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Structure of 5-Benzoyl-1-[4-(dimethylamino)phenylmethyleneamino]-4-phenyl-1*H*-pyrimidin-2-one

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Abstract. $C_{26}H_{22}N_4O_2$, $M_r = 422.485$, monoclinic, $P2_1/c$, $a = 9.564$ (2), $b = 11.305$ (1), $c = 20.389$ (3) Å, $\beta = 91.27$ (1)°, $V = 2203.95$ Å^3, $Z = 4$, $D_x = 1.273$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 6.3$ cm^{-1}, $F(000) = 888$, $T = 295$ K, $R = 0.050$ for 2816 observed reflections. The pyrimidine ring is significantly non-planar, the angles between the planes of atoms N1, C6, C5 and C2, N3, C4 (using standard numbering for pyrimidines) is 7.5°. In 1-amino-5-benzoyl-4-phenyl-1*H*-pyrimidine-2-thione, the corresponding angle is 0.74°.

Introduction. The pyrimidines are effective in antibacterial and insecticidal action (Cheng, 1969; McNair-Scott, Ulbricht, Rogers, Chu & Rose, 1959; Sankyo Co. Ltd & Ube Industries Ltd, 1984; Ziegler, Eder, Belegratis & Prewedorakis, 1967; Akçamur, Altural, Sarıpınar, Kollenz, Kappe, Peters, Peters & von Schnering, 1988; Özbey, Kendi, Akçamur, Yıldırım, Elerman & Soylu, 1991).

Experimental. An equimolar mixture of 4-benzoyl-5-phenyl-2,3-furandione and 4-(dimethylamino)benzaldehyde semicarbazone (2) was heated at 388 K for 15 min. The mixture was cooled to room temperature, and the product (3) extracted with ether (Sarıpınar, 1988). Yellow parallelepiped transparent

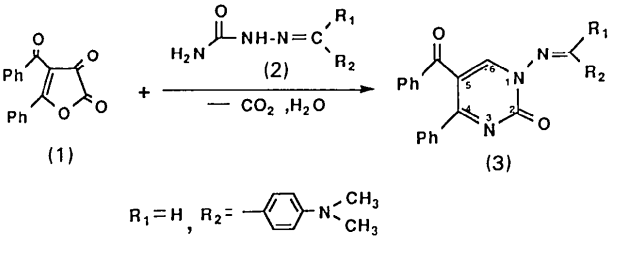

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Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$.

	x	y	z	B_{eq}
O2	0.1538 (4)	0.2592 (3)	0.0152 (2)	5.53 (8)
O7	0.5450 (3)	−0.1292 (3)	−0.0780 (2)	3.94 (6)
N1	0.1844 (3)	0.0583 (3)	0.0185 (2)	2.95 (6)
N3	0.2892 (4)	0.1761 (3)	−0.0633 (2)	3.64 (7)
N8	0.0978 (3)	0.0413 (3)	0.0735 (2)	3.47 (7)
N10	−0.1359 (4)	0.0790 (4)	0.3662 (2)	4.60 (9)
C2	0.2065 (5)	0.1721 (4)	−0.0093 (2)	3.65 (9)
C4	0.3533 (4)	0.0804 (4)	−0.0849 (2)	3.00 (8)
C5	0.3334 (4)	−0.0336 (3)	−0.0564 (2)	2.60 (7)
C6	0.2431 (4)	−0.0406 (4)	−0.0053 (2)	2.82 (8)
C7	0.4188 (4)	−0.1395 (4)	−0.0706 (2)	2.69 (7)
C9	0.1393 (4)	0.1022 (4)	0.1237 (2)	3.22 (8)
C11	−0.1075 (7)	0.1702 (6)	0.4171 (3)	7.0 (2)
C12	−0.2386 (6)	−0.0131 (6)	0.3799 (3)	5.9 (1)
C41	0.4447 (4)	0.0988 (4)	−0.1421 (2)	3.46 (9)
C42	0.5241 (6)	0.2015 (5)	−0.1463 (3)	5.3 (1)
C43	0.6133 (6)	0.2184 (5)	−0.1979 (3)	6.8 (1)
C44	0.6237 (6)	0.1337 (6)	−0.2462 (3)	6.8 (1)
C45	0.5446 (6)	0.0309 (5)	−0.2437 (2)	5.5 (1)
C46	0.4554 (5)	0.0134 (4)	−0.1919 (2)	4.2 (1)
C71	0.3514 (4)	−0.2574 (3)	−0.0735 (2)	2.56 (7)
C72	0.2094 (4)	−0.2715 (4)	−0.0887 (2)	3.33 (8)
C73	0.1531 (5)	−0.3847 (4)	−0.0935 (2)	4.6 (1)
C74	0.2367 (6)	−0.4828 (4)	−0.0828 (3)	5.0 (1)
C75	0.3774 (5)	−0.4687 (4)	−0.0675 (2)	4.6 (1)
C76	0.4348 (5)	−0.3571 (4)	−0.0629 (2)	3.80 (9)
C91	0.0663 (4)	0.0935 (4)	0.1853 (2)	2.97 (8)
C92	0.1067 (5)	0.1667 (4)	0.2371 (2)	4.1 (1)
C93	0.0417 (5)	0.1637 (4)	0.2966 (2)	4.4 (1)
C94	−0.0685 (4)	0.0837 (4)	0.3073 (2)	3.33 (8)
C95	−0.1064 (4)	0.0077 (4)	0.2554 (2)	3.72 (9)
C96	−0.0420 (4)	0.0135 (4)	0.1962 (2)	3.53 (9)

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

N1—C2	1.424 (5)	C71—C72	1.395 (5)
N1—C6	1.345 (6)	C72—C73	1.391 (6)
N1—N8	1.421 (4)	C73—C74	1.382 (7)
C2—O2	1.220 (5)	C74—C75	1.383 (8)
C2—N3	1.367 (5)	C75—C76	1.378 (6)
N3—C4	1.325 (5)	C76—C71	1.396 (6)
C4—C5	1.429 (6)	N8—C9	1.290 (5)
C4—C41	1.488 (6)	C9—C91	1.454 (6)
C41—C42	1.391 (7)	C91—C92	1.389 (6)
C42—C43	1.382 (8)	C92—C93	1.377 (6)
C43—C44	1.380 (9)	C93—C94	1.409 (6)
C44—C45	1.389 (9)	C94—C95	1.405 (6)
C45—C46	1.386 (7)	C95—C96	1.369 (7)
C46—C41	1.406 (6)	C96—C91	1.396 (6)
C5—C6	1.371 (5)	N10—C94	1.375 (5)
C5—C7	1.482 (5)	N10—C11	1.483 (7)
C7—O7	1.225 (4)	N10—C12	1.465 (8)
C7—C71	1.481 (5)		
C2—N1—C6	122.6 (3)	C42—C41—C46	118.7 (5)
C2—N1—N8	122.1 (3)	C41—C42—C43	120.6 (5)
C6—N1—N8	115.3 (3)	C42—C43—C44	120.2 (5)
N1—C2—O2	119.9 (4)	C43—C44—C45	120.4 (5)
N1—C2—N3	116.4 (4)	C44—C45—C46	119.5 (5)
O2—C2—N3	123.7 (5)	C41—C46—C45	120.6 (5)
O2—C2—N1	119.9 (4)	C7—C71—C72	122.2 (3)
C2—N3—C4	121.3 (4)	C7—C71—C76	118.3 (3)
N3—C4—C5	122.3 (3)	C72—C71—C76	119.5 (4)
N3—C4—C41	115.7 (4)	C71—C72—C73	119.7 (4)
C5—C4—C41	122.1 (4)	C72—C73—C74	120.4 (4)
C4—C5—C6	117.0 (3)	C73—C74—C75	120.0 (4)
C4—C5—C7	124.8 (3)	C74—C75—C76	120.3 (4)
C6—C5—C7	117.5 (3)	C75—C76—C71	120.2 (5)
N1—C6—C5	120.0 (4)	C9—C91—C96	123.7 (4)
C5—C7—O7	119.8 (4)	C9—C91—C92	119.1 (4)
C5—C7—C71	119.6 (4)	C9—C91—C96	123.7 (4)
O7—C7—C71	120.6 (3)	C92—C91—C96	117.4 (4)
N1—N8—C9	112.3 (3)	C91—C92—C93	122.1 (4)
N8—C9—C91	120.3 (4)	C92—C93—C94	120.4 (4)
C11—N10—C12	118.2 (5)	C93—C94—C95	117.2 (4)
C11—N10—C94	120.2 (5)	N10—C94—C93	121.7 (4)
C12—N10—C94	121.5 (4)	N10—C94—C95	121.2 (4)
C4—C41—C42	119.7 (4)	C94—C95—C96	121.4 (4)
C4—C41—C46	121.6 (4)	C95—C96—C91	121.4 (5)

crystals were recrystallized from *n*-butanol by slow evaporation at room temperature for a week; m.p. 477 K. Intensity data were collected for a crystal of *ca* 0.55 × 0.20 × 0.10 mm, mounted in a glass capillary on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Unit-cell parameters were obtained from 25 reflections with $17 \le \theta \le 24°$. Intensity data were collected in ω/θ scan mode with $6 \le \theta \le 65°$; $0 \le h \le 9$, $0 \le k \le 14$, $0 \le l \le 14$. Two standard reflections (054 and 344), measured every hour, showed no intensity variation. $R_{int} = 0.0135$. A total of 4217 reflections with $I \ge 3\sigma(I)$ were considered observed. Data were corrected for Lorentz–polarization effects, but not for absorption. The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1986) and refined by full-matrix least squares on F (*SHELX*76; Sheldrick, 1976) including anisotropic thermal parameters for non-H atoms. H atoms were geometrically positioned 1.0 Å from C atoms with $B = 0.05$ Å^2. 290 parameters were refined. $R = wR = 0.050$ ($w = 1/\sigma_F^2$). In the last cycle, $(\Delta/\sigma)_{max} = 0.001$. Maximum residual density was 0.17, minimum −0.14 e Å^{-3}. $S = 1.026$. Scattering factors for neutral atoms and f' and f'' were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computer programs used: Enraf–Nonius *SDP* package (B. A. Frenz & Associates, Inc., 1985), *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1986); *SCHAKAL* (Keller, 1988). Computer used: DEC MicroVAX 3500.

Discussion. Atomic positions and equivalent isotropic temperature factors for non-H atoms are listed in Table 1.* Bond lengths and angles are given in Table 2. The structure of the title compound is shown in Fig. 1. The formula suggested on the basis of NMR and IR spectroscopy is confirmed. It is interesting to note the relative orientations of the substituents attached to the pyrimidine ring. In the pyrimidine ring, the C2=O2 double-bond length of 1.220 (5) Å, and in the benzoyl group, the C7=O7 double-bond length of 1.225 (4) Å are almost the same as that found for the C=O bond [1.222 (3) Å] in 1-amino-5-benzoyl-4-phenyl-1*H*-pyrimidine-2-thione (Akkurt, Güldeste, Soylu, Altural & Sarı-

* Lists of anisotropic thermal parameters, positional and thermal parameters for the H atoms, bond distances and angles involving the H atoms and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55754 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1001]

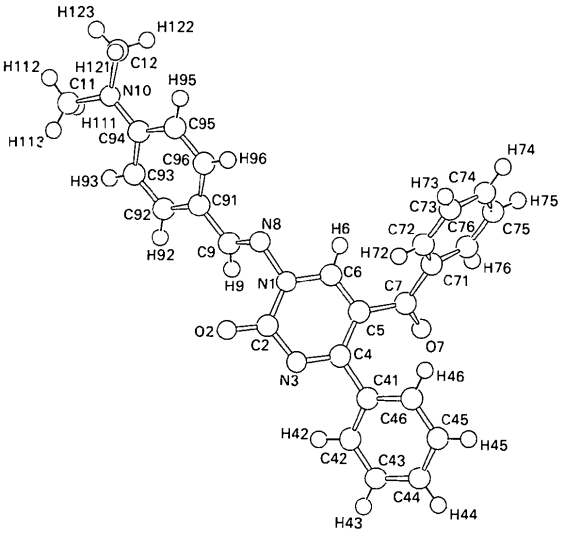

Fig. 1. A *SCHAKAL* (Keller, 1988) drawing of the title molecule with the atom-numbering scheme.

pınar, 1992). The N3—C2 and N1—C6 bond distances are respectively 1.367 (5) and 1.345 (6) Å, which are exceptional compared with the above compound [1.357 (3) and 1.332 (3) Å] and with 5-benzoyl-1-methyl-4-phenylpyrimidine-2-thione [1.385 (4) and 1.316 (4) Å (Özbey *et al.*, 1991)]. In the pyrimidine ring, the angle between the planes N1,C6,C5 and C2,N3,C4 is 7.5°; this angle appears sensitive to the type of substituents present on N(1) since in 5-benzoyl-1-(diphenylmethyleneamino)-4-phenyl-1*H*-pyrimidine-2-one it is 5.3° (Akçamur *et al.*, 1988) and in 1-amino-5-benzoyl-4-phenyl-1*H*-pyrimidine-2-thione it is 0.74° (Akkurt *et al.*, 1992).

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SHORT-FORMAT PAPERS

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Acta Cryst. (1993). C**49**, 749–751

Structures of Trimethyloxosulfonium Salts. VII. The Dichromate: $[(CH_3)_3SO]_2Cr_2O_7$

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Abstract. Trimethyloxosulfonium dichromate, $C_3H_9OS^+.Cr_2O_7^-$, $M_r = 402.32$, monoclinic, $P2_1$, $a = 8.917$ (2), $b = 10.319$ (2), $c = 8.418$ (1) Å, $\beta = 102.30$ (5)°, $V = 756.8$ (6) Å^3, $Z = 2$, $D_m = 1.75$ (3), $D_x = 1.765$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.698$ mm^{-1}, $F(000) = 412$, $T = 293$ K, final $R =$

0108-2701/93/040749-03$06.00