3-hydroxy-2-phospholenes. The distances 2·59 and 2·61 Å have been found respectively by Washecheck, Helm, Purdum & Berlin (1974) and GG (1980*b*). It appears that the acidity of the hydroxyl H atom is the most important factor affecting both the hydrogen-bond distance and its strength. Table 4 shows the hydrogen-bonding geometry for (I).

The same configurations at P and C(2) and the opposite configuration at C(5) have been suggested (Musierowicz, Waszkuć & Krawczyk, 1979) and confirmed in this study (Fig. 3).

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References

DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, pp. 16–22. New York: Plenum.

GAŁDECKI, Z. & GŁÓWKA, M. L. (1980*a*). *Acta Cryst.* B**36**, 2191–2193.

GAŁDECKI, Z. & GŁÓWKA, M. L. (1980*b*). *Acta Cryst.* B**36**, 2188–2190.

GAŁDECKI, Z. & GŁÓWKA, M. L. (1980*c*). *Acta Cryst.* B**36**, 1495–1497.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

GŁÓWKA, M. L. (1977). Thesis, Technical Univ. of Łódź.

GŁÓWKA, M. L. & GAŁDECKI, Z. (1980). *Acta Cryst.* B**36**, 2312–2315.

International Tables for X-ray Crystallography (1962). Vol. III, pp. 202–203. Birmingham: Kynoch Press.

MUSIEROWICZ, S., WASZKUĆ, W. T. & KRAWCZYK, H. W. (1980). *Phosphorus Sulfur.* In the press.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

TAKAYANAGI, H., SEO, K., YAMASHITA, M., YOSHIDA, H., OGATA, T. & INOKAWA, S. (1978). *Carbohydr. Res.* **63**, 105–113.

WASHECHECK, D. M., HELM, D., PURDUM, W. R. & BERLIN, K. D. (1974). *J. Org. Chem.* **39**, 3305–3307.

YAMASHITA, M., NAKATSUKASA, Y., YOSHIKANE, M., YOSHIDA, H., OGATA, T. & INOKAWA, S. (1977). *Carbohydr. Res.* **59**, C12–C14.

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Structure of 2-(4′-Chloro-2′-hydroxybenzoyl)benzoic Acid

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Abstract. $C_{14}H_9ClO_4$, $M_r = 276{\cdot}7$, monoclinic, $P2_1/c$, $a = 10{\cdot}838\ (3)$, $b = 15{\cdot}205\ (3)$, $c = 7{\cdot}347\ (1)$ Å, $\beta = 91{\cdot}18\ (1)°$, $V = 1210{\cdot}46$ Å^3, $D_x = 1{\cdot}52$, $D_m = 1{\cdot}50$ Mg m^{-3}, $Z = 4$, $F(000) = 568$. The structure was solved by the heavy-atom method and refined by least-squares calculations to $R = 0{\cdot}064$ for 622 visually estimated reflexions. The crystal structure consists of centrosymmetric dimers linked by hydrogen bonds [2·627 (13) Å] between the carboxyl groups.

Introduction. This work forms part of the studies on the structures of substituted 2-aroylbenzoic acids. Crystals of the title compound were synthesized and kindly supplied by Professor J. Gronowska of the Organic Chemistry Department of N. Copernicus University.

Cell dimensions were obtained from rotation and Weissenberg photographs and were confirmed by measurements on a Syntex 2_1 diffractometer. From systematic absences the space group was determined as $P2_1/c$. Intensities of 623 reflexions were collected photographically with the equi-inclination Weissenberg technique about the c axis (zones $hk0$–$hk5$), and about the a axis (zones $0kl$–$1kl$), using Ni-filtered Cu $K\alpha$ radiation. Intensities were corrected for Lorentz and polarization factors and for spot shape, but not for absorption.

The structure was solved by the heavy-atom method. The phases for the first Fourier synthesis were based on the structure factor calculation ($R = 0{\cdot}568$) from the position of the Cl atom. All non-hydrogen atoms were then located in two successive Fourier syntheses ($R = 0{\cdot}252$). Full-matrix least-squares refinements with isotropic and then anisotropic thermal parameters reduced R from 0·252 to 0·073. At this stage, seven of the nine H atoms were located from a difference Fourier map. The H atoms were given the isotropic

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Table 1. *Atomic coordinates* ($\times 10^4$, *for* H *atoms* $\times 10^3$) *with estimated standard deviations, and* B_{eq} *values*

	x	y	z	B_{eq} (Å^2)
Cl	6871 (3)	2919 (2)	5274 (4)	4·2
O(1)	5041 (6)	5552 (5)	2073 (9)	4·2
O(2)	1195 (7)	4814 (5)	3656 (9)	4·8
O(3)	2900 (5)	5447 (5)	566 (9)	4·2
O(4)	−732 (7)	4318 (5)	3342 (10)	5·3
C(1)	3804 (9)	4236 (7)	2117 (13)	3·1
C(2)	4881 (9)	4708 (8)	2574 (13)	3·5
C(3)	5834 (9)	4294 (8)	3524 (14)	3·9
C(4)	5677 (10)	3439 (9)	4054 (14)	4·3
C(5)	4600 (10)	2942 (8)	3701 (15)	4·5
C(6)	3681 (10)	3364 (8)	2694 (14)	4·1
C(7)	2840 (9)	4661 (9)	1005 (14)	3·8
C(8)	1788 (9)	4126 (7)	178 (15)	3·5
C(9)	632 (9)	4028 (6)	973 (14)	3·2
C(10)	−305 (9)	3585 (7)	51 (16)	3·9
C(11)	−99 (11)	3209 (7)	−1653 (18)	4·7
C(12)	1057 (12)	3302 (7)	−2457 (15)	4·5
C(13)	1993 (9)	3751 (7)	−1544 (14)	3·7
C(14)	405 (12)	4432 (7)	2774 (16)	4·0
H(1)	275	299	245	
H(2)	682	461	390	
H(3)	−123	339	53	
H(4)	304	373	200	
H(5)	−76	454	475	
H(7)	129	302	−400	
H(8)	−93	291	−211	

temperature factors of the atoms to which they are bonded. The final least-squares refinement was performed with anisotropic thermal parameters for non-hydrogen atoms and the inclusion of H atoms at fixed positions with fixed isotropic thermal parameters. The final $R = 0{\cdot}064$ and $R_w = 0{\cdot}083$ for 622 reflexions. Cruickshank, Pilling, Bujosa, Lovell & Truter's (1961) type of weighting scheme was used; $w = (A + B|F_o| + C|F_o|^2)^{-1}$ with $A = 5{\cdot}86$, $B = 0{\cdot}017$, $C = 0$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The average and maximum values of the shift/error for the atomic parameters after refinement were 0·027 and 0·152 repectively. A final difference map showed no peaks greater than 0·27 e Å^{-3}. All computations were carried out using the XRAY 70 system (Stewart, Kundell & Baldwin, 1970).

The final atomic coordinates and their estimated standard deviations are listed in Table 1.*

Discussion. Bond lengths and angles are given in Table 2. The structure projected along c is shown in Fig. 1 with the numbering of the atoms. The average C–C

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35412 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Cl C(4)	1·748 (18)	C(7) -C(8)	1·517 (17)
C(1) C(2)	1·405 (16)	C(8)- C(9)	1·401 (18)
C(2) -C(3)	1·386 (18)	C(9)–C(10)	1·384 (17)
C(3)–C(4)	1·369 (18)	C(10)–C(11)	1·399 (18)
C(4)- C(5)	1·410 (17)	C(11)–C(12)	1·404 (21)
C(5)–C(6)	1·386 (19)	C(12)–C(13)	1·385 (18)
C(6)- C(1)	1·400 (15)	C(13)--C(8)	1·409 (15)
O(1)–C(2)	1·347 (14)	C(9)–C(14)	1·483 (16)
C(1)- C(7)	1·463 (19)	C(14)- O(2)	1·212 (17)
O(3) -C(7)	1·240 (15)	C(14) O(4)	1·320 (17)

C(1)--C(2)–C(3)	119·7 (1·0)	C(7) -C(8)--C(13)	116·4 (0·9)
C(2)–C(3) C(4)	118·6 (1·0)	C(8)–C(9) C(10)	120·0 (0·9)
C(3)- C(4)–C(5)	124·3 (1·0)	C(9)- C(10) -C(11)	120·6 (1·0)
C(4) -C(5)–C(6)	115·7 (1·1)	C(10) C(11)–C(12)	119·8 (1·0)
C(5)–C(6) C(1)	121·9 (1·0)	C(11)–C(12) C(13)	119·8 (1·0)
C(6)–C(1) C(2)	119·7 (0·9)	C(12) -C(13) C(8)	120·4 (1·0)
C(6) -C(1)--C(7)	121·0 (1·0)	C(13) -C(8)- C(9)	119·4 (0·9)
C(7)–C(1)–C(2)	119·2 (1·0)	C(8) -C(9)–C(14)	119·6 (0·9)
C(1) -C(2)–O(1)	122·1 (0·9)	C(10) -C(9)–C(14)	120·3 (1·0)
C(3) C(2) -O(1)	118·1 (0·9)	O(2) C(14) -O(4)	123·2 (1·0)
C(1)–C(7) C(8)	120·6 (1·0)	O(2)–C(14) C(9)	123·1 (1·1)
C(1) C(7)--O(3)	122·0 (0·9)	O(4)–C(14)–C(9)	113·6 (1·0)
O(3) -C(7)--C(8)	117·1 (0·9)	Cl–C(4) C(3)	118·7 (0·8)
C(7) -C(8)- C(9)	124·1 (0·9)	Cl C(4)–C(5)	117·0 (0·9)

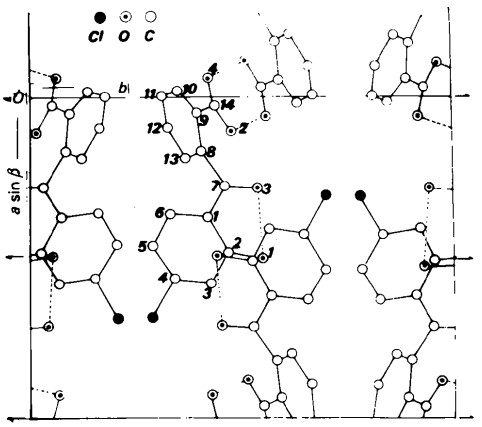

Fig. 1. Projection of the structure along the *c* axis. Hydrogen bonds are shown by broken lines.

bond lengths in the two benzene rings are 1·393 (17) and 1·396 (19) Å respectively and agree with the accepted value of the aromatic C–C bond length of 1·397 Å. The significant angular deviation in the benzene ring at the substituted C(4) is in agreement with recent studies (Domenicano, Mazzeo & Vaciago, 1976). The length of the C–OH bond [1·347 (14) Å] is within the limits observed in various hydroxyl compounds reported by Andersen & Andersen (1975).

Although the position of the H atom was not determined, no doubt the close contact of 2·55 (2) Å between the phenolic O(1) and the carbonyl O(3) represents an intramolecular hydrogen bond. This intramolecular hydrogen-bond length agrees well with the value found in 2,4-dihydroxybenzophenone (Liebich, 1979). There is also a strong intermolecular

Table 3. *Intermolecular distances* (Å) *less than* 3·5 Å *between the non-hydrogen atoms, and hydrogen bonding*

C(3)···O(1^{i})	3·40 (2)	C(2)···O(1^{ii})	3·440 (12)
C(2)···C(3^{i})	3·35 (2)	O(3)···C(3^{ii})	3·35 (3)
O(1)···C(4^{i})	3·34 (2)	O(1)···C(7^{ii})	3·27 (3)
O(1)···O(1^{ii})	3·477 (10)	O(3)···O(1^{ii})	3·35 (3)
C(13)···O(1^{ii})	3·415 (14)	C(10)···O(3^{iii})	3·199 (14)
C(2)···O(3^{ii})	3·38 (4)	C(13)···O(4^{iii})	3·487 (17)
O(1)···C(1^{ii})	3·36 (3)		

Hydrogen bond: O(4)–H(5)···O(2^{iv}) 2·627 (13); O(4)–H(5) 1·09; H(5)···O(2^{iv}) 1·61 Å; ∠O(4)–H(5)–O(2) 153°.

Symmetry code: (i) $1-x, 1-y, 1-z$; (ii) $1-x, 1-y, \bar{z}$; (iii) $\bar{x}, 1-y, \bar{z}$; (iv) $\bar{x}, 1-y, 1-z$. Short intermolecular distances due to dimerization (iv) have been omitted.

hydrogen bond, which binds the molecules into centrosymmetric dimers, through carboxyl groups. The O(2)···O(4) hydrogen-bond distance of 2·627 (13) Å is in the range normally observed for aromatic carboxylic acid dimers. The carboxyl group is approximately coplanar with the benzene ring to which it is bonded. The dihedral angle between the least-squares plane of the ring and the carboxyl group is only about 2°. Both benzene rings are planar within experimental error and are nearly perpendicular to each other, making a dihedral angle of 89°.

Fig. 1 shows the packing in the crystal viewed along **c**. C(2) and O(1) in the molecules related by the symmetry operations $1-x, 1-y, 1-z$ and $1-x, 1-y, \bar{z}$ are located above O(1) and C(2) respectively. Because of this overlapping many close intermolecular contacts occur (Table 3), but none of these is substantially shorter than the sum of the van der Waals radii of the atoms concerned.

The author would like to thank Professor J. Gronowska for supplying the crystal before the publication of her experimental results and for a valuable discussion, Dr M. Główka (Institute of General Chemistry, Lódź) for advice on the use of the XRAY system of crystallographic programs, Mgr T. Cieplak for installing the XRAY system on a Riad-32 computer, and Mgr B. Walentynowicz for technical assistance.

References

ANDERSEN, E. K. & ANDERSEN, I. G. K. (1975). *Acta Cryst.* B**31**, 387–390.

CRUICKSHANK, D. W. J., PILLING, D. E., BUJOSA, A., LOVELL, F. M. & TRUTER, M. R. (1961). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, edited by R. PEPINSKY, J. M. ROBERTSON & J. C. SPEAKMAN. Oxford: Pergamon Press.

DOMENICANO, A., MAZZEO, P. & VACIAGO, A. (1976). *Tetrahedron Lett.* pp. 1029–1032.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

LIEBICH, B. W. (1979). *Acta Cryst.* B**35**, 1186–1190.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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Structure of a Diastereoisomer of 1,9-Dimethyl-8-azabicyclo[4.3.0]nonane-3,7-dione. Proof of the Stereochemistry of a Synthetic Intermediate in the Synthesis of Vitamin B_{12}

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Abstract. $C_{10}H_{15}NO_2$, monoclinic, $P2_1$, $a = 7{\cdot}060\ (3)$, $b = 6{\cdot}510\ (3)$, $c = 10{\cdot}240\ (4)$ Å, $\beta = 100{\cdot}51\ (5)°$, $U = 462{\cdot}74$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$ Mg m^{-3}, $F(000) = 196$, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}091$ mm^{-1}. The structure was solved by direct methods and refined to $R = 0{\cdot}056$ for 809 observed reflections. Molecules are linked by hydrogen bonds to form columns down the screw axis.

Introduction. During studies of the synthesis of vitamin B_{12} the anion of nitroethane was added by a Michael reaction to a Hageman's ester (2-methyl-4-oxo-2-cyclohexene-1-carboxylate) (1) to give a racemic mixture of diastereoisomers (2) in 65% yield (Begbie, 1970).

The relative stereochemistry (*trans*) of the Michael addition was proved by synthetic comparison with a

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