Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-(3-Chlorophenyl)-*α*-(2-hydroxyphenyl)-nitrone: supramolecular aggregation through *π*–*π* and C–H···*π* interactions

Ebrahim Mothi Mohamed, Srinivasan Chandrasekar and Krishnaswamy Panchanatheswaran*

Department of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, India

Correspondence e-mail:
panch_45@yahoo.co.in

Received 2 January 2003
Accepted 15 January 2003
Online 31 January 2003

Key indicators

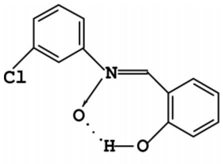
Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.097
Data-to-parameter ratio = 12.9

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

The title compound, $C_{13}H_{10}ClNO_2$, is identified to be a nitrone which is stabilized by an intramolecular O—H···O hydrogen bond. In the crystal structure, the molecules are packed in layers. The closest distance between the centroids of chlorophenyl and hydroxyphenyl rings in adjacent layers is 3.178 (2) Å.

Comment

The reaction of salicylaldehyde with 1-chloro-3-nitrobenzene yielded a solid. Its ^{1}H NMR spectrum revealed the presence of phenyl groups. This investigation was undertaken to assign the structure and the configuration of the title compound, (I).



(I)

The ellipsoid plot (Fig. 1) and the C5—N—C7—C8 torsion angle show that the two rings are oriented *trans* with respect to the C═N bond, as observed in many non-cyclic nitrones (Hamer & Macaluso, 1964). The multiplicity of the C═N indicates that the (I) exists as a nitrone with a C═N double bond, rather than as the isomeric oxaziridine. The molecule is non-planar, but the chlorophenyl and hydroxyphenyl rings are each planar, the r.m.s. deviations from planarity for the two rings being 0.0012 and 0.0109 Å, respectively. The dihedral angles between these two planes and the C7/N/O1 plane are 35.77 (7) and 33.93 (7)°, respectively. The two aromatic rings are nearly parallel, with a dihedral angle of 1.85 (8)°. The C═N and N—O bond lengths are unexceptional and are very similar to the corresponding lengths observed in similar nitrones (Chandrasekar & Panchanatheswaran, 2000; Bedford & Chaloner, 1991; Pritchard *et al.*, 1991).

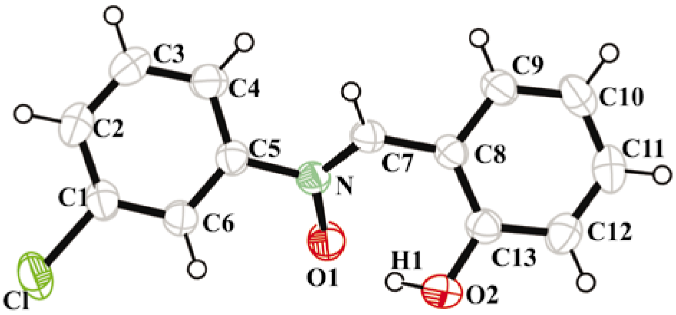


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-labelling scheme.

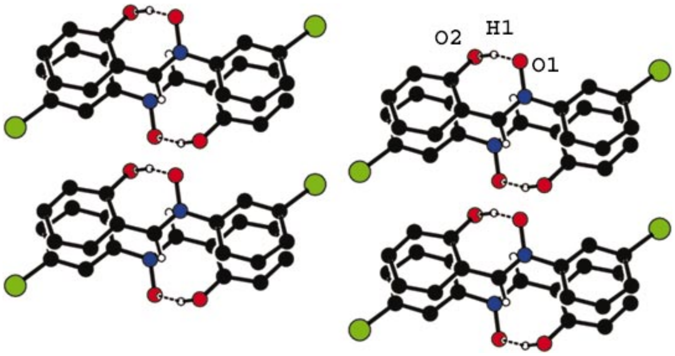


Figure 2
A packing diagram, viewed down the *b* axis. Hydrogen-bonding is indicated by dashed lines. Only those H atoms involved in hydrogen bonding are shown.

The OH group is intramolecularly hydrogen bonded to the O atom of the nitrone moiety, leading to an $R_1^1(7)$ (Bernstein *et al.*, 1995) arrangement (Fig. 2 and Table 2). In the crystal, the molecules are stacked in layers, held together by π–π interactions, with a distance of 3.718 (2) Å between the centroids of adjacent chlorophenyl and hydroxyphenyl rings (symmetry code: $1 - x$, $2 - y$, $1 - z$). The two molecules are also held together by C—H$\cdots\pi$ interactions between the H atoms on C3 and C9 and the hydroxyphenyl and chlorophenyl rings, respectively (Table 2). These π–π and C—H$\cdots\pi$ interactions compare well with the corresponding distances of 3.563 (2) and 3.543 (3) Å observed in 3-methyl-1,4-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine (Low *et al.*, 2002). The centroids of the chlorophenyl and hydroxyphenyl rings in the opposite direction (symmetry code: $-\frac{1}{2} + x$, $\frac{3}{2} - y$, $-\frac{1}{2} + z$) are separated by 4.612 (3) Å, leading to a segregated stacked arrangement (Desiraju, 1989).

Experimental

The title compound was prepared by the reductive coupling of 1-chloro-3-nitrobenzene with salicylaldehyde, using anhydrous tin(II) chloride in tetrahydrofuran as solvent. The product was extracted with diethyl ether and obtained as diffraction quality crystals.

Crystal data

$C_{13}H_{10}ClNO_2$
$M_r = 247.67$
Monoclinic, $P2_1/n$
$a = 5.9476$ (8) Å
$b = 14.613$ (8) Å
$c = 12.850$ (6) Å
$\beta = 97.436$ (6)°
$V = 1107.4$ (8) Å^3
$Z = 4$

$D_x = 1.486$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 2093 reflections
$\theta = 2$–12°
$\mu = 2.96$ mm^{-1}
$T = 293$ (2) K
Block, pale yellow
0.10 × 0.05 × 0.05 mm

Data collection

Enraf–Nnius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.835$, $T_{max} = 0.862$
2300 measured reflections
2093 independent reflections
1779 reflections with $I > 2\sigma(I)$

$R_{int} = 0.044$
$\theta_{max} = 69.9°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 17$
$l = -15 \rightarrow 15$
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.097$
$S = 1.06$
2093 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.3447P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0081 (6)

Table 1
Selected geometric parameters (Å, °).

Cl—C1	1.7412 (18)	N—C7	1.298 (2)
O1—N	1.3260 (16)	N—C5	1.448 (2)
O2—C13	1.3488 (19)	C7—C8	1.450 (2)
C7—N—O1	122.91 (13)	C2—C1—Cl	119.41 (13)
C7—N—C5	122.38 (13)	C9—C8—C7	116.60 (14)
O1—N—C5	114.70 (12)	O2—C13—C12	118.09 (15)
C6—C1—Cl	118.48 (13)	O2—C13—C8	122.38 (15)
Cl—C1—C2—C3	−179.66 (13)	C5—N—C7—C8	−179.37 (14)
O1—N—C5—C6	35.03 (19)	N—C7—C8—C9	147.22 (16)
O1—N—C5—C4	−143.26 (15)	N—C7—C8—C13	−35.1 (3)
O1—N—C7—C8	0.4 (3)		

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

*Cg*1 and *Cg*2 are the centroids of the chlorophenyl and hydroxyphenyl rings, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H1$\cdots$O1	0.95 (3)	1.57 (3)	2.495 (2)	163 (2)
C3—H3$\cdots Cg2^{i}$	0.93	2.87	3.549 (3)	131
C9—H9$\cdots Cg1^{ii}$	0.93	2.98	3.536 (3)	120

Symmetry codes: (i) $x + \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$; (ii) $x + \frac{1}{2}, \frac{3}{2} - y, z + \frac{1}{2}$.

Atoms H1 and H7 were located from a difference Fourier map and their positional parameters were refined. The displacement parameter of H1 was fixed as $1.5U_{eq}$(O2), while that of atom H7 was refined. The O2—H1 and C7—H7 distances are 0.95 (3) and 0.94 (2) Å, respectively. The H atoms of the aromatic rings were included in the refinement at calculated positions, with $U_{iso} = 1.2U_{eq}$(C).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 1998); software used to prepare material for publication: *SHELXL*97.

References

Bedford, R. B. & Chaloner, P. A. (1991). *Acta Cryst.* C**47**, 2484–2485.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Chandrasekar, S. & Panchanatheswaran, K. (2000). *Acta Cryst.* C**56**, 1442–1443.
Desiraju, G. R. (1989). *Crystal Engineering: The Design of Organic Solids*, p. 269. Amsterdam: Elsevier.

Acta Cryst. (2003). E**59**, o234–o236 Ebrahim Mothi Mohamed *et al.* • $C_{13}H_{10}ClNO_2$ **o235**

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Hamer, J. & Macaluso, A. (1964). *Chem. Rev.* **64**, 473–495.

Low, J. N., Cobo, J., Nogueras, M., Sanchez, A., Torres, H. & Insuasty, B. (2002). *Acta Cryst.* C**58**, o298–o300.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Pritchard, R. G., Banks, R. E., DuBoisson, R. A. & Tipping, A. E. (1991). *Acta Cryst.* C**47**, 230–232.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Spek, A. L. (1998). *PLATON*. Utrecht University, The Netherlands.

o236 Ebrahim Mothi Mohamed *et al.* • $C_{13}H_{10}ClNO_2$ *Acta Cryst.* (2003). E**59**, o234–o236