(35.2; 31.6), $\Sigma$ (233.2; 48.2), $\delta$ (233.3; 57.0), $\alpha_1$ (233.3; 52.6) and $\alpha_2$ (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.

We thank Professor M. Lora Tamayo and coworkers for suggesting the problem and supplying the material. We also thank the Centro de Proceso de Datos del Ministerio de Educación y Ciencia (Madrid) for allowing us to use the 1108 Univac computer. Most of the computations required in this analysis, were performed with the XRAY system (Stewart, Kundell & Baldwin, 1970).

References


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

BY RICCARDO SPAGNA AND ALESSANDRO VACIAGO

CNR-Laboratorio di Strutturistica Chimica 'Giordano Giacomello', Area della Ricerca di Montelibretti, CP No. 10, 00016 Monterotondo Stazione, Roma, Italy

(Received 19 July 1977; accepted 10 November 1977)

Abstract. C$_{30}$H$_{32}$N$_4$, triclinic, $P\bar{1}$, $a = 7.016$ (2), $b = 9.796$ (2), $c = 10.444$ (2) Å, $\alpha = 92.94$ (2), $\beta = 107.37$ (2), $\gamma = 109.18$ (2)$^\circ$, $D_m = 1.17$ (1), $Z = 1$, $D_e = 1.167$ g cm$^{-3}$. The structure was solved by direct methods and refined by block-diagonal least squares to $R = 0.063$. 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$^\circ$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 x 0.55 x 0.15 mm) mounted on the diffractometer (Mo $K\alpha$, monochromatized, $\theta$-2$\theta$ scan, $3^\circ < 2\theta < 52^\circ$). Of the 2487 unique reflections recorded, 1632 with $F_\sigma > 3\sigma(F_\sigma)$ were considered observed and used in the analysis. No absorption correction was applied [$\mu$(Mo $K\alpha$) = 0.65 cm$^{-1}$].

A statistical analysis of the intensities indicated that the space group was centrosymmetric (|$E$|) = 0.761, |$E^2 - 1$| = 1.021, $|E^2| = 1.000$, |$E$| |$E^2$| = 0.580, |$E$| > 1 = 30.20%, |$E$| > 2 = 4.75%, |$E$| > 3 = 0.68%) 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
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 $\Sigma w([F_o^2 - F_c^2]^2)$ 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.

---

* Tables of anisotropic thermal parameters for non-hydrogen atoms, positional and thermal parameters for hydrogen atoms, and a list of structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33166 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.
Table 3. Torsion angles (°), with estimated standard deviations in parentheses

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

<table>
<thead>
<tr>
<th>Torsion Angle</th>
<th>Value (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C(1)–N(1)–N(2)–C(1')</td>
<td>59.5 (4)</td>
</tr>
<tr>
<td>N(1)–N(2)–C(1')–N(1')</td>
<td>-62.4 (3)</td>
</tr>
<tr>
<td>N(2)–C(1')–N(1')–N(2')</td>
<td>-62.4 (3)</td>
</tr>
<tr>
<td>C(2)–N(1)–N(2)–C(1)</td>
<td>-63.6 (4)</td>
</tr>
<tr>
<td>C(2)–N(1)–N(2)–C(9)</td>
<td>-66.0 (4)</td>
</tr>
<tr>
<td>C(1)–N(1)–N(2)–C(9)</td>
<td>-170.5 (3)</td>
</tr>
<tr>
<td>C(1)–N(1)–N(2)–C(10)</td>
<td>76.7 (3)</td>
</tr>
<tr>
<td>N(2)–C(1)–N(2)–C(9)</td>
<td>-163.3 (3)</td>
</tr>
<tr>
<td>N(1)–C(2)–C(3)–C(4)</td>
<td>-109.7 (5)</td>
</tr>
<tr>
<td>N(1)–C(2)–C(3)–C(8)</td>
<td>68.9 (5)</td>
</tr>
<tr>
<td>N(1)–N(2)–C(9)–C(10)</td>
<td>159.0 (3)</td>
</tr>
<tr>
<td>C(1')–N(2)–C(9)–C(10)</td>
<td>-72.2 (4)</td>
</tr>
<tr>
<td>N(2)–C(9)–C(10)–C(11)</td>
<td>-70.1 (4)</td>
</tr>
<tr>
<td>N(2)–C(9)–C(10)–C(15)</td>
<td>108.1 (4)</td>
</tr>
</tbody>
</table>

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.

This work is part of a joint research project with the School of Chemical Sciences of the University of East Anglia, Norwich, England (Professor A. R. Katritzky), and the specimen was prepared by Dr V. J. Baker.

References


1,2β-Dimethoxy carbonyl-3α-hydroxy-2β,3,3,8,8α-pentahydropyrrolo[2,3-b]indole

By Judith L. Flippen

Naval Research Laboratory, Washington, DC 20375, USA

(Received 16 September 1977; accepted 11 November 1977)

Abstract. C_{14}H_{16}N_{2}O_{5}, P_{2}_1/c, a = 10.331 (6), b = 11.898 (6), c = 15.148 (8) Å, β = 131.7 (2)°, Z = 4, d_{c} = 1.4 g cm^{-3}. The compound is a photo-oxidation product of a derivative of DL-tryptophan (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

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