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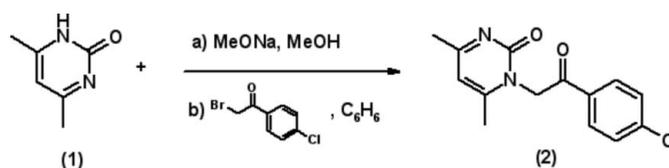
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.036
 wR factor = 0.086
Data-to-parameter ratio = 15.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1-[(4-Chlorobenzoyl)methyl]-4,6-dimethyl-2(1H)-pyrimidone

In the title molecule, $\text{C}_{14}\text{H}_{13}\text{ClN}_2\text{O}_2$, all bond lengths and angles show normal values. The mean planes of the heterocyclic ring and the carbonyl group make a dihedral angle of $81.38(5)^\circ$.Received 3 July 2006
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Comment

We have previously described the crystal structures of a series of *N*-phenacyl-2-pyridones (Albov *et al.*, 2004*a,b*, 2005) and the structure of *N*-phenacyl-2-pyrimidone (Rybakov *et al.*, 2006). In this communication, we report the synthesis and crystal structure of a homologue of these compounds, the title compound, (2), *N*-phenacyl-4,6-dimethyl-2-pyrimidone.

Interestingly, in the study of the phenacylation of sterically hindered 4,6-dimethylpyrimidone, only the *N*-isomer has been isolated, in poor yield (Ivanov & Reznik, 1983; Buchan *et al.*, 1978). We have found that, in the reaction of 4,6-dimethyl-2-pyrimidone, (1), with phenacyl bromide in the presence of K_2CO_3 , a mixture of two products is formed, namely *N*-phenacylpyrimidone (*N*-isomer) and *O*-phenacylpyrimidone (*O*-isomer). With the goal of decreasing the yield of the *O*-isomer, we have used the sodium salt in the reaction with *p*-chlorophenacyl bromide. The only product observed in this reaction was the title compound, (2).

In compound (2) (Fig. 1), all bond lengths and angles show normal values (Cambridge Structural Database; Version 5.27; Allen, 2002). In the pyrimidone (P) ring, N1/C2/N3/C4–C6, the single and double bonds alternate (Table 1), allowing some degree of conjugation. The mean planes of P and the carbonyl group C7/C8/O8/C9 make a dihedral angle of $81.38(5)^\circ$. The torsion angle O8–C8–C9–C14 = $4.1(3)^\circ$ indicates conjugation with the benzoyl fragment.

Experimental

4,6-Dimethyl-2-pyrimidone hydrochloride (5 g, 0.031 mol) and NaOH (1.25 g 0.031 mol) were dissolved in water (30 ml), stirred for 5 min and evaporated *in vacuo*. The residue was dissolved in CHCl_3 (50 ml), refluxed for 5 min and filtered. The filtrate was evaporated *in vacuo* to give the dimethylpyrimidone as the free base (3.8 g, 100%). The resulting 4,6-dimethyl-2-pyrimidone was added to a solution of sodium methylate (prepared by dissolving 0.031 mol sodium in 15 ml

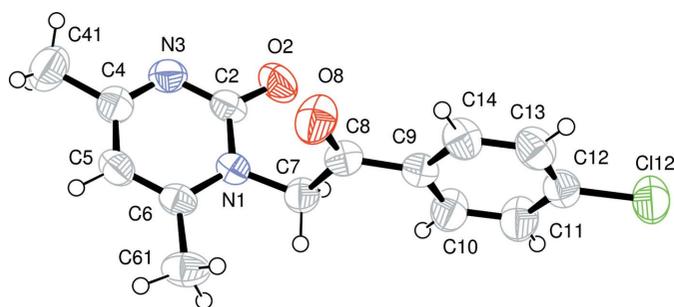


Figure 1
The structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

of anhydrous methanol), and the mixture was stirred for 30 min. The resulting precipitate of the sodium salt of dimethylpyrimidone was filtered off (4.40 g, 98%). This sodium salt of 2-pyrimidone (1.5 g, 0.01 mol) was suspended in benzene (10 ml). *p*-Chlorophenacyl bromide was then added (1.9 g, 0.0067 mol) and the mixture was stirred for 6 d at room temperature. The precipitate which formed was filtered off, and washed with water and then with diethyl ether. The product was isolated by suction and recrystallized from acetonitrile (yield 33%, m.p. 445–447 K).

Crystal data

$C_{14}H_{13}ClN_2O_2$
 $M_r = 276.71$
Monoclinic, $P2_1/n$
 $a = 7.1975$ (8) Å
 $b = 9.3817$ (10) Å
 $c = 19.422$ (3) Å
 $\beta = 92.314$ (9)°
 $V = 1310.4$ (3) Å³

$Z = 4$
 $D_x = 1.403$ Mg m⁻³
Cu $K\alpha$ radiation
 $\mu = 2.58$ mm⁻¹
 $T = 293$ (2) K
Prism, colourless
 $0.2 \times 0.2 \times 0.2$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer
 ω scans
Absorption correction: none
2760 measured reflections
2683 independent reflections

1907 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.090$
 $\theta_{max} = 74.9^\circ$
1 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.086$
 $S = 0.96$
2683 reflections
174 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0422P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.003$
 $\Delta\rho_{max} = 0.12$ e Å⁻³
 $\Delta\rho_{min} = -0.14$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N1–C6	1.3607 (18)	N3–C4	1.307 (2)
N1–C2	1.4116 (19)	C4–C5	1.402 (2)
N1–C7	1.4648 (16)	C4–C41	1.502 (2)
C2–O2	1.2210 (18)	C5–C6	1.3586 (19)
C2–N3	1.3645 (18)	C6–C61	1.493 (2)
C6–N1–C2	122.06 (12)	C4–N3–C2	119.31 (15)
C6–N1–C7	123.33 (13)	N3–C4–C5	123.56 (14)
C2–N1–C7	114.45 (12)	C6–C5–C4	118.71 (15)
O2–C2–N3	123.27 (16)	C5–C6–N1	118.13 (15)
O2–C2–N1	118.50 (14)	N1–C7–C8	110.70 (12)
N3–C2–N1	118.23 (14)		

All H atoms were positioned geometrically ($C-H = 0.93-0.97$ Å) and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ ($1.5U_{eq}$ for CH_3) of the parent atom.

Data collection: *CAD-4 EXPRESS*; cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, (1995)); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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