

Watkin, D. J., Prout, C. K., Carruthers, R. J. & Betteridge, P. (1996). *CRYSTALS*. Issue 10. Chemical Crystallography Laboratory, University of Oxford, England.

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N-(*p*-Hydroxybenzylidene)phenylamine *N*-Oxide

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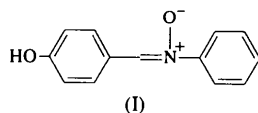
(Received 26 November 1996; accepted 10 April 1997)

Abstract

The crystal structure of the title compound, C₁₃H₁₁NO₂, confirms that it exists as a nitron. The geometry about C=N is *Z*. The torsion angles O8—N8—C7—H7 [174.55 (2)°] and C8—N8—C7—H7 [−0.89 (4)°] indicate the *trans* and *cis* positions around the nitron moiety.

Comment

The title compound, (I), has been analysed as part of crystallographic studies on substituted *N*-phenylnitrones.



The molecule exists as a nitron. The C=N distance of 1.304 (3) Å is within 3σ of 1.279 (8) Å reported by Allen *et al.* (1987). The dihedral angle between the C8, N8, C7 and N8, C7, C1 planes is 2.61 (2)°. The nitron group formed by atoms C8, N8, C7 and C1 attached to two phenyl rings is nearly planar. Atoms N8, C8, C7 and C1 deviate from the least-squares plane by −0.008 (2), −0.013 (2), −0.017 (2) and −0.015 (2) Å, respectively. The C1—C7 distance of 1.440 (3) Å is nearly equal to the value of 1.438 (4) Å reported by Bedford, Chaloner & Hitchcock (1991). The phenyl

bond lengths and angles are in agreement with the expected values reported by Allen *et al.* (1987). The dihedral angle between the two phenyl rings is 55.3 (1)°.

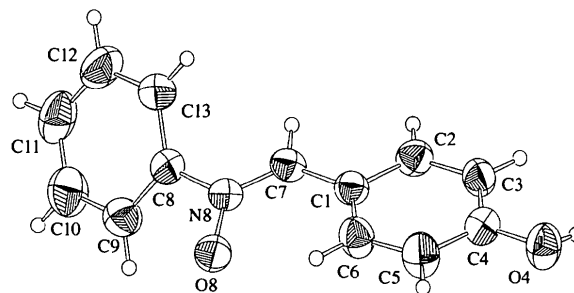


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

Experimental

A solution of 4-hydroxybenzaldehyde (1.22 g) in ethanol (25 ml) was added dropwise with stirring to a solution of *N*-phenylhydroxylamine (1.09 g) in ethanol (10 ml). The mixture was warmed for about 1 h at 333 K. On cooling, crude crystals of the title compound separated and were recrystallized from ethanol [m.p. 487 K; yield 1.76 g (83%)]. The title compound was prepared in the Department of Industrial Chemistry, Alagappa University, Karaikudi, India.

Crystal data

C₁₃H₁₁NO₂
M_r = 213.23
Orthorhombic
Pbca
a = 18.033 (9) Å
b = 14.019 (9) Å
c = 8.973 (5) Å
V = 2269 (2) Å³
Z = 8
D_x = 1.249 Mg m^{−3}
D_m not measured

Mo Kα radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 5–25°
μ = 0.085 mm^{−1}
T = 293 K
Rectangular
0.25 × 0.20 × 0.10 mm
Yellow

Data collection

Rigaku AFC-7S diffractometer
ω–2θ scans
Absorption correction:
ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.922, T_{max} = 1.000
1875 measured reflections
1874 independent reflections
843 reflections with
I > 2σ(I)

R_{int} = 0.032
θ_{max} = 24.87°
h = 0 → 21
k = 0 → 16
l = −10 → 0
3 standard reflections every 100 reflections
frequency: 150 min
intensity decay: negligible

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.035
wR(F²) = 0.115

(Δ/σ)_{max} = 0.048
Δρ_{max} = 0.127 e Å^{−3}
Δρ_{min} = −0.117 e Å^{−3}

$S = 0.972$
 1874 reflections
 145 parameters
 H atoms not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0407P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

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4-Chlorophenyl 2,6-Dimethylphenyl Sulfone

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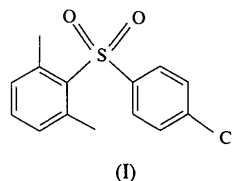
(Received 18 December 1996; accepted 14 April 1997)

Abstract

The phenyl rings in molecules of the title compound, C₁₄H₁₃ClO₂S, are perpendicular to one another. The crystal is stabilized by weak C—H···O intermolecular hydrogen bonding.

Comment

Like many sulfonamides and sulfones, the title compound, (I), has medicinal value (Krishnaiah, Narayana Raju, Lu, Chen & Narasinga Rao, 1995). It displays bond lengths which agree well with accepted values [*International Tables for Crystallography* (1992, Vol. C, Table 9.5.1.1)]. The C1—S1—C1' angle of 103.2(1)° is markedly less than the tetrahedral angle, 109.5°. This may be due to the short O2···C7 contact of 2.730(4) Å. The phenyl rings are perpendicular [dihedral angle 91.2(1)°] to one another.



The title structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). All non-H atoms were refined with anisotropic displacement parameters. All H atoms were located from difference Fourier maps and were included in the structure-factor calculations with isotropic displacement parameters equal to 1.1 U_{eq} of the respective carrier atom, but the parameters were not refined (*SHELXL93*; Sheldrick, 1993). The geometrical calculations were performed using *PARST* (Nardelli, 1983).

Data collection: *TEXSAN* (Molecular Structure Corporation, 1989). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1054). Services for accessing these data are described at the back of the journal.

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The crystal packing is stabilized by a weak intermolecular hydrogen bond [C2'—H2' 0.94(2), C2'···O2' 3.452(4), H2'···O2' 2.72(3) Å and C2'—H2'···O2' 136(2)°; symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, z + \frac{3}{2}$].

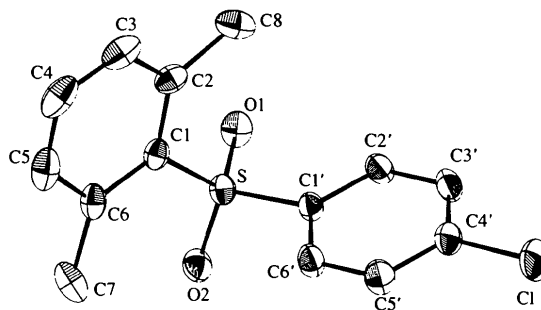


Fig. 1. The 50% probability displacement ellipsoid plot of the title molecule with the atom-numbering scheme. H atoms have been omitted.