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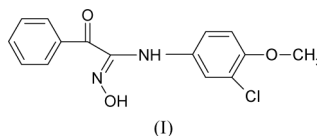
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.032
 wR factor = 0.078
Data-to-parameter ratio = 12.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N*-(3-Chloro-4-methoxyphenyl)-*N'*-
hydroxy-2-oxo-2-phenylacetamide**

The title compound, $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_3\text{Cl}$, consists of two aromatic groups linked through the $\text{C}=\text{N}-\text{OH}$ monoxime group. Intermolecular $\text{O}-\text{H}\cdots\text{N}$, $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds [$\text{O}\cdots\text{N}$ 2.773 (2), $\text{N}\cdots\text{Cl}$ 3.719 (2) Å] are highly effective in forming polymeric chains, thereby stabilizing the crystal structure. The compound has an *E* configuration.

Comment

Compounds containing oxime groups are crucially important in the chemical industry and numerous studies on their derivatives have been performed (Bertolasi *et al.*, 1982; Gilli *et al.*, 1983; Hökelek *et al.*, 2001; Büyükgüngör *et al.*, 2003; Saarinen *et al.*, 1978; Mokhr *et al.*, 2002). It is well known that the oximato group ($\text{C}=\text{N}-\text{OH}$) can function as a bridge to bind two metal ions through the imino nitrogen and deprotonated oxygen, which can then coordinate with metal ions in diverse ways. With the aim of investigating the versatility of oximes in coordination chemistry and further researching the supramolecular polymer chemistry, successful syntheses with oximato groups were carried out (Liu *et al.*, 2002). Oxime groups possess stronger hydrogen-bonding capabilities than alcohols, phenols and carboxylic acids (Hökelek *et al.*, 2001). In this paper, we report the synthesis, molecular and crystal structure of the title compound, (I). The crystal structure determination was carried out in order to investigate the strength of the hydrogen-bonding capabilities of the oxime group, and to compare the geometry of the oxime moiety with those found in *N*-hydroxy-2-oxo-2,*N'*-diphenylacetamide (II) (Büyükgüngör *et al.*, 2003), *N*-(3,4-dichlorophenyl)-*N'*-hydroxy-2-oxo-2-phenylacetamide, (III) (Hökelek *et al.*, 2003) and 1-(2,6-dimethylphenylamino)propane-1,2-dione dioxime, (IV) (Hökelek *et al.*, 2001). Comparative geometrical parameters in the oxime moiety of (I) and in the related compounds (II), (III) and (IV) are summarized in Table 3.



The title compound, (I) (Fig. 1), consists of two aromatic groups, linked through a monoxime group. The dihedral angles between the oxime plane *A* (O1/N1/C1) and the non-coplanar benzene rings *B* (C3–C8) and *C* (C9–C14) are $A/B = 63.7(1)^\circ$, $A/C = 37.2(1)^\circ$ and $B/C = 86.56(5)^\circ$. Ring *B* is linked to the $\text{C}=\text{N}-\text{OH}$ group by atom C2(O2). The other

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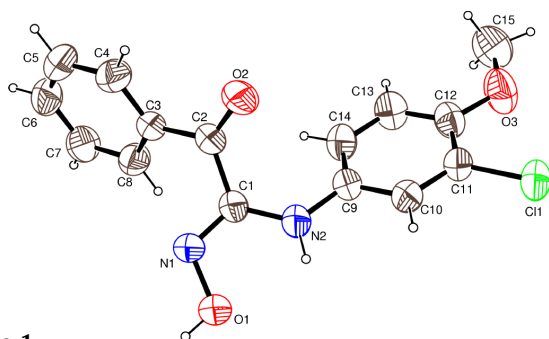


Figure 1
ORTEP drawing of the title molecule, with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

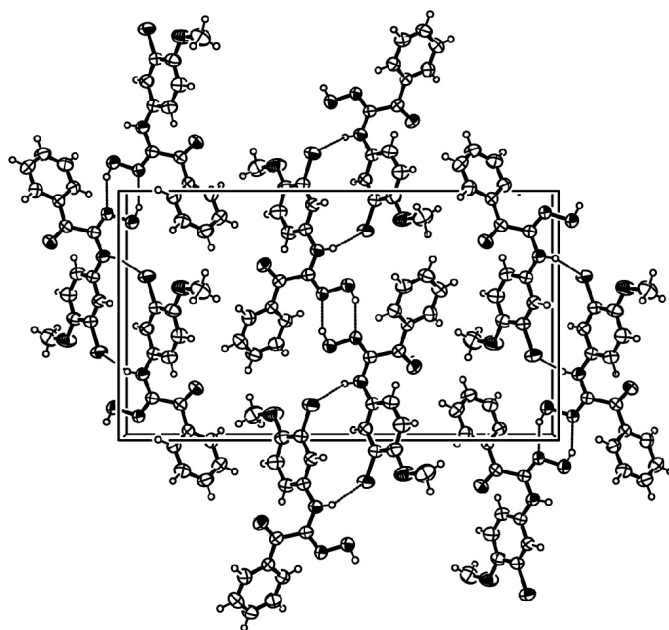


Figure 2
Packing diagram for (I), viewed along the *a* axis.

ring (C) is linked to the same functional group by atom N2(H2).

The oxime moiety has an *E* configuration [C2–C1=N1–O1 = –169.97 (14)°] (Hökelek *et al.*, 2001; Ellis *et al.*, 1997). In this configuration, the oxime group is involved as a donor in intermolecular hydrogen bonding [O1–H1...N1ⁱ 2.773 (2) Å; symmetry code: (i) 2 – *x*, 1 – *y*, 1 – *z*]. There is also an intermolecular N–H...Cl hydrogen bond [N2...Cl1ⁱⁱ 3.71 (18) Å; symmetry code: (ii) 2 – *x*, –*y*, 1 – *z*] (Table 2). These intermolecular hydrogen bonds are highly effective in forming polymeric chains, thereby stabilizing the crystal structure (Fig. 2). As shown in Fig. 2, the chains extend parallel to the *b* axis. Dipole–dipole and van der Waals interactions are also effective in the molecular packing in the crystal structure.

Experimental

The title compound was prepared from a mixture of *o*-chloroisitroacetophenone (2.75 g, 0.015 mol) and 3-chloro-4-methoxyaniline (2.36 g, 0.03 mol) in dichloromethane (40 ml). The precipitate

was filtered and was then heated at 293 K for 72 h. The crystallized product was filtered off, washed with ethyl alcohol and dried.

The IR spectrum of the complex was recorded with an IASCO FT/IR-430 spectrophotometer; IR spectroscopic data ν 3392 (N–H); 3223 (O–H); 1668 (C=O); 1633 (C=N); 972 (N–O). Elemental analyses: calc. C, 59.12; H, 4.30; N, 9.19; found: C, 59.21; H, 4.42; N, 9.31. From UV–VIS measurements the UV transitions of $\pi \rightarrow \pi^*$ and of $n \rightarrow H^*$ were observed at 236 ($\epsilon = 75888$)–253 ($\epsilon = 51824$) nm and at 253 ($\epsilon = 4580$) nm, respectively. The resonance values of the (C=N–OH) proton at 11.25 p.p.m., the (N–H) proton at 8.64 p.p.m., the aromatic protons between 7.99–6.65 p.p.m. and the OCH₃ protons at 3.74 p.p.m. were obtained from the ¹H NMR spectrum (AC-200 Bruker FT NMR).

Crystal data

C ₁₅ H ₁₃ ClN ₂ O ₃	$D_x = 1.408 \text{ Mg m}^{-3}$
$M_r = 304.72$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8473 reflections
$a = 5.0135$ (7) Å	$\theta = 0.0\text{--}29.4^\circ$
$b = 12.7483$ (11) Å	$\mu = 0.28 \text{ mm}^{-1}$
$c = 22.495$ (3) Å	$T = 293$ (2) K
$\beta = 91.553$ (11)°	Prism, yellow
$V = 1437.2$ (3) Å ³	0.45 × 0.32 × 0.30 mm
$Z = 4$	

Data collection

Stoe IPDS 2 diffractometer	$R_{\text{int}} = 0.058$
φ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -6 \rightarrow 6$
13425 measured reflections	$k = 0 \rightarrow 17$
2518 independent reflections	$l = 0 \rightarrow 30$
1622 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.078$	$(\Delta\rho)_{\text{max}} = 0.001$
$S = 0.83$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
2518 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
199 parameters	Extinction correction: <i>SHELXL</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0186 (18)

Table 1

Selected geometric parameters (Å, °).

Cl1–C11	1.7313 (17)	O3–C15	1.411 (2)
N1–C1	1.282 (2)	C1–C2	1.502 (2)
N1–O1	1.4063 (17)	C2–C3	1.479 (2)
N2–C1	1.353 (2)	C3–C8	1.382 (2)
N2–C9	1.412 (2)	C3–C4	1.386 (2)
O2–C2	1.2117 (19)	C9–C14	1.356 (2)
O3–C12	1.364 (2)	C9–C10	1.382 (2)
C1–N1–O1	111.00 (14)	C8–C3–C2	122.58 (15)
C1–N2–C9	128.97 (16)	C4–C3–C2	118.25 (16)
N1–C1–N2	123.02 (15)	C14–C9–N2	123.78 (16)
N1–C1–C2	113.75 (15)	C10–C9–N2	117.15 (16)
N2–C1–C2	122.32 (15)	C10–C11–Cl1	119.55 (14)
O2–C2–C3	122.37 (15)	C12–C11–Cl1	119.44 (13)
O2–C2–C1	119.18 (15)	O3–C12–C11	116.61 (16)
C3–C2–C1	118.36 (14)	O3–C12–C13	125.28 (18)
O1–N1–C1–C2	–169.97 (14)	O2–C2–C3–C8	151.55 (18)
C9–N2–C1–N1	167.99 (19)	O2–C2–C3–C4	–26.7 (3)
C9–N2–C1–C2	–23.6 (3)	C1–N2–C9–C14	–29.5 (3)
N2–C1–C2–O2	–34.0 (3)	C1–N2–C9–Cl1	153.3 (2)
N1–C1–C2–C3	–41.1 (2)	Cl1–C11–C12–O3	–0.6 (2)
N2–C1–C2–C3	149.54 (18)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N1 ⁱ	0.88 (2)	1.95 (2)	2.773 (2)	154 (2)
N2—H2 \cdots Cl1 ⁱⁱ	0.865 (18)	2.985 (18)	3.7186 (18)	143.9 (15)

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $2-x, -y, 1-z$.**Table 3**
Comparative geometrical parameters (Å, °) in the oxime moiety of (I) and in the related compounds (II), (III) and (IV).

	(I)	(II)	(III)	(IV)
N1—O1	1.4063 (17)	1.4167 (10)	1.429 (4)	1.423 (3)
N1—C1	1.282 (2)	1.2897 (12)	1.241 (6)	1.290 (3)
C1—C2	1.502 (2)	1.5098 (13)	1.551 (7)	1.489 (3)
C2—C1—N1	113.75 (15)	114.32 (8)	118.3 (5)	116.59 (19)
C1—N1—O1	111.00 (14)	110.66 (8)	112.2 (4)	109.37 (18)

H atoms on N and O atoms were found in a difference map and their coordinates were refined. All other H atoms were placed in calculated positions (C—H 0.93–0.96 Å) and refined isotropically ($U_{\text{iso}} = 1.2U_{\text{eq}}$ of the parent atom).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s)

used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*III Farrugia (1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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