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N-(3-Chlorophenyl)-*α*-phenylnitrone: association with benzoic acid through hydrogen bonding

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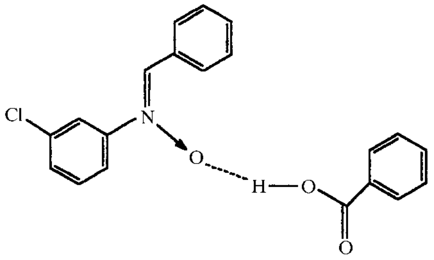
The identity of the title complex, $C_{13}H_{10}ClNO{\cdot}C_7H_6O_2$, is confirmed to be a hydrogen-bonded adduct of benzoic acid and *N*-(3-chlorophenyl)-*α*-phenylnitrone. The two aromatic rings in the nitrone are *trans* about the C=N bond.

Comment

The title complex, (I), was isolated during our research on the organic functional group transformations mediated by anhydrous tin(II) chloride. Its IR and ^{1}H NMR spectra indicated the presence of the nitrone as well as the carboxylic acid group. This investigation was undertaken to assign the stereochemistry of the nitrone and to precisely determine the nature of its bonding with the COOH group.



(I)

As found by this investigation, the asymmetric unit is made up of one molecule each of benzoic acid and the nitrone. In the latter, the 3-chlorophenyl and phenyl groups are bonded to nitrogen and carbon, respectively. The *ZORTEP* (Zsolnai, 1997) plot (Fig. 1) and the C4—N8—C11—C12 torsion angle, 178.6 (2)°, show that the two rings are oriented *trans* around the C=N bond. That the non-acid part of (I) exists as a nitrone and not as the isomeric oxaziridine is indicated by the multiplicity of the N8=C11 bond. Further, the nitrone molecule is non-planar, the dihedral angle between the two aromatic rings being 11.1 (1)°. This precludes appreciable delocalization between them. As expected, the C4—N8—O9—C11—C12 part of the molecule is nearly planar, the deviations of the atoms from the least-squares plane being −0.005 (1), 0.000 (1), 0.002 (1), 0.012 (2) and −0.008 (1) Å, respectively. The C=N and N—O bond lengths are comparable to similar bond lengths in *N*-methyl(phenyl)nitrone and *N*-(4-fluorophenyl)diphenylnitrone (Bedford *et al.*, 1991; Pritchard *et al.*, 1991). The benzoic acid molecule is oriented away from the planes of the phenyl and chlorophenyl groups, with dihedral angles of 75.1 (2) and 76.1 (2)°, respectively. The two molecules are held together by an O18—H18···O9 hydrogen bond [O18···O9 = 2.580 (3) Å, O18—H18···O9 = 171°].

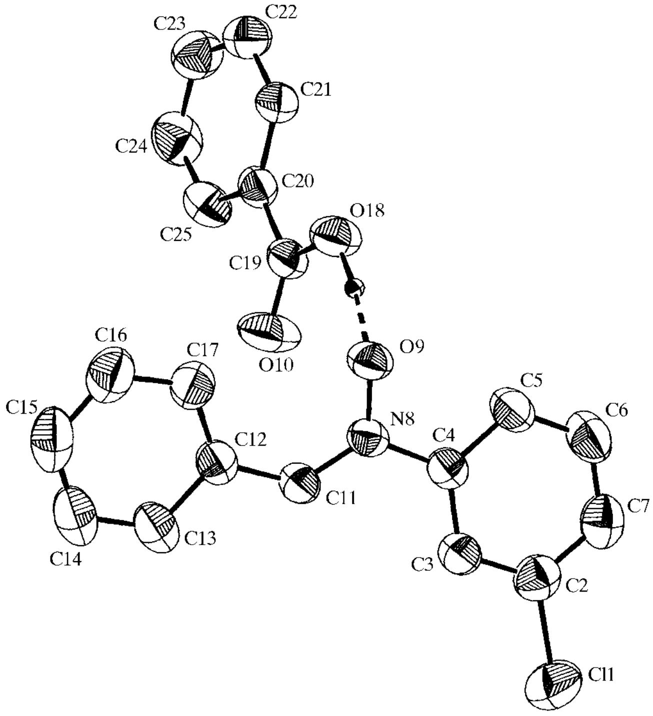


Figure 1
ZORTEP (Zsolnai, 1997) plot of the title compound. Displacement ellipsoids of 30% probability were used. The H atoms of the aromatic rings have been omitted for clarity.

Experimental

To the equimolar mixture of 1-chloro-3-nitrobenzene and anhydrous tin(II) chloride in tetrahydrofuran, benzaldehyde was added. The mixture was stirred for 24 h and decomposed with dilute hydrochloric acid. The organic product was extracted with diethyl ether. Yellow crystals were obtained by slow evaporation of the solvent.

Crystal data

$C_{13}H_{10}ClNO{\cdot}C_7H_6O_2$	$Z = 2$
$M_r = 353.79$	$D_x = 1.350$ Mg m^{-3}
Triclinic, $P\bar{1}$	Cu $K\alpha$ radiation
$a = 7.609$ (3) Å	Cell parameters from 25 reflections
$b = 9.630$ (2) Å	
$c = 12.858$ (2) Å	$\theta = 5$–$30°$
$\alpha = 110.14$ (1)°	$\mu = 2.098$ mm^{-1}
$\beta = 94.80$ (2)°	$T = 293$ (2) K
$\gamma = 96.83$ (2)°	Plate, light yellow
$V = 870.5$ (4) Å^3	0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.594$, $T_{\max} = 0.811$
3424 measured reflections
3163 independent reflections
2515 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.0104$
$\theta_{\max} = 67.88°$
$h = 0 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$
2 standard reflections every 100 reflections intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.146$
$S = 1.052$
3163 reflections
227 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0770P)^2 + 0.2612P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0045 (9)

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.743 (2)	O10—C19	1.209 (3)
C4—N8	1.455 (3)	C11—C12	1.446 (3)
N8—C11	1.304 (3)	O18—C19	1.313 (3)
N8—O9	1.318 (2)		
C7—C2—Cl1	118.9 (2)	O9—N8—C4	115.4 (2)
C3—C2—Cl1	118.3 (2)	O10—C19—O18	121.9 (2)
C11—N8—O9	122.9 (2)	O10—C19—C20	123.3 (2)
C11—N8—C4	121.7 (2)	O18—C19—C20	114.8 (2)
C4—N8—C11—C12	178.6 (2)		

All the H atoms were located from the difference map. Their positions were fixed in the subsequent cycles of refinement and their displacement parameters were fixed as $1.1U_{\rm eq}$ of the respective carrier atom, except for the hydrogen attached to O18 which was fixed as $1.5U_{\rm eq}$ of the respective carrier atom. Geometrical calculations were performed using *PARST*96 (Nardelli, 1983, 1995). C—H distances are in the range 0.94–1.11 Å and the O—H distance is 1.04 Å.

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*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1113). Services for accessing these data are described at the back of the journal.

References

Bedford, R. B., Chaloner, P. A. & Hitchcock, P. B. (1991). *Acta Cryst.* C**47**, 2484–2485.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
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. (1985). *SHELXS*86. University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL*93. University of Göttingen, Germany.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.