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Hydrogen-bonded $R_2^2(8)$ dimers in (*E*)-{[2-(phenyldiazenyl)phenyl]ethylideneaminooxy}acetic acid

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Molecules of the title compound, $C_{16}H_{15}N_3O_3$, are linked by paired O—H···O hydrogen bonds [H···O = 1.84 Å, O···O = 2.655 (3) Å and O—H···O = 175°] into centrosymmetric $R_2^2(8)$ dimers.

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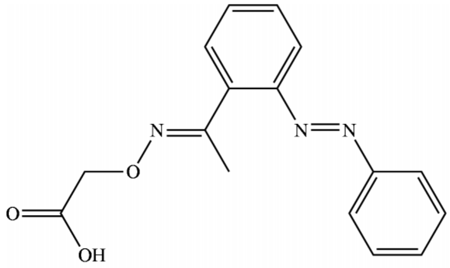
Key indicators

Single-crystal X-ray study
T = 120 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.069
wR factor = 0.187
Data-to-parameter ratio = 16.2

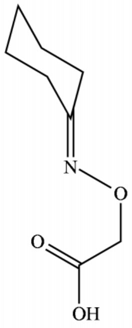
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

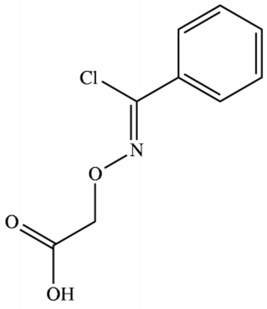
Persulfate oxidation of iminooxyacetic acids, *R*(*R*′)C═N-OCH$_2$COOH, provides a useful route to iminyl radicals (Forrester *et al.*, 1979). The subsequent reactions of the iminyl radicals thus generated depend greatly on the substituents, and important species including nitrogen-containing heterocycles can result. We report here the crystal structure of the title compound, (I) (Fig. 1), and we compare (I) with the simpler analogues (II) and (III), whose structures we reported recently (Glidewell *et al.*, 2004).



(I)



(II)



(III)

The C—O distances in the carboxyl group of (I) (Table 1) are consistent with the fully ordered location of the carboxyl H atom as deduced from a difference map. The distance O3—N4 corresponds exactly with the mean value for the —O—N═ bond in oximes (Allen *et al.*, 1987). The other distances are all typical of their types. The interbond angle at O3 is less than the idealized tetrahedral value, and those at N4, N11 and N12

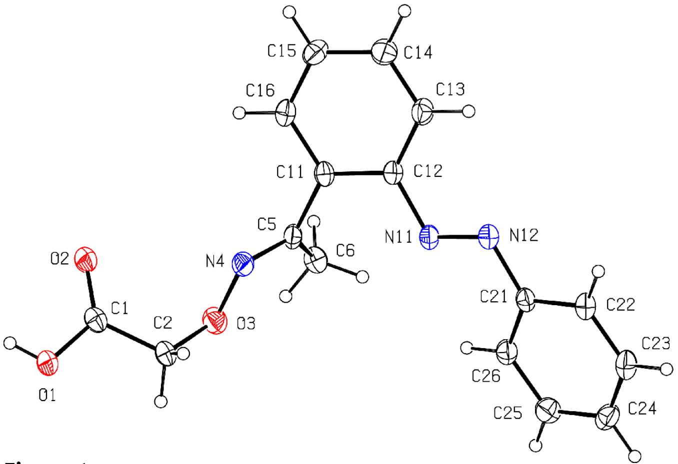


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

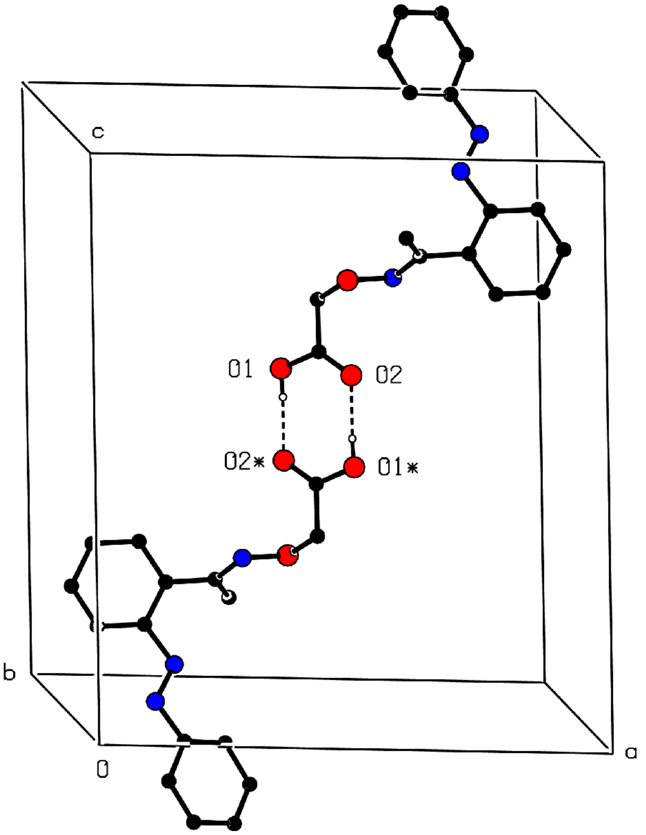


Figure 2
Part of the crystal structure of (I), showing the formation of an $R_2^2(8)$ dimer centred at (0.5, 0.5, 0.5). For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($1 - x$, $1 - y$, $1 - z$).

are all significantly less than 120°, pointing in every case to the stereochemical influence of the unshared pairs of electrons. The angles at C5 indicate planarity at this atom. While the azobenzene fragment is effectively planar, as indicated by the key torsion angles (Table 1), the conformation of the side chain between O1 and C11 (Fig. 1) is not amenable to simple explanation. Compounds (I) and (III) have configurations at the C═N double bond [*viz*. (*E*) in (I) and (*Z*) in (III)] such that the sterically smaller substituent at C5 is on the same side as the OCH_2COOH substituent.

The molecules of (I) are linked by paired, and nearly linear, O—H···O hydrogen bonds (Table 2) into a centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimer, selected for the sake of convenience to lie across the inversion centre at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) (Fig. 2). In this respect, the primary aggregation in (I) is the same at that in (II) and (III). The structure of (I) differs from that of (III), however, in that there are no direction-specific interactions between the dimers in (I), whereas those in (III) are linked into chains by a single aromatic π–π stacking interaction.

Experimental

The title compound was prepared by reaction of $ClCH_2CO_2H$ with the oxime derived from 2-(PhN═N)$C_6H_4COCH_3$ (Forrester *et al.*, 1979), following the general procedure described recently (Glidewell *et al.*, 2004). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in ethanol (m.p. 420–421 K).

Crystal data

$C_{16}H_{15}N_3O_3$
$M_r = 297.31$
Monoclinic, $P2/c$
$a = 16.927$ (2) Å
$b = 4.5244$ (5) Å
$c = 19.673$ (2) Å
$\beta = 105.828$ (5)°
$V = 1449.5$ (3) Å^3
$Z = 4$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3256 reflections
$\theta = 3.7$–27.6°
$\mu = 0.10$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.24 × 0.18 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{\min} = 0.967$, $T_{\max} = 0.990$
13 640 measured reflections
3256 independent reflections
1443 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.114$
$\theta_{\max} = 27.6°$
$h = -21 \rightarrow 21$
$k = -5 \rightarrow 5$
$l = -25 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.187$
$S = 0.95$
3256 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.306 (4)	C2—O3	1.419 (4)
C1—O2	1.216 (4)	O3—N4	1.416 (3)
C1—C2	1.497 (5)	N4—C5	1.321 (4)
C2—O3—N4	107.6 (2)	N4—C5—C6	126.8 (3)
O3—N4—C5	107.8 (2)	N4—C5—C11	114.1 (3)
N12—N11—C12	114.1 (2)	C6—C5—C11	119.0 (3)
N11—N12—C21	113.9 (3)		
O1—C1—C2—O3	157.5 (3)	N4—C5—C11—C12	114.9 (3)
C1—C2—O3—N4	83.0 (3)	C11—C12—N11—N12	−171.6 (3)
C2—O3—N4—C5	174.9 (3)	C12—N11—N12—C21	−177.0 (2)
O3—N4—C5—C11	176.4 (2)	N11—N12—C21—C22	−169.2 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.84	2.655 (3)	175

Symmetry code: (i) $1-x, 1-y, 1-z$.

All H atoms were located in difference maps and then treated as riding atoms in idealized positions, with distances C—H = 0.95 (aromatic), 0.98 (CH_3) or 0.99 Å (CH_2), and O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,O)$ or $1.5U_{eq}(C_{methyl})$. The low proportion (*ca* 44%) of the reflections labelled observed, even at 120 (2) K, together with the rather high merging index (0.11), are suggestive of poorly diffracting crystals, which may in turn be associated with the very limited intermolecular aggregation.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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