

(Cyclohexylideneamino)oxyacetic acid and [chloro(phenyl)methyleneamino]oxyacetic acid: hydrogen-bonded $R_2^2(8)$ dimers and aromatic π - π stacking interactions

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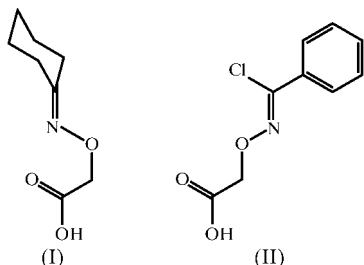
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Molecules of (cyclohexylideneamino)oxyacetic acid, $C_8H_{13}NO_3$, (I), are linked into centrosymmetric dimers by pairs of $O-H\cdots O$ hydrogen bonds [$H\cdots O = 1.84$ Å, $O\cdots O = 2.6782$ (12) Å and $O-H\cdots O = 178^\circ$]. In [chloro(phenyl)methyleneamino]oxyacetic acid, $C_9H_8ClNO_3$, (II), the molecules are similarly linked into centrosymmetric dimers by pairs of $O-H\cdots O$ hydrogen bonds [$H\cdots O = 1.79$ Å, $O\cdots O = 2.6329$ (17) Å and $O-H\cdots O = 176^\circ$], and these dimers are weakly linked into chains by a single type of aromatic π - π stacking interaction.

Comment

Persulfate oxidation of iminoxyacetic acids, $R(R')C=NOCH_2COOH$, 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 molecular and supra-molecular structures of two representative examples of such



precursors, *viz.* (cyclohexylideneamino)oxyacetic acid, (I), and [chloro(phenyl)methyleneamino]oxyacetic acid, (II).

In both (I) and (II) (Figs. 1 and 2), the C—O distances in the carboxy groups (Tables 1 and 3) are consistent with the fully ordered locations of the carboxy H atoms as deduced from difference maps. In the side chains of (I) and (II), the corresponding distances show very similar values, apart from the $O3-N4$ distances, which are significantly different; for comparison, the mean value for the $-O-N=$ bond in oximes is 1.416 Å (Allen *et al.*, 1987). The other distances are typical for bonds of their types.

The conformations of the side chains both exhibit near-planar fragments, *viz.* $O1-C1-C2-O3$ and $C2-O3-N4-Cn$ [$n = 11$ in (I) and $n = 5$ in (II); see Figs. 1 and 2], but while the intervening $C1-C2-O3-N4$ torsion angles have similar magnitudes in (I) and (II), they have opposite signs, and it is this difference that determines the different overall conformations of these two molecules. For the cyclohexylidene ring in (I), the ring-puckering parameters (Cremer & Pople, 1975) corresponding to the atom sequence $C11-C16$ [$\varphi = 174.0$ (2°) and $\theta = 12$ (2°)] indicate a conformation close to the chair form (Evans & Boeyens, 1989), despite the planarity at atom C11.

In the structures of both (I) and (II), the molecules are linked into centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimers (Figs. 3 and 4) by paired $O-H\cdots O$ hydrogen bonds, which are fairly short and effectively linear (Tables 2 and 4). In

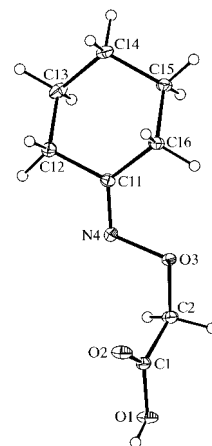


Figure 1

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

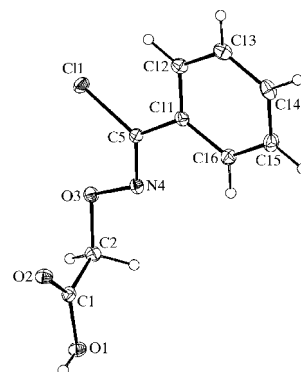


Figure 2

The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

each structure, the reference molecule has been selected so that the hydrogen-bonded dimer in which this molecule participates is centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. In the structure of (I), there are no further direction-specific interactions between the molecules, but in (II), the $R_2^2(8)$ dimers are weakly linked into

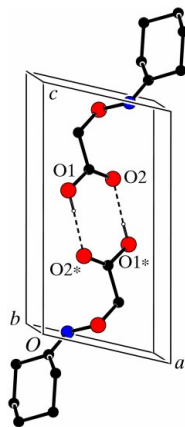


Figure 3
Part of the crystal structure of (I), showing the formation of an $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

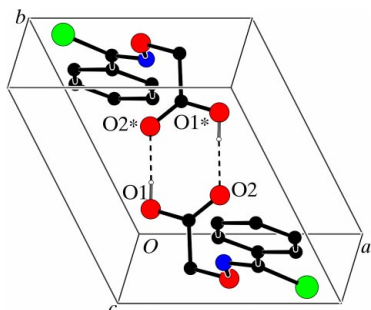


Figure 4
Part of the crystal structure of (II), showing the formation of an $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

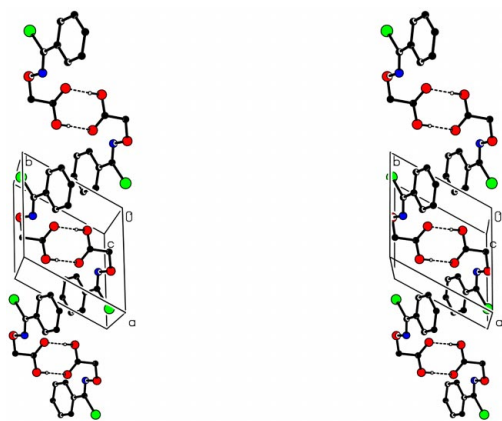


Figure 5
A stereoview of part of the crystal structure of (II), showing the formation of a π -stacked [101] chain of hydrogen-bonded dimers. For clarity, H atoms bonded to C atoms have been omitted.

chains by a single aromatic π - π stacking interaction. The aryl rings in the molecules at (x, y, z) and $(2-x, 1-y, 2-z)$ are parallel, with an interplanar spacing of 3.422 (2) Å; the ring-centroid separation is 3.856 (2) Å, corresponding to a centroid offset of 1.777 (2) Å. The molecules at (x, y, z) and $(2-x, 1-y, 2-z)$ are components of hydrogen-bonded dimers centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and $(1, \frac{1}{2}, 1)$, respectively, and propagation by inversion of the two intermolecular interactions generates a chain running parallel to the [101] direction (Fig. 5).

In neither (I) nor (II) do atoms O3 and N4 act as acceptors of C-H...X (X = O or N) hydrogen bonds; there are no intermolecular H...X contact distances involving O3 or N4 that are less than 2.60 Å in (I), and none of less than 2.70 Å in (II).

Experimental

Benzhydroxamoyl chloride, Cl(Ph)C=NOH, was prepared from H(Ph)C=NOH according to the method of Baruah *et al.* (1988). Compounds (I) and (II) were prepared by reaction of chloroacetic acid with either cyclohexanone oxime [for (I)] or Cl(Ph)C=NOH [for (II)], using the following modification of the procedure of Forrester *et al.* (1979). A solution of the oxime (0.10 mol), chloroacetic acid (0.20 mol) and sodium hydroxide (0.40 mol) in a mixture of water (100 ml) and ethanol (50 ml) was heated under reflux overnight. The cooled solution was poured on to ice and acidified with dilute hydrochloric acid. The precipitate was collected, washed with water and dissolved in NaHCO₃ solution (100 ml of 1 mol dm⁻³). This solution was extracted with diethyl ether and acidified with dilute hydrochloric acid. The resulting solid was collected, washed with water and recrystallized from ethanol, yielding crystals suitable for single-crystal X-ray diffraction; m.p.: (I) 365–367 K, (II) 381–383 K.

Compound (I)

Crystal data

C₈H₁₃NO₃
M_r = 171.19
 Triclinic, $P\bar{1}$
a = 5.0776 (1) Å
b = 8.6533 (3) Å
c = 10.7804 (3) Å
 α = 111.2609 (15)°
 β = 102.437 (2)°
 γ = 93.597 (2)°
V = 425.88 (2) Å³

Z = 2
D_x = 1.335 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 1945 reflections
 θ = 3.9–27.5°
 μ = 0.10 mm⁻¹
T = 120 (2) K
 Block, colourless
 0.40 × 0.30 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
 φ scans, and ω scans with κ offsets
 Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)
T_{min} = 0.917, *T_{max}* = 0.968
 9176 measured reflections
 1945 independent reflections

1621 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
 θ_{\max} = 27.5°
h = -5 → 6
k = -11 → 11
l = -13 → 13

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.038
 $wR(F^2)$ = 0.098
S = 1.04
 1945 reflections
 110 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.1199P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e \AA}^{-3}$

Table 1
Selected geometric parameters (Å, °) for (I).

C1—O1	1.3186 (14)	O3—N4	1.4331 (12)
C1—O2	1.2134 (14)	N4—C11	1.2775 (15)
O1—C1—O2	124.23 (11)	C2—O3—N4	107.87 (8)
O1—C1—C2	111.52 (10)	O3—N4—C11	111.71 (9)
O2—C1—C2	124.24 (10)		
O1—C1—C2—O3	−177.14 (9)	C2—O3—N4—C11	−175.25 (9)
O2—C1—C2—O3	1.83 (17)	O3—N4—C11—C12	179.11 (9)
C1—C2—O3—N4	−83.62 (11)	O3—N4—C11—C16	1.24 (16)

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O2 ⁱ	0.84	1.84	2.6782 (12)	178

Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*.

Compound (II)

Crystal data

C₉H₈CINO₃
M_r = 213.61
 Triclinic, *P* $\bar{1}$
a = 6.7961 (1) Å
b = 7.3857 (2) Å
c = 10.8173 (3) Å
 α = 98.4852 (9)°
 β = 93.4156 (10)°
 γ = 117.0191 (12)°
V = 473.44 (2) Å³
Z = 2
D_x = 1.498 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 2133 reflections
 θ = 3.2–27.4°
 μ = 0.38 mm^{−1}
T = 120 (2) K
 Plate, colourless
 0.28 × 0.20 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
 φ scans, and ω scans with κ offsets
 Absorption correction: multi-scan
 (SORTAV; Blessing, 1995, 1997)
T_{min} = 0.922, *T_{max}* = 0.981
 3988 measured reflections
 2133 independent reflections
 1980 reflections with *I* > 2σ(*I*)
R_{int} = 0.031
 θ_{\max} = 27.4°
h = −8 → 8
k = −9 → 9
l = −13 → 13

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.040
wR (*F*²) = 0.141
S = 1.20
 2132 reflections
 128 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0766P)^2 + 0.1738P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.48 \text{ e } \text{Å}^{-3}$

Crystals of (I) and (II) are triclinic; space group *P* $\bar{1}$ was selected for each and confirmed in both cases by the subsequent analyses. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic) or 0.99 Å (CH₂), and O—H distances of 0.84 Å.

For both compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to

Table 3
Selected geometric parameters (Å, °) for (II).

C5—C11	1.738 (2)	O3—N4	1.405 (2)
C1—O1	1.311 (2)	N4—C5	1.273 (2)
C1—O2	1.226 (2)		
O1—C1—O2	124.64 (16)	C2—O3—N4	107.45 (13)
O1—C1—C2	112.36 (14)	O3—N4—C5	113.12 (14)
O2—C1—C2	123.00 (16)		
O1—C1—C2—O3	−171.61 (14)	C2—O3—N4—C5	−173.61 (14)
C1—C2—O3—N4	75.87 (17)		

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O2 ⁱ	0.84	1.79	2.6329 (17)	176

Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*.

solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants that have provided computing facilities for this work. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG1209). Services for accessing these data are described at the back of the journal.

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supporting information

Acta Cryst. (2004). C60, o270–o272 [doi:10.1107/S0108270104004299]

(Cyclohexylideneamino)oxyacetic acid and [chloro(phenyl)methyleneamino]-oxyacetic acid: hydrogen-bonded $R[2^2](8)$ dimers and aromatic π – π stacking interactions

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Computing details

For both compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) (Cyclohexylideneamino)oxyacetic acid

Crystal data

$C_8H_{13}NO_3$	$Z = 2$
$M_r = 171.19$	$F(000) = 184$
Triclinic, $P\bar{1}$	$D_x = 1.335 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 5.0776 (1) \text{ \AA}$	Cell parameters from 1945 reflections
$b = 8.6533 (3) \text{ \AA}$	$\theta = 3.9\text{--}27.5^\circ$
$c = 10.7804 (3) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 111.2609 (15)^\circ$	$T = 120 \text{ K}$
$\beta = 102.437 (2)^\circ$	Block, colourless
$\gamma = 93.597 (2)^\circ$	$0.40 \times 0.30 \times 0.30 \text{ mm}$
$V = 425.88 (2) \text{ \AA}^3$	

Data collection

Nonius KappaCCD diffractometer	9176 measured reflections
Radiation source: rotating anode	1945 independent reflections
Graphite monochromator	1621 reflections with $I > 2\sigma(I)$
φ scans, and ω scans with κ offsets	$R_{\text{int}} = 0.042$
Absorption correction: multi-scan (<i>SORTAV</i> ; Blessing, 1995, 1997)	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.9^\circ$
$T_{\text{min}} = 0.917$, $T_{\text{max}} = 0.968$	$h = -5 \rightarrow 6$
	$k = -11 \rightarrow 11$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	1945 reflections
Least-squares matrix: full	110 parameters
$R[F^2 > 2\sigma(F^2)] = 0.038$	0 restraints
$wR(F^2) = 0.098$	Primary atom site location: structure-invariant
$S = 1.04$	direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.1199P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.25509 (18)	0.43807 (11)	0.58143 (9)	0.0258 (2)
O2	0.63107 (18)	0.63065 (11)	0.66261 (9)	0.0253 (2)
O3	0.53648 (16)	0.75100 (10)	0.92037 (8)	0.0198 (2)
N4	0.76773 (19)	0.68931 (12)	0.97793 (10)	0.0191 (2)
C1	0.4331 (2)	0.56279 (14)	0.67960 (12)	0.0180 (2)
C2	0.3569 (2)	0.61259 (14)	0.81506 (11)	0.0197 (3)
C11	0.9316 (2)	0.80606 (14)	1.08118 (11)	0.0169 (2)
C12	1.1796 (2)	0.75439 (15)	1.15083 (13)	0.0229 (3)
C13	1.2053 (3)	0.81203 (15)	1.30557 (13)	0.0243 (3)
C14	1.1915 (2)	0.99871 (15)	1.36960 (12)	0.0202 (3)
C15	0.9271 (2)	1.03967 (15)	1.29856 (12)	0.0204 (3)
C16	0.9009 (2)	0.98760 (14)	1.14378 (11)	0.0188 (3)
H1	0.2940	0.4191	0.5059	0.031*
H2A	0.1694	0.6406	0.8025	0.024*
H2B	0.3569	0.5159	0.8435	0.024*
H12A	1.3448	0.8042	1.1357	0.027*
H12B	1.1665	0.6307	1.1098	0.027*
H13A	1.0561	0.7477	1.3210	0.029*
H13B	1.3810	0.7889	1.3509	0.029*
H14A	1.3495	1.0637	1.3611	0.024*
H14B	1.2017	1.0318	1.4688	0.024*
H15A	0.9237	1.1617	1.3408	0.024*
H15B	0.7696	0.9802	1.3122	0.024*
H16A	0.7204	1.0051	1.0985	0.023*
H16B	1.0435	1.0579	1.1294	0.023*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0266 (5)	0.0269 (5)	0.0144 (4)	-0.0078 (4)	0.0022 (3)	0.0008 (3)
O2	0.0272 (5)	0.0249 (5)	0.0169 (4)	-0.0064 (4)	0.0039 (4)	0.0027 (3)
O3	0.0203 (4)	0.0184 (4)	0.0150 (4)	0.0042 (3)	-0.0013 (3)	0.0030 (3)
N4	0.0183 (5)	0.0195 (5)	0.0178 (5)	0.0051 (4)	0.0020 (4)	0.0065 (4)
C1	0.0192 (5)	0.0148 (5)	0.0160 (6)	0.0016 (4)	0.0000 (4)	0.0041 (4)
C2	0.0190 (6)	0.0206 (6)	0.0149 (6)	-0.0002 (4)	0.0012 (4)	0.0037 (5)
C11	0.0185 (5)	0.0176 (5)	0.0147 (5)	0.0025 (4)	0.0047 (4)	0.0063 (4)
C12	0.0202 (6)	0.0181 (6)	0.0239 (6)	0.0033 (4)	0.0002 (5)	0.0037 (5)
C13	0.0222 (6)	0.0246 (6)	0.0245 (6)	-0.0012 (5)	-0.0034 (5)	0.0135 (5)
C14	0.0172 (5)	0.0263 (6)	0.0138 (5)	-0.0020 (5)	0.0013 (4)	0.0064 (5)
C15	0.0178 (6)	0.0244 (6)	0.0146 (5)	0.0024 (4)	0.0039 (4)	0.0027 (4)

C16	0.0234 (6)	0.0157 (5)	0.0145 (5)	0.0024 (4)	0.0016 (4)	0.0046 (4)
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Geometric parameters (Å, °)

C1—O1	1.3186 (14)	C12—H12B	0.99
C1—O2	1.2134 (14)	C13—C14	1.5221 (17)
C1—C2	1.5087 (17)	C13—H13A	0.99
O1—H1	0.84	C13—H13B	0.99
C2—O3	1.4092 (13)	C14—C15	1.5261 (16)
C2—H2A	0.99	C14—H14A	0.99
C2—H2B	0.99	C14—H14B	0.99
O3—N4	1.4331 (12)	C15—C16	1.5351 (16)
N4—C11	1.2775 (15)	C15—H15A	0.99
C11—C16	1.5015 (15)	C15—H15B	0.99
C11—C12	1.5022 (16)	C16—H16A	0.99
C12—C13	1.5309 (18)	C16—H16B	0.99
C12—H12A	0.99		
O1—C1—O2	124.23 (11)	C12—C13—H13A	109.4
O1—C1—C2	111.52 (10)	C14—C13—H13B	109.4
O2—C1—C2	124.24 (10)	C12—C13—H13B	109.4
C1—O1—H1	109.5	H13A—C13—H13B	108.0
O3—C2—C1	112.73 (10)	C13—C14—C15	110.75 (10)
O3—C2—H2A	109.0	C13—C14—H14A	109.5
C1—C2—H2A	109.0	C15—C14—H14A	109.5
O3—C2—H2B	109.0	C13—C14—H14B	109.5
C1—C2—H2B	109.0	C15—C14—H14B	109.5
H2A—C2—H2B	107.8	H14A—C14—H14B	108.1
C2—O3—N4	107.87 (8)	C14—C15—C16	111.18 (10)
O3—N4—C11	111.71 (9)	C14—C15—H15A	109.4
N4—C11—C16	127.65 (10)	C16—C15—H15A	109.4
N4—C11—C12	116.10 (10)	C14—C15—H15B	109.4
C16—C11—C12	116.22 (10)	C16—C15—H15B	109.4
C11—C12—C13	110.86 (10)	H15A—C15—H15B	108.0
C11—C12—H12A	109.5	C11—C16—C15	109.79 (9)
C13—C12—H12A	109.5	C11—C16—H16A	109.7
C11—C12—H12B	109.5	C15—C16—H16A	109.7
C13—C12—H12B	109.5	C11—C16—H16B	109.7
H12A—C12—H12B	108.1	C15—C16—H16B	109.7
C14—C13—C12	111.15 (10)	H16A—C16—H16B	108.2
C14—C13—H13A	109.4		
O1—C1—C2—O3	-177.14 (9)	C16—C11—C12—C13	51.20 (14)
O2—C1—C2—O3	1.83 (17)	C11—C12—C13—C14	-52.29 (13)
C1—C2—O3—N4	-83.62 (11)	C12—C13—C14—C15	57.08 (13)
C2—O3—N4—C11	-175.25 (9)	C13—C14—C15—C16	-58.22 (13)
O3—N4—C11—C12	179.11 (9)	N4—C11—C16—C15	126.14 (12)
O3—N4—C11—C16	1.24 (16)	C12—C11—C16—C15	-51.73 (13)

N4—C11—C12—C13 -126.92 (11) C14—C15—C16—C11 53.91 (13)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O2 ⁱ	0.84	1.84	2.6782 (12)	178

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

(II) [Chloro(phenyl)methyleneamino]oxyacetic acid

Crystal data

C₉H₈ClNO₃

M_r = 213.61

Triclinic, *P*1̄

Hall symbol: -P 1

a = 6.7961 (1) Å

b = 7.3857 (2) Å

c = 10.8173 (3) Å

α = 98.4852 (9)°

β = 93.4156 (10)°

γ = 117.0191 (12)°

V = 473.44 (2) Å³

Z = 2

F(000) = 220

D_x = 1.498 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2133 reflections

θ = 3.2–27.4°

μ = 0.38 mm⁻¹

T = 120 K

Plate, colourless

0.28 × 0.20 × 0.05 mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: rotating anode

Graphite monochromator

φ scans, and ω scans with κ offsets

Absorption correction: multi-scan
(*SORTAV*; Blessing, 1995, 1997)

T_{min} = 0.922, *T_{max}* = 0.981

3988 measured reflections

2133 independent reflections

1980 reflections with *I* > 2σ(*I*)

R_{int} = 0.031

θ_{max} = 27.4°, θ_{min} = 3.2°

h = -8→8

k = -9→9

l = -13→13

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.040

wR(*F*²) = 0.141

S = 1.20

2132 reflections

128 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0766*P*)² + 0.1738*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.34 e Å⁻³

Δρ_{min} = -0.48 e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> [*] / <i>U_{eq}</i>
Cl1	0.82919 (7)	0.00737 (7)	0.78680 (4)	0.02459 (19)
O1	0.2370 (2)	0.27726 (19)	0.50408 (13)	0.0235 (3)
O2	0.5944 (2)	0.36247 (19)	0.56736 (12)	0.0229 (3)
O3	0.4685 (2)	0.00535 (19)	0.65729 (11)	0.0211 (3)

N4	0.5117 (3)	0.1192 (2)	0.78071 (14)	0.0197 (3)
C1	0.3942 (3)	0.2414 (3)	0.55267 (16)	0.0193 (4)
C2	0.3016 (3)	0.0310 (3)	0.58876 (17)	0.0219 (4)
C5	0.6749 (3)	0.1242 (2)	0.84810 (16)	0.0175 (4)
C11	0.7394 (3)	0.2326 (2)	0.98149 (16)	0.0183 (4)
C12	0.9281 (3)	0.2563 (3)	1.05444 (17)	0.0219 (4)
C13	0.9880 (3)	0.3636 (3)	1.17899 (18)	0.0264 (4)
C14	0.8604 (3)	0.4479 (3)	1.23152 (18)	0.0276 (4)
C15	0.6704 (3)	0.4242 (3)	1.15935 (19)	0.0272 (4)
C16	0.6092 (3)	0.3170 (3)	1.03528 (18)	0.0230 (4)
H1	0.2951	0.3949	0.4848	0.028*
H2A	0.1816	0.0141	0.6408	0.026*
H2B	0.2353	-0.0780	0.5113	0.026*
H12	1.0167	0.1987	1.0190	0.026*
H13	1.1172	0.3790	1.2281	0.032*
H14	0.9020	0.5217	1.3165	0.033*
H15	0.5823	0.4819	1.1954	0.033*
H16	0.4790	0.3007	0.9867	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0273 (3)	0.0281 (3)	0.0234 (3)	0.0181 (2)	0.00411 (19)	0.00161 (19)
O1	0.0176 (6)	0.0243 (7)	0.0273 (7)	0.0094 (5)	-0.0010 (5)	0.0045 (5)
O2	0.0174 (6)	0.0228 (6)	0.0254 (7)	0.0076 (5)	-0.0016 (5)	0.0039 (5)
O3	0.0230 (6)	0.0223 (6)	0.0172 (6)	0.0109 (5)	0.0008 (5)	0.0014 (5)
N4	0.0228 (7)	0.0193 (7)	0.0160 (7)	0.0091 (6)	0.0037 (6)	0.0030 (5)
C1	0.0188 (8)	0.0226 (8)	0.0149 (8)	0.0097 (7)	0.0009 (6)	-0.0004 (6)
C2	0.0176 (8)	0.0241 (8)	0.0216 (8)	0.0079 (7)	0.0005 (6)	0.0044 (7)
C5	0.0185 (8)	0.0143 (7)	0.0210 (8)	0.0078 (6)	0.0058 (6)	0.0053 (6)
C11	0.0211 (8)	0.0134 (7)	0.0193 (8)	0.0063 (6)	0.0050 (6)	0.0059 (6)
C12	0.0245 (9)	0.0174 (8)	0.0229 (9)	0.0092 (7)	0.0021 (7)	0.0043 (7)
C13	0.0287 (9)	0.0225 (9)	0.0241 (9)	0.0097 (7)	-0.0019 (7)	0.0036 (7)
C14	0.0360 (10)	0.0202 (9)	0.0196 (9)	0.0080 (8)	0.0038 (7)	0.0018 (7)
C15	0.0326 (10)	0.0246 (9)	0.0258 (10)	0.0145 (8)	0.0091 (8)	0.0029 (7)
C16	0.0243 (9)	0.0232 (8)	0.0232 (9)	0.0124 (7)	0.0050 (7)	0.0048 (7)

Geometric parameters (Å, °)

C5—C11	1.738 (2)	C11—C12	1.390 (3)
C1—O1	1.311 (2)	C11—C16	1.402 (3)
C1—O2	1.226 (2)	C12—C13	1.391 (3)
C1—C2	1.510 (2)	C12—H12	0.95
O1—H1	0.84	C13—C14	1.381 (3)
C2—O3	1.418 (2)	C13—H13	0.95
C2—H2A	0.99	C14—C15	1.393 (3)
C2—H2B	0.99	C14—H14	0.95
O3—N4	1.405 (2)	C15—C16	1.386 (3)

N4—C5	1.273 (2)	C15—H15	0.95
C5—C11	1.477 (2)	C16—H16	0.95
O1—C1—O2	124.64 (16)	C16—C11—C5	119.14 (16)
O1—C1—C2	112.36 (14)	C11—C12—C13	120.46 (18)
O2—C1—C2	123.00 (16)	C11—C12—H12	119.8
C1—O1—H1	109.5	C13—C12—H12	119.8
O3—C2—C1	111.90 (14)	C14—C13—C12	120.27 (18)
O3—C2—H2A	109.2	C14—C13—H13	119.9
C1—C2—H2A	109.2	C12—C13—H13	119.9
O3—C2—H2B	109.2	C13—C14—C15	119.69 (18)
C1—C2—H2B	109.2	C13—C14—H14	120.2
H2A—C2—H2B	107.9	C15—C14—H14	120.2
C2—O3—N4	107.45 (13)	C16—C15—C14	120.40 (18)
O3—N4—C5	113.12 (14)	C16—C15—H15	119.8
N4—C5—C11	120.30 (15)	C14—C15—H15	119.8
N4—C5—C11	121.60 (14)	C15—C16—C11	120.00 (17)
C11—C5—C11	118.09 (13)	C15—C16—H16	120.0
C12—C11—C16	119.17 (17)	C11—C16—H16	120.0
C12—C11—C5	121.68 (16)		
O2—C1—C2—O3	8.7 (2)	C11—C5—C11—C16	-176.09 (12)
O1—C1—C2—O3	-171.61 (14)	C16—C11—C12—C13	-0.5 (3)
C1—C2—O3—N4	75.87 (17)	C5—C11—C12—C13	178.41 (15)
C2—O3—N4—C5	-173.61 (14)	C11—C12—C13—C14	0.0 (3)
O3—N4—C5—C11	-178.38 (13)	C12—C13—C14—C15	0.3 (3)
O3—N4—C5—C11	2.2 (2)	C13—C14—C15—C16	-0.1 (3)
N4—C5—C11—C12	-174.38 (15)	C14—C15—C16—C11	-0.4 (3)
C11—C5—C11—C12	5.0 (2)	C12—C11—C16—C15	0.6 (3)
N4—C5—C11—C16	4.5 (2)	C5—C11—C16—C15	-178.26 (15)

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots O2 ⁱ	0.84	1.79	2.6329 (17)	176

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