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4,4,5,5-Tetramethyl-2-(*p*-pyridyl)-imidazoline-1-oxyl 3-oxide

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.152
Data-to-parameter ratio = 22.2

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

The title compound, $C_{12}H_{16}N_3O_2$, has axial twofold symmetry and features a twist of 23.31 (8)° between the five- and six-membered rings. The N—O bond distance is 1.2777 (13) Å.

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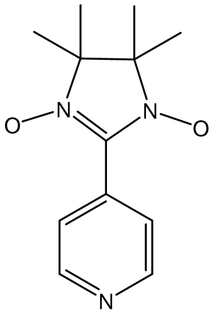
Comment

Interest in the the organic radical compound, (I), arises as a result of the interesting magnetic properties exhibited by its metal complexes (*e.g.* Fettouhi *et al.*, 1999). The structure of the title compound was solved and refined in the monoclinic space group *C*2/*c* so that a crystallographic twofold axis bisects the molecule, with atoms N1, C3 and C4 lying on the axis (Fig. 1). This symmetry imposes equivalence on the N—O bonds and on the C4—N bonds (Table 1). To investigate whether there was ordering in the structure, leading to inequivalent N—O bond distances, for example, refinement was also performed in the space group *Cc*. This revealed equivalence in the N—O bonds [1.275 (4) and 1.281 (4) Å] but non-sensible variations in the N—C bond distances of the pyridine ring. Furthermore, the equivalence in the above-mentioned geometric parameters matches that found for related structures having the 4,4,5,5-tetramethylimidazoline-1-oxyl 3-oxide entity (Sugano *et al.*, 1999). Thus, results for the high-symmetry structure are presented here. The five- and six-membered rings are each essentially planar and the N2—C4—C3—C2 torsion angle is 23.31 (8)°.



(I)

Experimental

A suspension of 2,3-bis(hydroxylamino)-2,3-dimethylbutane (1.48 g) and pyridine-4-carboxaldehyde (1 ml) in methanol (20 ml) was stirred for 24 h and then evaporated to dryness *in vacuo*. The residue was dissolved in benzene (30 ml) and stirred for 5 min with lead dioxide (10 g). After filtration, the deep-blue solution was evaporated. Single crystals were obtained by slow evaporation of a concentrated benzene solution at room temperature.

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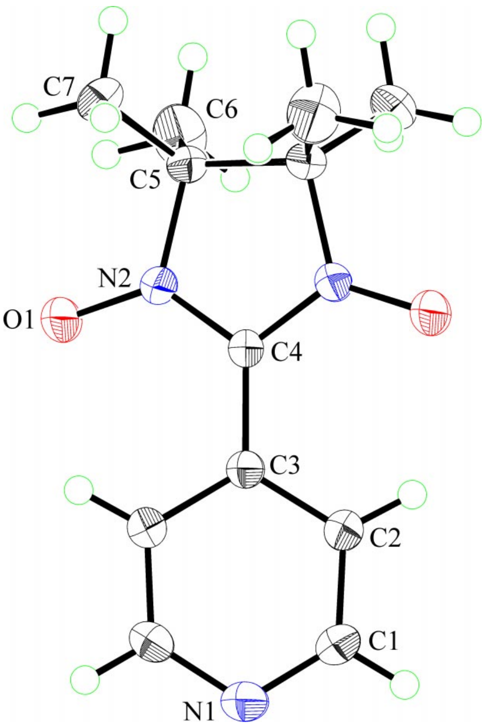

Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level (Johnson, 1976). The unlabeled half of the molecule is generated by the symmetry operator ($1 - x, y, \frac{1}{2} - z$).

Crystal data

$C_{12}H_{16}N_3O_2$
$M_r = 234.28$
Monoclinic, $C2/c$
$a = 11.0922$ (5) Å
$b = 10.3891$ (5) Å
$c = 10.8714$ (6) Å
$\beta = 107.632$ (2)°
$V = 1193.94$ (10) Å^3
$Z = 4$

$D_x = 1.303$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1882 reflections
$\theta = 2.8$–30.0°
$\mu = 0.09$ mm^{-1}
$T = 223$ (2) K
Block, brown
0.31 × 0.26 × 0.26 mm

Data collection

Bruker AXS SMART CCD diffractometer
ω scans
Absorption correction: none
4911 measured reflections
1753 independent reflections

1476 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\rm max} = 30.0°$
$h = -15 \rightarrow 15$
$k = -14 \rightarrow 11$
$l = -13 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.152$
$S = 1.06$
1753 reflections
79 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 0.5851P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.46$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N2	1.2777 (13)	N2—C5	1.5021 (14)
N1—C1	1.3358 (16)	C1—C2	1.3829 (17)
N2—C4	1.3444 (13)		
C1—N1—C1$^{\rm i}$	116.19 (15)	O1—N2—C5	120.30 (10)
O1—N2—C4	126.39 (11)	C4—N2—C5	113.20 (10)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

The C—H atoms were included in the riding model approximation, with C—H distances of 0.94 Å (0.97 Å for methyl), $U_{\rm iso}$(phenyl H) = $1.2U_{\rm eq}$(parent atom) and $U_{\rm iso}$(methyl H) = $1.5U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHEXLTL*.

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