

2,3-Cyclohexanopyrrolo[2,3-*b*]tropone

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Key indicators

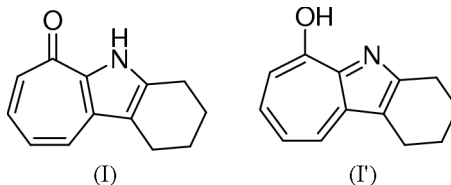
Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.062
wR factor = 0.194
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the solid state, the title compound, $\text{C}_{13}\text{H}_{13}\text{NO}$, exists in its keto form rather than in the enol form. The pyrrole NH group participates in an intermolecular hydrogen bond of the $\text{N}-\text{H}\cdots\text{O}$ type, leading to the formation of infinite chains.

Comment

Pyrrolo[*b*]tropones (Nozoe *et al.*, 1954) are isoelectronic with 8-quinolinols. 3-Phenylpyrrolo[2,3-*b*]tropone (Kubo *et al.*, 2001) gives a dark-purple coloration when treated with aqueous ferric chloride in methanol and the color fades upon addition of mineral acid. This suggests that pyrrolo[2,3-*b*]tropones can be used as an analytical reagent for metal cations. 3-Phenylpyrrolo[2,3-*b*]tropone exists as the keto tautomer in the solid state (Kubo *et al.*, 2001) and we now report the structure of the title compound, (I), in order to determine its form.



In (I), the C–C, C–O and C–N bond lengths of the pyrrolotropone ring are similar to those found in unsubstituted tropone (Barrow *et al.*, 1973), unsubstituted pyrrole (Goddard *et al.*, 1997), and 3-phenylpyrrolo[2,3-*b*]tropone (Kubo *et al.*, 2001). The location of the H1 atom bonded to N1, rather than to the O1 atom, as well as the tropone-like distribution of bonds, allows the unambiguous assignment of the keto form (I) rather than the enol form (I').

The pyrrole NH group participates in an intermolecular hydrogen bond of the $\text{N}-\text{H}\cdots\text{O}$ type: $\text{N1}-\text{H1}$ 0.91 (3), $\text{N1}\cdots\text{O1}^i$ 2.781 (4) and $\text{H1}\cdots\text{O1}^i$ 1.89 (4) Å; symmetry code: (i) $2-x, 1-y, 1-z$. The hydrogen-bonding scheme links molecules into infinite chains orientated along a bisector between the *b* and *c* axes of the unit cell. A similar mode of association was found in the structure of 3-phenylpyrrolo[2,3-*b*]tropone (Kubo *et al.*, 2001).

Experimental

The title compound, (I), was prepared by condensation of 2-hydra-zinotropone with cyclohexanone accompanied by cyclization. The single crystals of (I) were obtained by recrystallization from a mixture of ethyl acetate and hexane.

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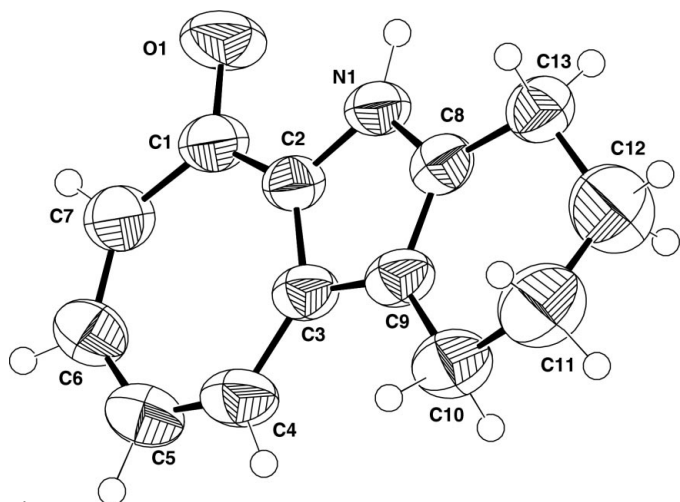


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids (Johnson, 1976).

Crystal data

$C_{13}H_{13}NO$
 $M_r = 199.24$
Monoclinic, $P2_1/a$
 $a = 8.778$ (2) Å
 $b = 15.3778$ (19) Å
 $c = 8.442$ (2) Å
 $\beta = 113.590$ (18)°
 $V = 1044.3$ (4) Å³
 $Z = 4$

$D_x = 1.267$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 16 reflections
 $\theta = 8.9$ – 18.2°
 $\mu = 0.08$ mm⁻¹
 $T = 296$ (2) K
Prism, yellow
 $0.23 \times 0.13 \times 0.13$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω – 2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.962$, $T_{\max} = 0.999$
2677 measured reflections
2519 independent reflections
828 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.059$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -11 \rightarrow 0$
 $k = 0 \rightarrow 20$
 $l = -10 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: 0.6%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.194$
 $S = 0.94$
2519 reflections
140 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0777P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

All C-bound H atoms were included in the refinement at calculated positions as riding models with C–H distances set to 0.93 Å for aromatic and 0.97 Å for CH₂ H atoms. The H atom of the NH group

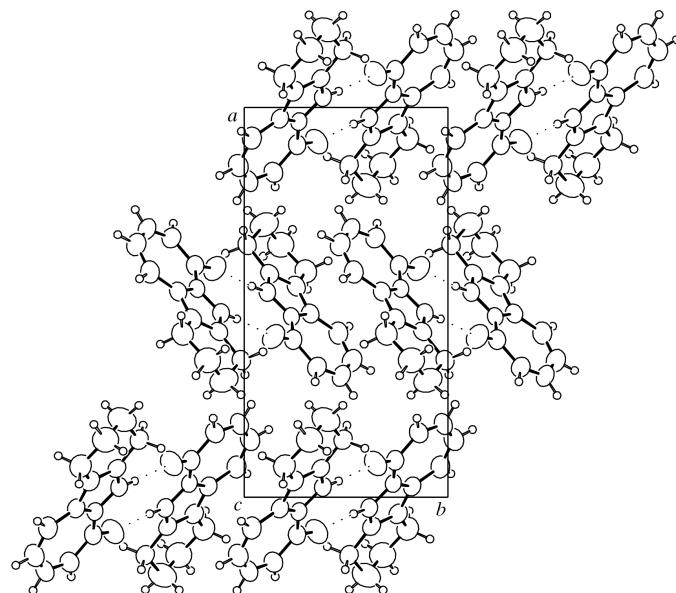


Figure 2
Packing diagram of (I) viewed down the c axis. Dotted lines represent hydrogen bonds.

was located from a difference synthesis and both positional and displacement parameters were refined for this atom.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MOLLEN* (Fair, 1990); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Xtal_GX* (Hall & du Boulay, 1995); software used to prepare material for publication: *SHELXL97*.

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Barrow, M. J., Mills, O. S. & Filippini, G. (1973). *J. Chem. Soc. Chem. Commun.* pp. 66–67.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MOLLEN*. Enraf–Nonius, Delft, The Netherlands.
Goddard, R., Heinemann, O. & Krüger, C. (1997). *Acta Cryst.* **C53**, 1846–1850.
Hall, S. R. & du Boulay, D. (1995). *Xtal_GX*. University of Western Australia, Australia.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kubo, K., Tsujimoto, T. & Mori, A. (2001). *Acta Cryst.* **E57**, o225–227.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Nozoe, T., Kitahara, Y. & Arai, T. (1954). *Proc. Jpn Acad.* **30**, 478–481.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.