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(2,3-Dichloro-4-formylphenoxy)acetic Acid

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Abstract. $C_9H_6Cl_2O_4$, $M_r = 249{\cdot}05$, orthorhombic, *Pnam*, $a = 11{\cdot}8480\ (5)$, $b = 12{\cdot}7840\ (6)$, $c = 6{\cdot}4678\ (6)$ Å, $V = 979{\cdot}7\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}69$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 5{\cdot}9$ cm^{-1}, $F(000) = 504$, $T = 296$ K, final $R(F) = 0{\cdot}047$ for 700 unique observed reflections. The molecule is planar and lies on the mirror plane of space group *Pnam*. The terminal oxyacetic acid moiety is oriented in a synperiplanar–synperiplanar manner. The molecules form chains along the *a* axis *via* hydrogen bonds with distance O(3)···O(1) being 2·668 (5) Å and an O(3)—H···O(O1) angle of 168·5°.

Experimental. The title compound was crystallized from ethanol at room temperature. Data were collected on a Rigaku AFC-5 diffractometer equipped with a rotating-anode X-ray source. Data collection at 32° min^{-1}, 2θ–ω scans. Crystal dimensions 0·2 × 0·3 × 0·3 mm. Cell parameters from 20 reflections in the 2θ range 68–78°. Range of indices: h 0 to 13, k 0 to 14, l 0 to 7 ($\theta < 60°$). Three standard reflections were measured after every 150 reflections and showed no decrease in intensity ($1\bar{4}1$, $1\bar{4}0$, $2\bar{4}0$; with a maximum reduction in intensity of 0·4% at the end of data collection). Empirical absorption correction was applied based on ψ scans of three reflections ($0\bar{1}1$, $0\bar{2}2$, $1\bar{2}2$: with minimum and maximum transmission coefficients of 0·7 and 1·0 respectively). Of the 893 reflections measured, 700 had $I > 2{\cdot}0\sigma(I)$. Direct methods (*MITHRIL*; Gilmore, 1983) were used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. Function minimized was $\sum[w(k|F_o| - |F_c|)^2]$, where $w = 1/\sigma(|F_o|)^2$ (*UPALS*; Lundgren, 1978). $R(F) = 0{\cdot}043$, $wR(F) = 0{\cdot}065$, $S = 1{\cdot}78$, for 700 reflections and 159 refined parameters in space group $Pna2_1$. Refinement also carried out in the non-isomorphic supergroup *Pnam*, with all atoms on the mirror plane along **c**. The *z* coordinate of all atoms was fixed and the B_{13} and B_{23} components of their anisotropic temperature factor tensors were set at zero. $R(F) = 0{\cdot}047$, $wR(F) = 0{\cdot}070$, $S = 1{\cdot}84$, max.

Table 1. *Fractional atomic coordinates and* B_{eq} (Å^2) *with their e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl(1)	0·2781 (1)	0·2022 (1)	0·75000	5·09 (5)
Cl(2)	0·0185 (1)	0·1916 (1)	0·75000	4·92 (5)
O(1)	−0·1315 (3)	−0·1139 (3)	0·75000	5·0 (1)
O(2)	0·3897 (3)	0·0054 (3)	0·75000	4·0 (1)
O(3)	0·6444 (3)	−0·1373 (3)	0·75000	5·1 (1)
O(4)	0·6144 (3)	0·0353 (3)	0·75000	5·0 (1)
C(1)	0·2767 (4)	−0·0085 (4)	0·75000	3·5 (1)
C(2)	0·2120 (4)	0·0828 (4)	0·75000	3·6 (1)
C(3)	0·0955 (4)	0·0771 (4)	0·75000	3·6 (1)
C(4)	0·0400 (4)	−0·0194 (4)	0·75000	3·7 (1)
C(5)	0·1069 (4)	−0·1096 (4)	0·75000	4·0 (1)
C(6)	0·2227 (4)	−0·1053 (4)	0·75000	4·2 (2)
C(7)	−0·0826 (4)	−0·0301 (5)	0·75000	4·1 (1)
C(8)	0·4590 (4)	−0·0866 (4)	0·75000	3·8 (1)
C(9)	0·5800 (4)	−0·0529 (4)	0·75000	3·7 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with their e.s.d.'s in parentheses*

C(2)—Cl(1)	1·714 (5)	C(3)—Cl(2)	1·723 (5)
C(1)—O(2)	1·349 (6)	C(1)—C(2)	1·395 (7)
C(1)—C(6)	1·392 (7)	C(2)—C(3)	1·380 (7)
C(3)—C(4)	1·396 (7)	C(4)—C(5)	1·398 (7)
C(4)—C(7)	1·458 (8)	C(5)—C(6)	1·370 (7)
C(7)—O(1)	1·216 (7)	C(8)—O(2)	1·432 (6)
C(8)—C(9)	1·495 (7)	C(9)—O(3)	1·320 (6)
C(9)—O(4)	1·198 (6)		

O(2)—C(1)—C(6)	124·9 (4)	O(2)—C(1)—C(2)	115·8 (4)
C(2)—C(1)—C(6)	119·3 (4)	C(1)—C(2)—C(3)	120·3 (4)
Cl(1)—C(2)—C(1)	119·6 (4)	C(2)—C(3)—C(4)	121·1 (5)
Cl(2)—C(3)—C(4)	120·0 (4)	C(3)—C(4)—C(5)	117·4 (5)
C(4)—C(5)—C(6)	122·2 (5)	C(1)—C(6)—C(5)	119·7 (5)
O(1)—C(7)—C(4)	123·8 (5)	O(2)—C(8)—C(9)	108·2 (4)
O(3)—C(9)—O(4)	124·8 (5)	C(8)—C(9)—O(4)	126·6 (5)
C(8)—C(9)—O(3)	108·6 (4)	C(1)—O(2)—C(8)	117·4 (4)

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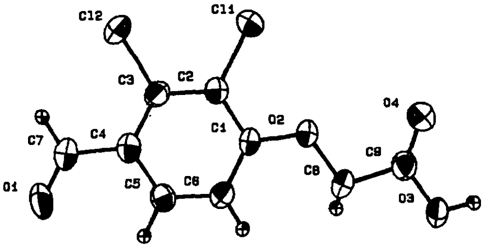

Fig. 1. Molecular structure and atomic labelling scheme of (2,3-dichloro-4-formylphenoxy)acetic acid (*ORTEP*, Johnson, 1965).

$\Delta/\sigma = 0{\cdot}03$, for 107 refined parameters. Maximum peak height in the final difference Fourier map = 0·17 e Å^{-3} and minimum peak height was −0·17 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148).

Atomic parameters from the *Pnam* refinement are given in Table 1,* selected bond lengths and bond angles are presented in Table 2. An *ORTEP* (Johnson, 1965) plot of the molecule is given in Fig. 1, and a cell packing diagram in Fig. 2.

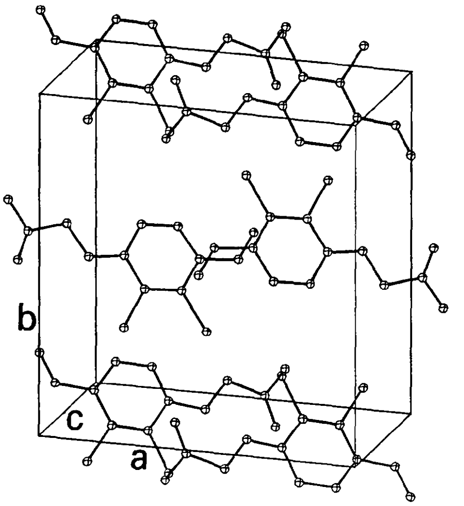

Fig. 2. Cell packing in the crystal structure of (2,3-dichloro-4-formylphenoxy)acetic acid (*ORTEP*, Johnson, 1965).

Related literature. The structures of several phenoxyalkanoic acids were reported by Kennard, Smith & White (1982). Packing modes and conformational features are discussed by Leiserowitz (1976). The acid groups in the title compound do not form cyclic dimers as in the structures of these related molecules. No disorder is observed in the carboxyl group.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53471 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 8-Hydroxy-2-(1-hydroxyethyl)naphtho[2,3-*b*]furan-4,9-dione

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Abstract. $C_{14}H_{10}O_5$, $M_r = 258{\cdot}23$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}851\ (3)$, $b = 35{\cdot}89\ (2)$, $c = 4{\cdot}618\ (1)$ Å, $V = 1135{\cdot}4\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}511$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 8{\cdot}48$ cm^{-1}, $F(000) = 536$, $T = 293$ K, $R = 0{\cdot}068$ for 987 observed reflections with $|F_o| > 3\sigma(F_o)$. The phenolic hydroxyl group

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