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3-Carboxyphenoxyacetic acid monohydrate

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Key indicators

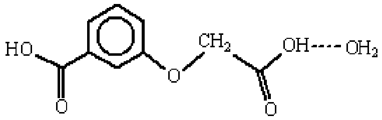
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.117
Data-to-parameter ratio = 13.8

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

The asymmetric unit of the title compound, $C_9H_{10}O_6 \cdot H_2O$, consists of one 3-carboxyphenoxyacetic acid (3-$CPOAH_2$) and one water molecule. The intermolecular interactions link symmetry-related molecules into hydrogen-bonded dimers. A supramolecular hydrogen-bonding network structure is formed *via* further intermolecular hydrogen bonds involving the water molecule.

Comment

Carboxyphenoxyacetic acids, which have been known to show biological activities and are widely used in agriculture, are a family of excellent ligands with versatile binding modes. To the best of our knowledge, several metal complexes with 4-carboxyphenoxyacetic acid and 2-carboxyphenoxyacetic acid have been reported (Wai *et al.*, 1990; Kennard *et al.*, 1986; Gao *et al.*, 2004). However, there has been little structural information on 3-carboxyphenoxyacetic acid to date. The reaction of chloroacetic acid with 3-hydroxybenzoic acid under basic conditions yielded the title compound, (3-$CPOAH_2$)·H_2O, (I), whose structure is reported here.



(I)

As shown in Fig. 1, the asymmetric unit consists of a molecule of 3-carboxyphenoxyacetic acid and one water molecule, which are linked by two hydrogen bonds, one involving atom O1 and the other involving O5 (see Table 2), leading to a supramolecular network structure. The bond lengths of the carboxyl O atoms of the oxyacetic acid group are slightly shorter than those of benzoic acid. The C3—O3—C2 and O3—C2—C1 bond angles are 118.9 (1) and 107.6 (1) Å, respectively. The oxyacetic acid group and the benzene ring

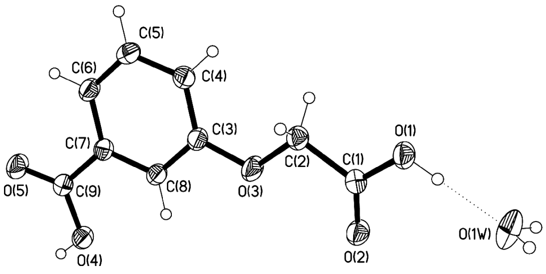


Figure 1
View of the title compound, with 30% probability displacement ellipsoids. The dashed line indicates a hydrogen bond.

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are almost coplanar, with a mean deviation of 0.066 Å. A dimeric unit, analogous to a crown ether ring, is formed by an O—H···O bond between the carbonyl O atom of the oxyacetic acid group and the carboxyl O atom of an adjacent molecule (see Fig. 2 and Table 2).

Experimental

The title compound was prepared by the addition of 3-hydroxybenzoic acid (0.05 mol) to an aqueous solution of chloroacetic acid (15 mmol), the pH being adjusted to 9–10 with 0.2 *M* NaOH solution. The resulting mixture was refluxed for 4 h and then cooled to room temperature. The pH was adjusted to 2 by the addition of 0.1 *M* HCl with subsequent filtration. Colorless plate-like single crystals were obtained from the filtrate at room temperature over a period of several days. IR spectroscopic analysis: (KBr, ν cm^{-1}): 3376, 1725, 1377, 1256, 1093; analysis calculated for $C_9H_{10}O_6$: C 50.47, H 4.71%; found: C 50.61, H 4.89%.

Crystal data

$C_9H_8O_5\cdot H_2O$
$M_r = 214.17$
Monoclinic, $P2_1/n$
$a = 8.728\ (2)$ Å
$b = 4.847\ (1)$ Å
$c = 21.935\ (4)$ Å
$\beta = 97.22\ (3)^\circ$
$V = 920.6\ (3)$ Å^3
$Z = 4$

$D_x = 1.545$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6464 reflections
$\theta = 3.9\text{–}27.4^\circ$
$\mu = 0.13$ mm^{-1}
$T = 293\ (2)$ K
Plate, colorless
$0.38 \times 0.26 \times 0.11$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.960$, $T_{\max} = 0.986$
3435 measured reflections

2097 independent reflections
1640 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 27.5^\circ$
$h = -11 \rightarrow 11$
$k = -6 \rightarrow 6$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.117$
$S = 1.02$
2097 reflections
152 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.296 (2)	O4—C9	1.319 (2)
O2—C1	1.199 (2)	O5—C9	1.211 (2)
O1—C1—C2	110.8 (1)	O4—C9—C7	113.3 (1)
O2—C1—O1	125.0 (2)	O5—C9—O4	123.4 (2)
O2—C1—C2	124.2 (2)	O5—C9—C7	123.3 (2)
O3—C2—C1	107.6 (1)	C3—O3—C2	118.9 (1)

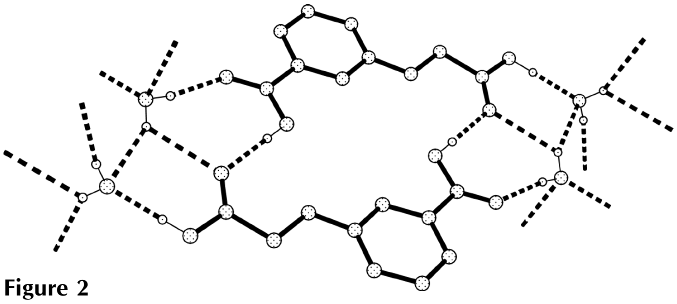

Figure 2
The hydrogen-bonded framework. The dashed lines indicate hydrogen bonds.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H10···O1*W*	0.86 (2)	1.71 (2)	2.563 (2)	179 (3)
O4—H11···O2$^{\rm i}$	0.84 (2)	1.93 (2)	2.765 (2)	179 (2)
O1*W*—H12*A*···O2$^{\rm ii}$	0.82 (2)	2.43 (3)	2.967 (2)	124 (2)
O1*W*—H12*A*···O1*W*$^{\rm ii}$	0.82 (2)	2.34 (2)	3.057 (2)	147 (2)
O1*W*—H12*B*···O5$^{\rm iii}$	0.84 (2)	1.91 (2)	2.740 (2)	169 (3)

Symmetry codes: (i) $2 - x, 3 - y, -z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $x - \frac{1}{2}, \frac{5}{2} - y, \frac{1}{2} + z$.

The H atoms attached to C were placed in calculated positions, with C—H = 0.93 or 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, and were included in the refinement in the riding-model approximation. The H atoms of the water molecule and carboxyl groups were located in Fourier difference maps and refined isotropically, with the O—H distances restrained to 0.82 (1) Å.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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