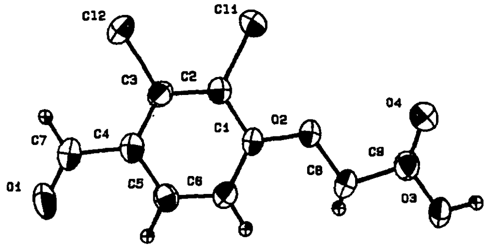

Fig. 1. Molecular structure and atomic labelling scheme of (2,3-dichloro-4-formylphenoxy)acetic acid (*ORTEP*, Johnson, 1965).

$\Delta/\sigma = 0{\cdot}03$, for 107 refined parameters. Maximum peak height in the final difference Fourier map = $0{\cdot}17\ \text{e}\ \text{Å}^{-3}$ and minimum peak height was $-0{\cdot}17\ \text{e}\ \text{Å}^{-3}$. 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.

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.

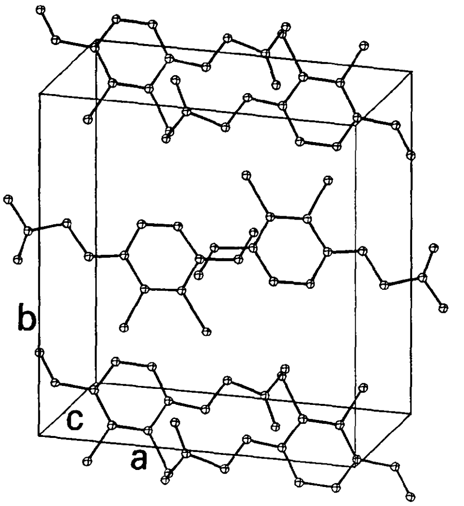

Fig. 2. Cell packing in the crystal structure of (2,3-dichloro-4-formylphenoxy)acetic acid (*ORTEP*, Johnson, 1965).

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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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Acta Cryst. (1991). C**47**, 1127–1129

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

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(*Received* 15 *October* 1990; *accepted* 6 *November* 1990)

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}, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}5418$ 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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Table 1. *Final atomic coordinates* (× 10^4) *with e.s.d.'s, and equivalent isotropic thermal parameters*, B_{eq}(Å^2), *for non*-H *atoms*

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

	x	y	z	B_{eq}
O(1)	−3741 (5)	−1077 (1)	−5162 (8)	3·1
O(4)	2286 (7)	−973 (1)	−931 (10)	4·8
O(8)	−3124 (10)	−2261 (2)	1474 (12)	5·7
O(9)	−4356 (7)	−1773 (1)	−2272 (10)	4·6
O(14)	−2837 (8)	−199 (1)	−8854 (9)	3·7
C(2)	−2863 (9)	−753 (2)	−6055 (11)	3·0
C(3)	−1067 (9)	−716 (2)	−4810 (12)	2·9
C(4)	780 (8)	−1152 (2)	−1134 (13)	3·2
C(5)	1848 (9)	−1625 (2)	2447 (14)	3·8
C(6)	1567 (12)	−1950 (2)	4030 (14)	4·6
C(7)	−59 (12)	−2159 (2)	3658 (14)	4·3
C(8)	−1538 (10)	−2046 (2)	1732 (13)	3·8
C(9)	−2851 (9)	−1596 (2)	−1854 (12)	3·3
C(10)	−830 (8)	−1032 (1)	−3017 (12)	2·8
C(11)	437 (8)	−1505 (2)	524 (11)	3·0
C(12)	−1285 (9)	−1713 (2)	154 (12)	2·9
C(13)	−2438 (9)	−1246 (2)	−3304 (12)	3·1
C(14)	−4030 (10)	−505 (1)	−7980 (12)	3·1
C(15)	−5872 (10)	−376 (2)	−6536 (16)	4·2

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

O(1)—C(2)	1·370 (7)	C(5)—C(6)	1·391 (9)
O(1)—C(13)	1·379 (6)	C(5)—C(11)	1·381 (8)
O(4)—C(4)	1·219 (7)	C(6)—C(7)	1·354 (10)
O(8)—C(8)	1·340 (8)	C(7)—C(8)	1·408 (10)
O(9)—C(9)	1·224 (7)	C(8)—C(12)	1·409 (9)
O(14)—C(14)	1·427 (8)	C(9)—C(12)	1·479 (8)
C(2)—C(3)	1·365 (8)	C(9)—C(13)	1·453 (8)
C(2)—C(14)	1·491 (8)	C(10)—C(13)	1·348 (7)
C(3)—C(10)	1·415 (7)	C(11)—C(12)	1·406 (8)
C(4)—C(10)	1·468 (7)	C(14)—C(15)	1·500 (9)
C(4)—C(11)	1·501 (8)		

C(2)—O(1)—C(13)	106·0 (4)	C(12)—C(9)—C(13)	113·2 (5)
O(1)—C(2)—C(3)	110·7 (5)	C(3)—C(10)—C(4)	131·8 (5)
O(1)—C(2)—C(14)	116·7 (5)	C(3)—C(10)—C(13)	107·7 (5)
C(3)—C(2)—C(14)	132·5 (5)	C(4)—C(10)—C(13)	120·4 (5)
C(2)—C(3)—C(10)	105·7 (5)	C(4)—C(11)—C(5)	118·7 (5)
O(4)—C(4)—C(10)	121·9 (5)	C(4)—C(11)—C(12)	121·2 (5)
O(4)—C(4)—C(11)	122·6 (5)	C(5)—C(11)—C(12)	120·1 (5)
C(10)—C(4)—C(11)	115·6 (4)	C(8)—C(12)—C(9)	118·4 (5)
C(6)—C(5)—C(11)	120·1 (6)	C(8)—C(12)—C(11)	119·3 (5)
C(5)—C(6)—C(7)	120·8 (7)	C(9)—C(12)—C(11)	122·3 (5)
C(6)—C(7)—C(8)	120·8 (7)	O(1)—C(13)—C(9)	122·8 (5)
O(8)—C(8)—C(7)	118·2 (6)	O(1)—C(13)—C(10)	109·9 (4)
O(8)—C(8)—C(12)	122·9 (6)	C(9)—C(13)—C(10)	127·3 (5)
C(7)—C(8)—C(12)	118·8 (6)	O(14)—C(14)—C(2)	108·7 (5)
O(9)—C(9)—C(12)	124·2 (5)	O(14)—C(14)—C(15)	111·7 (5)
O(9)—C(9)—C(13)	122·6 (5)	C(2)—C(14)—C(15)	111·8 (5)

takes part in an intramolecular hydrogen bond with the carbonyl group, and the ethanolic hydroxyl group forms a hydrogen bond and connects the molecules along the *c* axis.

Experimental. Crystal dimensions: 0·4 × 0·2 × 0·05 mm. Rigaku AFC-4 diffractometer with Cu $K\alpha$ radiation, graphite monochromator. Lattice parameters refined by least-squares method from 15 reflections in the 2θ range 45–51°. 1151 integrated intensities measured up to $2\theta_{max} = 125°$, h,k,l (max. range 8, 41, 5). ω–2θ-scan mode $\Delta\omega = (1{\cdot}0 + 0{\cdot}15\tan\theta)°$. Standard reflections showed intensity variations less than 2·5%. No absorption correction. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984). The non-H atoms refined anisotropically. The H atoms except H(141), H(151), H(152) and H(153) refined isotropically. The above four H atoms were placed on calculated positions riding on their bonded atoms and their isotropic thermal parameters were refined. 206 parameters were refined on F with full-matrix least squares using *SHELX76* (Sheldrick, 1976). Convergence at $R = 0{\cdot}068$ and $wR = 0{\cdot}071$, where $w = [\sigma^2(|F|) + 0{\cdot}004|F|^2]^{-1}$. $\Delta/\sigma_{max} = 0{\cdot}11$ for non-H-atom parameters, final difference electron density within $\Delta\rho = \pm 0{\cdot}35$ e Å^{-3}. Scattering factors from *SHELX76*. Final atomic parameters are given in Table 1,* and bond lengths and angles in Table 2. Fig. 1 shows the molecular conformation and the atom-numbering scheme. Fig. 2 shows the crystal structure viewed

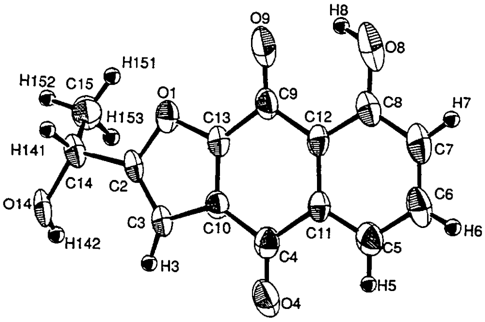

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule with the numbering of the atoms. Thermal ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

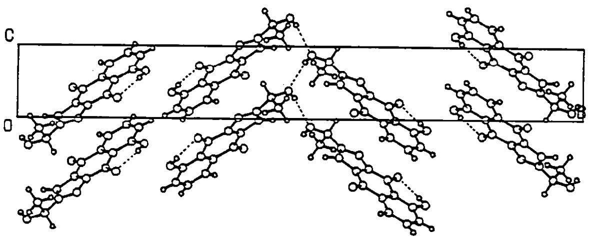

Fig. 2. Crystal structure viewed along the *a* axis. Hydrogen bonds are drawn with dotted lines.

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along the *a* axis. The intramolecular O···O and intermolecular O···O distances in the hydrogen bonds are 2·605 (8) and 2·755 (11) Å, respectively.

Related literature. The isolation and identification of the title compound have been reported (Inoue, Inoue & Chen, 1981; Rao & Kingston, 1982). However, the structure proposed was erroneous, since the hydroxyl group in the naphthoquinone ring was bonded to C(5) instead of C(8).

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Acta Cryst. (1991). C**47**, 1129–1131

Structure of a New Electrically Conducting Charge-Transfer Complex: 1,4-Phenylenebis(diazenecarbonitrile)–3,3′4,4′-Tetramethyl-2,2′,5,5′-tetrathiafulvalene (1/1)

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(*Received* 28 *August* 1990; *accepted* 9 *November* 1990)

Abstract. 1,4-Phenylenebis(diazenecarbonitrile) (1) [Kachkurova, Ashkinadze, Shamsutdinova & Kazitsyna (1987). *Zh. Org. Khim.* **23**, 1629–1634] and tetramethyltetrathiafulvalene (2) [Ferraris, Poehler, Bloch & Cowan (1973). *Tetrahedron Lett.* **27**, 2553–2556] form a charge-transfer complex, $C_8H_4N_6.C_{10}H_{12}S_4$, $M_r = 444·62$, triclinic, $P\bar{1}$, $a = 7·656\,(2)$, $b = 8·074\,(2)$, $c = 9·236\,(2)$ Å, $\alpha = 89·67\,(2)$, $\beta = 88·16\,(2)$, $\gamma = 62·58\,(2)°$, $V = 506·5$ Å^3, $Z = 1$, $D_x = 1·46$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70930 Å, $\mu = 4·7$ cm^{-1}, $F(000) = 230$, $T = 296$ K, $R = 0·036$ for 1919 reflections with $F_o^2 > 3·0\sigma(F_o^2)$. The planar π-electron systems of both organic molecules form a mixed stack with the double bond of (2) lying above the benzene ring of (1) at an intermolecular distance of 3·45 Å. The two N=N—CN groups of (1) are in an *anti* configuration; the stereochemistry of the N=N—CN group is *trans* with an N—N—C angle of 113·0 (1)° and an N—C≡N angle of 170·7 (3)°. The thermally activated single-crystal conductivity is 10^{-3}–10^{-5} S cm^{-1} between 300 and 180 K.

Experimental. Crystals of the title compound (1–2) were obtained by slowly cooling a solution of (1) and (2) in dichloromethane. The crystal used for data collection was a black needle with dimensions 0·02 × 0·05 × 0·40 mm. Preliminary examination and data collection were performed on an Enraf–Nonius CAD-4 computer-controlled diffractometer. Cell constants and the orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $15 < \theta < 17°$, measured by a computer-controlled diagonal slit method of centering. There were no systematic absences. Intensities were measured using ω–2θ scans of 1–4° min^{-1} in ω and a scan width of $(0·9 + 0·4\tan\theta)°$. Data were collected to a maximum of 56·0° in 2θ. No absorption correction was applied. The range of reflections was $-10 < h < 10$, $-10 < k < 10$, $0 < l < 12$. Three representative reflections were measured every 120 min and remained constant within experimental error. A total of 2582 reflections were collected, of which 2434 were unique. $R_{\rm int} = 0·02$. 1919 reflections having intensities greater than three times their standard deviation were used in the

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