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Structure of 4-Amino-4′-methylbenzophenone

BY SU GENBO, GUO SHOUWU, PAN FENG AND HE YOUPING

Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian, People's Republic of China

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Abstract. $C_{14}H_{13}NO$, $M_r = 211.26$, orthorhombic, $P2_12_12_1$, $a = 8.331$ (4), $b = 24.066$ (8), $c = 5.663$ (2) Å, $V = 1135.4$ (8) Å^3, $Z = 4$, $D_x = 1.24$ g cm^{-3}, λ(Mo $K\alpha$) = 0.710690 Å, $\mu = 0.73$ cm^{-1}, $F(000) = 448$, $T = 296$ K, $R = 0.045$ for 834 observed unique reflections with $I \geq 3\sigma(I)$. The molecules are linked by N—H···O hydrogen bonds [N···O ($\frac{1}{2} - x$, $-y$, $\frac{1}{2} + z$) 2.89 (5) and NH···O 1.98 Å, ∠N—H···O 162°]. The dihedral angles between the plane of the carbonyl group and those of the amino- and methyl-substituted phenyl groups are 21.1 and 49.5° respectively.

Experimental. A light yellow transparent crystal, with dimensions 0.4 × 1.2 × 0.2 mm, was grown from alcohol solution and mounted in a random orientation on a glass fiber. Data were collected with a Rigaku AFC5R diffractometer (*CONTROL* software; Molecular Structure Corporation, 1986). Cell constants were obtained by least-squares analysis of 20 diffraction maxima ($5 < 2\theta < 45°$). $\omega/2\theta$ scans, scan speed varied between 2 and 8° min^{-1} (in ω) on the basis of *SEARCH* intensity, the scan width was $(1.523 + 0.35\tan\theta)°$, maximum 2θ was 50° ($0 \leq h \leq 10$, $0 \leq k \leq 29$, $0 \leq l \leq 7$). The intensities of 1217 unique reflections were measured. Three standard reflections were measured periodically, corrections were applied to the intensities to allow for the drop of 1.0% in the mean standard intensity during data collection. $I = C - \frac{1}{2}(t_c/t_b)(b_1 + b_2)$ where C = total number of counts, t_c = time spent counting peak intensity, t_b = time spent counting one side of the background, b_1 = high-angle background counts and b_2 = low-angle background counts; $\sigma(F^2) = [C + \frac{1}{4}(t_c/t_b)^2(b_1 + b_2) + (pI)^2]^{1/2}$ where p, the factor that downweights strong reflections, was taken to be 0.03. An empirical absorption correction, based on azimuthal scans of three reflections, was applied. *DIFABS* (Walker & Stuart, 1983) correction was also applied (transmission factor ranges from 0.7018 to 1.1887). The data were corrected for Lorentz and polarization factors. 834 reflections with $I \geq 3\sigma(I)$ were obtained and used in the refinement.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983), the C, N and O atoms being located from the E map. H atoms were placed in geometrically calculated positions with C—H and N—H 0.95 Å, but not included in the refinement. The structure was refined on F by the full-matrix least-squares technique with anisotropic thermal parameters for C, N and O atoms. Final $R = 0.045$, $wR = 0.063$ and $S = 1.573$, $w = 1/\sigma^2(F_o)$. $(\Delta/\sigma)_{max} = 0.0027$, 145 parameters; in the final difference electron density synthesis largest and smallest heights were 0.14 and -0.15 e Å^{-3}. Anomalous-dispersion corrections were not applied. All calculations were performed on a VAX-11/785 computer using the *TEXSAN* (Molecular Structure Corporation, 1987) program package, the scattering factors were taken from Cromer & Waber (1974). The views of the molecule and unit cell were produced by the *ORTEP*II program (Johnson, 1976), as shown in

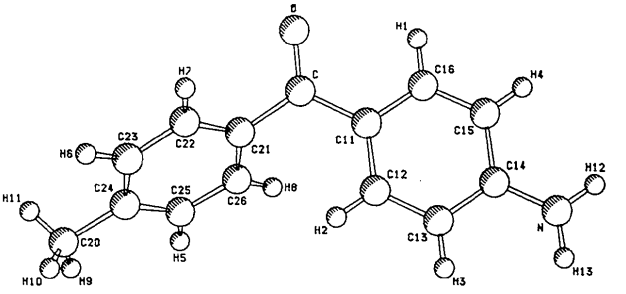

Fig. 1. The AMBP molecule.

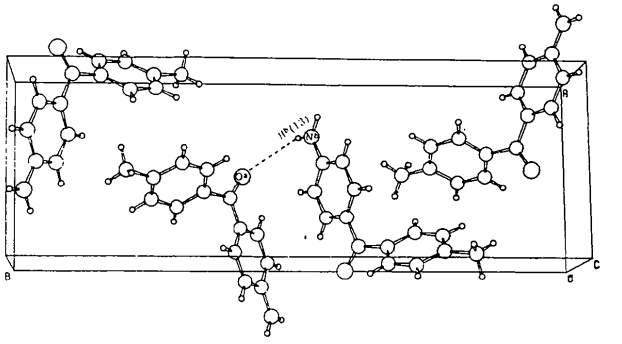

Fig. 2. Packing of AMBP molecules in the unit cell. The O···H—N hydrogen bond is indicated by a dashed line.

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33})$.

	x	y	z	B_{eq}
O	0.4784 (4)	0.0872 (1)	0.6692 (8)	5.4 (2)
N	1.2092 (5)	0.0344 (2)	0.9017 (9)	5.3 (2)
C	0.6000 (5)	0.1108 (2)	0.6025 (9)	3.7 (2)
C(11)	0.7606 (5)	0.0921 (2)	0.6801 (8)	3.4 (2)
C(12)	0.8966 (6)	0.1039 (2)	0.5519 (8)	3.9 (2)
C(13)	1.0466 (5)	0.0847 (2)	0.6199 (9)	3.8 (2)
C(14)	1.0618 (6)	0.0529 (2)	0.8277 (9)	3.7 (2)
C(15)	0.9223 (6)	0.0401 (2)	0.9560 (9)	4.1 (2)
C(16)	0.7762 (5)	0.0591 (2)	0.8816 (9)	3.7 (2)
C(20)	0.5165 (7)	0.2990 (2)	−0.0260 (1)	5.5 (3)
C(21)	0.5845 (5)	0.1588 (2)	0.4375 (8)	3.4 (2)
C(22)	0.4865 (6)	0.1548 (2)	0.2455 (1)	4.8 (3)
C(23)	0.4683 (7)	0.1992 (2)	0.0947 (1)	5.2 (3)
C(24)	0.5405 (6)	0.2502 (2)	0.1368 (9)	4.1 (2)
C(25)	0.6345 (6)	0.2538 (2)	0.3331 (1)	4.5 (2)
C(26)	0.6619 (6)	0.2088 (2)	0.4801 (9)	4.0 (2)

Figs. 1 and 2, respectively. The atomic parameters are listed in Table 1;* important bond lengths and bond angles are given in Table 2.

Related literature. 4-Aminobenzophenone (ABP) has a large second-harmonic generation efficiency (Frazier & Cockerham 1987) and also belongs to the monoclinic system with space group $P2_1$ (Su, Pan, Li, He & Huang, 1991). However, in ABP the carbonyl/aminophenyl dihedral angle of 6° is much smaller than the corresponding angle of 21.1° in 4-amino-4′-methylbenzophenone (AMBP) found here.

* 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 54516 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0212]

Table 2. *Intramolecular distances* (Å) *and angles* (°)

C—O	1.221 (5)	C(15)—C(16)	1.366 (6)
C—C(11)	1.479 (6)	C(21)—C(22)	1.363 (7)
C—C(21)	1.490 (6)	C(21)—C(26)	1.387 (6)
C(11)—C(12)	1.375 (6)	C(22)—C(23)	1.377 (7)
C(11)—C(16)	1.396 (6)	C(23)—C(24)	1.388 (7)
C(12)—C(13)	1.387 (6)	C(24)—C(20)	1.506 (7)
C(13)—C(14)	1.409 (7)	C(24)—C(25)	1.362 (6)
C(14)—N	1.372 (6)	C(25)—C(26)	1.384 (6)
C(14)—C(15)	1.405 (7)		

O—C—C(11)	121.1 (4)	C(14)—C(15)—C(16)	120.3 (4)
O—C—C(21)	118.8 (4)	C(15)—C(16)—C(11)	121.7 (4)
C(11)—C—C(21)	120.1 (4)	C(16)—C(11)—C(12)	118.2 (4)
C—C(11)—C(12)	121.7 (4)	C(21)—C(22)—C(23)	120.4 (5)
C—C(11)—C(16)	120.0 (4)	C(22)—C(23)—C(24)	122.2 (5)
C—C(21)—C(22)	119.8 (4)	C(23)—C(24)—C(20)	121.8 (5)
C—C(21)—C(26)	121.5 (4)	C(25)—C(24)—C(20)	121.8 (5)
C(11)—C(12)—C(13)	121.8 (4)	C(23)—C(24)—C(25)	116.4 (5)
C(12)—C(13)—C(14)	119.6 (4)	C(24)—C(25)—C(26)	122.5 (4)
N—C(14)—C(13)	120.8 (4)	C(25)—C(26)—C(21)	119.8 (4)
N—C(14)—C(15)	120.8 (4)	C(26)—C(21)—C(22)	118.6 (4)
C(13)—C(14)—C(15)	118.5 (4)		

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Structure of Hymenoratin

BY PAUL W. BAURES, KUAI-LIN SUN, JAMES A. CHAN AND DRAKE S. EGGLESTON*

Departments of Biomolecular Discovery and Physical and Structural Chemistry, SmithKline Beecham Pharmaceuticals, L-950, Box 1539, King of Prussia, PA 19406-0939, USA

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Abstract. Hymenoratin, (3aα,4aβ,5β,6α,7aα,8α,9aα)-decahydro-5,6-dihydroxy-4a,8-dimethyl-3-methyleneazuleno[6,5-*b*]furan-2(3*H*)-one, $C_{15}H_{22}O_4$, M_r = 266.34, monoclinic, $P2_1$, a = 13.525 (7), b = 7.549 (4), c = 13.646 (7) Å, β = 90.02 (4)°, V = 1393.2 (11) Å^3, Z = 4, D_x = 1.270 g cm^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, μ = 0.849 cm^{-1}, $F(000)$ = 576, T = 225 K, R = 0.046 for 2838 unique observed reflections, $I \geq 3\sigma(I)$. The sesquiterpene lactone has its seven-membered ring *cis* fused to the α-methylene-

* To whom all correspondence should be addressed.

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