C(20)—C(21)	1·372 (5)	C(23)—C(24)	1·505 (4)
C(24)—C(25)	1·383 (4)	C(24)—C(29)	1·382 (4)
C(25)—C(26)	1·375 (4)	C(26)—C(27)	1·380 (4)
C(27)—C(28)	1·386 (5)	C(27)—C(30)	1·513 (6)
C(28)—C(29)	1·371 (5)		
C(2)—N(1)—C(6)	109·4 (2)	C(2)—N(1)—C(7)	115·2 (2)
C(6)—N(1)—C(7)	112·7 (2)	N(1)—C(2)—N(3)	116·8 (2)
C(2)—N(3)—C(4)	109·0 (2)	C(2)—N(3)—C(15)	115·6 (2)
C(4)—N(3)—C(15)	112·4 (2)	N(3)—C(4)—N(5)	112·0 (2)
C(4)—N(5)—C(6)	108·0 (2)	C(4)—N(5)—C(23)	110·4 (2)
C(6)—N(5)—C(23)	110·6 (2)	N(1)—C(6)—N(5)	111·5 (2)
N(1)—C(7)—C(8)	113·1 (2)	C(7)—C(8)—C(9)	120·6 (3)
C(7)—C(8)—C(13)	121·6 (3)	C(9)—C(8)—C(13)	117·7 (3)
C(8)—C(9)—C(10)	121·2 (3)	C(9)—C(10)—C(11)	121·5 (3)
C(10)—C(11)—C(12)	116·9 (3)	C(10)—C(11)—C(14)	121·2 (3)
C(12)—C(11)—C(14)	121·9 (3)	C(11)—C(12)—C(13)	122·0 (3)
C(8)—C(13)—C(12)	120·6 (3)	N(3)—C(15)—C(16)	112·4 (2)
C(15)—C(16)—C(17)	121·6 (3)	C(15)—C(16)—C(21)	120·7 (3)
C(17)—C(16)—C(21)	117·6 (3)	C(16)—C(17)—C(18)	120·9 (3)
C(17)—C(18)—C(19)	122·3 (3)	C(18)—C(19)—C(20)	116·1 (3)
C(18)—C(19)—C(22)	120·7 (4)	C(20)—C(19)—C(22)	123·1 (4)
C(19)—C(20)—C(21)	122·4 (4)	C(16)—C(21)—C(20)	120·7 (3)
N(5)—C(23)—C(24)	113·8 (2)	C(23)—C(24)—C(25)	122·2 (2)
C(23)—C(24)—C(29)	120·5 (3)	C(25)—C(24)—C(29)	117·3 (3)
C(24)—C(25)—C(26)	121·5 (3)	C(25)—C(26)—C(27)	121·2 (3)
C(26)—C(27)—C(28)	117·3 (3)	C(26)—C(27)—C(30)	121·3 (4)
C(28)—C(27)—C(30)	121·4 (4)	C(27)—C(28)—C(29)	121·5 (3)
C(24)—C(29)—C(28)	121·3 (3)		

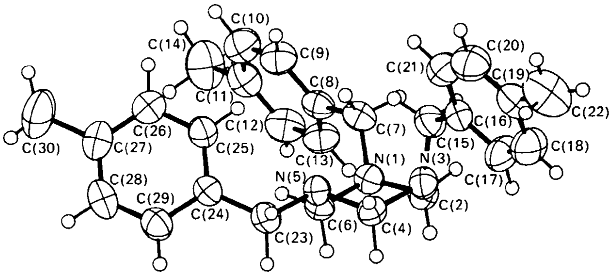

Fig. 1. Molecular structure and atomic numbering. The thermal ellipsoids of the C and N atoms are drawn at the 50% probability level and the H atoms are represented by spheres of radius 0·1 Å.

absence of any unambiguous evidence for the *eee* conformation in these compounds emphasizes the important role of the lone pairs of electrons on the N atoms.

The N atoms have distinctly pyramidal bonding geometry, the exocyclic N—C bonds being inclined at 45·3 (2), 45·5 (2) and 53·5 (2)° to the CH_2—N—CH_2 planes at N(1), N(3) and N(5), respectively. The out-of-plane angle for the equatorial N(5)—C(23) bond is close to the value of 54·7° for tetrahedrally arranged bonds whereas the out-of-plane angles for the axial N(1)—C(7) and N(3)—C(15) bonds are distinctly smaller and these bonds are bent outwards from ideal axial positions by *ca* 9°, a displacement that relieves 1,3-diaxial repulsion. Other geometrical responses to the repulsion between the axial substituents of N(1) and N(3) involve the N(1)—C(2)—N(3) ring angle of 116·8 (2)° being distinctly larger than the other N—C—N ring angles of 111·5 (2) and 112·0 (2)° and the N(1)—C(2) and N(3)—C(2) ring torsion angles of −50·6 (2) and 50·6 (3)° being substantially smaller than the N(5)—C(4) and N(5)—C(6) ring torsion angles of 61·1 (2) and −60·7 (2)°. These molecular adjustments result in unexceptional 1,3-diaxial H···H and C···C separations: H(7*A*)···H(15*B*) 2·33 (3), H(7*B*)···H(15*A*) 2·65 (3), C(7)···C(15) 3·246 (3) Å.

The flatter pyramids at N(1) and N(3) indicate some rehybridization to give more *s* character to the bonds at these atoms and in accord with this the N—C ring bonds at N(1) and N(3), 1·449–1·456 (3) Å, are slightly shorter than those at N(5), 1·466–1·477 (3) Å. Similar minor differences between the ring bonds at equatorial and axial sites occur in the tribenzyl (Sim, 1987) and tricyclohexyl (Bouchemma *et al.*, 1988) compounds.

The CH_2—N—CH_2 angles in the triazacyclohexane ring are all slightly smaller than tetrahedral, 108·0 (2)–109·4 (2)°, and the N—CH_2—N angles are all larger than tetrahedral, 111·5 (2)–116·8 (2)°. The N—CH_2—C angles of the substituent groups are greater than tetrahedral, 112·4 (2)–113·8 (2)°. The torsion angles around the exocyclic N—CH_2 bonds deviate from ideal values of 60 and 180° to reduce steric interactions: *e.g.* C(6)—N(1)—C(7)—C(8) = −76·4 (3), C(4)—N(3)—C(15)—C(16) = 81·6 (3), C(6)—N(5)—C(23)—C(24) = −71·7 (3)°.

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A Centric Thiuram Disulfide. Structure of Tetraisopropylthiuram Disulfide

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Abstract. $C_{14}H_{28}N_2S_4$, $M_r = 352{\cdot}6$, monoclinic, $P2_1/n$, $a = 6{\cdot}137\,(1)$, $b = 11{\cdot}392\,(2)$, $c = 13{\cdot}446\,(2)$ Å, $\beta = 91{\cdot}98\,(1)^\circ$, $V = 939{\cdot}4\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}30\,(2)$, $D_x = 1{\cdot}25$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 4{\cdot}41$ mm^{-1}, $F(000) = 380$, $T = 293$ K, final $R = 0{\cdot}041$ for 1631 observed reflections. The unusual structural features are (i) the C—S—S—C torsion angle of 180·0° and (ii) the S—S bond length of 2·069 (1) Å which is the longest observed in thiuram disulfides. The molecule is centrosymmetric. In the central part of the molecule, the C—S—S—C moiety adopts a strictly *trans* planar conformation. No intermolecular short contacts are observed.

Introduction. Thiuram disulfides are the immediate oxidation products of dithiocarbamic acids and are known to be formed as one of the products during redox complexation reactions of dithiocarbamates with metal ions like Te$^{\text{IV}}$, Se$^{\text{IV}}$ *etc.* (Bode, Tusche & Wahrhausen, 1962; Fabiani, Spagna, Vaciago & Zambonelli, 1971; Schnabel, Deuten & Klar, 1980). In the course of our investigation on the diisopropyldithiocarbamate complexes of Te$^{\text{IV}}$, we noticed the formation of the thiuram disulfide. The structure determination was carried out to study the structural features of the disulfide, namely the S—S bond and the two torsion angles, C—S—S—C and N—C—S—S. Other structures of thiuram disulfide which have been reported are those of tetramethylthiuram disulfide (Maroy, 1967), tetraethylthiuram disulfide (Karle, Estlin & Britts, 1967), dipyrrolidyl disulfide (Geoffrey, Statham & White, 1983) and bis(4-morpholinethiocarbonyl) disulfide (Rout, Seshasayee & Aravamudan, 1982). For the first time we report a torsion angle of 180° in the free disulfide.

Experimental. The title compound was prepared by the controlled oxidation of sodium diisopropyldithiocarbamate by iodine dissolved in an aqueous solution of potassium iodide. The yellow solid formed was redissolved in ethyl acetate. This on slow evaporation at room temperature gave pale yellow needles, dimensions $0{\cdot}975 \times 0{\cdot}450 \times 0{\cdot}225$ mm; D_m by flotation in acetone/carbon tetrachloride; $P2_1/n$ from systematic absences; Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator; cell parameters by least squares from setting angles of 15 reflections with $60^\circ \leq 2\theta \leq 100^\circ$. 2032 reflections with $2^\circ \leq 2\theta \leq 140^\circ$ and $0 \leq h \leq 7$, $0 \leq k \leq 13$, $-16 \leq l \leq 16$ were collected using ω–2θ scan, two standard reflections measured every hour showed no significant change; correction for Lorentz, polarization and absorption effects, maximum and minimum transmission factors are 0·4301 and 0·1548 respectively; crystal bound by the faces (100), $(0\bar{1}1)$, (012); 1631 reflections with $I > 3\sigma(I)$ used for structure determination; structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986); non-H atoms refined on F with anisotropic and H atoms with isotropic temperature factors; maximum electron density less than $0{\cdot}3$ e Å^{-3}, $R = 0{\cdot}041$, $wR = 0{\cdot}049$, $w = 1/[\sigma^2(F_o) + 0{\cdot}0037|F_o|^2]$, $(\Delta/\sigma)_{\max} = 0{\cdot}008$ and $S = 0{\cdot}8382$. Scattering factors used were those in *SHELXS*86.

Discussion. Table 1 contains the atomic coordinates and the thermal parameters of all non-H atoms.

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