

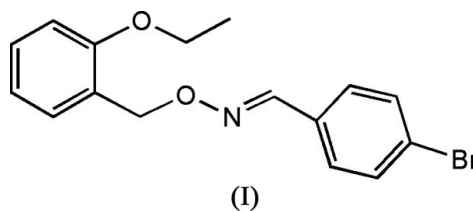
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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.034
 wR factor = 0.087
Data-to-parameter ratio = 14.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-Bromobenzaldehyde *O*-(2-ethoxybenzyl)oximeThe title compound, $\text{C}_{16}\text{H}_{16}\text{BrNO}_2$, is a potential new herbicide containing an oxime $\text{C}=\text{N}$ double bond. X-ray crystallographic analysis reveals that the double bond is in the *anti* form.Received 16 March 2006
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Comment

Saligenin derivatives exhibit good herbicidal activity. In our research on the bioactivities of these compounds, a series of *o*-benzyl-*anti*-benzaloxime derivatives has been synthesized from the reaction of 2-substituted benzyl chloride and 4-substituted benzaloxime. The crystal structure of the title compound, (I), will be helpful in the investigation of the relationship between structure and herbicidal activity.

The molecule of the title compound, (I), contains a central oxime ($-\text{C}=\text{N}-\text{O}$) unit (Fig. 1). The bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987), and in agreement with the corresponding values in 2,3-dimethylquinoxaline-dimethylglyoxime (1/1), (II) (Hökelek, Batı *et al.*, 2001) and 1-(2,6-dimethylphenylamino)propane-1,2-dione dioxime, (III) (Hökelek, Zülfikaroğlu & Batı, 2001). The central oxime unit *A* (O1/N1/C1) and benzene rings *B* (C2–C7) and *C* (C9–C14) are each planar; the dihedral angles between them are *A/B* 1.69 (3), *A/C* 5.77 (4) and *B/C* 7.40 (3)°.

In the oxime unit, the O1–N1 bond length and the O1–N1–C1 angle reflect the type and electron-withdrawing or electron-donating properties of the substituent bonded to atom C1, as in compounds (II) and (III). The C1=N double-bond length in the oxime unit is shorter than the corresponding bonds in (II) [1.2811 (18) and 1.2813 (19) Å] and (III) [1.290 (3) and 1.282 (3) Å], showing that the C1=N1 bond is conjugated with ring *B*; it is in the *anti* form.

There is an intramolecular hydrogen bond, C14–H14···O1 (Table 2) and, as a result, a pseudo-five-membered ring (C14–H14···O1–C8–C9) is formed (Fig. 1).

Experimental

2-Ethoxybenzyl chloride (1.5 mmol) was added to a solution of 4-bromobenzaldehyde (0.50 g, 2.0 mmol) and potassium hydroxide

(1.0 g, 18 mmol) in water (1 ml) and DMSO (3 ml) with stirring. The mixture was stirred at room temperature for an additional 2 h. After the reaction was complete, brine (20 ml) was added to the reaction mixture, and the product was extracted with CH₂Cl₂ three times. After drying, the solvent was removed and the title compound was separated through silica gel (yield 65%, m.p. 333–334 K). Recrystallization from CH₂Cl₂/petroleum ether (1:7) yielded single crystals suitable for X-ray analysis.

Crystal data

C₁₆H₁₆BrNO₂ $Z = 2$
 $M_r = 334.21$ $D_x = 1.488 \text{ Mg m}^{-3}$
 Triclinic, $P\bar{1}$ Mo $K\alpha$ radiation
 Cell parameters from 1769 reflections
 $a = 7.626 (3) \text{ \AA}$ $\theta = 2.9\text{--}26.3^\circ$
 $b = 7.833 (3) \text{ \AA}$ $\mu = 2.76 \text{ mm}^{-1}$
 $c = 13.934 (6) \text{ \AA}$ $T = 294 (2) \text{ K}$
 $\alpha = 91.501 (7)^\circ$ Prism, colorless
 $\beta = 92.925 (7)^\circ$ $0.24 \times 0.22 \times 0.18 \text{ mm}$
 $\gamma = 115.968 (6)^\circ$
 $V = 746.2 (5) \text{ \AA}^3$

Data collection

Bruker SMART CCD area-detector 2614 independent reflections
 diffractometer 2033 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{\text{int}} = 0.022$
 Absorption correction: multi-scan $\theta_{\text{max}} = 25.0^\circ$
 (SADABS; Sheldrick, 1996) $h = -9 \rightarrow 9$
 $T_{\text{min}} = 0.519$, $T_{\text{max}} = 0.607$ $k = -9 \rightarrow 9$
 3800 measured reflections $l = -16 \rightarrow 8$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.2986P]$
 $R[F^2 > 2\sigma(F^2)] = 0.034$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.088$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 $S = 1.03$ $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 2614 reflections $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
 182 parameters
 H-atom parameters constrained

Table 1

Selected geometric parameters (\AA , $^\circ$).

Br1—C5	1.894 (3)	O2—C10	1.361 (3)
N1—C1	1.262 (4)	O2—C15	1.432 (3)
N1—O1	1.399 (3)	C1—C2	1.454 (4)
O1—C8	1.425 (3)	C8—C9	1.500 (4)
C1—N1—O1	111.7 (2)	N1—C1—C2	121.4 (3)
N1—O1—C8	108.34 (19)	O1—C8—C9	109.0 (2)
C1—N1—O1—C8	179.6 (3)	C15—O2—C10—C9	177.3 (2)
O1—N1—C1—C2	−179.5 (2)	C8—C9—C10—O2	−1.6 (4)
N1—O1—C8—C9	−178.6 (2)	C10—O2—C15—C16	−177.9 (3)

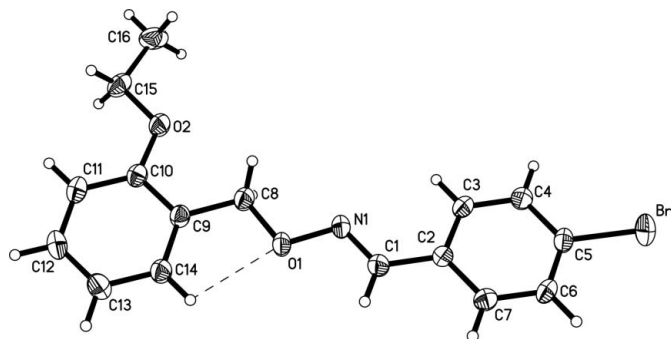


Figure 1

The structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

Table 2

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

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C14—H14 \cdots O1	0.93	2.37	2.723 (4)	102

H atoms were positioned geometrically, with C—H = 0.93, 0.96 and 0.97 \AA for aromatic, methyl and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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