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Ethyl 4-(2-chloro-4-nitrophenoxy)benzoate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.126
Data-to-parameter ratio = 17.0

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

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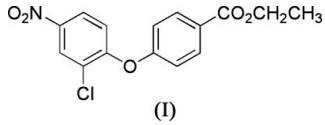
The title compound, $C_{15}H_{12}ClNO_5$, was synthesized by the reaction of ethyl 4-hydroxybenzoate and 1,2-dichloro-4-nitrobenzene in the presence of potassium hydroxide. In the V-shaped molecule, the dihedral angle between the two benzene rings is 72.90 (5)°.

Comment

Diphenyl ether analogs have been used successfully as herbicides (Harrington *et al.*, 1995). The molecular structure of the title compound, (I), is shown in Fig. 1 and selected bond angles are given in Table 1.



(I)

The molecule has a V-shaped conformation and the dihedral angle between the two benzene ring planes is 72.90 (5)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 2) connect molecules into extended chains.

Experimental

A mixture of ethyl 4-hydroxybenzoate (17.0 g, 102 mmol), KOH (6.0 g, 106 mmol) in dimethylformamide (50 ml) and toluene (80 ml) was heated to reflux for 6 h with a Dean–Stark apparatus to remove the water. 1,2-Dichloro-4-nitrobenzene (19 g, 99 mmol) was added and the resulting mixture was refluxed for 24 h. The desired product was extracted with ethyl acetate (120 ml). The organic layer was washed successively with 10% aqueous NaOH (50 ml × 3), saturated $NaHCO_3$ (50 ml) and brine (50 ml), dried over anhydrous $MgSO_4$ and concentrated to give a yellow solid (25 g, yield 78%). Single crystals suitable for X-ray analysis were obtained by recrystallization from ethanol.

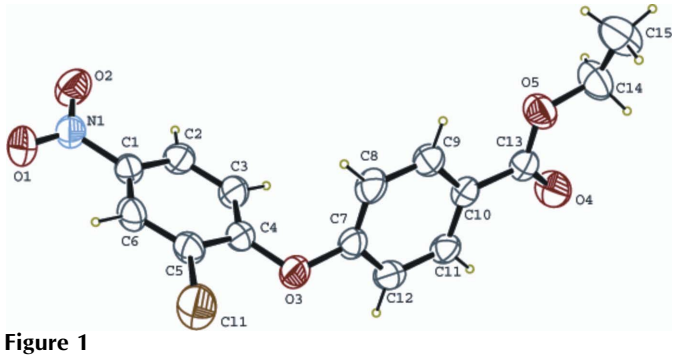


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 40% probability level.

Crystal data

$C_{15}H_{12}ClNO_5$
M_r = 321.71
Monoclinic, $P2_1/n$
a = 12.890 (6) Å
b = 8.846 (3) Å
c = 13.967 (5) Å
β = 111.230 (16)°
V = 1484.5 (10) Å^3
Z = 4
D_x = 1.439 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10694 reflections
θ = 3.1–27.5°
μ = 0.28 mm^{-1}
T = 296 (1) K
Block, colorless
0.38 × 0.28 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.891, T_{max} = 0.946
14122 measured reflections
3398 independent reflections
2406 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°
$h = -16 \rightarrow 16$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.06
3398 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.1257P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected bond angles (°).

C13—O5—C14	117.94 (14)	O1—N1—O2	123.27 (16)
C4—O3—C7	117.63 (12)	O4—C13—O5	123.36 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.58	3.418 (3)	151
C8—H8···O4^{ii}	0.93	2.47	3.362 (3)	162

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

The H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.96 (methylene) and 0.97 Å (methyl), and were included in a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl C atoms..

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Harrington, P. M., Singh, B. K., Szamosia, I. T. & Birk, J. H. (1995). *J. Agric. Food. Chem.* **43**, 804–808.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Version 3.00. Rigaku/MSC, 900 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.