Crystal structure of 2-(3,4-dimethylphenyl)-2-cyclohexene-1-one oxime, $C_{14}H_{17}NO$

C. Meenakshi^I, V. Ramamoorthy^I, S. Muthusubramanian^I, S. Sivasubramanian^I, T. Ziegler^{II} and J. Lex^{*,III}^I Madurai Kamaraj University, School of Chemistry, Madurai, 625021, India^{II} University of Tübingen, Institute of Organic Chemistry, D-72076, Tübingen, Germany^{III} University of Cologne, Institute of Organic Chemistry, Greinstrasse 4, D-50939 Cologne, Germany

Received August 1, 2001, CCDC-No. 1267713

Abstract

$C_{14}H_{17}NO$, triclinic, $P\bar{1}$ (No. 2), $a = 6.731(1)$ Å, $b = 8.757(1)$ Å, $c = 11.348(1)$ Å, $\alpha = 67.69(1)^\circ$, $\beta = 80.71(1)^\circ$, $\gamma = 79.29(1)^\circ$, $V = 605.0$ Å³, $Z = 2$, $R_g(F) = 0.041$, $wR_{ref}(F^2) = 0.117$, $T = 293$ K.

Source of material

To a mixture of 1-(3,4-dimethyl phenyl)-cyclohexene (62 g), *n*-butyl nitrite (35 g) in glacial acetic acid (65 ml) kept at 258 K, a cold solution of 40 ml of hydrochloric acid was added dropwise. The stirring was continued for a further period of 30 minutes and treated with 100 ml of ice cold methanol. The precipitate was removed by filtration, washed twice with ice cold methanol (25 ml) and dried to afford 1-(3,4-dimethylphenyl)-1-chloro-2-nitrosocyclohexane (57 g, 68%); mp 288 K (dec.).

The above nitroschloride (28 g) was suspended in pyridine (60 ml) and warmed to 343 K whereupon the solid dissolved. The mixture was kept at 343 K for a period of 10 minutes, cooled and acidified with dilute hydrochloric acid. The resultant solid was filtered, washed with water and crystallized from ethanol to give 2-(3,4-dimethylphenyl)-2-cyclohexene-1-one oxime (17 g, 71%); mp 428 K.

Discussion

The corresponding molecule parameters referring to those found in the two preceding derivatives [1,2] have comparable values: dihedral angles from 2.4° up to 56.5° , a twist angle between the two carbon rings of 45.5° and N...H hydrogen bonds in the dimer with N...H distances of 1.99 Å.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size $0.15 \times 0.15 \times 0.15$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.74 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, ϕ/ω
$2\theta_{\text{max}}$:	53.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5012, 2615
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2149
$N(\text{param})_{\text{refined}}$:	213
Programs:	SHELXS-97 [1], SHELXL-97 [2], SCHAKAL 99 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3)	2i	0.232(2)	0.903(2)	-0.123(1)	0.052(4)
H(4A)	2i	0.060(3)	0.821(2)	0.088(2)	0.072(5)
H(4B)	2i	0.199(3)	0.947(2)	0.086(2)	0.069(5)
H(5A)	2i	0.282(2)	0.586(2)	0.196(2)	0.071(5)
H(5B)	2i	0.259(3)	0.717(2)	0.273(2)	0.077(5)
H(6A)	2i	0.610(2)	0.616(2)	0.238(2)	0.060(4)
H(6B)	2i	0.572(3)	0.811(2)	0.165(2)	0.077(5)
H(7A)	2i	0.492(3)	0.644(2)	-0.520(2)	0.083(5)
H(7B)	2i	0.292(3)	0.674(2)	-0.429(2)	0.091(6)
H(7C)	2i	0.378(3)	0.821(3)	-0.553(2)	0.101(7)
H(8A)	2i	0.843(3)	0.703(3)	-0.592(2)	0.104(7)
H(8B)	2i	0.973(4)	0.848(3)	-0.607(2)	0.126(8)
H(8C)	2i	0.741(4)	0.888(3)	-0.646(2)	0.121(8)
H(2')	2i	0.342(2)	0.711(2)	-0.242(1)	0.048(3)
H(5')	2i	0.998(3)	0.864(2)	-0.405(2)	0.065(4)
H(6')	2i	0.866(2)	0.845(2)	-0.195(2)	0.059(4)
HO	2i	1.015(3)	0.477(2)	0.102(2)	0.088(6)

* Correspondence author (e-mail: johann.lex@uni-koeln.de)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	2i	0.6250(2)	0.6839(1)	0.0429(1)	0.0378(6)	0.0439(6)	0.0361(6)	−0.0044(5)	−0.0021(4)	−0.0150(5)
C(2)	2i	0.5014(2)	0.7685(1)	−0.0665(1)	0.0401(6)	0.0399(6)	0.0365(6)	−0.0018(4)	−0.0034(4)	−0.0136(5)
C(3)	2i	0.3110(2)	0.8392(2)	−0.0462(1)	0.0451(7)	0.0488(7)	0.0443(7)	0.0045(5)	−0.0065(5)	−0.0161(5)
C(4)	2i	0.2059(2)	0.8351(2)	0.0817(1)	0.0446(7)	0.0630(9)	0.0534(8)	0.0067(6)	0.0008(6)	−0.0262(6)
C(5)	2i	0.3117(2)	0.7010(2)	0.1908(1)	0.0504(8)	0.072(1)	0.0428(7)	0.0024(6)	0.0034(6)	−0.0218(7)
C(6)	2i	0.5402(2)	0.7029(2)	0.1686(1)	0.0497(7)	0.0671(9)	0.0411(7)	0.0011(6)	−0.0050(5)	−0.0257(6)
C(1')	2i	0.5895(2)	0.7774(1)	−0.1979(1)	0.0416(6)	0.0392(6)	0.0352(6)	0.0028(4)	−0.0053(4)	−0.0102(4)
C(2')	2i	0.4759(2)	0.7461(2)	−0.2766(1)	0.0422(6)	0.0467(7)	0.0429(6)	0.0023(5)	−0.0071(5)	−0.0158(5)
C(3')	2i	0.5493(2)	0.7566(2)	−0.4012(1)	0.0551(7)	0.0452(7)	0.0406(6)	0.0086(5)	−0.0124(5)	−0.0163(5)
C(4')	2i	0.7445(2)	0.7997(2)	−0.4495(1)	0.0594(8)	0.0522(7)	0.0365(6)	0.0044(6)	−0.0017(5)	−0.0128(5)
C(5')	2i	0.8571(2)	0.8318(2)	−0.3713(1)	0.0500(7)	0.0617(8)	0.0425(7)	−0.0067(6)	0.0027(6)	−0.0119(6)
C(6')	2i	0.7824(2)	0.8216(2)	−0.2479(1)	0.0469(7)	0.0522(7)	0.0394(6)	−0.0068(5)	−0.0041(5)	−0.0123(5)
C(7')	2i	0.4192(3)	0.7219(3)	−0.4818(2)	0.074(1)	0.079(1)	0.0577(9)	0.0041(9)	−0.0198(8)	−0.0351(9)
C(8')	2i	0.8327(3)	0.8109(3)	−0.5833(2)	0.087(1)	0.095(1)	0.0439(8)	−0.005(1)	0.0064(8)	−0.0269(9)
N	2i	0.7965(1)	0.5996(1)	0.02391(9)	0.0371(5)	0.0523(6)	0.0361(5)	−0.0011(4)	−0.0066(4)	−0.0125(4)
O	2i	0.9015(1)	0.5252(1)	0.13440(9)	0.0415(5)	0.0763(7)	0.0408(5)	0.0056(4)	−0.0114(4)	−0.0170(4)

References

1. Meenakshi, C.; Ramamoorthy, V.; Muthusubramanian, S.; Sivasubramanian, S.; Ziegler, T.; Lex, J.: Crystal structure of 2-phenyl-2-cyclohexene-1-one oxime, C₁₂H₁₃NO. *Z. Kristallogr. NCS* **216** (2001) 649–650.
2. Meenakshi, C.; Ramamoorthy, V.; Muthusubramanian, S.; Sivasubramanian, S.; Ziegler, T.; Lex, J.: Crystal structure of 2-(4-methoxyphenyl)-2-cyclohexene-1-one oxime, C₁₃H₁₅NO₂. *Z. Kristallogr. NCS* **216** (2001) 651–652.
3. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Keller, E.: SCHAKAL 99. University of Freiburg, Germany 1999.