Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isoquinoline 1-oxide–2-nitrobenzoic acid (1/1)

Rodolfo Moreno-Fuquen,[a]* Angela Marcela Montaño[a] and Reinaldo Atencio[b]

[a]Departamento de Química, Facultad de Ciencias, Universidad del Valle, Apartado 25360, Santiago de Cali, Colombia, and [b]Centro de Química, Instituto Venezolano de Investigaciones, Científicas, IVIC, Apartado 21827, Caracas, Venezuela

Correspondence e-mail: rodimo26@yahoo.es

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.104
Data-to-parameter ratio = 11.9

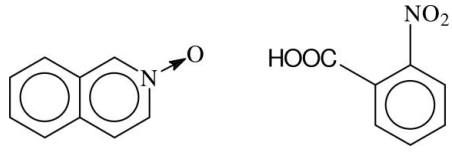
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 August 2005
Accepted 18 August 2005
Online 31 August 2005

The title molecular complex, $C_9H_7NO{\cdot}C_7H_5NO_4$, owes its formation to an intermolecular hydrogen bond between the O—H and N—O groups, with an O···O distance of 2.514 (2) Å. The dihedral angle between the planes of the isoquinoline N-oxide and nitrobenzoic acid rings of the complex is 49.91 (4)°. The crystal structure exhibits overlap between the aromatic rings of the molecules in the [101] direction.

Comment

In order to give continuity to the structural studies on molecular complexes formed by hydrogen bonds, with potential applications in non-linear optics (Moreno-Fuquen *et al.*, 1998), the synthesis of co-crystals with greater conjugation has been undertaken. To this end, the isoquinoline 1-oxide (IQNO) molecule, as an acceptor of hydrogen bond, was complexed with a 2-nitrobenzoic acid (ONBA) molecule, giving the title complex, (I). Two structures related to (I) were found in the Cambridge Structural Database (Version5.25; Allen, 2002), namely isoquinoline (Hensen *et al.*, 1999) and 2-nitrobenzoic acid (Tavale & Pant, 1973). These two systems were used as reference standards to analyse the structural characteristics of title complex, (I).



IQNO (I) ONBA

The title complex is held together by a strong intermolecular hydrogen bond (Emsley, 1984) between the O—H group of the ONBA molecule and the N—O group of the IQNO molecule. As is shown in Fig. 1, the O1···O4 bond length is 2.514 (2) Å and the O1···H*O*4—O4 bond angle is 167 (3)°. The dihedral angle between the planes of the IQNO and ONBA rings of the complex is 49.91 (4)°. In the title complex there is a decrease in N—C bond length of the isoquinoline ring. In isoquinoline itself (Hensen et al., 1999), N—C bonds are 1.36 (4) and 1.39 (4) Å, , whereas in (I), N1—C8 is 1.325 (3) Å. This change can be attributed to the formation of the intermolecular hydrogen bond. Other distances and bond angles are similar to those values found in the free isoquinoline molecule. Otherwise bond distances and angles in (I) are very similar to those found in ONBA molecule (Tavale, & Pant, 1973). The molecules stack, with an

overlap between IQNO rings alternating in opposite directions, at a distance of 3.336 (2) Å. This overlap is in the [101] direction.

Experimental

Reagents and solvents for the synthesis were purchased from Aldrich Chemical Co. and were used without additional purification. Pale-yellow single crystals of the title complex suitable for X-ray analysis were obtained by slow evaporation of an equimolar solution of IQNO and ONBA in acetonitrile. The crystals of the IQNO–ONBA molecular complex have a melting point of 387 (1) K.

Crystal data

$C_9H_7NO\cdot C_7H_5NO_4$
M_r = 312.28
Monoclinic, $P2_1/c$
a = 11.639 (3) Å
b = 7.235 (4) Å
c = 17.206 (3) Å
β = 96.399 (18)°
V = 1439.9 (9) Å^3
Z = 4
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 1.0–25.0°
μ = 0.11 mm^{-1}
T = 293 K
Prism, pale yellow
0.23 × 0.20 × 0.12 mm

Data collection

Rigaku AFC-7S diffractometer
$\omega/2\theta$ scans
2532 measured reflections
2532 independent reflections
1847 reflections with $I > 2\sigma(I)$
θ_{max} = 25.0°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = -20 \rightarrow 20$
2 standard reflections
frequency: 150 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.08
2532 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.6379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0214 (15)

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.333 (2)	N2—O3	1.222 (3)
N1—C8	1.325 (3)	N2—C2	1.458 (3)
N1—C16	1.380 (3)	C1—C7	1.501 (3)
O4—H*O*4	1.03 (3)	C7—O5	1.205 (2)
N2—O2	1.216 (2)	C7—O4	1.297 (3)
O1—N1—C16	119.29 (17)	O2—N2—C2	118.0 (2)
O2—N2—O3	123.9 (2)		
O3—N2—C2—C3	−35.8 (3)	C2—C1—C7—O5	130.2 (2)
C6—C1—C7—O5	−51.5 (3)		

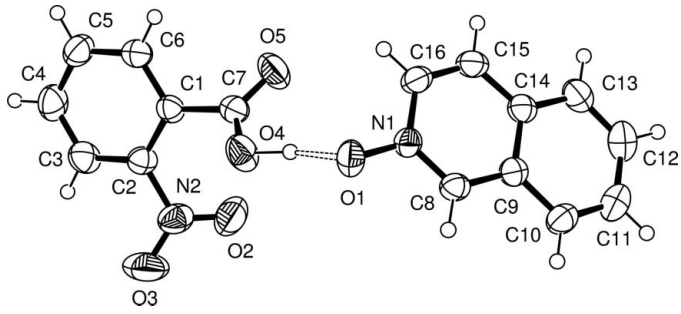


Figure 1
An *ORTEP3* (Farrugia, 1997) plot of the title compound, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level for the non-H atoms. H atoms are shown as spheres of an arbitrary radius. The dashed line indicates a hydrogen bond.

Carbon-bound H-atom positions were idealized (C—H = 0.93 Å), with H atoms riding on the atoms to which they were attached. These idealized H atoms had their isotropic displacement parameters fixed at 1.2 times the U_{eq} of the carrier atom. Atom H*O*4 atom was located in a difference map and its coordinates were refined freely.

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1993); cell refinement: MSC/AFC Diffractometer Control Software; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

MAM acknowledges the Instituto Venezolano de Investigaciones Cientificas (IVIC), Venezuela, for the diffraction analysis. The authors also thank the Colombian Institute of Science (COLCIENCIAS) for financial support.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Emsley, J. (1984). *Complex Chemistry. Structure and Bonding*, Vol. 57, pp. 147–191. Berlin: Springer-Verlag.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hensen, K., Mayr-Stein, R. & Bolte, M. (1999). *Acta Cryst.* C**55**, 1565–1567.
Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software.* Version 5.1.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1995). *TEXSAN/TEXRAY*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Moreno-Fuquen, R., Gambardella, M. T. do P. & Valderrama-N, J. (1998). *Acta Cryst.* C**54**, 515–517.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Tavale, S. S. & Pant, L. M. (1973). *Acta Cryst.* B**29**, 2979–2980.