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3-Hydroxyimino-1-methyl-5-phenylcyclohexane-1-carbonitrile

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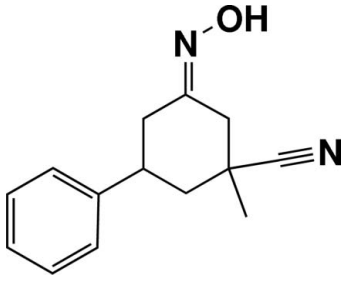
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.002 Å; R factor = 0.059; wR factor = 0.165; data-to-parameter ratio = 22.6.

In the title molecule, $C_{14}H_{16}N_2O$, the cyclohexane ring adopts a chair conformation. The oxime group has an equatorial orientation. The cyano group and the methyl group have axial and equatorial orientations, respectively. The phenyl ring has an equatorial orientation. The structure is stabilized by intermolecular O—H···N hydrogen bonds and intramolecular C—H···O interactions.

Related literature

For related literature, see: Gurumani, *et al.* (1997); Pandiarajan *et al.* (1984).



Experimental

Crystal data

$C_{14}H_{16}N_2O$
M_r = 228.29
Monoclinic, $P2_1/n$
a = 6.6394 (2) Å
b = 14.6658 (5) Å
c = 12.6653 (3) Å
β = 95.146 (2)°
V = 1228.28 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 160 (1) K
0.25 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
33562 measured reflections
3588 independent reflections
2568 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.165
S = 1.06
3588 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.97 (3)	1.88 (3)	2.7814 (17)	154 (2)
C2—H2B···O1	0.97	2.30	2.7070 (18)	104

Symmetry code: (i) $-x, -y+1, -z$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

The data collection was carried out by Dr A. Linden of the Institute of Organic Chemistry at the University of Zurich. The help is gratefully acknowledged by AT.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2282).

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supplementary materials

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3-Hydroxyimino-1-methyl-5-phenylcyclohexane-1-carbonitrile

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Comment

Related literature were reported by Pandiarajan *et al.*, 1984 and Gurumani *et al.*, 1997.

The present X-ray diffraction study was undertaken to determine how the conformation of the system is affected by the substitution of an oxime moiety at position 3, cyano and methyl groups at position 1 and phenyl ring at position 5 of the cyclohexane. The molecular structure of (I), with atomic numbering scheme is shown in Fig. 1. The cyclohexane ring adopts a chair conformation. The oxime moiety at position 1 is planar and has an equatorial orientation. The cyano group and the methyl group at position 1 have an axial and equatorial orientations, respectively. The phenyl ring at position 5 has an equatorial orientation. The mean plane of atoms C2/C3/C5/C6 and phenyl ring make dihedral angle of 79.92 (6)°. In the crystal structure, the molecules are stabilized by intermolecular O1–H1···N1 hydogen bonds and intramolecular C2–H2B···O1 interactions (Fig. 2).

Experimental

A mixture of 3-cyano-3-methyl-5-phenylcyclohexanone (2.13 g, 0.01 mol), sodium acetate trihydrate (4.08 g, 0.03 mol), hydroxylamine hydrochloride (1.39 g, 0.02 mol) and ethanol (50 ml) was heated with reflux for 20 m. The reaction mixture was cooled to room temperature and poured into water. The separated solid was filtered off and it was purified by column chromatography (Benzene-EtOAc, 9.5:0.5 v/v). The yield of the isolated product was 2.17 g (87%).

Refinement

H atom bonded to O was located in a difference Fourier map and refined freely. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C–H = 0.93-0.98 Å and U_{iso}=1.2 or 1.5 times U_{eq}(C).

Figures

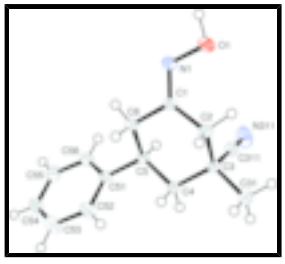

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

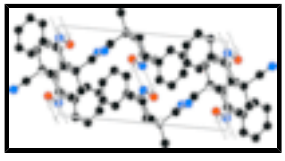

Fig. 2. The molecular packing of (I), viewed down the b axis. Dashed lines indicate hydrogen bonds.

3-Hydroxyimino-1-methyl-5-phenylcyclohexane-1-carbonitrile

Crystal data

$C_{14}H_{16}N_2O$	F_{000} = 488
M_r = 228.29	D_x = 1.235 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 449 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.6394 (2) Å	Cell parameters from 3701 reflections
b = 14.6658 (5) Å	θ = 2.0–30.0º
c = 12.6653 (3) Å	μ = 0.08 mm^{-1}
β = 95.146 (2)º	T = 160 (1) K
V = 1228.28 (6) Å^3	Block, colourless
Z = 4	0.25 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer	3588 independent reflections
Radiation source: Nonius FR590 sealed tube generator	2568 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	R_{int} = 0.082
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 30.0º
T = 160(1) K	θ_{min} = 2.1º
φ and ω scans with κ offsets	h = −9→9
Absorption correction: none	k = −20→20
33562 measured reflections	l = −17→17

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.3782P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.165	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
3588 reflections	Extinction correction: none
159 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Experimental. Solvent used: n-hexane / EtOAc Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.678 (2) Frames collected: 361 Seconds exposure per frame: 28 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.20084 (18)	0.49328 (7)	−0.06439 (9)	0.0284 (3)
N1	0.13724 (19)	0.41986 (8)	−0.00310 (9)	0.0215 (3)
N311	0.2380 (2)	0.23128 (12)	−0.26976 (11)	0.0412 (5)
C1	0.2597 (2)	0.35272 (9)	0.00221 (11)	0.0194 (4)
C2	0.4533 (2)	0.34800 (10)	−0.04978 (12)	0.0233 (4)
C3	0.4888 (2)	0.25392 (9)	−0.09912 (11)	0.0196 (4)
C4	0.4514 (2)	0.17716 (9)	−0.01999 (11)	0.0192 (4)
C5	0.2397 (2)	0.18121 (9)	0.01813 (10)	0.0178 (4)
C6	0.2112 (2)	0.27419 (9)	0.07144 (11)	0.0209 (4)
C31	0.7058 (2)	0.25006 (11)	−0.13174 (13)	0.0276 (4)
C51	0.1935 (2)	0.10561 (9)	0.09409 (11)	0.0191 (4)
C52	0.3403 (3)	0.06703 (11)	0.16485 (12)	0.0299 (5)
C53	0.2892 (3)	0.00186 (12)	0.23784 (14)	0.0376 (5)
C54	0.0913 (3)	−0.02518 (11)	0.24113 (13)	0.0319 (5)
C55	−0.0553 (3)	0.01230 (12)	0.17107 (14)	0.0346 (5)
C56	−0.0048 (2)	0.07656 (11)	0.09776 (13)	0.0298 (5)
C311	0.3463 (2)	0.24197 (11)	−0.19548 (12)	0.0254 (4)
H1	0.097 (4)	0.5381 (16)	−0.0536 (16)	0.054 (6)*
H2A	0.56521	0.36168	0.00241	0.0279*
H2B	0.45126	0.39411	−0.10477	0.0279*
H4A	0.46953	0.11866	−0.05358	0.0230*
H4B	0.55085	0.18172	0.04073	0.0230*
H5	0.14139	0.17711	−0.04420	0.0213*
H6A	0.07228	0.27958	0.08869	0.0250*
H6B	0.29793	0.27701	0.13721	0.0250*
H31A	0.72880	0.19193	−0.16337	0.0414*
H31B	0.79959	0.25823	−0.07026	0.0414*
H31C	0.72469	0.29761	−0.18200	0.0414*
H52	0.47452	0.08484	0.16364	0.0358*
H53	0.38959	−0.02360	0.28471	0.0451*

H54	0.05733	−0.06836	0.29030	0.0382*
H55	−0.18938	−0.00555	0.17280	0.0415*
H56	−0.10563	0.10071	0.05012	0.0357*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0305 (6)	0.0167 (5)	0.0388 (6)	0.0023 (4)	0.0084 (5)	0.0075 (4)
N1	0.0258 (6)	0.0148 (6)	0.0242 (6)	0.0002 (5)	0.0035 (5)	0.0015 (4)
N311	0.0411 (9)	0.0545 (10)	0.0267 (8)	−0.0099 (7)	−0.0040 (7)	0.0064 (7)
C1	0.0225 (7)	0.0158 (6)	0.0197 (7)	0.0011 (5)	0.0014 (5)	−0.0018 (5)
C2	0.0225 (7)	0.0195 (7)	0.0287 (8)	−0.0002 (6)	0.0068 (6)	0.0003 (6)
C3	0.0195 (7)	0.0208 (7)	0.0187 (7)	0.0024 (5)	0.0032 (5)	−0.0007 (5)
C4	0.0204 (7)	0.0181 (7)	0.0192 (6)	0.0049 (5)	0.0019 (5)	0.0006 (5)
C5	0.0193 (7)	0.0164 (6)	0.0175 (6)	0.0031 (5)	0.0012 (5)	0.0007 (5)
C6	0.0247 (7)	0.0175 (7)	0.0211 (7)	0.0033 (5)	0.0057 (5)	0.0005 (5)
C31	0.0228 (7)	0.0314 (8)	0.0297 (8)	0.0027 (6)	0.0083 (6)	0.0010 (6)
C51	0.0244 (7)	0.0151 (6)	0.0182 (6)	0.0032 (5)	0.0037 (5)	−0.0009 (5)
C52	0.0301 (8)	0.0288 (8)	0.0295 (8)	−0.0012 (7)	−0.0039 (7)	0.0078 (6)
C53	0.0451 (10)	0.0335 (9)	0.0324 (9)	0.0018 (8)	−0.0065 (8)	0.0132 (7)
C54	0.0474 (10)	0.0205 (7)	0.0291 (8)	0.0012 (7)	0.0108 (7)	0.0059 (6)
C55	0.0318 (9)	0.0300 (9)	0.0439 (10)	0.0012 (7)	0.0141 (7)	0.0088 (7)
C56	0.0251 (8)	0.0294 (8)	0.0350 (9)	0.0040 (6)	0.0036 (7)	0.0112 (7)
C311	0.0266 (8)	0.0274 (8)	0.0226 (7)	−0.0014 (6)	0.0051 (6)	0.0036 (6)

Geometric parameters (Å, °)

O1—N1	1.4139 (16)	C54—C55	1.372 (3)
O1—H1	0.97 (3)	C55—C56	1.385 (2)
N1—C1	1.2749 (18)	C2—H2A	0.9700
N311—C311	1.143 (2)	C2—H2B	0.9700
C1—C6	1.4999 (19)	C4—H4A	0.9700
C1—C2	1.4975 (19)	C4—H4B	0.9700
C2—C3	1.542 (2)	C5—H5	0.9800
C3—C4	1.5421 (19)	C6—H6A	0.9700
C3—C31	1.5351 (19)	C6—H6B	0.9700
C3—C311	1.486 (2)	C31—H31A	0.9600
C4—C5	1.5277 (19)	C31—H31B	0.9600
C5—C6	1.5408 (19)	C31—H31C	0.9600
C5—C51	1.5172 (19)	C52—H52	0.9300
C51—C56	1.3886 (19)	C53—H53	0.9300
C51—C52	1.384 (2)	C54—H54	0.9300
C52—C53	1.393 (2)	C55—H55	0.9300
C53—C54	1.377 (3)	C56—H56	0.9300
O1···N1^{i}	2.7814 (17)	H4A···H31A	2.5500
O1···H2B	2.3000	H4B···C52	2.7700
O1···H1^{i}	2.62 (2)	H4B···H6B	2.5800
O1···H2A^{ii}	2.7100	H4B···H31B	2.5300

O1···H53^{iii}	2.7200	H4B···H52	2.2000
N1···O1^{i}	2.7814 (17)	H4B···H56^{ix}	2.5600
N1···N1^{i}	2.9797 (17)	H4B···N311^{x}	2.9000
N1···H53^{iv}	2.9100	H5···C311	2.6300
N1···H1^{i}	1.88 (3)	H5···H31B^{xi}	2.5600
N311···H4B^{iii}	2.9000	H5···H56	2.3900
C56···C56^{v}	3.348 (2)	H6A···C56	3.0300
C1···H54^{iv}	3.0200	H6B···H4B	2.5800
C1···H1^{i}	2.98 (3)	H6B···H54^{iv}	2.6000
C4···H52	2.6800	H6B···C31^{vii}	3.0700
C31···H6B^{vi}	3.0700	H31A···H4A	2.5500
C52···H4B	2.7700	H31A···C53^{viii}	2.9900
C52···H31C^{vii}	2.9300	H31A···C54^{viii}	2.9300
C52···H4A	3.0600	H31B···H2A	2.4100
C53···H31A^{viii}	2.9900	H31B···H4B	2.5300
C54···H2B^{vii}	2.9500	H31B···H5^{ix}	2.5600
C54···H31A^{viii}	2.9300	H31C···H2B	2.5600
C56···H6A	3.0300	H31C···C52^{vi}	2.9300
C311···H5	2.6300	H52···C4	2.6800
H1···O1^{i}	2.62 (2)	H52···H4B	2.2000
H1···N1^{i}	1.88 (3)	H52···H55^{ix}	2.5900
H1···C1^{i}	2.98 (3)	H53···N1^{xii}	2.9100
H1···H1^{i}	2.25 (3)	H53···O1^{x}	2.7200
H1···H53^{iii}	2.3700	H53···H1^{x}	2.3700
H2A···H31B	2.4100	H54···C1^{xii}	3.0200
H2A···O1^{ii}	2.7100	H54···H6B^{xii}	2.6000
H2B···O1	2.3000	H55···H52^{xi}	2.5900
H2B···H31C	2.5600	H56···H4B^{xi}	2.5600
H2B···C54^{vi}	2.9500	H56···H5	2.3900
H4A···C52	3.0600		
N1—O1—H1	100.8 (14)	C3—C4—H4A	109.00
O1—N1—C1	113.37 (12)	C5—C4—H4B	109.00
N1—C1—C2	125.52 (13)	C3—C4—H4B	109.00
N1—C1—C6	117.04 (12)	C5—C4—H4A	109.00
C2—C1—C6	117.34 (12)	H4A—C4—H4B	108.00
C1—C2—C3	112.84 (11)	C51—C5—H5	108.00
C2—C3—C4	110.52 (11)	C4—C5—H5	108.00
C2—C3—C31	108.87 (11)	C6—C5—H5	108.00
C4—C3—C31	110.96 (11)	C1—C6—H6A	109.00
C4—C3—C311	108.66 (11)	C5—C6—H6B	109.00
C2—C3—C311	109.22 (12)	C1—C6—H6B	109.00
C31—C3—C311	108.57 (12)	C5—C6—H6A	109.00
C3—C4—C5	112.54 (11)	H6A—C6—H6B	108.00

C4—C5—C51	114.59 (11)	C3—C31—H31C	109.00
C6—C5—C51	109.22 (11)	C3—C31—H31A	109.00
C4—C5—C6	109.00 (11)	C3—C31—H31B	109.00
C1—C6—C5	112.47 (11)	H31B—C31—H31C	109.00
C5—C51—C52	122.58 (13)	H31A—C31—H31B	109.00
C5—C51—C56	119.45 (12)	H31A—C31—H31C	109.00
C52—C51—C56	117.88 (14)	C51—C52—H52	120.00
C51—C52—C53	120.73 (17)	C53—C52—H52	120.00
C52—C53—C54	120.52 (17)	C54—C53—H53	120.00
C53—C54—C55	119.26 (16)	C52—C53—H53	120.00
C54—C55—C56	120.39 (17)	C55—C54—H54	120.00
C51—C56—C55	121.23 (15)	C53—C54—H54	120.00
C1—C2—H2B	109.00	C54—C55—H55	120.00
C1—C2—H2A	109.00	C56—C55—H55	120.00
H2A—C2—H2B	108.00	C51—C56—H56	119.00
C3—C2—H2A	109.00	C55—C56—H56	119.00
C3—C2—H2B	109.00	N311—C311—C3	178.81 (17)
O1—N1—C1—C2	−0.2 (2)	C4—C5—C6—C1	52.56 (14)
O1—N1—C1—C6	176.13 (11)	C51—C5—C6—C1	178.44 (11)
N1—C1—C2—C3	−138.80 (14)	C4—C5—C51—C52	32.02 (19)
C6—C1—C2—C3	44.89 (17)	C4—C5—C51—C56	−151.53 (13)
N1—C1—C6—C5	135.74 (13)	C6—C5—C51—C52	−90.56 (16)
C2—C1—C6—C5	−47.64 (16)	C6—C5—C51—C56	85.88 (15)
C1—C2—C3—C4	−47.54 (15)	C5—C51—C52—C53	175.88 (14)
C1—C2—C3—C31	−169.65 (12)	C56—C51—C52—C53	−0.6 (2)
C1—C2—C3—C311	71.95 (15)	C5—C51—C56—C55	−175.43 (14)
C2—C3—C4—C5	56.63 (14)	C52—C51—C56—C55	1.2 (2)
C31—C3—C4—C5	177.51 (11)	C51—C52—C53—C54	−0.2 (3)
C311—C3—C4—C5	−63.20 (14)	C52—C53—C54—C55	0.5 (3)
C3—C4—C5—C6	−58.77 (14)	C53—C54—C55—C56	0.1 (3)
C3—C4—C5—C51	178.53 (11)	C54—C55—C56—C51	−0.9 (3)

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*+1, −*y*+1, −*z*; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) −*x*, −*y*, −*z*; (vi) *x*+1/2, −*y*+1/2, *z*−1/2; (vii) *x*−1/2, −*y*+1/2, *z*+1/2; (viii) −*x*+1, −*y*, −*z*; (ix) *x*+1, *y*, *z*; (x) *x*+1/2, −*y*+1/2, *z*+1/2; (xi) *x*−1, *y*, *z*; (xii) −*x*+1/2, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.97 (3)	1.88 (3)	2.7814 (17)	154 (2)
C2—H2B···O1	0.97	2.30	2.7070 (18)	104

Symmetry codes: (i) −*x*, −*y*+1, −*z*.

Fig. 1

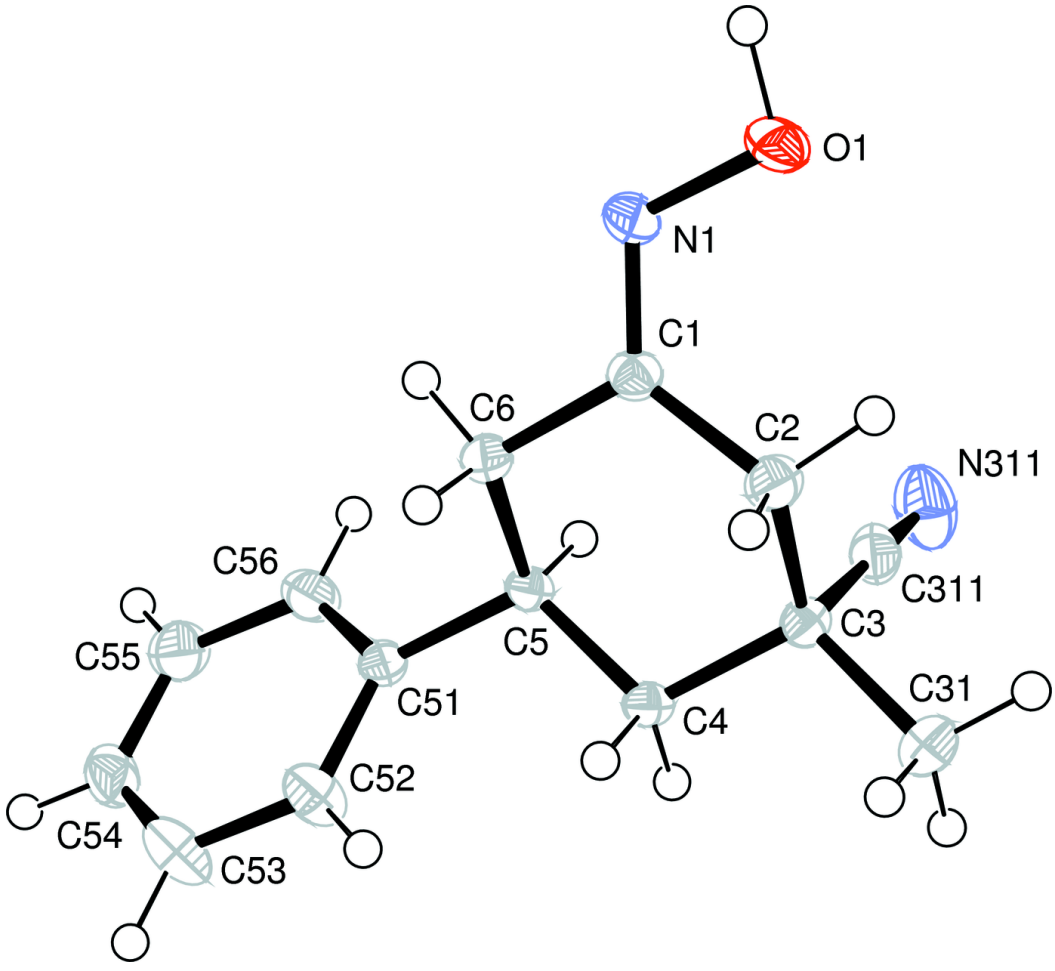

Fig. 2