

**Discussion.** The final atomic coordinates are given in Table 1.\* Fig. 1 gives the atom numbering as well as the final bond distances and angles in the structure. The pyridine ring is essentially planar and the maximum deviation of the ring atoms from the least-squares plane is 0.009 Å. The C(2)—N(1)—C(6) bond angle of 116.8 (2)° agrees with the values found in nicotinamide (Wright & King, 1954), isonicotinic acid (Takusagawa & Shimada, 1976), nicotinic acid (Wright & King, 1953; Gupta & Kumar, 1975; Kutoglu & Scheringer, 1983) and other non-protonated pyridine compounds (~117°) (Singh, 1965). The dihedral angle between the planes of the pyridine ring and of the methylcarboxamide groups is 22.0°.

Fig. 2 gives a stereoview of the packing of the molecules in the unit cell. Molecules related by screw axes are connected by an N(7)—H(N7)···O(7) hydrogen bond involving the amino N(7) and the carbonyl O(7) atoms [N(7)···O(7) 2.869 (1), H(N7)···O(7) 1.98 (2) Å and N(7)—H(N7)···O(7) 163 (2)°]. There is no intermolecular stacking observed in the crystal structure.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52949 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of Xanthone

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**Abstract.** 9H-Xanthen-9-one, C<sub>13</sub>H<sub>8</sub>O<sub>2</sub>, *M*<sub>r</sub> = 196.21, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 14.072 (10), *b* = 13.627 (8), *c* = 4.878 (3) Å, *V* = 935.4 (10) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.393, *D*<sub>m</sub> = 1.391 (6) g cm<sup>-3</sup>, *Mo* *K*α, λ = 0.70926 Å, μ = 0.875 cm<sup>-1</sup>, *F*(000) = 408, room temperature, *R* = 0.077 for 648 independent reflections. The molecule is planar except for the carbonyl O atom (0.13 Å out of the plane). The benzene rings are not regular hexagons but the distortion is sym-

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metric and the molecule has approximate *C*<sub>2v</sub> symmetry. There are no abnormally short distances between molecules.

**Introduction.** The molecular structure of xanthone has been studied in the gas and the solid phases. In the gas phase xanthone is planar and has *C*<sub>2v</sub> symmetry. The benzene rings were assumed to be regular hexagons in the structure analysis, but the symmetry of the ring appeared to be lower (Iijima, Misu, Ohnishi & Onuma, 1989). On the other hand, the

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Table 1. Atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2$ )

	x	y	z	$B_{\text{eq}}^*$
O(1)	0.2896 (4)	0.3462 (4)	0.270 (1)	4.3
O(2)	0.5192 (4)	0.1679 (5)	0.357 (2)	6.3
C(11)	0.2957 (6)	0.2622 (7)	0.123 (2)	4.4
C(12)	0.2229 (6)	0.2451 (8)	-0.065 (2)	4.9
C(13)	0.2259 (7)	0.1617 (8)	-0.215 (3)	6.3
C(14)	0.2981 (8)	0.0948 (7)	-0.201 (3)	6.4
C(15)	0.3710 (7)	0.1113 (6)	-0.019 (3)	5.6
C(16)	0.3709 (6)	0.1963 (6)	0.148 (2)	3.5
C(7)	0.4480 (6)	0.2188 (6)	0.335 (2)	4.2
C(21)	0.3580 (6)	0.3669 (6)	0.460 (2)	4.0
C(22)	0.3455 (7)	0.4546 (6)	0.605 (3)	5.5
C(23)	0.4103 (8)	0.4799 (7)	0.796 (2)	5.9
C(24)	0.4890 (7)	0.4229 (8)	0.845 (2)	6.0
C(25)	0.5030 (7)	0.3371 (7)	0.704 (2)	5.1
C(26)	0.4370 (5)	0.3092 (6)	0.501 (2)	3.6
H(12)	0.175 (6)	0.304 (6)	-0.09 (2)	7 (3)
H(13)	0.174 (4)	0.151 (5)	-0.39 (2)	3 (2)
H(14)	0.289 (6)	0.043 (6)	-0.29 (2)	8 (3)
H(15)	0.419 (5)	0.073 (6)	0.04 (2)	5 (2)
H(22)	0.303 (6)	0.497 (8)	0.49 (2)	10 (3)
H(23)	0.406 (7)	0.543 (7)	0.90 (3)	10 (3)
H(24)	0.531 (5)	0.430 (5)	1.04 (2)	5 (2)
H(25)	0.552 (5)	0.282 (5)	0.73 (2)	4 (2)

\*  $B_{\text{eq}}$  is defined as  $(4/3)(B_{11}a^2 + B_{22}b^2 + B_{33}c^2)$ .

crystal structure analysis could not provide a definite conclusion about the planarity of the molecule (Biswas & Sen, 1982). The C—C distances of the benzene rings vary widely between 1.33 and 1.43 Å, and no systematic trend was found in these distances with respect to the molecular symmetry. This seems to be due to the inaccurate intensity data from the visual method of crystal structure analysis. Thus we undertook a redetermination of the crystal structure of xanthone with diffractometer data.

**Experimental.** Single crystals were grown from a benzene solution. A crystal of dimensions 0.40 × 0.30 × 0.25 mm was selected for study. Crystal density was measured by the flotation method. Rigaku diffractometer, graphite-monochromated Mo  $K\alpha$ . Cell constants from setting angles of 20 reflections ( $18 < 2\theta < 25^\circ$ ). Intensity measurement by  $\omega$ - $2\theta$  scan. The scan speed was  $8^\circ \text{ min}^{-1}$  in  $2\theta$ . Standard reflections, 180, 701 and 402, observed every 100 measurements, no appreciable trends. 1323 reflections measured, 648 reflections [ $h \leq 17, k \leq 16, l \leq 6, 2\theta < 55.0^\circ$  with  $F > 3\sigma(F)$ ] used to solve and refine the structure. Correction for Lorentz-polarization, absorption ignored. The structure was solved by direct methods using *MULTAN* (Main, Lessinger, Woolfson Germain & Declercq, 1977) and refined by a block-diagonal least-squares method with anisotropic temperature factors to  $R = 0.099$ . Unit weights were adopted. The function minimized was  $\sum \Delta F^2$ . The H atoms were placed at C—H = 1.08 Å and included in the subsequent refinement with isotropic temperature factors. Final  $R = 0.077$ ,  $wR = 0.075$  and  $S = 1.14$  obtained using 169 parameters.

$(\Delta/\sigma)_{\text{max}} = 0.44$  in final cycle,  $\Delta\rho < 0.24 \text{ e \AA}^{-3}$  in final difference map. A full-matrix least-squares analysis could not lower the  $R$  factor. The relatively large  $R$  value appeared to be attributable to the poorly shaped crystal hindering accurate intensity measurements. The final coordinates and temperature factors are given in Table 1.\* The labelling of the atoms is shown in Fig. 1. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The analysis was carried out using *UNICSIII* programs (Sakurai & Kobayashi, 1979) in the Computer Center of the University of Tokyo.

**Discussion.** Fig. 1 shows the perspective view of the unit cell. There is one molecule in the asymmetric unit. The shortest intermolecular contact (excluding H atoms) is 3.23 Å for O(2)···C(23<sup>i</sup>) [(i):  $1 - x, y - 0.5, 1.5 - z$ ] as shown in Fig. 1, and there are no abnormally short distances between molecules. The molecule is planar except for the O(2) atom, which deviates by 0.13 Å from the plane. The deviation of the O(2) atom is probably due to the repulsion from the C(23<sup>i</sup>) atom mentioned above and the H atom attached to C(23<sup>i</sup>).

Bond distances and angles are given in Table 2. In the central ring the COC angle is  $119^\circ$ , and both the C—O bonds are shorter than those in dimethyl ether (1.415 Å) (Tamagawa, Takemura, Konaka & Kimura, 1984). The two C(7)—C bonds in the central ring are shorter than the corresponding bond in acetone (1.520 Å) (Iijima, 1972). This trend is similar

\* Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52931 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

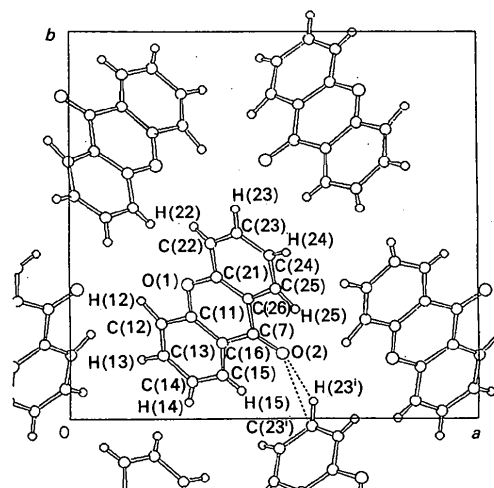


Fig. 1. Structure of xanthone projected along the  $c$  axis.

Table 2. Bond distances (Å) and angles (°) for xanthone with *e.s.d.*'s in parentheses

C(11)—C(12)	1.39 (1)	C(21)—C(22)	1.40 (1)
C(12)—C(13)	1.35 (2)	C(22)—C(23)	1.35 (2)
C(13)—C(14)	1.37 (2)	C(23)—C(24)	1.37 (2)
C(14)—C(15)	1.38 (2)	C(24)—C(25)	1.37 (2)
C(15)—C(16)	1.42 (1)	C(25)—C(26)	1.41 (1)
C(16)—C(11)	1.39 (1)	C(26)—C(21)	1.38 (1)
O(1)—C(11)	1.36 (1)	O(1)—C(21)	1.37 (1)
C(7)—C(16)	1.45 (1)	C(7)—C(26)	1.48 (1)
C(7)—O(2)	1.22 (1)		
C(11)—C(12)—C(13)	118.1 (9)	C(21)—C(22)—C(23)	118.9 (9)
C(12)—C(13)—C(14)	124.0 (10)	C(22)—C(23)—C(24)	121.4 (10)
C(13)—C(14)—C(15)	118.4 (10)	C(23)—C(24)—C(25)	120.8 (10)
C(14)—C(15)—C(16)	120.3 (9)	C(24)—C(25)—C(26)	119.2 (9)
C(15)—C(16)—C(11)	118.4 (8)	C(25)—C(26)—C(21)	118.7 (8)
C(16)—C(11)—C(12)	120.7 (9)	C(26)—C(21)—C(22)	121.0 (9)
O(1)—C(11)—C(12)	116.3 (8)	O(1)—C(21)—C(22)	115.6 (8)
O(1)—C(11)—C(16)	123.0 (8)	O(1)—C(21)—C(26)	123.3 (8)
C(11)—C(16)—C(7)	119.3 (8)	C(21)—C(26)—C(7)	118.6 (8)
C(15)—C(16)—C(7)	122.3 (8)	C(25)—C(26)—C(7)	122.7 (8)
C(16)—C(7)—O(2)	123.4 (8)	C(26)—C(7)—O(2)	120.5 (8)
C(11)—O(1)—C(21)	119.4 (6)	C(16)—C(7)—C(26)	116.2 (7)

to that in the gas phase (Iijima, Misu, Ohnishi & Onuma, 1989), and we conclude that the  $p_z$  electrons of atoms O(1) and C(7) are used for conjugation in

the central ring and this conjugation makes the skeleton of the molecule planar.

The benzene rings are apparently not regular hexagons in shape. This distortion, however, appears to be symmetric, and the whole molecule has approximate  $C_{2v}$  symmetry.

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## Structure of 2,4,4,6-Tetraphenyl-4H-pyran

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**Abstract.**  $C_{29}H_{22}O$ ,  $M_r = 386.49$ , monoclinic,  $P2_1/c$ ,  $a = 12.128$  (5),  $b = 12.372$  (5),  $c = 14.599$  (5) Å,  $\beta = 105.57$  (3)°,  $V = 2110$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.21$  (1),  $D_x = 1.217$  (1) Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.561$  mm<sup>-1</sup>,  $F(000) = 816$ ,  $T = 298$  K,  $R = 0.039$  for 2729 unique observed reflections. The 4H-pyran ring is roughly planar with the phenyl rings substituted on the C( $sp^2$ ) atoms C2 and C6. The ring shows a slightly distorted boat conformation with the boat angles 4.3 (1) and 7.7 (1)° on C( $sp^2$ ) and O, respectively. The observed conformation is compared with three other structures containing similar pyran rings.

**Introduction.** The structure of 2,4,4,6-tetraphenyl-4H-pyran has been determined by the X-ray method because by analogy with three similar structures which were available from the Cambridge Structural Database (Allen *et al.*, 1979; release 1988), neither

the conformation of the 4H-pyran moiety nor the orientation of its substituents could be predicted unambiguously. The photochromic behaviour is similar to that of differently substituted 1,4-dihydropyridines observed by Iwasaki, Watanabe & Maeda (1987). The molecular geometry is considered to be significant for the explanation of photochromic properties.

**Experimental.** 2,4,4,6-Tetraphenyl-4H-pyran crystallized as colourless, well developed crystals from an oxygen-free benzene solution. The crystals turn blue in light and X-rays, but no changes in diffracted intensities were observed. The density was determined by flotation in an aqueous KBr solution.

The crystal used was a prism with dimensions 0.6 × 0.3 × 0.3 mm. Weissenberg photographs confirmed the good quality of the crystal and indicated