ETHYL 6-(p-CHLOROPHENYL)-3-PHENYLIMIDAZO[2,1-b]THIAZOL-2-YLACETATE

The short intermolecular contact (C—H(3)···O(24), 2.47 (5) Å [\(\frac{1}{2} + x, \frac{1}{2} - y, z\)], with \(d = 0.23\) Å (Taylor & Kennard, 1982) probably forms a weak hydrogen bond; otherwise intermolecular distances correspond to or are greater than the relevant van der Waals radii.

Thanks are due to Professor S. N. Sawhney of this Department for the crystal sample and to Professor W. H. Watson, Chemistry Department, Texas Christian University, Fort Worth, USA, for his kind help.

References


Structures of Two Isomeric Phenylethanolamine Analogs Containing the Benzobicyclo-[3.2.1]octane Skeleton

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(Received 20 May 1987; accepted 3 September 1987)

Abstract. endo-8-Amino-6,7,8,9-tetrahydro-5,8-methano-5H-benzocyclohepten-9-ol hydrochloride, C\(_{12}\)H\(_{13}\)NO\(_x\)+Cl\(_-\), \(M_f = 225.72\), tetragonal, \(I\_4 / a\), \(a = b = 24.641\) (3), \(c = 7.709\) (1) Å, \(V = 4681\) (1) Å\(^3\), \(Z = 16\), \(D_x = 1.281\) Mg m\(^{-3}\), \(\lambda (Mo\_K\alpha) = 0.71069\) Å, \(\mu = 0.3032\) mm\(^{-1}\), \(F(000) = 1920\), \(T = 297\) K, \(R = 23.032\) for 1543 independent reflections collected. exo-8-Amino-6,7,8,9-tetrahydro-5,8-methano-5H-benzocyclohepten-9-ol, C\(_{12}\)H\(_{13}\)NO, \(M_f = 189.26\), monoclinic, \(P2_1 / c\), \(a = 13.863\) (3), \(b = 6.910\) (2), \(c = 13.437\) (3) Å, \(V = 1018.2\) (5) Å\(^3\), \(Z = 4\), \(D_x = 1.235\) Mg m\(^{-3}\), \(\lambda (Mo\_K\alpha) = 0.71069\) Å, \(\mu = 0.08456\) mm\(^{-1}\), \(F(000) = 408\), \(T = 297\) K, \(R = 0.0378\) for 1340 independent reflections collected. The torsion angles N—C(8)—C(9)—C(9a) of the two isomers are 0108-2701/87/122418-03$01.50


Introduction. In the continuation of our effort in studying the relationship between conformation and biological activities in phenylethanolamine analogs (Grunewald, Reitz, Hallett, Rutledge, Vollmer, Archuleta & Ruth, 1980; Rafferty & Grunewald, 1982; Grunewald, Pleiss & Rafferty, 1982), we obtained endo- and exo-8-amino-6,7,8,9-tetrahydro-5,8-methano-5H-benzocyclohepten-9-ols (1) and (2). They are both conformationally defined analogs of phenyl-
ethanolamine. In order to assign the stereochemistry of the hydroxyl group in the two isomers unambiguously, we carried out X-ray crystallographic studies on each isomer.

Experimental. endo-8-Amino-6,7,8,9-tetrahydro-5,8-methano-5H-benzocyclohepten-9-ol hydrochloride [compound (1)] was obtained by recrystallization from ethanol and ether as colorless tetragonal prisms and exo-8-amino-6,7,8,9-tetrahydro-5,8-methano-5H-benzocyclohepten-9-ol [compound (2)] was recrystallized from methylene chloride as colorless plates. Each crystal was mounted on a glass fiber oriented approximately along [001] for (1) or [100] for (2). Cell constants were determined using 15 centered reflections widely scattered throughout reciprocal space. Full spheres of data out to $2\theta = 45^\circ$ were collected using a Syntex $P_2_1$ diffractometer (Mo Ka, graphite monochromator) with $\theta$-2$\theta$ scan mode. Corrections were made in intensity of two [for (2)] or three [for (1)] standard reflections monitored every 100 measurements. Independent $F_2^2$ data were obtained by merging equivalent reflections. The reflections with $F_2^2 < 0.2\sigma(F_2^2)$ were reset to $F_2^2 = 0.2\sigma(F_2^2)$. All reflections were used in subsequent calculations. The structures were solved by the direct method using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All H atoms were located from the difference Fourier maps. Full-matrix refinement, anisotropic for non-H and isotropic for H atoms. Function minimized $\sum w(F_0^2 - |F_1|)^2$ with $w = 1/\sigma^2(F_0^2)$. The final difference Fourier maps were featureless [±0.17 eÅ$^{-3}$ for (1) and ±0.26 eÅ$^{-3}$ for (2)]. The $\Delta/\sigma$ of the last cycle of least squares was less than 0.13 for (1) and 0.76 for (2). Atomic scattering factors were taken from International Tables for X-ray Crystallography.

![Diagram](image)

Fig. 1. ORTEPII (Johnson, 1976) drawings of (1) and (2). Non-H atoms are displayed as principal ellipses at the 50% probability level, H atoms as small spheres of arbitrary size.
Table 3. Bond lengths (\(\AA\)) and bond angles (°) involving non-H atoms, with their e.s.d.'s in parentheses

<table>
<thead>
<tr>
<th>Compound</th>
<th>Bond length ((\AA))</th>
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<th>Compound</th>
<th>Bond length ((\AA))</th>
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<tbody>
<tr>
<td>N—C(8)</td>
<td>1.495 (2)</td>
<td>C(4)—C(9a)</td>
<td>1.386 (4)</td>
<td>C(6)—C(7)</td>
<td>1.354 (3)</td>
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<tr>
<td>C(9)—C(9a)</td>
<td>1.425 (2)</td>
<td>C(4a)—C(9a)</td>
<td>1.389 (3)</td>
<td>C(7)—C(8)</td>
<td>1.355 (3)</td>
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<tr>
<td>C(1)—C(2)</td>
<td>1.384 (3)</td>
<td>C(4a)—C(9a)</td>
<td>1.400 (3)</td>
<td>C(8)—C(9)</td>
<td>1.520 (2)</td>
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<td></td>
</tr>
<tr>
<td>C(1)—C(9a)</td>
<td>1.395 (3)</td>
<td>C(1)—C(2)—C(3)</td>
<td>1.512 (3)</td>
<td>C(8)—C(9)</td>
<td>1.540 (3)</td>
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<tr>
<td>C(1)—C(3)</td>
<td>1.375 (4)</td>
<td>C(1)—C(2)—C(3)</td>
<td>1.553 (3)</td>
<td>C(9)—C(10)</td>
<td>1.519 (3)</td>
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<tr>
<td>C(1)—C(4)</td>
<td>1.373 (4)</td>
<td>C(1)—C(2)—C(3)</td>
<td>1.553 (3)</td>
<td>C(9)—C(10)</td>
<td>1.517 (3)</td>
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</table>

Discussion. Final atomic parameters are given in Table 2. Bond lengths, bond angles and torsion angles are listed in Table 3.* Views of (1) and (2) with the atomic numbering are presented in Fig. 1.

(1974). All calculations were performed on a Honeywell 66/6000 computer or a VAX 8600 computer at the University of Kansas using programs in the KUDNA system (Takusagawa, 1984). Other experimental details are summarized in Table 1.

Financial support from the National Institutes of Health (Grants HL34193 and GM22988) is gratefully acknowledged.

References


* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44339 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.