

$V = 1678.2 (7) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.236 \text{ Mg m}^{-3}$

Parallelepiped  
 $0.30 \times 0.20 \times 0.15 \text{ mm}$   
 Colorless

**Data collection**

Siemens P4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  
 none  
 1304 measured reflections  
 1100 independent reflections  
 582 observed reflections  
 $[F > 4.0\sigma(F)]$

$\theta_{\max} = 22.5^\circ$   
 $h = 0 \rightarrow 12$   
 $k = 0 \rightarrow 9$   
 $l = -16 \rightarrow 0$   
 3 standard reflections  
 monitored every 47  
 reflections  
 intensity decay: 1.3%

**Refinement**

Refinement on  $F^2$   
 $R = 0.0446$   
 $wR = 0.0529$   
 $S = 1.10$   
 582 reflections  
 103 parameters  
 H atoms riding on associated  
 C atoms except for the  
 carboxyl H atom which  
 was not located  
 $w = 1/[\sigma^2(F) + 0.00070F^2]$

$(\Delta/\sigma)_{\max} = 0.006$   
 $\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = 0.00 \text{ e \AA}^{-3}$   
 Extinction correction:  
 empirical isotropic  
 Extinction coefficient:  
 0.0059  
 Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

	x	y	z	$U_{\text{eq}}$
O(1)	0.0314 (3)	-0.0761 (3)	0.7415 (2)	0.088 (1)
O(2)	0.0253 (3)	0.1223 (3)	0.5785 (2)	0.097 (1)
O(3)	0.1234 (3)	-0.0767 (4)	0.5416 (2)	0.107 (1)
C(1)	0.1504 (3)	0.1337 (4)	0.7372 (2)	0.066 (2)
C(2)	0.0636 (3)	0.0341 (5)	0.7765 (3)	0.063 (2)
C(3)	0.0213 (4)	0.0778 (5)	0.8634 (3)	0.084 (2)
C(4)	0.1181 (4)	0.1118 (5)	0.9246 (3)	0.095 (2)
C(5)	0.1984 (4)	0.2225 (5)	0.8846 (3)	0.099 (2)
C(6)	0.2447 (4)	0.1688 (5)	0.8000 (3)	0.089 (2)
C(7)	0.1958 (4)	0.0700 (5)	0.6549 (3)	0.088 (2)
C(8)	0.1075 (4)	0.0380 (6)	0.5884 (3)	0.084 (2)

Table 2. Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O(1)—C(2)	1.204 (5)	O(2)—C(8)	1.243 (6)
O(3)—C(8)	1.289 (6)	C(1)—C(2)	1.496 (5)
C(1)—C(6)	1.518 (6)	C(1)—C(7)	1.513 (6)
C(2)—C(3)	1.507 (6)	C(3)—C(4)	1.519 (6)
C(4)—C(5)	1.516 (7)	C(5)—C(6)	1.517 (7)
C(7)—C(8)	1.501 (6)	O(3)···O(2 <sup>i</sup> )	2.604 (3)
C(2)—C(1)—C(6)	110.9 (3)	C(2)—C(1)—C(7)	111.1 (3)
C(6)—C(1)—C(7)	112.3 (3)	O(1)—C(2)—C(1)	122.0 (3)
O(1)—C(2)—C(3)	122.0 (4)	C(1)—C(2)—C(3)	116.0 (3)
C(2)—C(3)—C(4)	112.3 (3)	C(3)—C(4)—C(5)	109.8 (3)
C(4)—C(5)—C(6)	111.8 (4)	C(1)—C(6)—C(5)	112.1 (4)
C(1)—C(7)—C(8)	115.2 (3)	O(2)—C(8)—O(3)	122.7 (4)
O(2)—C(8)—C(7)	120.3 (4)	O(3)—C(8)—C(7)	116.9 (4)
C(8)—O(2)···O(3 <sup>i</sup> )	120.9 (2)	C(8)—O(3)···O(2 <sup>i</sup> )	116.4 (2)

Symmetry code: (i)  $-x, -y, 1 - z$ .

The structure was solved by direct methods. Integrated intensities were corrected for Lorentz and polarization effects. Full-matrix least-squares refinement was performed using *SHELXTL/PC* (Sheldrick, 1990). All non-H atoms were refined anisotropically. Although all non-carboxyl H atoms were found in difference maps, they were nonetheless replaced by H

atoms in calculated positions and refined with a riding model, which gave a slightly better goodness of fit.

Data collection, cell refinement, data reduction, structure solution, structure refinement, molecular graphics and preparation of material for publication: *SHELXTL/PC*.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PT1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### *cis*-(±)-1,2,3,6,11,11a-Hexahydro-6-methyl-4*H*-benzo[*b*]quinolizin-4-one

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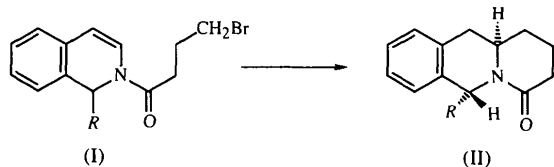
**Abstract**

In the title compound,  $C_{14}H_{17}NO$ , the methyl substituent and the H atom at the ring junction are *cis* and both occupy pseudo-axial positions. The angles at the N atom sum to  $360^\circ$  indicating that there is no pyramidalization of this atom. Both rings of the quinolizidine moiety are in half-chair conformations.

**Comment**

During our studies on the diastereoselective synthesis of alkaloids containing the benzo[*b*]quinolizidine nucleus, it was found that dihydroisoquinolines of the general

structure (I), where *R* is an alkyl or aryl substituent, when treated with tributylstannane in benzene, smoothly undergo radical cyclization with very high diastereoselectivity (Beckwith, Joseph & Mayadunne, 1993).



## Experimental

Crystals of the title compound were recrystallized from dichloromethane/hexane.

### Crystal data

C<sub>14</sub>H<sub>17</sub>NO  
*M<sub>r</sub>* = 215.30  
 Monoclinic  
*C*2/*c*  
*a* = 18.541 (1) Å  
*b* = 8.291 (2) Å  
*c* = 15.366 (1) Å  
 $\beta$  = 97.26 (1)°  
*V* = 2343.2 (6) Å<sup>3</sup>  
*Z* = 8  
*D<sub>r</sub>* = 1.221 Mg m<sup>-3</sup>

Cu K $\alpha$  radiation  
 $\lambda$  = 1.5418 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 42–47°  
 $\mu$  = 0.56 mm<sup>-1</sup>  
*T* = 298 K  
 Multi-faceted  
 0.27 × 0.21 × 0.15 mm  
 Colourless

### Data collection

Rigaku AFC-6R diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction: analytical  
 $T_{\min}$  = 0.87,  $T_{\max}$  = 0.93  
 2017 measured reflections  
 1752 independent reflections  
 1260 observed reflections  
 $|I| > 3\sigma(I)$

$R_{\text{int}}$  = 0.022  
 $\theta_{\text{max}}$  = 60.04°  
 $h = -20 \rightarrow 20$   
 $k = 0 \rightarrow 9$   
 $l = 0 \rightarrow 17$   
 3 standard reflections monitored every 150 reflections  
 intensity decay: 8%

### Refinement

Refinement on *F*<sup>2</sup>  
*R* = 0.047  
 $wR$  = 0.063  
 $S$  = 1.555  
 1253 reflections  
 198 parameters  
 Only coordinates of H atoms refined  
 $w = 1/[\sigma^2(F) + 0.0009F^2]$

$(\Delta/\sigma)_{\text{max}}$  = 0.052  
 $\Delta\rho_{\text{max}}$  = 0.167 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.218 e Å<sup>-3</sup>  
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

The relative stereochemistry of the products was predicted to be as shown by (II) from their <sup>1</sup>H and <sup>13</sup>C NMR spectra and by molecular-mechanics calculations on the transition structures. For (II) with *R* = CH<sub>3</sub>, the present single-crystal X-ray analysis confirms that the newly formed bond is *trans* to the methyl substituent. Electron delocalization between the N atom and the carbonyl centre leads to a short N—C13 distance of 1.343 (3) Å and to atoms C1, C9, C12, C13, N and O being close to coplanar. A further consequence is for the newly formed ring of N, C9, C10, C11, C12 and C13 to assume a half-chair conformation, as shown by the Cremer & Pople (1988) puckering parameters calculated using *RING88* (Cremer, 1988):  $q_2 = 0.374$ ,  $q_3 = -0.310$  Å,  $\varphi_2 = 340.2$ ,  $\theta_2 = 129.6^\circ$ . Bond lengths and angles in this section of the molecule resemble the corresponding dimensions in matrine alkaloids and related compounds (e.g. Ibragimov, Talipov, Tishchenko, Kushmuradov & Aripov, 1978; Ibragimov, Tishchenko, Talipov, Kushmuradov & Aripov, 1981; Chen, Brown & Gonnella, 1986). The central ring (N, C1, C2, C7, C8 and C9) is also in a half-chair conformation ( $q_2 = 0.381$ ,  $q_3 = 0.313$  Å,  $\varphi_2 = 325.1$ ,  $\theta_2 = 50.6^\circ$ ), in this case arising from the aromatic character of the C2—C7 bond. There is no significant pyramidalization at the N atom or C13:  $\theta_N = 1.0$  and  $\theta_C = 1.7^\circ$  (Winkler & Dunitz, 1971; Cieplak, 1994).

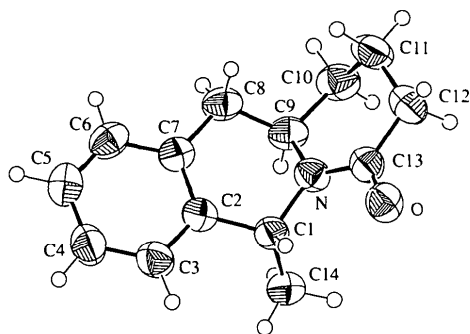


Fig. 1. View of C<sub>14</sub>H<sub>17</sub>NO showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level; H atoms are drawn as small circles of arbitrary radii.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
C1	0.4184 (1)	0.2854 (3)	0.6379 (1)	0.051 (1)
C2	0.3544 (1)	0.4006 (3)	0.6315 (1)	0.050 (1)
C3	0.3341 (1)	0.4652 (4)	0.7080 (2)	0.063 (1)
C4	0.2772 (1)	0.5738 (4)	0.7059 (2)	0.073 (2)
C5	0.2397 (1)	0.6193 (4)	0.6268 (2)	0.070 (2)
C6	0.2593 (1)	0.5557 (3)	0.5507 (2)	0.060 (1)
C7	0.3163 (1)	0.4451 (3)	0.5510 (1)	0.051 (1)
C8	0.3365 (1)	0.3801 (3)	0.4661 (1)	0.060 (1)
C9	0.3843 (1)	0.2294 (3)	0.4793 (1)	0.054 (1)
C10	0.4132 (2)	0.1740 (4)	0.3961 (2)	0.069 (2)
C11	0.4798 (2)	0.2676 (4)	0.3801 (2)	0.073 (2)
C12	0.5387 (2)	0.2413 (4)	0.4555 (2)	0.066 (2)
C13	0.5146 (1)	0.2683 (3)	0.5448 (2)	0.053 (1)
C14	0.4030 (2)	0.1249 (4)	0.6799 (2)	0.067 (2)
N	0.4432 (1)	0.2617 (2)	0.5517 (1)	0.051 (1)
O	0.56041 (8)	0.2916 (2)	0.6090 (1)	0.065 (1)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.518 (3)	C7—C8	1.501 (3)
C1—C14	1.522 (4)	C8—C9	1.530 (4)
C1—N	1.470 (3)	C9—C10	1.518 (4)
C2—C3	1.388 (3)	C9—N	1.481 (3)
C2—C7	1.394 (3)	C10—C11	1.506 (5)
C3—C4	1.384 (4)	C11—C12	1.504 (4)
C4—C5	1.375 (4)	C12—C13	1.513 (4)
C5—C6	1.372 (4)	C13—N	1.343 (3)
C6—C7	1.399 (3)	C13—O	1.232 (3)
C2—C1—C14	113.0 (2)	C7—C8—C9	112.5 (2)
C2—C1—N	110.8 (2)	C8—C9—C10	113.3 (2)
C14—C1—N	111.3 (2)	C8—C9—N	108.1 (2)
C1—C2—C3	118.8 (2)	C10—C9—N	112.4 (2)
C1—C2—C7	121.9 (2)	C9—C10—C11	111.5 (2)
C3—C2—C7	119.3 (2)	C10—C11—C12	109.3 (2)
C2—C3—C4	121.2 (2)	C11—C12—C13	114.1 (2)
C3—C4—C5	119.9 (3)	C12—C13—N	118.2 (2)
C4—C5—C6	119.3 (3)	C12—C13—O	119.8 (2)
C5—C6—C7	122.0 (2)	N—C13—O	122.0 (2)
C2—C7—C6	118.3 (2)	C1—N—C9	114.6 (2)
C2—C7—C8	121.4 (2)	C1—N—C13	119.3 (2)
C6—C7—C8	120.3 (2)	C9—N—C13	126.1 (2)
N—C1—C2—C7	13.2 (3)	C10—C9—N—C13	10.2 (3)
C2—C1—N—C9	-47.0 (3)	C9—C10—C11—C12	61.0 (3)
C1—C2—C7—C8	0.4 (3)	C10—C11—C12—C13	-50.4 (3)
C2—C7—C8—C9	17.4 (3)	C11—C12—C13—N	20.4 (4)
C7—C8—C9—N	-47.4 (3)	C11—C12—C13—O	-161.3 (2)
N—C9—C10—C11	-40.7 (3)	C12—C13—N—C9	0.3 (3)
C8—C9—N—C1	65.3 (2)	O—C13—N—C9	-178.0 (2)

The  $\theta$ -scan width was  $(1.3 + 0.346 \tan \theta)^\circ$ , with  $\theta$ -scan rate  $32^\circ \text{ min}^{-1}$ ; up to four scans on weak reflections and background counts for one quarter of the scan time on each end of every scan were made. H atoms were refined with one common isotropic displacement factor.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *Xtal3.0 DIFDAT*, *ABSORB*, *SORTRF* and *ADDREF* (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *Xtal3.0 CRYLSQ*. Molecular graphics: *Xtal3.0*. Software used to prepare material for publication: *Xtal3.0 BONDLA* and *CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Screw-Chain Structure of 1,10-Phenanthroline Hydrate, C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>·H<sub>2</sub>O

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## Abstract

The X-ray analysis revealed that the title compound is an infinite  $3_1$  screw-chain structure with a repeat distance of 8.453 Å. The O atoms of the water connect to one another around the screw axis to form the core of the chain and the *o*-phenanthroline molecules connect through intermolecular hydrogen bonds to these water molecules. The structure can be regarded as a screw supermolecule assembled by hydrogen bonds and is a displacive modulation of an idealized  $P3_121$  parent structure.

## Comment

*o*-Phenanthroline (*o*-phen) is well known as a bidentate ligand in coordination chemistry. Many metal complexes involving *o*-phen as a ligand have special properties and in order to obtain a better understanding of structures of the transition metal to *o*-phen chelating systems, many authors have investigated the crystal structures of *o*-phen and *o*-phen·H<sub>2</sub>O (Donnay, Donnay & Harding, 1965; Sen, 1974; Nishigaki, Yoshioka & Nakatsu, 1975, 1978). However, no detailed structure determination of *o*-phen·H<sub>2</sub>O has been reported. Moreover, it is of interest to compare the crystal and molecular structure of *o*-phen·H<sub>2</sub>O with that of *o*-phen and to study the effect of hydrogen bonding on the crystal structure. In this paper, an unrestrained crystal structure analysis is reported. The molecular geometry and unit cell are illustrated in Figs. 1 and 2, respectively.