

Chun-Hua Diao* and Ming Yu

College of Sciences, Tianjin University of
Science and Technology, Tianjin 300222,
People's Republic of ChinaCorrespondence e-mail:
diao_chunhua@163.com

Key indicators

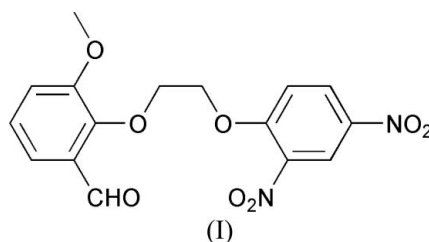
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.043
 wR factor = 0.119
Data-to-parameter ratio = 14.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.2-[2-(2,4-Dinitrophenoxy)ethoxy]-3-methoxy-
benzaldehyde

In the title compound, $\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_8$, the *o*-vanillin group makes a dihedral angle of $15.75(8)^\circ$ with the dinitrobenzene ring. The crystal structure is stabilized by weak non-classical intermolecular $\text{C}-\text{H}\cdots\text{O}=\text{N}$ hydrogen-bonding interactions, forming one-dimensional extended chains.

Received 16 October 2006
Accepted 31 October 2006

Comment

There has been a steady growth of interest in the structure and reactivity of Schiff bases due to their potential biological activities, such as antibacterial and antitumour properties (Klayman *et al.*, 1979). As part of our interest in the coordination properties of Schiff bases functioning as ligands, we have investigated the title compound, (I), which is used as a precursor in the preparation of Schiff bases.



In the title molecule (Fig. 1), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The *o*-vanillin group (atoms C2–C8/O1/O3) is essentially planar, with an r.m.s. deviation for fitted atoms of 0.0185 Å. This group makes a dihedral angle of $15.75(8)^\circ$ with the benzene ring (C11–C16). The two nitro groups and their attached aromatic ring are not coplanar; the dihedral angles between them are $19.40(15)^\circ$ and $7.91(16)^\circ$ for O5/N1/O6 and O7/N2/O8, respectively.

The crystal structure is stabilized by weak non-classical intermolecular $\text{C}-\text{H}\cdots\text{O}=\text{N}$ hydrogen-bonding interactions (Table 1), forming one-dimensional extended chains (Fig. 2).

Experimental

An anhydrous acetonitrile solution (50 ml) of 2-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) was added dropwise to a solution (100 ml) of 1-(2-bromoethoxy)-2,4-dinitrobenzene (2.91 g, 10 mmol) and potassium carbonate (1.38 g, 10 mmol) in acetonitrile over a period of 30 min. The mixture was then refluxed for 24 h under a nitrogen atmosphere. The solvent was removed and the resulting mixture poured into ice–water (100 ml). The yellow precipitate was isolated and recrystallized from acetonitrile, then dried in a vacuum to give the pure compound in 81% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{16}H_{14}N_2O_8$
 $M_r = 362.29$
 Monoclinic, $P2_1/c$
 $a = 13.192(2) \text{ \AA}$
 $b = 8.4397(16) \text{ \AA}$
 $c = 14.673(3) \text{ \AA}$
 $\beta = 94.990(3)^\circ$
 $V = 1627.5(5) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.479 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 294(2) \text{ K}$
 Block, yellow
 $0.32 \times 0.24 \times 0.20 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.943$, $T_{\max} = 0.976$

8940 measured reflections
 3339 independent reflections
 2001 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 26.4^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.119$
 $S = 1.02$
 3339 reflections
 236 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.1477P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C16-H16\cdots O6^i$	0.93	2.56	3.378 (3)	147

Symmetry code: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C–H bond lengths and isotropic $U_{\text{iso}}(\text{H})$ parameters: 0.93 \AA and $1.2U_{\text{eq}}(\text{C})$ for Csp^2 , 0.97 \AA and $1.2U_{\text{eq}}(\text{C})$ for methylene, and 0.96 \AA and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); 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 (1999). SMART (Version 5.0) and SAINT (Version 4.0) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Klayman, D. L., Bartosevich, J. F., Griffin, T. S., Mason, C. J. & Scovill, J. P. (1979). *J. Med. Chem.* **22**, 855–862.

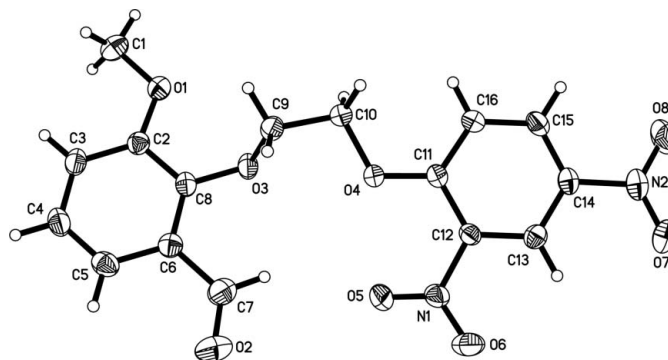


Figure 1

The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

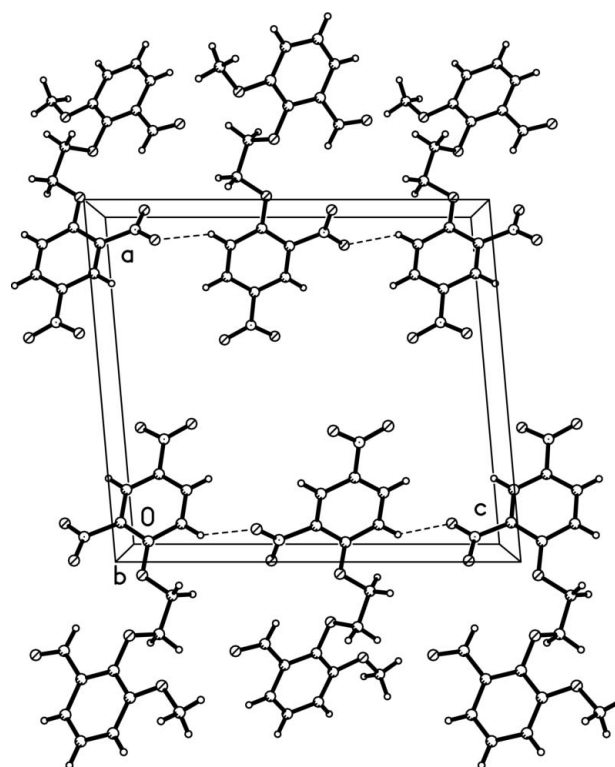


Figure 2

Intermolecular hydrogen-bonding interactions (dashed lines) in (I).

Sheldrick, G. M. (1996). SHELXS97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.10 for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.