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2-Hydroxy-4-methoxybenzophenone

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Abstract. $C_{14}H_{12}O_3$, orthorhombic, *Pbca*, $a=12{\cdot}069(7)$, $b=7{\cdot}198(4)$, $c=26{\cdot}37(2)$ Å, $U=2290{\cdot}8$ Å^3, $Z=8$, F.W. 228·2, $D_x=1{\cdot}322$ g cm^{-3}, $F(000)=960$. Counter technique, direct methods, least-squares refinement. $R=0{\cdot}051$ for 937 reflexions measured at room temperature (20°C). An intramolecular asymmetrical hydrogen bond with O(1)–O(2) = 2·55 Å is present.

Introduction. 2-Hydroxy-4-methoxybenzophenone is used as an ultraviolet absorber in cosmetics and plastics. The fluorescent properties of its complex with boron in sulphuric acid may be used to determine trace amounts of boron (Liebich, 1972). The product is commercially available from EGA Chemie K.G. in Steinheim (Germany).

Reciprocal-lattice explorer photographs of a needle-shaped single crystal (diameter 0·1 mm, length 0·4 mm) permitted the determination of the unique centrosymmetric space group *Pbca* (No. 61) with systematic absences for $0kl$ with $k\neq 2n$, $h0l$ with $l\neq 2n$ and $hk0$ with $h\neq 2n$. 1362 intensities were collected at room temperature (20°C) on a Philips PW 1100 automatic diffractometer (ω–2θ scan, Mo $K\alpha$ radiation, $\lambda=0{\cdot}7107$ Å, graphite monochromator).

The crystal structure was solved by direct methods with the computer program *LSAM* (Main, Woolfson & Germain, 1972). The atomic positions and anisotropic thermal parameters of the heavy atoms, given in Table 1, were refined by full-matrix least-squares calculation, the function minimized being $\sum\omega(\Delta F_o^2)$ where $\omega=1/\sigma^2(F_o)$ (*ORXFLS*3 programme by Busing *et al.*, 1971*b*). A list of structure factors is available.*

The positions of the hydrogen atoms were found from a difference map, were subsequently refined with isotropic thermal parameters, and are given in Table 2.

Table 2. *Positional and thermal parameters of the hydrogen atoms*

	x	*y*	*z*	*B*
H(O1)	0·358 (3)	0·300 (5)	0·129 (2)	6 (1)
H(C3)	0·399 (3)	0·411 (4)	0·002 (1)	2·3 (8)
H(C5)	0·059 (2)	0·116 (4)	0·474 (1)	1·8 (7)
H(C6)	0·027 (2)	0·325 (4)	0·065 (1)	1·8 (7)
H(C2′)	0·091 (3)	0·055 (5)	0·205 (1)	5 (1)
H(C3′)	0·424 (2)	0·068 (5)	0·253 (1)	4·4 (9)
H(C4′)	0·294 (3)	0·315 (5)	0·270 (1)	5·2 (9)
H(C5′)	0·165 (3)	0·064 (5)	0·333 (1)	4·6 (9)
H(C6′)	0·489 (2)	0·062 (4)	0·127 (1)	2·7 (7)
H(C8)	0·164 (3)	0·506 (5)	0·372 (1)	4·5 (9)
H′(C8)	0·383 (3)	0·151 (5)	0·409 (1)	6 (1)
H″(C8)	0·110 (3)	0·430 (5)	0·430 (1)	3·6 (9)

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Table 1. *Positional and thermal parameters for 2-hydroxy-4-methoxybenzophenone* ($\times 10^4$)

Space group *Pbca*. All atoms are in equipoint 8(*c*). The thermal parameters are of the form $\exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^3+2b_{12}hk+2b_{13}hl+2b_{23}kl)]$. The standard deviations are in parentheses.

	x	*y*	*z*	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	1953 (1)	3356 (4)	843 (1)	62 (4)	171 (4)	10 (1)	7 (4)	−4 (1)	4 (1)
C(2)	3029 (3)	3571 (4)	647 (1)	56 (4)	165 (10)	17 (1)	8 (5)	−9 (2)	−1 (2)
C(3)	3215 (3)	3938 (5)	139 (1)	58 (3)	195 (10)	14 (1)	7 (5)	−5 (2)	1 (2)
C(4)	2321 (3)	917 (4)	4821 (1)	77 (4)	154 (9)	9 (1)	−18 (5)	−1 (2)	−2 (2)
C(5)	1243 (3)	3813 (5)	9993 (1)	45 (3)	209 (11)	14 (1)	4 (4)	1 (1)	2 (2)
C(6)	1073 (3)	3466 (5)	500 (1)	62 (3)	165 (10)	14 (1)	8 (5)	−2 (1)	5 (2)
C(7)	1785 (3)	2988 (5)	1377 (1)	73 (4)	195 (11)	17 (1)	1 (5)	−6 (2)	−1 (3)
C(8)	3472 (4)	188 (8)	4103 (2)	80 (3)	377 (14)	16 (1)	−12 (6)	10 (1)	12 (3)
C(1′)	666 (3)	3046 (5)	1624 (1)	79 (4)	218 (12)	11 (1)	−15 (6)	−5 (1)	4 (3)
C(2′)	409 (3)	1698 (6)	1977 (1)	112 (5)	290 (14)	14 (1)	−16 (7)	−2 (2)	10 (3)
C(3′)	4397 (4)	1720 (7)	2774 (1)	129 (5)	374 (16)	13 (1)	−36 (8)	1 (2)	−14 (3)
C(4′)	3658 (4)	3131 (7)	2870 (1)	95 (5)	461 (18)	11 (1)	−44 (8)	−4 (2)	14 (3)
C(5′)	3901 (3)	4515 (6)	3217 (1)	94 (4)	328 (15)	13 (1)	−3 (7)	3 (2)	15 (3)
C(6′)	4911 (3)	4462 (5)	3478 (1)	82 (4)	235 (12)	14 (1)	−14 (6)	2 (1)	8 (3)
O(1)	3931 (2)	3437 (4)	951 (1)	58 (2)	315 (8)	18 (1)	6 (4)	−9 (1)	3 (2)
O(2)	2586 (2)	2607 (4)	1662 (1)	85 (3)	387 (9)	17 (1)	11 (4)	−8 (1)	19 (2)
O(3)	2398 (2)	488 (3)	4321 (1)	66 (2)	309 (8)	14 (1)	−8 (4)	4 (1)	−2 (2)

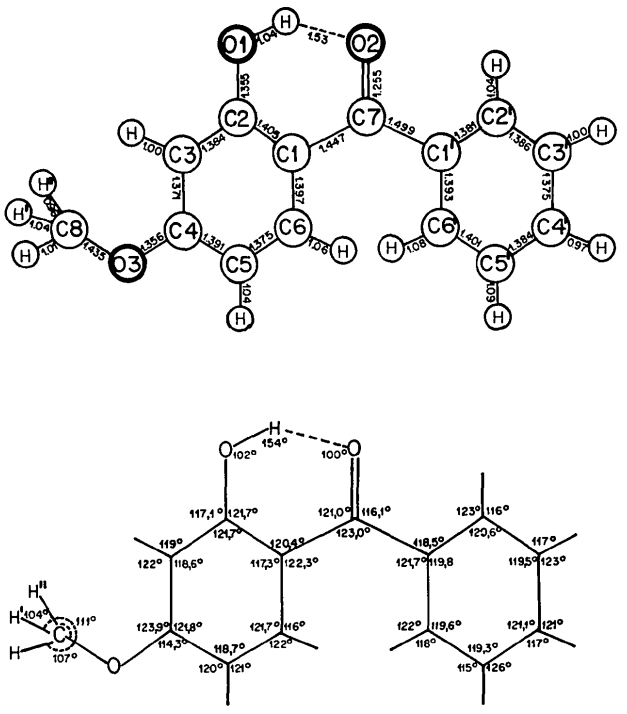

Fig. 1. The molecule, the numbering scheme, the intramolecular distances and the angles in 2-hydroxy-4-methoxybenzophenone. The errors for distances between the heavy atoms are 0·005 Å but for bonds to hydrogen atoms 0·05 Å. The error of the angles between heavy atoms is 0·3°, but for those including hydrogen 2°.

The final $R=\sum|\Delta F|/\sum|F_o|$ is 5·1% for 937 reflexions with $I>3\sigma$.

Discussion. The bond distances and angles, calculated with the program *ORFFE3* (Busing *et al.*, 1971*a*), are given in Fig. 1 and seem normal.

The molecule is characterized by an intramolecular hydrogen bond between O(1) and O(2). Hydrogen bonds with similar environment have been reported for dibenzoylmethane (Williams, 1966), ω-(*p*-toluoyl)-acetophenone enol (Kato, 1971), benzoylacetone (Semmingsen, 1972), 1,5-dihydroxyanthraquinone (Hall & Nobbs, 1966) and naphthazarin (Cradwick & Hall, 1971) to mention only a few. In all these cases the hydrogen atom is asymmetrically located. A stereographic drawing of the molecule is shown in Fig. 2. The dihedral angle between the two benzene rings of one molecule is −49°. Least-squares calculations show that the ring formed by O(1)–O(2)–C(7)–C(1)–C(2) is inclined by 4° with respect to the neighbouring benzene ring. The O(1)–O(2) distance is 2·55 Å.

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References

BUSING, W. R., MARTIN, K. O., LEVY, H. A., BROWN, G. M., JOHNSON, C. K. & THIESSEN, W. E. (1971*a*). *ORFFE3*. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

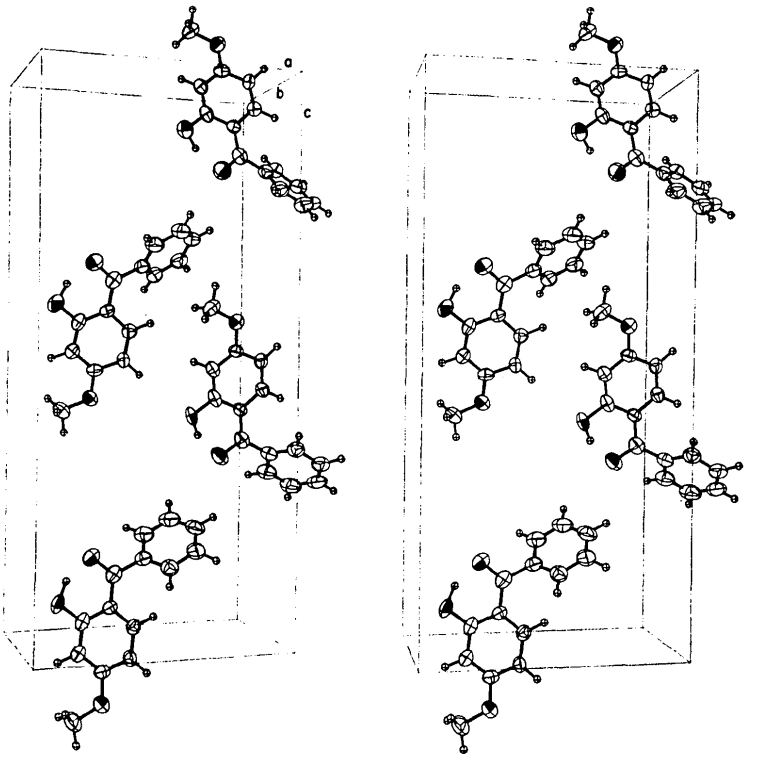

Fig. 2. Stereo view of the arrangement of $C_{14}H_{12}O_3$ molecules in the (010) plane. Only the four molecules with centres between $0<y<\frac{1}{2}$ are shown (*ORTEP*, Johnson, 1965).

BUSING, W. R., MARTIN, K. O., LEVY, H. A., ELLISON, R. D., HAMILTON, W. C., IBERS, J. A., JOHNSON, C. K. & THIESSEN, W. E. (1971*b*). *ORXFLS3*. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
CRADWICK, P. D. & HALL, D. (1971). *Acta Cryst.* B**27**, 1990–1997.
HALL, D. & NOBBS, C. L. (1966). *Acta Cryst.* **21**, 927–934.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
KATO, K. (1971). *Acta Cryst.* B**27**, 2028–2034.
LIEBICH, B. W. (1971). *Etude du Système Luminescent* HMCB–H_3BO_3–H_2SO_4 *(conc.) et ses applications au dosage de traces de bore dans les eaux, sols et plantes*. Ph. D. dissertation, Université de Genève, Genève.
MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1972). *LSAM*, a System of Computer Programs for Automatic Solution of Centrosymmetric Crystal Structures. Department of Physics, Univ. of York, York, England.
SEMMINGSEN, D. (1972). *Acta Chem. Scand.* **26**, 143–154.
WILLIAMS, D. E. (1966). *Acta Cryst.* **21**, 340–349.

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Crystal data for iodostrychninesulphonic acid.

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Crystal data for iodostrychninesulphonic acid ($IC_{21}H_{22}N_2O_2SO_3$) are: orthorhombic, $P2_12_12$, $Z=4$, $F(000)=1140$; $a=12{\cdot}53$ (2), $b=15{\cdot}67$ (1), $c=10{\cdot}81$ (1) Å, $D_m=1{\cdot}72$, $D_c=1{\cdot}69$ g cm^{-3}.

The structure of strychninesulphonic acid tetrahydrate has been solved by direct methods (Sake Gowda, Cartz & Natarajan, 1973). If this structure had not been solved by this method, an alternative approach was to have been by the use of a heavy-atom derivative of strychninesulphonic acid. For this purpose, iodostrychninesulphonic acid in powder form was kindly supplied by Professor John T. Edward, McGill University, Canada. Crystals were grown from a solution of dilute ammonium hydroxide and were very tiny, needle-like, and yellow-white in colour. The space group was determined on a single-crystal diffractometer with the crystal mounted about the needle axis (*c* axis) and Cu $K\alpha$ radiation. The extinction conditions were $h00$ ($h=2n$), $0k0$ ($k=2n$) with no other systematic absence, so that the space group is $P2_12_12$. The cell dimensions were determined from a least-squares analysis of 10 high-angle reflections. The crystal data are given in Table 1.

The density was measured by the flotation technique in bromobenzene and bromoform. The agreement between the measured and the calculated density indicates the absence of water of crystallization in iodostrychninesulphonic acid whereas four water molecules of crystallization were found to be present in crystals of strychninesulphonic acid. The molecular packing is likely to be different for the iodo derivative, because of the different crystal symmetry, with the sulphur oxygen atoms involved in the intermolecular bonding. The detailed crystal structure of the iodo derivative is not being determined.

Table 1. *Crystal data for iodostrychninesulphonic acid*

Chemical formula: $IC_{21}H_{22}N_2O_2SO_3$
Crystal system and space group: orthorhombic, $P2_12_12$
$Z=4$
$F(000)=1140$
$a=12{\cdot}53$ (2) Å
$b=15{\cdot}67$ (1)
$c=10{\cdot}81$ (1)
$D_m=1{\cdot}72$ g cm^{-3}
$D_c=1{\cdot}69$

Reference

SAKE GOWDA, D. S., CARTZ, L. & NATARAJAN, S. (1973). *Acta Cryst.* B**29**, 2760–2770.