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1-Ethyl-3-hydroxy-4,4-dimethyl-2-pyrrolidone

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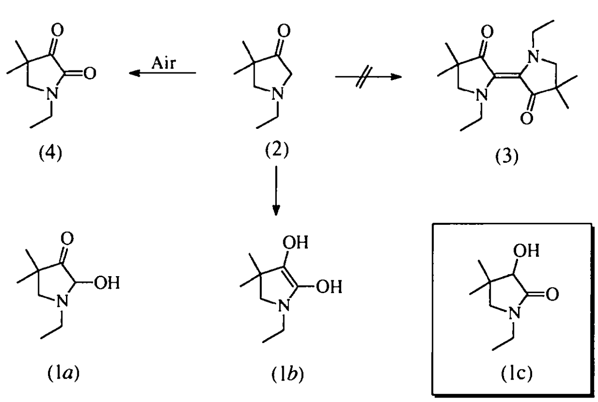
Abstract

The title compound, $C_8H_{15}NO_2$, crystallizes as a racemate. The five-membered ring adopts an envelope conformation with C4 out of the plane. The molecules are linked into pairs by O—H···O hydrogen bonding over an inversion centre.

Comment

1-Ethyl-4,4-dimethyl-3-pyrrolidone, (2) (Fitjer & Lüttke, 1972), is a potential precursor of the dye (3) (Wille & Lüttke, 1971) with the fundamental chromophore of the well known indigo (Klessinger & Lüttke, 1963). The indoxyl analogue (2) is readily oxidized by atmospheric oxygen to give the isatin analogue 1-ethyl-4,4-dimethylpyrrolidin-2,3-dione, (4), but not the desired indigoid dye (3). Oxidation of (2) with potassium hexacyanoferrate(III) under exclusion of oxygen again does not result in (3), but instead a hydroxylated derivative, (1), which can potentially exist as any of three tautomers: the hemiaminal (1*a*), the enediol (1*b*) or the hydroxylactam (1*c*). NMR data indicate one of the two racemates (1*a*) or (1*c*). To distinguish between these, an X-ray study was undertaken.



The X-ray results provide unambiguous evidence for form (1*c*) in the solid state (Fig. 1). The compound crystallizes as a racemate with normal bond lengths and angles. The five-membered ring adopts an envelope conformation in which C4 lies 0.521 (2) Å out of the plane of the other atoms (mean deviation 0.007 Å).

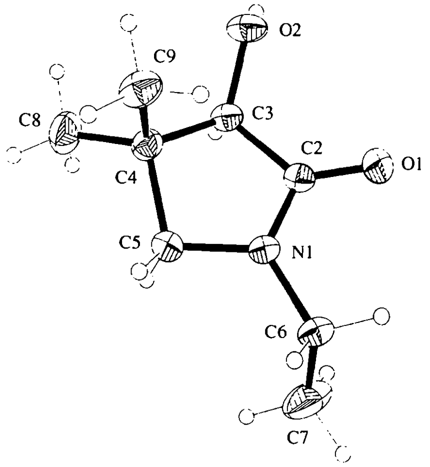


Fig. 1. The title compound in the crystal. Ellipsoids correspond to the 50% probability level and H-atom radii are arbitrary.

Molecules of (1) are linked into dimers by O2—H02···O1($-x$, $-y$, $1-z$) hydrogen bonds, with O···O 2.699 (1), H···O 1.86 Å and O—H···O 174°.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed only four examples of 3-hydroxy-2-pyrrolidones obeying the following conditions: no further annelation, $R < 0.1$, no other double-bonded substituents, only C or H substituents at nitrogen, and coordinates present. Of these, three possessed an essentially identical ring conformation to (1) and all four displayed O—H···O hydrogen bonding with O···O *ca* 2.7 Å (although not exclusively over inversion centres). A typical example is FOBKEM (Hartwig & Born, 1987); the other refcodes are TEFFOZ, YOPPUO and ZUBCAA.

Experimental

The title compound, (1), was obtained by treatment of (2) in CCl_4 with a solution of potassium hexacyanoferrate(III) in 2 *M* aqueous sodium hydroxide under exclusion of oxygen. The mixture was refluxed for 8 h. After separation of the phases, the aqueous phase was extracted with diethyl ether. The organic phases were combined, dried with sodium sulfate, and the solvent removed. The resulting solid was recrystallized by slow evaporation from dichloromethane–pentane as pale yellow rhombs (m.p. 335 K) in 90% yield.

Crystal data

$C_8H_{15}NO_2$
$M_r = 157.21$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 9.0207\,(6)$ Å
$b = 10.1127\,(8)$ Å
$c = 10.5364\,(10)$ Å
$\beta = 111.809\,(6)°$
$V = 892.38\,(13)$ Å^3
$Z = 4$
$D_x = 1.170$ Mg m^{-3}
D_m not measured

Cell parameters from 65 reflections
$\theta = 5.0–12.5°$
$\mu = 0.083$ mm^{-1}
$T = 173\,(2)$ K
Flattened pyramid
$0.45 \times 0.45 \times 0.30$ mm
Pale yellow

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
3733 measured reflections
2048 independent reflections
1648 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$

$\theta_{\rm max} = 27.5°$
$h = -11 \rightarrow 7$
$k = 0 \rightarrow 13$
$l = -13 \rightarrow 13$
3 standard reflections every 247 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.050$
2048 reflections
104 parameters
H atoms: rigid methyls and OH, others riding

$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.0536P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.244$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.192$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.3373 (13)	C3—C4	1.5412 (14)
N1—C5	1.4633 (14)	C4—C5	1.5405 (15)
C2—C3	1.5294 (15)		
C2—N1—C5	112.59 (9)	C5—C4—C3	101.10 (8)
N1—C2—C3	108.20 (9)	N1—C5—C4	103.68 (8)
C2—C3—C4	103.49 (8)		
C5—N1—C2—C3	1.69 (12)	C2—N1—C5—C4	−21.76 (12)
N1—C2—C3—C4	19.05 (11)	C3—C4—C5—N1	31.41 (11)
C2—C3—C4—C5	−30.37 (10)		

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1349). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.

Fait, J. (1991). *XSCANS Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Fitjer, L. & Lüttke, W. (1972). *Chem. Ber.* **105**, 907–918.

Hartwig, W. & Born, L. (1987). *J. Org. Chem.* **52**, 4352–4358.

Klessinger, M. & Lüttke, W. (1963). *Tetrahedron*, **19**, Suppl. **2**, 315–335.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1994). *XP. Interactive Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Wille, E. & Lüttke, W. (1971). *Angew. Chem.* **83**, 853–854; *Angew. Chem. Int. Ed. Engl.* **10**, 803–804.

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A New Modification of Bis(diphenylphosphino)methane Diselenide

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Abstract

In the title compound, $C_{25}H_{22}P_2Se_2$ (dppmSe$_2$), the methylene C atom lies on a crystallographic twofold axis, in contrast to the previously known modification, which had no imposed symmetry. The conformation is expressed by the torsion angle Se—P···P—Se 148.12 (7)°. Bond lengths and angles are similar to those in other dppmX_2 molecules.

Comment

Bis(diphenylphosphino)methane (dppm) and its dichalcogenide derivatives (with O, S, Se) are well known compounds (Grim & Walton, 1980, and references therein). Structures are known for dppm (Schmidbaur *et al.*, 1988), dppmO$_2$ (Antipin *et al.*, 1980), dppmS$_2$ (Carmalt *et al.*, 1996), and dppmSe$_2$ (isostructural with dppmS$_2$) (Carroll & Titus, 1977), and also for the fluorinated derivative $(Ph_2PS)_2CF_2$ (dppfmS$_2$) (Jones & Bembenek, 1996).

We have fortuitously obtained a new modification of dppmSe$_2$, (1), crystallizing in the space group *C*2/*c*

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