

MARSH, R. E. & DONOHUE, J. (1967). *Adv. Protein Chem.* **22**, 235–256.
 MASON, R. & ROBERTSON, G. B. (1966). In *Advances in Structural Research by Diffraction Methods*, Vol. 2, edited by R. BRILL & R. MASON. New York: Wiley-Interscience.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 ONDETTI, M. A., RUBIN, B. & CUSHMAN, D. W. (1977). *Science*, **196**, 441–444.
 STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination*, pp. 410–412. New York: Macmillan.

Acta Cryst. (1980). **B36**, 3199–3201

10,10-Divanillyl-9(10H)-anthracenone

BY KEVIN L. BROWN

Chemistry Division, Department of Scientific and Industrial Research, Private Bag, Petone, New Zealand

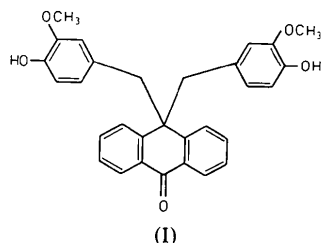
AND TERRY J. FULLERTON

Forest Research Institute, Private Bag, Rotorua, New Zealand

(Received 21 July 1980; accepted 16 September 1980)

Abstract. $C_{30}H_{26}O_5$, $M_r = 466.5$, monoclinic, $P2_1/n$, $a = 11.205$ (1), $b = 9.332$ (2), $c = 22.804$ (3) Å, $\beta = 100.70$ (1)°, λ (Cu $K\alpha$) = 1.5418 Å, $Z = 4$, $D_c = 1.32$ Mg m $^{-3}$, $\mu = 0.64$ mm $^{-1}$. $R = 0.065$ for 1759 diffractometer data. Strong shielding effects in the proton NMR spectrum are explained by the disposition of the substituted vanillyl rings with respect to the anthrone ring.

Introduction. During a study of the mechanism of anthraquinone wood pulping (Fullerton, 1978), the title compound (I) was isolated from the reaction of anthrahydroquinone with vanillyl alcohol (Fullerton, 1980). The proton NMR spectrum was unusual, with none of the expected resonances of a vanillyl substituent. The methoxy and aromatic protons showed strong shielding, which was assumed to be because the vanillyl rings have a preferred orientation above and below the anthrone ring. To elucidate the exact nature of this orientation, an X-ray analysis was undertaken.



Crystals obtained from $CHCl_3$ were found to contain occluded chloroform and subsequently proved unsuitable for data collection. Suitable crystals were then obtained from toluene. The crystals are thin colourless

plates and intensities and cell dimensions were obtained from a plate $0.52 \times 0.25 \times 0.03$ mm. The systematic absences $0k0$, $k \neq 2n$ and $h0l$, $h + l \neq 2n$ indicated space group $P2_1/n$. Cell dimensions were determined by least squares from the parameters of 12 reflections centred on a Hilger & Watts automated diffractometer. Data were collected to a maximum of $\theta = 57^\circ$ (Ni-filtered Cu $K\alpha$ radiation, θ - 2θ scan, scan width = 0.70° , scan time = 70 s). Three standard reflections measured periodically showed only random fluctuations of $\pm 2\%$.

Intensity measurements were obtained for 3959 reflections which, after averaging of equivalent forms, yielded 1759 reflections which were considered observed ($I > 2.5\sigma_I$). Absorption corrections were applied (de Meulenaer & Tompa, 1965), the correction ranging from 1.05 to 1.12. The structure was solved by direct methods and refined by full-matrix least squares with experimental weights ($W = 2.97/\sigma_F^2$; a weighting analysis confirmed the validity of this function). The non-hydrogen parameters were refined with anisotropic thermal parameters and the positional parameters of the H atoms with a common temperature factor [$U_H = 0.066$ (4) Å 2]. All the H atoms, except that bonded to O(25), were found from difference syntheses. It proved impossible to locate H(25), even on the final difference synthesis. The high-resolution mass spectrum confirmed the formula $C_{30}H_{26}O_5$; therefore it was assumed that H(25) is disordered to the point where the electron density did not show on a difference map. Consequently, H(25) has not been included in the final model which converged at $R = 0.065$, $R_w = 0.060$. A final difference synthesis showed no unusual features.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for the non-hydrogen atoms

The equivalent isotropic thermal parameters were calculated using

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C(1)	5346 (5)	7705 (6)	8003 (3)	47
C(2)	6478 (5)	8519 (6)	7992 (3)	40
C(3)	6775 (6)	8814 (7)	7434 (3)	57
C(4)	7792 (7)	9643 (8)	7403 (4)	66
C(5)	8484 (6)	10151 (7)	7934 (4)	59
C(6)	8172 (6)	9853 (7)	8474 (4)	55
C(7)	7165 (5)	9010 (6)	8522 (3)	43
C(8)	6850 (5)	8761 (6)	9123 (3)	46
C(9)	5759 (5)	7757 (7)	9108 (3)	45
C(10)	5400 (6)	7385 (8)	9639 (3)	59
C(11)	4389 (7)	6541 (8)	9649 (4)	68
C(12)	3695 (7)	6090 (8)	9115 (4)	71
C(13)	4021 (6)	6445 (8)	8593 (4)	64
C(14)	5054 (5)	7298 (6)	8578 (3)	47
O(15)	4648 (4)	7406 (5)	7525 (2)	69
C(16)	7977 (6)	8154 (7)	9575 (3)	50
C(17)	8455 (6)	6703 (7)	9407 (3)	52
C(18)	9252 (6)	6570 (8)	9017 (3)	57
C(19)	9753 (6)	5246 (8)	8911 (3)	56
C(20)	9412 (6)	4033 (6)	9196 (3)	62
C(21)	8616 (7)	4135 (8)	9566 (4)	63
C(22)	8109 (6)	5461 (8)	9674 (3)	62
O(23)	10572 (4)	5022 (5)	8556 (2)	71
C(24)	10637 (8)	6030 (10)	8092 (5)	84
O(25)	9938 (4)	2731 (4)	9085 (2)	80
C(26)	6515 (6)	10250 (7)	9375 (3)	54
C(27)	5508 (6)	11070 (7)	8981 (3)	46
C(28)	4302 (6)	10704 (7)	8969 (3)	49
C(29)	3370 (6)	11454 (7)	8602 (3)	52
C(30)	3635 (6)	12549 (7)	8241 (3)	53
C(31)	4830 (6)	12915 (7)	8258 (3)	60
C(32)	5769 (6)	12189 (7)	8623 (3)	60
O(33)	2166 (4)	11196 (5)	8576 (2)	77
C(34)	1781 (8)	9814 (11)	8674 (5)	105
O(35)	2754 (4)	13292 (5)	7866 (2)	80

Table 2. Bond lengths (\AA) and angles ($^\circ$) between the non-hydrogen atoms

E.s.d.'s are 0.006–0.008 \AA for bond lengths and 0.5–0.7 $^\circ$ for angles.

1–2	1.482	17–18	1.376
1–14	1.459	17–22	1.398
1–15	1.250	18–19	1.397
2–3	1.400	19–20	1.393
2–7	1.385	19–23	1.348
3–4	1.390	20–21	1.341
4–5	1.393	20–25	1.393
5–6	1.369	21–22	1.403
6–7	1.397	23–24	1.427
7–8	1.496	26–27	1.513
8–9	1.535	27–28	1.389
8–16	1.579	27–32	1.389
8–26	1.576	28–29	1.399
9–10	1.389	29–30	1.378
9–14	1.385	29–33	1.361
10–11	1.382	30–31	1.376
11–12	1.383	30–35	1.368
12–13	1.351	31–32	1.392
13–14	1.410	33–34	1.390
16–17	1.531		
2–1–14	119.0	8–16–17	115.4
2–1–15	119.9	16–17–18	122.7
14–1–15	121.1	16–17–22	119.0
1–2–3	117.7	18–17–22	118.2
1–2–7	119.5	17–18–19	121.4
3–2–7	122.7	18–19–20	118.9
2–3–4	119.4	18–19–23	125.6
3–4–5	118.3	20–19–23	115.5
4–5–6	121.2	19–20–21	120.5
5–6–7	122.0	19–20–25	117.5
6–7–8	119.4	21–20–25	122.0
6–7–2	116.3	20–21–22	120.8
2–7–8	124.1	21–22–17	120.0
7–8–9	113.4	19–23–24	118.5
7–8–16	110.9	8–26–27	115.6
7–8–26	108.1	26–27–28	120.2
9–8–16	109.4	26–27–32	120.9
9–8–26	107.7	28–27–32	118.9
16–8–26	107.1	27–28–29	120.3
8–9–10	119.5	28–29–30	120.6
8–9–14	122.1	28–29–33	124.1
10–9–14	118.3	30–29–33	115.3
9–10–11	121.8	29–30–31	118.9
10–11–12	119.2	29–30–35	122.6
11–12–13	120.1	31–30–35	118.5
12–13–14	121.2	30–31–32	121.3
13–14–1	119.4	31–32–27	120.0
13–14–9	119.4	29–33–34	119.4
9–14–1	121.2		

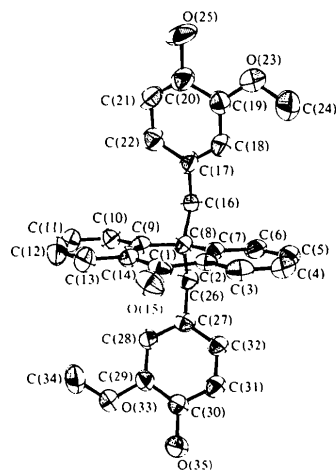


Fig. 1. A view of the molecule showing the numbering system. Thermal ellipsoids are drawn at the 50% probability level.

The atomic coordinates for the non-hydrogen atoms are given in Table 1,* bond lengths and angles in Table 2. A general view of the molecule is given in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters and H atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35581 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

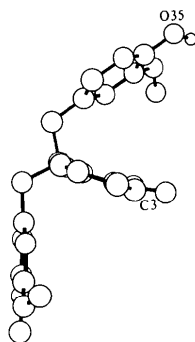


Fig. 2. A view of the molecule parallel to the anthraquinone ring showing the disposition of the vanillyl rings.

Discussion. The vanillyl substituents do indeed adopt positions above and below the plane of the anthrone ring (Fig. 2) which explains the NMR shielding effect. The planes of the vanillyl groups are tilted with respect to the anthrone ring, the methoxy group being nearest to the anthrone ring in both cases. Unlike 10-

bromoanthrone (Destro, D'Alfonso & Simonetta, 1973), the anthrone ring in the present compound has a minimum amount of puckering, the angle between the planes of the outer phenyl rings being only 1.6° . However, as in 10-bromoanthrone, the central ring adopts a boat-like conformation, the O atom being 0.21 \AA out of the plane through C(2), C(7), C(9) and C(14).

The molecules form hydrogen-bonded spirals parallel to *b*. The H atom on O(35) is hydrogen bonded to the carbonyl O atom of a screw-related molecule [$\text{H}(35)\cdots\text{O}(15) = 1.91 \text{ \AA}$, $\text{O}(15)\cdots\text{H}(35)-\text{O}(35) = 151.6^\circ$]. There is no other intermolecular bonding.

References

- DESTRO, R., D'ALFONSO, T. B. & SIMONETTA, M. (1973). *Acta Cryst.* B29, 2214–2220.
 FULLERTON, T. J. (1978). *Appita*, 32, 117–118.
 FULLERTON, T. J. (1980). To be published.
 MEULENAER, J. DE & TOMPA, H. (1965). *Acta Cryst.* 19, 1014–1018.

Acta Cryst. (1980). B36, 3201–3203

The Structure of 2-(5'-Chloro-2'-hydroxybenzoyl)benzoic Acid

BY ZOFIA SKRZAT

Institute of Chemistry, N. Copernicus University, ul. Gagarina 7, 87-100 Toruń, Poland

(Received 16 June 1980; accepted 8 September 1980)

Abstract. $\text{C}_{14}\text{H}_9\text{ClO}_4$, $M_r = 276.7$, monoclinic, $P2_1/c$, $a = 14.724(2)$, $b = 7.104(1)$, $c = 13.439(3) \text{ \AA}$, $\beta = 113.60(1)^\circ$, $Z = 4$, $V = 1288 \text{ \AA}^3$, $D_x = 1.426$, $D_m = 1.425 \text{ Mg m}^{-3}$, $F(000) = 568$. The structure was solved by direct methods, using three-dimensional photographic data, and refined to an R value of 0.069 for 1088 observed reflexions. In the crystal structure the molecules exist as hydrogen-bonded centrosymmetric dimers. The $\text{O}-\text{H}\cdots\text{O}$ bond is $2.623(7) \text{ \AA}$.

Introduction. This work is a continuation of the studies on the structures of substituted 2-aryloxybenzoic acids.

Crystals of $\text{C}_{14}\text{H}_9\text{ClO}_4$ grown from aqueous acetic acid solution were well formed colourless transparent plates. The unit-cell dimensions and the space group were determined from oscillation and Weissenberg photographs. (The unit-cell dimensions were confirmed by measurements on a Syntex $P2_1$ diffractometer.) According to systematic absences the space

group was $P2_1/c$. The diffraction data were collected on equi-inclination Weissenberg photographs, with Cu $K\alpha$ radiation, for the layers $h0l$ to $h6l$. The $hk0$ zonal data were also recorded for scaling purposes. The intensities of 1088 independent reflexions were visually estimated by comparison with calibrated strips. The data were corrected for Lorentz and polarization factors and for spot shape, but not for absorption.

After an unsuccessful attempt to solve the structure by the Patterson method, it was solved by direct methods using the Σ_2 and PHASE programs of the XRAY 70 system (Stewart, Kundell & Baldwin, 1970). An E map computed with 203 signs for $E > 1.5$ clearly revealed the positions of all 19 non-hydrogen atoms in the structure ($R = 0.337$). The structure was refined by the full-matrix least-squares program CRYLSQ first isotropically and then anisotropically to an R value of 0.082. At this stage the positions of eight of the nine H atoms were found on a difference Fourier map. The H