(35·2; 31·6), Σ (233·2; 48·2), δ (233·3; 57·0), α_1 (233·3; 52·6) and α_2 (−0·1; −4·4). These parameters show that the conformation for the *cis* compound is diplanar at the C(2)–C(3) and C(4)–N(13) bonds, distorted towards a half chair at the C(2)–C(3) bond. For the *trans* compound the conformation is envelope at N(13) slightly distorted towards a half-chair at the C(2)–C(3) bond.

The packing in the crystal is entirely due to van der Waals forces.

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1,2,4,5-Tetrabenzylhexahydro-s-tetrazine

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Abstract. $C_{30}H_{32}N_4$, triclinic, $P\bar{1}$, $a = 7\cdot016$ (2), $b = 9\cdot796$ (2), $c = 10\cdot444$ (2) Å, $\alpha = 92\cdot94$ (2), $\beta = 107\cdot37$ (2), $\gamma = 109\cdot18$ (2)°, $D_m = 1\cdot17$ (1), $Z = 1$, $D_c = 1\cdot167$ g cm^{-3}. The structure was solved by direct methods and refined by block-diagonal least squares to $R = 0\cdot063$. The centrosymmetric molecule has a chair conformation, with one of the two independent benzyl groups axial and the other equatorial.

Introduction. The preparation of the title compound has recently been described, together with PMR and CMR spectra and dipole-moment measurements (Baker, Katritzky, Majoral, Martin & Sullivan, 1976).

Crystals suitable for X-ray analysis were grown from petroleum ether (40–60 °C) solutions. Cell parameters were determined by a least-squares refinement of the setting angles of 15 reflections centred on a Syntex $P2_1$ automatic diffractometer with graphite-monochromatized Mo $K\alpha$ radiation.

Preliminary Weissenberg photographs revealed a triclinic lattice. Intensity data were obtained at room temperature from a crystal (0·30 × 0·55 × 0·15 mm) mounted on the diffractometer (Mo $K\alpha$, mono-chromatized, θ–2θ scan, $3° < 2\theta < 52°$). Of the 2487 unique reflections recorded, 1632 with $F_o > 3\sigma(F_o)$ were considered observed and used in the analysis. No absorption correction was applied [μ(Mo $K\alpha$) = 0·65 cm^{-1}].

A statistical analysis of the intensities indicated that the space group was centrosymmetric ($\langle |E| \rangle = 0\cdot761$, $\langle |E^2 - 1| \rangle = 1\cdot021$, $\langle |E^2| \rangle = 1\cdot000$, $\langle |E| \rangle^2 / \langle |E^2| \rangle = 0\cdot580$, $|E| > 1 = 30\cdot20\%$, $|E| > 2\cdot0 = 4\cdot75\%$, $|E| > 3\cdot0 = 0\cdot68\%$) and the space group $P\bar{1}$ with half a molecule in the asymmetric unit was assumed. The structure was determined by the application of direct methods with the weighted multisolution tangent refinement technique (Germain, Main & Woolfson, 1971).

The E map computed with the phases from the set with the highest figure of merit revealed the positions of all but one of the non-hydrogen atoms. This remaining atom was located by successive Fourier syntheses. The

Table 1. *Final coordinates for the non-hydrogen atoms ($\times 10^4$), with estimated standard deviations in parentheses*

	x	y	z
N(1)	1921 (4)	1262 (3)	667 (3)
N(2)	−127 (4)	1348 (3)	−94 (3)
C(1)	1947 (5)	−89 (4)	44 (3)
C(2)	2329 (6)	1314 (4)	2159 (4)
C(3)	4684 (5)	1668 (4)	2879 (3)
C(4)	5429 (8)	629 (5)	3457 (5)
C(5)	7601 (10)	953 (8)	4081 (6)
C(6)	9025 (8)	2293 (10)	4167 (5)
C(7)	8327 (8)	3334 (8)	3588 (5)
C(8)	6153 (7)	3041 (5)	2948 (4)
C(9)	−229 (6)	2785 (4)	278 (4)
C(10)	−1952 (5)	3047 (4)	−852 (4)
C(11)	−1666 (6)	3298 (4)	−2086 (4)
C(12)	−3245 (8)	3507 (5)	−3128 (5)
C(13)	−5113 (8)	3465 (5)	−2947 (5)
C(14)	−5419 (7)	3200 (6)	−1740 (6)
C(15)	−3835 (7)	3000 (5)	−687 (5)

refinement was carried out by block-diagonal least-squares methods and converged to $R = 0.15$ and $R = 0.063$ ($R_w = 0.080$) with isotropic and anisotropic temperature factors, respectively, for the non-hydrogen atoms. The H atoms were positioned geometrically, and readjusted after each cycle; their final isotropic thermal parameters are the final values obtained in the isotropic refinement of the atoms to which they are bonded.

The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ with weights $w = \sin \theta/\lambda$.

The atomic form factors used were those from *International Tables for X-ray Crystallography* (1974).

The computer programs used in this analysis were *MULTAN* (Germain, Main & Woolfson, 1971), on the Univac 1110 computer of the Università di Roma, and *CAOS* (Cerrini & Spagna, 1977), a crystallographic program system for the Hewlett-Packard HP 21MX minicomputer of the CNR Computing Centre in Montelibretti.

Final positional parameters for the non-hydrogen atoms are given in Table 1.*

Discussion. The structure is shown in Fig. 1, together with the numbering of the atoms. The centrosymmetric molecule has a chair conformation, with one of the two independent benzyl groups axial and the other equatorial.

Bond lengths and angles are given in Table 2. The bond lengths of the hexahydrotetrazine ring are in agreement with the accepted values for C—N and N—N bonds. For the latter, the value found [1·452 (5) Å] agrees well with the few literature values for a pure single N—N bond: 1·453 (5) Å by the IR technique (Yamaguchi, Ichishima, Shimanouchi & Mizushima, 1959), and 1·449 (4) Å by electron diffraction analysis (Morino, Iijima & Murata, 1960) for hydrazine; 1·447 (3) Å by X-ray analysis (Ansell & Erickson, 1975) for hexahydro-1,4-dimethyl-s-tetrazine.

The geometry of the phenyl rings is rather inaccurate since they are affected by strong librational motion and the atoms are not well located.
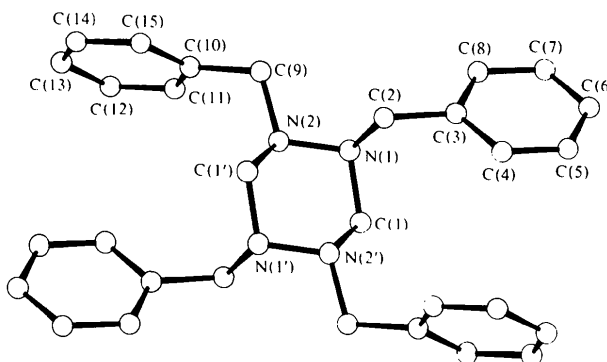

Fig. 1. Perspective view of the molecule. The numbering scheme of the atoms is indicated.

Table 2. *Intramolecular bond lengths (Å) and angles (°), with estimated standard deviations in parentheses*

(a) Distances

N(1)—N(2)	1·452 (5)	C(6)—C(7)	1·363 (10)
N(1)—C(1)	1·452 (5)	C(7)—C(8)	1·393 (9)
N(1)—C(2)	1·494 (5)	C(9)—C(10)	1·517 (6)
N(2)—C(1')	1·471 (5)	C(10)—C(11)	1·385 (6)
N(2)—C(9)	1·472 (5)	C(10)—C(15)	1·369 (6)
C(2)—C(3)	1·507 (7)	C(11)—C(12)	1·381 (8)
C(3)—C(4)	1·378 (6)	C(12)—C(13)	1·367 (7)
C(3)—C(8)	1·383 (8)	C(13)—C(14)	1·364 (8)
C(4)—C(5)	1·384 (11)	C(14)—C(15)	1·386 (9)
C(5)—C(6)	1·342 (14)		

(b) Angles

N(2')—C(1)—N(1)	110·3 (3)	C(5)—C(6)—C(7)	119·5 (7)
C(1)—N(1)—N(2)	105·7 (3)	C(6)—C(7)—C(8)	121·0 (8)
N(1)—N(2)—C(1')	112·3 (3)	C(7)—C(8)—C(3)	119·5 (5)
C(1)—N(1)—C(2)	111·6 (3)	N(2)—C(9)—C(10)	109·6 (4)
N(2)—N(1)—C(2)	114·4 (3)	C(9)—C(10)—C(11)	120·3 (3)
N(1)—N(2)—C(9)	111·6 (4)	C(9)—C(10)—C(15)	120·9 (4)
C(1')—N(2)—C(9)	114·3 (3)	C(11)—C(10)—C(15)	118·8 (4)
N(1)—C(2)—C(3)	109·2 (3)	C(10)—C(11)—C(12)	120·6 (4)
C(2)—C(3)—C(4)	121·1 (5)	C(11)—C(12)—C(13)	120·0 (4)
C(2)—C(3)—C(8)	120·4 (4)	C(12)—C(13)—C(14)	119·8 (6)
C(4)—C(3)—C(8)	118·5 (5)	C(13)—C(14)—C(15)	120·5 (5)
C(3)—C(4)—C(5)	120·6 (6)	C(14)—C(15)—C(10)	120·3 (4)
C(4)—C(5)—C(6)	121·0 (6)		

Table 3. *Torsion angles* (°), *with estimated standard deviations in parentheses*

The convention of Klyne & Prelog (1960) is adopted.

C(1)–N(1)–N(2)–C(1')	59·5 (4)
N(1)–N(2)–C(1')–N(1')	−62·4 (3)
N(2)–C(1')–N(1')–N(2')	58·4 (3)
C(2)–N(1)–N(2)–C(1')	−63·6 (4)
C(2)–N(1)–N(2)–C(9)	66·3 (4)
C(1)–N(1)–N(2)–C(9)	−170·5 (3)
C(1)–N(1)–C(2)–C(3)	76·7 (3)
N(2)–N(1)–C(2)–C(3)	−163·3 (3)
N(1)–C(2)–C(3)–C(4)	−109·7 (5)
N(1)–C(2)–C(3)–C(8)	68·9 (5)
N(1)–N(2)–C(9)–C(10)	159·0 (3)
C(1')–N(2)–C(9)–C(10)	−72·2 (4)
N(2)–C(9)–C(10)–C(11)	−70·1 (4)
N(2)–C(9)–C(10)–C(15)	108·1 (4)

In the tetrazine ring, the angle at C(1) [110·3 (3)°] is very near to the tetrahedral value (109·47°), whilst the angles at the N atoms are considerably different: 105·7 (3)° for N(1) where the benzyl group is axial and 112·3 (3)° for N(2) where the benzyl group is equatorial.

Relevant torsion angles are reported in Table 3. Those in the tetrazine ring are in good agreement with the ideal values for a chair conformation. The non-bonded interactions involving the substituents increase the value of the torsion angle C(2)–N(1)–N(2)–C(9) [66·3 (4)°] and lower that of the torsion angle C(1)–N(1)–N(2)–C(9) [−170·5 (3)°]. Such interactions could help to explain the flattening observed at the N

atoms, which, however, is more pronounced at N(2), where the benzyl group is equatorial. In fact, the angle of the N(2)–C(9) bond with respect to the N(1)–N(2)–C(1') plane is 45·6°, 9·1° lower than the tetrahedral value (54·7°), and the angle of the N(1)–C(2) bond with respect to the C(1)–N(1)–N(2) plane is 49·6°.

No short contacts between adjacent molecules are observed.

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1,2β-Dimethoxycarbonyl-3aα-hydroxy-2α,3,3,8,8aα-pentahydropyrrolo[2,3-*b*]indole

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Abstract. $C_{14}H_{16}N_2O_5$, $P2_1/c$, $a = 10·331$ (6), $b = 11·898$ (6), $c = 15·148$ (8) Å, $\beta = 131·7$ (2)°, $Z = 4$, $d_c = 1·4$ g cm^{-3}. The compound is a photo-oxidation production of a derivative of DL-tryptophan. There is a *cis* junction between the two five-membered rings and the hydroxyl group is *exo* to the ring system.

Introduction. The title compound is a photo-oxidation product of a DL-tryptophan derivative (Nakagawa *et al.*, 1977). The X-ray analysis was performed to determine unambiguously the stereoconfiguration of the molecule at C(9). The numbering system used throughout this paper was chosen arbitrarily. The chemical