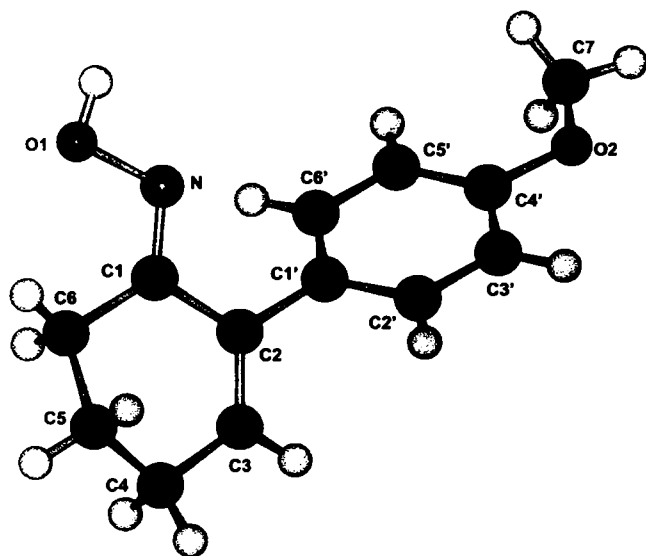


Crystal structure of 2-(4-methoxyphenyl)-2-cyclohexene-1-one oxime, $C_{13}H_{15}NO_2$

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. 1267712



Abstract

$C_{13}H_{15}NO_2$, triclinic, $P\bar{1}$ (No. 2), $a = 6.493(1)$ Å, $b = 9.195(1)$ Å, $c = 10.225(1)$ Å, $\alpha = 70.60(1)^\circ$, $\beta = 85.80(1)^\circ$, $\gamma = 83.49(1)^\circ$, $V = 571.7$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.038$, $wR_{\text{ref}}(F^2) = 0.108$, $T = 293$ K.

Source of material

A mixture of 1-(4-methoxyphenyl)-cyclohexene (31 g), 24 ml of hydrochloric acid in propionic acid (75 ml) was kept at 273 K and treated dropwise with a cold solution of *n*-butyl nitrite (18 g) in propionic acid (10 ml). 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-phenyl-1-chloro-2-nitrosocyclohexene (26 g, 62%); mp 362 K – 363 K.

The above nitrosocyclohexene (30 g) was suspended in pyridine (60 ml) and warmed to 353 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-(4-methoxyphenyl)-2-cyclohexene-1-one oxime (20 g, 74%); mp 442 K – 443 K.

Discussion

The puckered six-membered ring has – similar to the findings in the preceding compound [1] – dihedral angles between 3.6° and 55.1° . The torsion angle between the two ring systems is 52.8° , the hydrogen bonds forming the dimer have bond lengths of 1.96 Å.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size $0.15 \times 0.15 \times 0.20$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.85 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ/ω
$2\theta_{\text{max}}$:	54°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4824, 2478
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2166
$N(\text{param})_{\text{refined}}$:	205
Programs:	SHELXS-97 [2], SHELXL-97 [3], SCHAKAL 99 [4]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2i	−0.130(2)	0.171(2)	0.904(2)	0.054(4)
H(4A)	2i	−0.102(3)	0.320(2)	0.653(2)	0.068(5)
H(4B)	2i	−0.307(3)	0.373(2)	0.728(2)	0.083(5)
H(5A)	2i	−0.113(2)	0.591(2)	0.584(2)	0.059(4)
H(5B)	2i	−0.155(2)	0.590(2)	0.741(2)	0.060(4)
H(6A)	2i	0.190(2)	0.631(2)	0.681(2)	0.056(4)
H(6B)	2i	0.237(2)	0.478(2)	0.640(2)	0.059(4)
H(7A)	2i	0.621(3)	−0.120(2)	1.457(2)	0.063(4)
H(7B)	2i	0.553(3)	−0.260(2)	1.415(2)	0.070(5)
H(7C)	2i	0.513(3)	−0.260(2)	1.569(2)	0.073(5)
H(2')	2i	−0.127(2)	0.192(2)	1.153(1)	0.050(4)
H(3')	2i	−0.034(2)	0.014(2)	1.375(2)	0.054(4)
H(5')	2i	0.560(2)	−0.023(2)	1.222(2)	0.055(4)
H(6')	2i	0.468(2)	0.150(2)	1.006(1)	0.049(4)
HO(1)	2i	0.524(3)	0.600(2)	0.904(2)	0.071(5)

* 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	0.2038(2)	0.4342(1)	0.8491(1)	0.0340(5)	0.0352(5)	0.0348(5)	−0.0029(4)	−0.0048(4)	−0.0105(4)
C(2)	2i	0.0932(2)	0.2957(1)	0.9194(1)	0.0387(6)	0.0355(5)	0.0376(5)	−0.0055(4)	−0.0034(4)	−0.0128(4)
C(3)	2i	−0.0654(2)	0.2670(2)	0.8574(1)	0.0495(7)	0.0452(7)	0.0475(7)	−0.0143(5)	−0.0083(5)	−0.0141(5)
C(4)	2i	−0.1506(2)	0.3709(2)	0.7220(2)	0.0543(8)	0.0624(8)	0.0526(7)	−0.0148(6)	−0.0190(6)	−0.0162(6)
C(5)	2i	−0.0803(2)	0.5319(2)	0.6815(1)	0.0466(7)	0.0525(7)	0.0449(7)	−0.0027(5)	−0.0162(5)	−0.0100(6)
C(6)	2i	0.1501(2)	0.5278(2)	0.7023(1)	0.0446(6)	0.0447(6)	0.0357(6)	−0.0069(5)	−0.0071(5)	−0.0080(5)
C(7)	2i	0.5167(2)	−0.1934(2)	1.4749(2)	0.0565(8)	0.0558(8)	0.0538(8)	−0.0035(7)	−0.0151(6)	−0.0016(7)
C(1')	2i	0.1591(2)	0.1888(1)	1.0580(1)	0.0400(6)	0.0307(5)	0.0374(5)	−0.0072(4)	−0.0028(4)	−0.0117(4)
C(2')	2i	0.0131(2)	0.1466(1)	1.1679(1)	0.0360(6)	0.0429(6)	0.0434(6)	−0.0041(5)	−0.0017(4)	−0.0130(5)
C(3')	2i	0.0688(2)	0.0444(2)	1.2960(1)	0.0423(6)	0.0502(7)	0.0396(6)	−0.0095(5)	0.0039(5)	−0.0122(5)
C(4')	2i	0.2731(2)	−0.0194(1)	1.3177(1)	0.0455(6)	0.0348(5)	0.0367(6)	−0.0084(4)	−0.0046(4)	−0.0080(4)
C(5')	2i	0.4209(2)	0.0203(1)	1.2103(1)	0.0372(6)	0.0386(6)	0.0470(6)	−0.0023(5)	−0.0023(5)	−0.0099(5)
C(6')	2i	0.3626(2)	0.1246(1)	1.0822(1)	0.0395(6)	0.0388(6)	0.0404(6)	−0.0054(5)	0.0031(4)	−0.0099(5)
N	2i	0.3405(1)	0.4656(1)	0.91795(9)	0.0381(5)	0.0348(5)	0.0372(5)	−0.0097(4)	−0.0053(4)	−0.0051(4)
O(1)	2i	0.4386(1)	0.5987(1)	0.84108(9)	0.0464(5)	0.0433(5)	0.0435(4)	−0.0181(4)	−0.0100(4)	−0.0005(4)
O(2)	2i	0.3127(1)	−0.1193(1)	1.44860(9)	0.0527(5)	0.0543(5)	0.0402(5)	−0.0060(4)	−0.0067(4)	−0.0004(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. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
4. Keller, E.: SCHAKAL 99. University of Freiburg, Germany 1999.