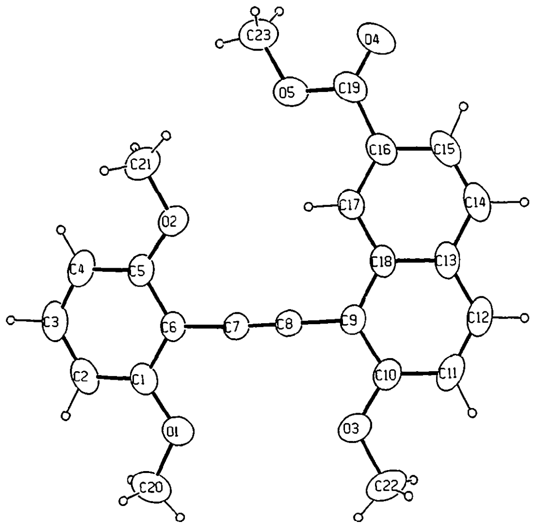

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with arbitrary radius.

methyl 2-[(2,6-dimethoxyphenyl)ethynyl]benzoate: Huang, Evans, Fronczek & Gandour (1991) and 1-ethynyl-2,7-dimethoxynaphthalene: Prince, Fronczek & Gandour (1990).

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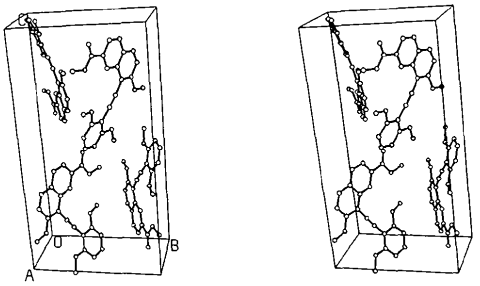

Fig. 2. Stereoview of the unit cell.

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Structure of 2-Cyanobenzophenone

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Abstract. 2-Benzoylbenzonitrile, $C_{14}H_9NO$, $M_r = 207.23$, monoclinic, $P2_1/c$, $a = 12.771$ (7), $b = 7.873$ (5), $c = 11.571$ (4) Å, $\beta = 112.47$ (3)°, $V = 1075$ (1) Å^3, $Z = 4$, $D_x = 1.280$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.08$ mm^{-1}, $F(000) = 432$, $T = 291$ (1) K, final $R = 0.048$ for 981 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The structure consists of discrete molecular units. All bond lengths, bond angles and dihedral angles are normal. The torsion angles O(1)—C(1)—C(8)—C(13) −42.6 (4) and C(1)—C(8)—C(13)—C(14) −2.8 (5)° indicate relatively strong repulsion between the C=O and C≡N groups. The planes of the two rings form a dihedral angle of 66.2 (1)°.

Experimental. The title compound was prepared by reaction of *o*-aminobenzophenone *via* the diazonium compound (Seidel & Bezner, 1932). Colourless crystals (m.p. 359.5 K) were obtained by multiple recrystallization from anhydrous ethanol. The vibrational spectrum shows a C≡N band at 2232 cm^{-1} and a C=O band at 1663 cm^{-1}. A crystal platelet of size ~ 0.52 × 0.14 × 0.09 mm was used. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 1.0–10.2° min^{-1} in θ, scan width 1.2° plus dispersion. A Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a symmetry-constrained least-squares fit of 24 reflections with $2\theta_{\max} = 21.96°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (200, 020, 003, $\bar{2}00$, $0\bar{2}0$, $00\bar{3}$) were recorded every 300 reflections, only random deviations during 85.0 h of X-ray exposure; 2531 reflections with $2.0 \le 2\theta \le 42.0°$ (4264 accessible), $0 \le h \le 12$, $-8 \le k \le 8$, $-13 \le l \le 13$, were measured. The data were corrected for Lorentz–polarization but not for absorption effects because of the small absorption coefficient and averaged ($R_{\rm int} = 0.041$) to 1135 unique reflections, 981 of which had $F \ge 4.0\sigma(F)$. The systematic absences ($h0l$) $l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/c$. The structure was solved *via* direct methods and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for the H atoms, which were placed in geometrically calcu-

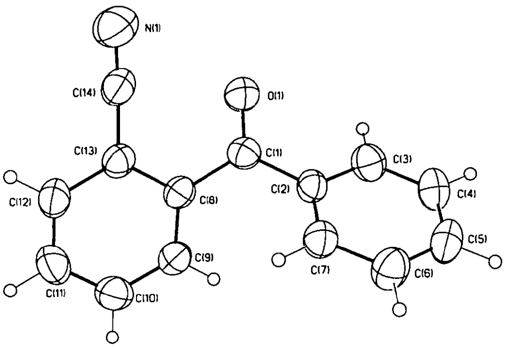

Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

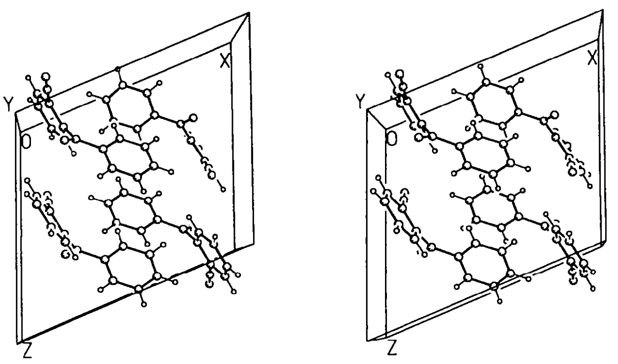

Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O(1)	0.7922 (2)	0.1571 (3)	0.8043 (2)	563
N(1)	0.8696 (3)	0.0960 (4)	1.1075 (3)	730
C(1)	0.7418 (2)	0.2910 (3)	0.7983 (2)	416
C(2)	0.6241 (2)	0.3147 (3)	0.7046 (2)	401
C(3)	0.5885 (3)	0.2208 (4)	0.5953 (3)	514
C(4)	0.4783 (3)	0.2340 (4)	0.5099 (3)	570
C(5)	0.4031 (3)	0.3364 (4)	0.5341 (3)	555
C(6)	0.4364 (3)	0.4279 (4)	0.6430 (3)	564
C(7)	0.5477 (3)	0.4192 (4)	0.7269 (3)	491
C(8)	0.7966 (2)	0.4303 (3)	0.8873 (3)	412
C(9)	0.7932 (3)	0.5969 (4)	0.8471 (3)	509
C(10)	0.8505 (3)	0.7235 (4)	0.9284 (3)	592
C(11)	0.9095 (3)	0.6873 (4)	1.0534 (3)	611
C(12)	0.9119 (3)	0.5249 (4)	1.0968 (3)	548
C(13)	0.8575 (2)	0.3950 (4)	1.0149 (3)	438
C(14)	0.8642 (3)	0.2258 (4)	1.0639 (3)	501

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes, a dihedral angle* (°) *and possible hydrogen bonds* (Å, °)

O(1)—C(1)	1.223 (4)	C(6)—C(7)	1.383 (4)
N(1)—C(14)	1.130 (5)	C(8)—C(9)	1.386 (4)
C(1)—C(2)	1.491 (3)	C(8)—C(13)	1.409 (4)
C(1)—C(8)	1.485 (4)	C(9)—C(10)	1.374 (4)
C(2)—C(3)	1.384 (4)	C(10)—C(11)	1.382 (5)
C(2)—C(7)	1.374 (5)	C(11)—C(12)	1.370 (5)
C(3)—C(4)	1.381 (4)	C(12)—C(13)	1.387 (4)
C(4)—C(5)	1.363 (5)	C(13)—C(14)	1.438 (4)
C(5)—C(6)	1.370 (5)		

O(1)—C(1)—C(8)	119.9 (3)	C(1)—C(8)—C(13)	120.4 (3)
O(1)—C(1)—C(2)	120.8 (3)	C(1)—C(8)—C(9)	121.3 (3)
C(2)—C(1)—C(8)	119.3 (3)	C(9)—C(8)—C(13)	118.2 (3)
C(1)—C(2)—C(7)	122.0 (3)	C(8)—C(9)—C(10)	121.1 (3)
C(1)—C(2)—C(3)	119.0 (3)	C(9)—C(10)—C(11)	120.1 (3)
C(3)—C(2)—C(7)	118.9 (3)	C(10)—C(11)—C(12)	120.2 (3)
C(2)—C(3)—C(4)	120.2 (3)	C(11)—C(12)—C(13)	120.2 (3)
C(3)—C(4)—C(5)	120.2 (3)	C(8)—C(13)—C(12)	120.1 (3)
C(4)—C(5)—C(6)	120.3 (3)	C(12)—C(13)—C(14)	118.4 (3)
C(5)—C(6)—C(7)	119.7 (3)	C(8)—C(13)—C(14)	121.5 (3)
C(2)—C(7)—C(6)	120.7 (3)	N(1)—C(14)—C(13)	176.8 (4)

O(1)—C(1)—C(8)—C(9)	135.0 (3)	C(2)—C(1)—C(8)—C(13)	135.6 (3)
O(1)—C(1)—C(8)—C(13)	−42.6 (4)	C(8)—C(1)—C(2)—C(7)	−28.3 (4)
O(1)—C(1)—C(2)—C(3)	−26.0 (5)	C(8)—C(1)—C(2)—C(3)	155.7 (3)
O(1)—C(1)—C(2)—C(7)	149.9 (3)	C(1)—C(8)—C(13)—C(14)	−2.8 (5)
C(2)—C(1)—C(8)—C(9)	−46.8 (4)		

No.	Plane through atoms	Equation of the plane	χ^2
1	C(2),C(3),C(4),C(5),C(6),C(7)	$0.466x + 0.7562y - 0.460z = 0.67$ Å	46.5
2	C(8), C(9),C(10),C(11),C(12),C(13)	$0.9670x - 0.175y - 0.186z = 3.68$ Å	40.1

Dihedral angle: 1, 2 66.2 (1)

Possible hydrogen bonds

Donor···acceptor	H···acceptor	Donor—H···acceptor
C(3)···O(1)	H(3)···O(1)	C(3)—H(3)···O(1)
2.839 (3)	2.589 (3)	95.0 (2)
C(11)···N(1$^{\rm i}$)	H(11)···N(1$^{\rm i}$)	C(11)—H(11)···N(1$^{\rm i}$)
3.353 (5)	2.708 (5)	125.1 (3)

Symmetry codes: none x, y, z; (i) $x, y + 1, z$.

lated positions (C—H 0.96 Å). 154 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + (0.005F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 0.92$, $R = 0.048$, $wR = 0.071$, $(\Delta/\sigma)_{max} = 0.033$ (no extinction correction). The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in final $\Delta\rho$ map were ± 0.1 (1) e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is in Fig. 2. Positional parameters and equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes, a dihedral angle and possible hydrogen bonds are given in Table 2.

Related literature. For further details concerning different methods of preparation, see de Diesbach & Bulliard (1924) and Seidel & Bezner (1932).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54782 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0513]

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Structure of Xanthoherquein Tetraacetate*

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Abstract. $C_{23}H_{20}O_{11}.0.25H_2O$, $M_r = 476.91$, monoclinic, $C2/c$, $a = 24.146$ (1), $b = 8.919$ (2), $c = 22.044$ (2) Å, $\beta = 107.69$ (1)°, $V = 4523$ (1) Å^3, $Z = 8$, D_m (flotation in aqueous KI) = 1.391, $D_x = 1.400$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54178$ Å, $\mu = 9.4$ cm^{-1}, $F(000) = 1988$, $T = 294$ K, $R = 0.060$ for 2810 reflections. The molecule contains an approximately planar perinaphthenone ring skeleton, with a delocalized π system and a symmetrical intramolecular O···H···O hydrogen bond [O···O = 2.483 (3), O···H = 1.29, 1.23 Å, O···H···O = 159°]. Small electron-density peaks on twofold (C_2) axes are interpreted as two partially occupied sites for water molecules, which are weakly hydrogen bonded to the carbonyl oxygen of an acetate group [O···O = 2.92, 2.95 (1) Å].

Experimental. Yellow prisms (Neill & Raistrick, 1956), 0.10 × 0.40 × 0.10 mm, Rigaku AFC-6S diffractometer, monochromatized Cu $K\alpha$ radiation, lattice parameters from 25 reflections with $2\theta = 55$–95°. Intensities for $2\theta \leq 155°$, hkl: −30 to 29, 0 to 11, 0 to 24, ω–2θ scan, ω-scan width $(1.10 + 0.20\tan\theta)°$ at 32° min^{-1}, up to nine scans, stationary background counts (peak:background time = 2:1), three standard reflections showed no significant variation, Lp and absorption corrections (ψ scan, relative transmission factors 0.63–1.0). 4556 unique reflections, 2810 with $I \geq 3\sigma(I)$. Structure by direct methods, refined by full-matrix least-squares procedures, H1 from a difference map, other H atoms in calculated posi-

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* 2,3,6,7-Acetoxy-9-hydroxy-8-methoxy-4-methylperinaphthen-1-one; 2,3,6,7-acetoxy-9-hydroxy-8-methoxy-4-methylphenalen-1-one.

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