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# 1-(4-Chlorobenzoylmethyl)pyrimidin-2(1*H*)-one

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**Key indicators**

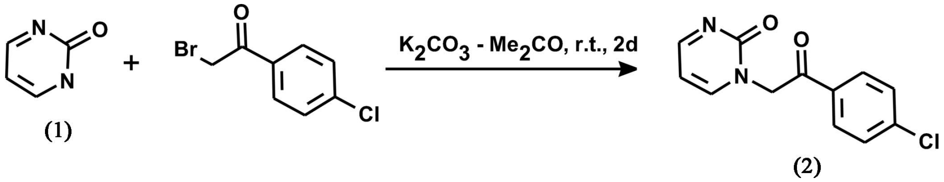
Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.054
*wR* factor = 0.161
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{12}H_9ClN_2O_2$, was synthesized and characterized by $^1$H NMR and IR spectrocopy and X-ray diffraction techniques. The chlorobenzoyl fragment in the structure is almost flat, whereas the carbonyl group is almost perpendicular to the pyrimidine ring plane.

## Comment

We have previously described the crystal structures of a series of *N*-phenacyl-2-pyridones (Albov *et al.*, 2004*a*,*b*, 2005). We report here the synthesis and crystal structure of their aza-analog, (2). In contrast to well investigated reactions of pyridones with α-halogenoketones, analogous reactions for pyrimidones have been little studied. There are only a few examples, namely the reaction of (1) with the acetal of α-bromoacetaldehyde (Holy *et al.*, 1984) or with chloroacetic acid derivatives (Gefenas & Vainilavichus, 2003) and phenacylation of the 5-chloro derivative of (1) (Benneche & Gandersen, 1988). In all of these cases, the products of *N*-alkylation were exclusively observed. Interestingly, in the study of the phenacylation of sterically hindered 4,6-dimethylpyrimidone, only the *N*-isomers have been isolated (Ivanov *et al.*, 1983; Buchan *et al.*, 1978). Usually these reactions are performed in aprotic bipolar solvents in the presense of bases, so that the alkylated species has been the pyrimidone anion.



We have found that reaction of (1) with *p*-chlorophenacyl bromide in the presense of $K_2CO_3$ in acetone yields the title compound, (2). The $^1$H NMR spectra were useless for distinguishing between *O*- and *N*-isomers. In the IR spectrum of (2), two C=O peaks were observed, one for the –N–C=O fragment of pyrimidone and another for the carbonyl group, thus clearly confirming selective *N*-alkylation (in the case of *O*-phenacylation no amide peak would be observed). The structure of the *N*-isomer was finally confirmed by X-ray crystallographic analysis.

In the pyrimidone ring of (2), the single and double bonds alternate (Table 1), though allowing some degree of conjugation. In the Cambridge Structural Database (CSD; Version 5.27; Allen, 2002), we found only one structure with the pyrimidone (2) fragment, namely the parent pyrimidone (1) (Furberg & Solbakk, 1970) with the same distribution of bond lengths. In the structure of (2), the dihedral angle between the

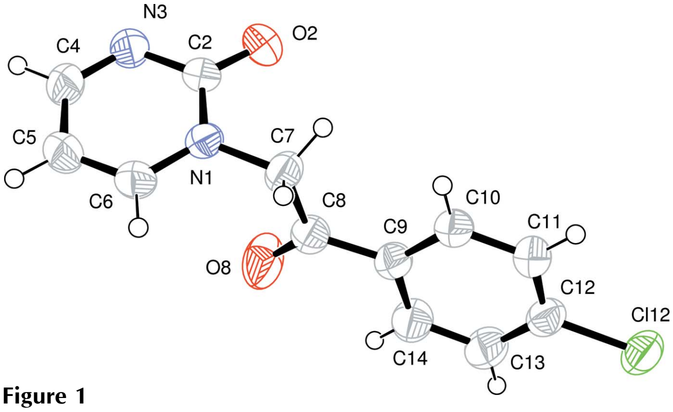


**Figure 1**
The structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, with H atoms shown as spheres of arbitrary radius.

pyrimidone ring and the C7/C8/O8/C9 unit is 81.57 (8)°. The O8—C8—C9—C14 torsion angle of −4.2 (2)° indicates conjugation in the benzoyl fragment.

## Experimental

The hydrochloride of (1) (26.5 g; 0.2 mol) was suspended in 500 ml of dry acetone. A solution of 0.1 mol of *p*-chlorophenacyl bromide in 100 ml acetone and 0.4 mol $K_2CO_3$ was added with stirring. The reaction mixture was stirred for 2 d at room temperature, and then the solvent was evaporated under vacuum. The residue was washed with water and then with ethyl acetate The product was isolated by suction and recrystallized from acetonitrile. (yield 95%, m.p. 497–499 K). $^1$H NMR (DMSO-$d_6$/TMS): $\delta$ $J$(Hz) 8.57 (*m*, 1H, H-6), 8.02–8.16 (*m*, 3H, 2H of *p*-ClPh, H-4), 7.57 (*m*, 2H, *p*-ClPh), 6.42 (*m*, 1H, H-5), 5.44 (*s*, 2H, $NCH_2$). IR (nujol) $\nu_{CO}$, cm$^{-1}$ 1695, 1615. Analysis calculated for $C_{12}H_9ClN_2O_2$: C 57.96, H 3.65, N 11.27%; found: C 57.67, H 3.41, N 11.29%.

### *Crystal data*

$C_{12}H_9ClN_2O_2$
$M_r$ = 248.66
Monoclinic, $P2_1/n$
$a$ = 10.2191 (10) Å
$b$ = 10.8421 (14) Å
$c$ = 11.3449 (15) Å
$\beta$ = 115.065 (11)°
$V$ = 1138.6 (3) Å$^3$
$Z$ = 4
$D_x$ = 1.451 Mg m$^{-3}$
Cu $K\alpha$ radiation
$\mu$ = 2.91 mm$^{-1}$
$T$ = 293 (2) K
Prism, colourless
0.2 × 0.2 × 0.2 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$ scans
Absorption correction: none
2327 measured reflections
2327 independent reflections
1842 reflections with $I > 2\sigma(I)$
$\theta_{max}$ = 77.9°
1 standard reflection frequency: 120 min intensity decay: 5%

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.161
$S$ = 1.14
2327 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0895P)^2 + 0.2167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.71 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.79 e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0192 (19)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| N1—C6 | 1.353 (3) | C8—O8 | 1.213 (3) |
| N1—C2 | 1.399 (3) | C8—C9 | 1.477 (3) |
| N1—C7 | 1.455 (3) | C9—C10 | 1.395 (3) |
| C2—O2 | 1.226 (3) | C9—C14 | 1.396 (3) |
| C2—N3 | 1.364 (3) | C10—C11 | 1.382 (3) |
| N3—C4 | 1.307 (3) | C11—C12 | 1.379 (3) |
| C4—C5 | 1.397 (3) | C12—C13 | 1.384 (4) |
| C5—C6 | 1.341 (4) | C12—Cl12 | 1.742 (2) |
| C7—C8 | 1.514 (3) | C13—C14 | 1.371 (4) |
| | | | |
| C6—N1—C2 | 121.49 (19) | O8—C8—C7 | 119.5 (2) |
| C6—N1—C7 | 120.52 (19) | C9—C8—C7 | 118.47 (19) |
| C2—N1—C7 | 117.84 (19) | C10—C9—C14 | 118.8 (2) |
| O2—C2—N3 | 122.8 (2) | C10—C9—C8 | 122.9 (2) |
| O2—C2—N1 | 119.7 (2) | C14—C9—C8 | 118.3 (2) |
| N3—C2—N1 | 117.53 (19) | C11—C10—C9 | 120.3 (2) |
| C4—N3—C2 | 119.42 (19) | C12—C11—C10 | 119.4 (2) |
| N3—C4—C5 | 124.3 (2) | C11—C12—C13 | 121.4 (2) |
| C6—C5—C4 | 116.6 (2) | C11—C12—Cl12 | 119.3 (2) |
| C5—C6—N1 | 120.5 (2) | C13—C12—Cl12 | 119.22 (19) |
| N1—C7—C8 | 111.92 (19) | C14—C13—C12 | 118.8 (2) |
| O8—C8—C9 | 122.0 (2) | C13—C14—C9 | 121.3 (2) |

All H atoms were refined using a riding model, with C—H = 0.93 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for aromatic H atoms C—H = 0.97 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for $CH_2$ H atoms, and C—H = 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for $CH_3$ H atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; 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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