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2,4,4-Trimethyl-2-phenyl-3-pentanone oxime

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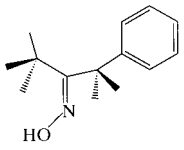
The title compound, $C_{14}H_{21}NO$, has two molecules in the asymmetric unit. Each molecule forms hydrogen-bonded dimers about inversion centers *via* O—H···N hydrogen bonds between oxime groups. The N—O distances in the oxime groups are 1.4160 (15) and 1.4131 (14) Å.

Comment

We had previously carried out spectral and kinetic measurements on a series of persistent iminoxyl radicals (Eisenhauer, Wang, Brown *et al.*, 1997). This study of the title compound, (I), was conducted in order to unequivocally establish the geometry about the C═N—O group, and to see if there would be any angular distortions induced by the bulky groups (*i.e.* ^{t}Bu and cumyl) surrounding the oxime group. The angle in question in both molecules, C2—C3—C4 at 123.39 (11)° and C12—C13—C14 at 123.85 (11)°, is similar, and not very much larger than that expected for an sp^2-hybridized C atom. Therefore, we conclude that neither molecule exhibits large distortions.



(I)

The two molecules constituting the asymmetric unit contain similar bond distances and angles, as is partly evident in Table 1. In both of these molecules, the oxime –C(═NOH)– group is staggered with respect to two of the methyl groups at the ^{t}Bu end of the molecule, with the H atom on each O atom pointing away from this ^{t}Bu group and almost eclipsed with respect to one of the methyl groups on the other end (Fig. 1). Additionally, in both molecules the oxime OH is located *anti* with respect to the larger cumyl group, as expected from steric considerations. The two conformers also contain short intramolecular H···H contacts, namely H1*B*···H5*C* and H11*B*···H15*C* of 1.97 (3) and 2.07 (3) Å, respectively. These H atoms are located on different methyl groups and this distance is noteworthy as non-bonding H···H contacts are normally about twice the van der Waals radius of H or 2.4 Å (Jeffrey & Saenger, 1991).

The crystal packing is assisted by hydrogen bonding (Table 2). These are conventional, almost-linear, hydrogen bonds from the H atom on the OH groups to the lone pair on the N atoms in the inversion-related molecule. This is a pattern usually observed in other oxime crystal structures (Hamilton, 1961; Bertolasi *et al.*, 1982).

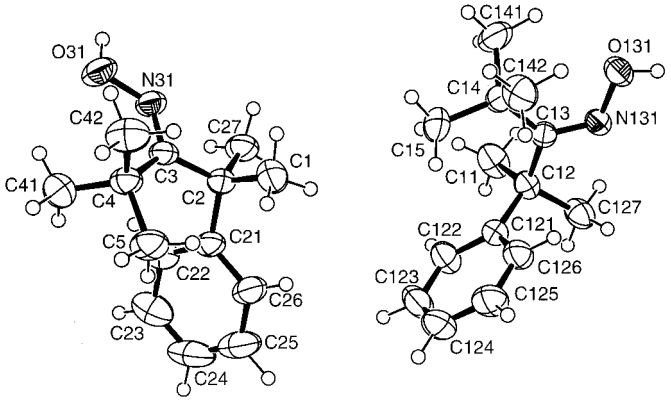


Figure 1
View of the two independent molecules of (I) with 50% probability displacement ellipsoids.

Experimental

The title compound was prepared as reported previously (Eisenhauer, Wang, Labaziewicz *et al.*, 1997). Crystals melting at 455–457 K were obtained by allowing the slow concentration of a benzene solution of (I) in a volumetric flask over a period of five years.

Crystal data

$C_{14}H_{21}NO$
M_r = 219.32
Triclinic, $P\overline{1}$
a = 6.2605 (7) Å
b = 12.0422 (8) Å
c = 18.193 (2) Å
α = 105.689 (7)°
β = 95.477 (9)°
γ = 95.672 (7)°
V = 1303.3 (2) Å^3
Z = 4
D_x = 1.118 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14–16°
μ = 0.069 mm^{-1}
T = 293 (2) K
Block, white
0.40 × 0.40 × 0.25 mm

Data collection

Enraf–Nonius TurboCAD4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.95, T_{max} = 0.98
5052 measured reflections
4591 independent reflections
3606 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 24.98°
$h = 0 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 250 min
intensity decay: 5%

Refinement

Refinement on F^2
$R(F)$ = 0.038
$wR(F^2)$ = 0.105
S = 1.014
4591 reflections
458 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.24P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.037 (3)

Table 1
Selected geometric parameters (Å, °).

O31—N31	1.4160 (15)	O131—N131	1.4131 (14)
N31—C3	1.2811 (17)	N131—C13	1.2770 (17)
C2—C3	1.5428 (19)	C12—C13	1.5451 (19)
C3—C4	1.5490 (19)	C13—C14	1.5494 (18)
C3—N31—O31	115.83 (11)	C13—N131—O131	116.25 (11)
N31—C3—C2	112.31 (12)	N131—C13—C12	112.05 (11)
N31—C3—C4	124.21 (12)	N131—C13—C14	123.97 (12)
C2—C3—C4	123.39 (11)	C12—C13—C14	123.85 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O31—H31···N31^{i}	0.92 (2)	1.97 (2)	2.8598 (18)	163.4 (19)
O131—H131···N131^{ii}	0.93 (2)	1.91 (2)	2.7973 (15)	160 (2)

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $-x, 2-y, 2-z$.

The C—H distances and U_{iso} values for the refined H atoms are in the ranges 0.94 (2)–1.04 (2) Å and 0.058 (5)–0.106 (7) Å^2, respectively.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for *Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1259). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Bertolasi, V., Gilli, G. & Veronese, A. C. (1982). *Acta Cryst.* B**38**, 502–511.
Eisenhauer, B. M., Wang, M., Brown, R. E., Labaziewicz, H., Ngo, M., Kettinger, K. W. & Mendenhall, G. D. (1997). *J. Phys. Org. Chem.* **10**, 737–746.
Eisenhauer, B. M., Wang, M., Labaziewicz, H., Ngo, M. & Mendenhall, G. D. (1997). *J. Org. Chem.* **62**, 2050–2053.
Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1998). *WinGX*. University of Glasgow, Scotland.
Hamilton, W. C. (1961). *Acta Cryst.* **14**, 95–100.
Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.
Jeffrey, G. A. & Saenger, W. (1991). In *Hydrogen Bonding in Biological Structures*. Berlin: Springer.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.