organic papers

3-(3-Hydroxyphenyl)-1-(4-nitrophenyl)-prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.100
wR factor = 0.185
Data-to-parameter ratio = 12.1

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

The molecule of the title compound, $C_{15}H_{11}NO_4$, is almost planar and displays a *trans* configuration with respect to the C=C double bond.

Comment

Chalcone derivatives have been of great interest for many years. In the title compound, (I) (Fig. 1), all bond lengths are within normal ranges (Allen *et al.*, 1987). The C8=C9 bond length conforms to the value for a double bond. The bond length between atoms C7 and C8 is greater than the value for a double bond and less than the value for a single bond, due to conjugation effects in the molecule. The dihedral angle between the least-squares planes of the two benzene rings is 4.0 (2)°. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds (Fig. 2 and Table 1).
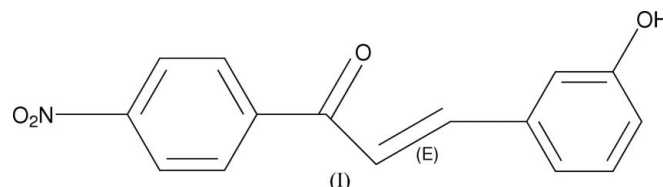
Experimental

The reagents were commercial products and were used without further purification. An aqueous solution of potassium hydroxide (20%, 1 ml) was added with stirring overnight to a solution of 3-hydroxybenzaldehyde (3 mmol, 0.37 g) and 4-nitroacetophenone (3 mmol, 0.50 g) in ethanol (95%, 15 ml) at room temperature. The reaction mixture was then poured into water (10 ml) and neutralized
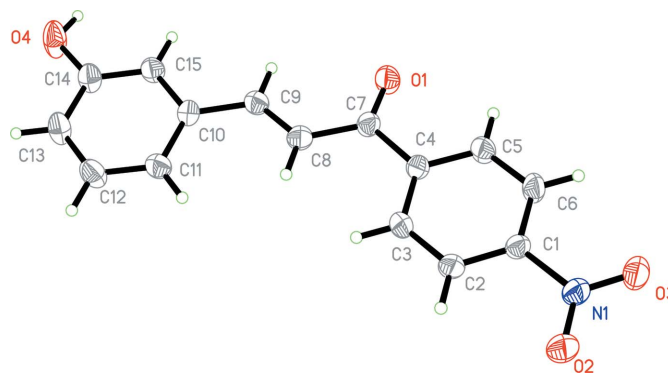

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

with hydrochloric acid (5%). A yellow solid was obtained on addition of ethanol. The solid was dissolved in acetone (12 ml) and stirred for about 10 min to give a clear solution. After allowing the solution to stand in air for 10 d, yellow block-shaped crystals were formed at the bottom of the vessel on slow evoporation of the solvent. These were collected, washed three times with acetone and dried in a vacuum desiccator using $CaCl_2$. The compound was isolated in 69% yield.

Crystal data

$C_{15}H_{11}NO_4$	$D_x = 1.408$ Mg m^{-3}
$M_r = 269.25$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2065
$a = 8.241$ (2) Å	reflections
$b = 12.698$ (3) Å	$\theta = 2.4$–$24.4°$
$c = 12.194$ (3) Å	$\mu = 0.10$ mm^{-1}
$\beta = 95.474$ (4)°	$T = 298$ (2) K
$V = 1270.3$ (5) Å^3	Block, yellow
$Z = 4$	$0.40 \times 0.40 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	1958 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.030$
Absorption correction: none	$\theta_{max} = 25.0°$
5093 measured reflections	$h = -9 \rightarrow 9$
2237 independent reflections	$k = -10 \rightarrow 15$
	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.100$	$+ 1.1432P]$
$wR(F^2) = 0.185$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.34$	$(\Delta/\sigma)_{max} < 0.001$
2237 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
185 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

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

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
O4—H1$\cdots$O1^i	0.86 (5)	1.86 (5)	2.706 (4)	165 (5)

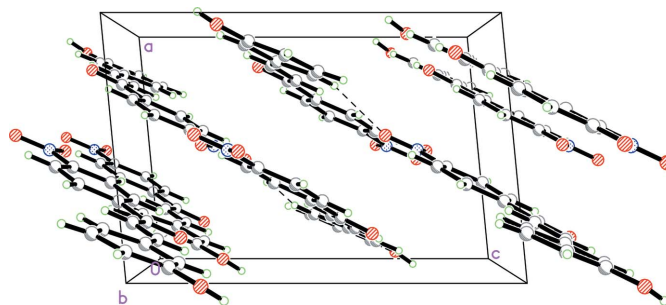Symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z - \frac{1}{2}$.

Figure 2
The crystal packing, viewed along the b axis, showing the intermolecular O—H$\cdots$O hydrogen bonds (dashed lines).

Atom H1 was located in a difference Fourier map and refined with an O—H distance restraint of 0.86 (5) Å. The other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å. They were treated as riding atoms with $U_{iso}(H)$ values set at $1.2U_{eq}(C)$ of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02), Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.