Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

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

	x	y	z	B_{eq}
C1	0.6918 (2)	0.5946 (1)	1.0794 (3)	3.88 (5)
C2	0.7083 (2)	0.5436 (1)	1.1332 (4)	4.85 (6)
C3	0.6065 (2)	0.5167 (1)	1.1125 (6)	5.89 (8)
C4	0.5472 (2)	0.5235 (1)	0.9284 (6)	5.50 (8)
C5	0.5270 (2)	0.5750 (1)	0.8951 (4)	3.86 (5)
C6	0.4540 (2)	0.5842 (1)	0.7205 (5)	4.69 (6)
C7	0.4206 (2)	0.6345 (1)	0.7190 (4)	4.06 (5)
C8	0.5165 (1)	0.6679 (1)	0.7119 (3)	2.85 (4)
C9	0.6010 (1)	0.6537 (1)	0.8691 (3)	2.68 (4)
C10	0.6328 (1)	0.6020 (1)	0.8831 (3)	3.05 (4)
C11	0.6950 (1)	0.6867 (1)	0.8564 (3)	2.95 (4)
C12	0.6699 (1)	0.7347 (1)	0.8153 (3)	3.03 (4)
C13	0.5720 (1)	0.7505 (1)	0.7747 (3)	2.52 (3)
C14	0.4765 (1)	0.7182 (1)	0.7601 (3)	2.84 (4)
C15	0.3956 (2)	0.7341 (1)	0.6010 (4)	4.14 (5)
C16	0.3786 (2)	0.7858 (1)	0.5926 (4)	4.33 (6)
C17	0.4839 (2)	0.8124 (1)	0.5716 (4)	3.45 (5)
C18	0.5491 (1)	0.8016 (1)	0.7606 (3)	2.88 (4)
C19	0.6479 (1)	0.8319 (1)	0.7823 (3)	3.13 (4)
C20	0.6213 (2)	0.8837 (1)	0.7744 (3)	3.49 (4)
C21	0.5561 (2)	0.8943 (1)	0.5884 (4)	4.50 (6)
C22	0.4579 (2)	0.8639 (1)	0.5689 (4)	4.62 (6)
O23	0.5744 (2)	0.4926 (1)	1.2469 (5)	9.75 (10)
O24	0.7876 (1)	0.67525 (4)	0.8844 (3)	4.09 (4)
C25	0.7044 (2)	0.5855 (1)	0.7129 (4)	4.07 (5)
C26	0.5656 (2)	0.6658 (1)	0.5032 (3)	4.02 (5)
C27	0.4205 (2)	0.7212 (1)	0.9637 (4)	4.06 (5)
C28	0.5433 (2)	0.7992 (1)	0.3843 (4)	4.48 (6)
C29	0.5635 (2)	0.8991 (1)	0.9623 (5)	5.36 (7)
C30	0.7264 (2)	0.9099 (1)	0.7662 (4)	3.96 (5)
O31	0.8133 (1)	0.8938 (1)	0.7897 (5)	7.16 (7)
O32	0.7125 (1)	0.95352 (5)	0.7245 (3)	5.37 (5)
C33	0.8071 (2)	0.9817 (1)	0.7171 (5)	5.70 (7)

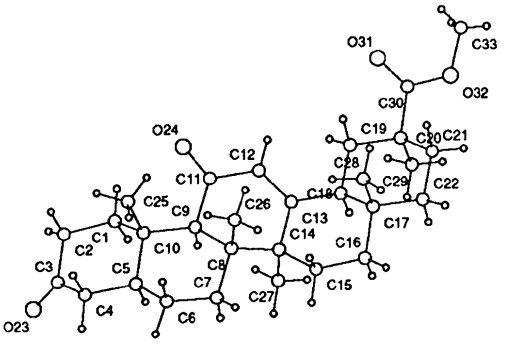


Fig. 1. Perspective view drawn by *PLUTO* (Motherwell & Clegg, 1978).

References

DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., TATE, C. & WOOLFSON, M. M. (1987). *MULTAN87. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

TERASAWA, T., OKADA, T., HARA, T. & ITOH, K. (1991). *Eur. J. Med. Chem. Chim. Ther.* To be submitted.

Acta Cryst. (1992). C**48**, 763–765

2-[(2,6-Dihydroxyphenyl)ethynyl]benzoic Acid

BY ENOCH T. HUANG, KEVIN L. EVANS, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, *USA*

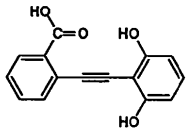
(*Received* 17 *July* 1991; *accepted* 5 *September* 1991)

Abstract. $C_{15}H_{10}O_4$, $M_r = 254.2$, monoclinic, $P2_1/n$, $a = 12.8584$ (13), $b = 5.0051$ (2), $c = 19.381$ (2) Å, $\beta = 109.141$ (9)°, $V = 1178.3$ (2) Å^3, $Z = 4$, $D_x = 1.431$ g cm^{-3} at 295 K, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 8.28$ cm^{-1}, $F(000) = 528$, 2316 unique data measured, final $R = 0.037$ for 2065 reflections with $I > 3.0\sigma(I)$. Maximum deviations of the two aromatic rings are 0.0031 (15) Å for the ring containing the carboxy substitutent and 0.0063 (13) Å for the ring containing two hydroxy substituents. These two rings are essentially coplanar, exhibiting mean and maximum deviations of 0.007 and 0.016 (1) Å, respectively, from the 12-atom best plane. The ethynyl C atoms lie 0.014 (1) and 0.017 (1) Å in the same direction out of this plane. The triple-bond distance is 1.195 (2) Å, and the bond angles at the ethynyl C atoms are 172.3 (2) and 174.1 (2)°, which results in a *trans* kink in the three bonds that link the two aryls. One hydroxy substituent forms an intramolecular hydrogen bond of length 2.973 (2) Å with the carbonyl O atom of the carboxy group, with angle at H of 155 (2)°. The carboxyl group forms centrosymmetric hydrogen-bonded dimers, with O···O distance 2.684 (2) Å and a 170 (2)° angle at H. The other hydroxy group of the dihydroxyphenyl group forms chains of intermolecular hydrogen bonds propagated by the screw axis, having O···O distance 2.793 (2) Å and angle at H of 163 (2)°.

* To whom correspondence should be addressed.

0108-2701/92/040763-03$03.00

Experimental. A pink crystal of (1) was isolated by recrystallization from methanol from the demethylation reaction of methyl 2-[(2,6-dimethoxyphenyl)-ethynyl]benzoate with boron tribromide. Crystal size



(1)

0.18 × 0.28 × 0.37 mm, space group from systematic absences $h0l$ with $h + l$ odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to maximum scan time = 120 s, scan rates varied 0.63–3.30° min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \le h \le 16$, $0 \le k \le 5$, $-24 \le l \le 22$ measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 400, 020, 006 varied randomly, and no decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0.9395 to 0.9994. 2714 total data were collected, and redundant $0kl$ and $0k\bar{l}$ data merged, $R_{int} = 0.010$, to yield 2316 unique data, 2065 observed with $I > 3\sigma(I)$. The structure is isomorphous with the analogous compound bearing no OH group at C5 (F. R. Fronczek, M. A. Oliver & R. D. Gandour, 1983; unpublished results), and coordinates from that structure were used as a starting model. The structure was refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using *Enraf–Nonius SDP/VAX* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located from difference maps and refined isotropically. Final $R = 0.037$ (0.040 for all data), $wR = 0.058$, $S = 3.545$ for 213 variables. Maximum shift 0.07σ in the final cycle, maximum residual density 0.12 e Å^{-3}, minimum -0.12 e Å^{-3}, extinction coefficient (Larson, 1969) $g = 5.9\,(3) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, maximum correction 16.4% for the $31\bar{1}$ reflection.

Coordinates* and equivalent isotropic thermal parameters are given in Table 1; bond distances and

* Lists of H-atom coordinates, bond distances and angles involving H atoms, torsion angles, anisotropic thermal parameters, least-squares-planes data, and the structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54643 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0544]

Table 1. *Coordinates and isotropic thermal parameters*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O1	0.69348 (8)	0.3771 (2)	0.49819 (5)	4.73 (2)
O2	0.74330 (8)	0.0633 (2)	0.28024 (5)	4.18 (2)
O3	0.53280 (7)	0.8145 (2)	0.43824 (5)	4.25 (2)
O4	0.40616 (9)	1.1330 (3)	0.41609 (5)	5.64 (3)
C1	0.7441 (1)	0.2130 (3)	0.46365 (7)	3.51 (3)
C2	0.8204 (1)	0.0304 (3)	0.50510 (8)	4.39 (3)
C3	0.8717 (1)	−0.1401 (3)	0.47040 (8)	4.40 (3)
C4	0.8490 (1)	−0.1360 (3)	0.39539 (8)	3.90 (3)
C5	0.7720 (1)	0.0456 (3)	0.35436 (6)	3.25 (2)
C6	0.71929 (9)	0.2240 (3)	0.38767 (6)	3.00 (2)
C7	0.63987 (9)	0.4113 (3)	0.34680 (6)	3.10 (2)
C8	0.5723 (1)	0.5785 (3)	0.32010 (6)	3.22 (2)
C9	0.4916 (1)	0.7688 (3)	0.28103 (6)	3.09 (2)
C10	0.4653 (1)	0.7786 (3)	0.20471 (7)	4.02 (3)
C11	0.3880 (1)	0.9555 (3)	0.16327 (7)	4.37 (3)
C12	0.3354 (1)	1.1284 (3)	0.19638 (8)	4.11 (3)
C13	0.3602 (1)	1.1241 (3)	0.27121 (7)	3.67 (3)
C14	0.43791 (9)	0.9460 (3)	0.31453 (6)	3.10 (2)
C15	0.4643 (1)	0.9545 (3)	0.39517 (6)	3.45 (2)

Table 2. *Bond distances* (Å) *and angles* (°)

O1—C1	1.353 (2)	C6—C7	1.421 (2)
O2—C5	1.364 (2)	C7—C8	1.195 (2)
O3—C15	1.217 (2)	C8—C9	1.429 (2)
O4—C15	1.312 (2)	C9—C10	1.406 (2)
C1—C2	1.388 (2)	C9—C14	1.406 (2)
C1—C6	1.402 (2)	C10—C11	1.376 (2)
C2—C3	1.381 (2)	C11—C12	1.380 (2)
C3—C4	1.386 (2)	C12—C13	1.379 (2)
C4—C5	1.387 (2)	C13—C14	1.395 (2)
C5—C6	1.400 (2)	C14—C15	1.487 (2)

O1—C1—C2	118.5 (1)	C8—C9—C10	117.6 (1)
O1—C1—C6	121.0 (1)	C8—C9—C14	123.8 (1)
C2—C1—C6	120.5 (1)	C10—C9—C14	118.6 (1)
C1—C2—C3	119.0 (1)	C9—C10—C11	121.0 (1)
C2—C3—C4	122.1 (1)	C10—C11—C12	120.2 (1)
C3—C4—C5	118.5 (1)	C11—C12—C13	120.0 (1)
O2—C5—C4	122.8 (1)	C12—C13—C14	121.1 (1)
O2—C5—C6	116.1 (1)	C9—C14—C13	119.2 (1)
C4—C5—C6	121.1 (1)	C9—C14—C15	121.7 (1)
C1—C6—C5	118.8 (1)	C13—C14—C15	119.1 (1)
C1—C6—C7	119.1 (1)	O3—C15—O4	122.5 (1)
C5—C6—C7	122.1 (1)	O3—C15—C14	124.7 (1)
C6—C7—C8	172.3 (1)	O4—C15—C14	112.8 (1)
C7—C8—C9	174.1 (1)		

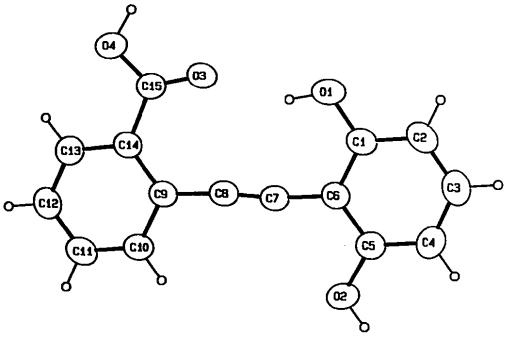


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 50% probability level.

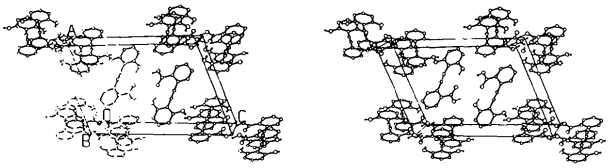


Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *b* axis with *c* horizontal.

angles are given in Table 2. The molecule is illustrated in Fig. 1. The unit cell is illustrated in Fig. 2.

Related literature. Structure of methyl 2-[(2,6-dimethoxyphenyl)ethynyl]benzoate: Huang, Evans, Fronczek & Gandour (1991). Structure of methyl 2-[(2,6-dimethoxyphenyl)ethynyl]-3-methoxybenzoate: Evans, Horn, Fronczek & Gandour (1990). Structure of diphenylacetylene: Mavridis & Moustakali-Mavridis (1977). Structure of bis(*m*-chlorophenyl)acetylene: Espiritu & White (1977). Structure of *p*-butyl-*p'*-methoxydiphenylacetylene: Cotrait (1977).

References

COTRAIT, D. T. (1977). *C. R. Acad. Sci. Sér. C*, **285**, 547–550.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

ESPIRITU, A. A. & WHITE, J. G. (1977). *Acta Cryst.* B**33**, 3899–3901.

EVANS, K. L., HORN, G. W., FRONCZEK, F. R. & GANDOUR, R. D. (1990). *Acta Cryst.* C**46**, 502–504.

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf-Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HUANG, E. T., EVANS, K. L., FRONCZEK, F. R. & GANDOUR, R. D. (1991). *Acta Cryst.* C**47**, 2727–2729.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

LARSON, A. C. (1969). In *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–299. Copenhagen: Munksgaard.

MAVRIDIS, A. & MOUSTAKALI-MAVRIDIS, I. (1977). *Acta Cryst.* B**33**, 3612–3615.

Acta Cryst. (1992). C**48**, 765–767

3-(2,6-Dihydroxyphenyl)-1*H*-2-benzopyran-1-one

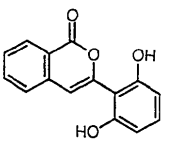
BY ENOCH T. HUANG, KEVIN L. EVANS, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, *USA*

(*Received* 17 *July* 1991; *accepted* 5 *September* 1991)

Abstract. $C_{15}H_{10}O_4$, $M_r = 254.2$, monoclinic, $P2_1$, $a = 5.9476$ (4), $b = 8.1253$ (10), $c = 12.0198$ (12) Å, $\beta = 100.938$ (7)°, $V = 570.3$ (2) Å^3, $Z = 2$, $D_x = 1.480$ g cm^{-3} at 297 K, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 8.56$ cm^{-1}, $F(000) = 264$, 1268 unique data, final $R = 0.035$ for 1113 reflections with $I > 3.0\sigma(I)$. Maximum deviations from planarity of the two aromatic rings are 0.008 (3) Å for the ring containing the two hydroxy substituents and 0.006 (4) Å for the aromatic ring of the benzopyran. The two rings form a dihedral angle of 113.3 (1)°. The C=C double bond distance is 1.330 (3) Å. Two distinct intermolecular hydrogen bonds are observed. One hydroxy group donates an intermolecular hydrogen bond to the carbonyl O atom of a second molecule, and also accepts a second intermolecular hydrogen bond from a hydroxy group of a third molecule. The former hydrogen bond has O···O distance 2.680 (3) Å and angle at H of 175 (4)°, while the latter has O···O distance 2.763 (3) Å and angle at H of 152 (3)°.

Experimental. The title compound was isolated as a side product of the demethylation of methyl 2-[(2,6-dimethoxyphenyl)ethynyl]benzoate using boron tribromide. Colorless plates of (1) were isolated by



(1)

slow evaporation from methanol. Crystal size 0.02 × 0.25 × 0.30 mm, space group from systematic absences $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $20 < \theta < 25°$. Data collection on Enraf–Nonius CAD-4 diffrac-

* To whom correspondence should be addressed.

0108-2701/92/040765-03$03.00