Crystal data

$C_{20}H_{22}O_8$
M_r = 390.38
Monoclinic
$C2/c$
a = 21.644 (9) Å
b = 5.398 (2) Å
c = 16.765 (5) Å
β = 101.97 (2)°
V = 1916.1 (12) Å^3
Z = 4
D_x = 1.353 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 28 reflections
θ = 3.80–10.75°
μ = 0.105 mm^{-1}
T = 200 (2) K
Plate
0.80 × 0.30 × 0.08 mm
Yellow

Data collection

Siemens $P4$ diffractometer
Profile data from ω scans
Absorption correction: none
2265 measured reflections
1693 independent reflections
923 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

θ_{max} = 25°
$h = -1 \rightarrow 25$
$k = -1 \rightarrow 6$
$l = -19 \rightarrow 19$
3 standard reflections
every 100 reflections
intensity decay: 2.42%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.191
S = 1.026
1693 reflections
129 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0846P)^2 + 0.9902P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.035
$\Delta\rho_{max}$ = 0.317 e Å^{-3}
$\Delta\rho_{min}$ = −0.201 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C4	1.364 (5)	C2—C3	1.406 (6)
O1—C1	1.385 (4)	C3—C4	1.363 (5)
O2—C10	1.218 (4)	C4—C5	1.479 (5)
O3—C8	1.339 (5)	C5—C6	1.507 (6)
O3—C6	1.459 (5)	C6—C7	1.501 (6)
O4—C8	1.196 (5)	C8—C9	1.477 (6)
C1—C2	1.351 (5)	C10—C10^i	1.552 (7)
C1—C10	1.445 (5)		
C3—C4—C5	134.0 (4)	O2—C10—C1	122.5 (3)
O1—C4—C5	116.9 (3)	O2—C10—C10^i	120.8 (5)

Symmetry code: (i) $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$.

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9C···O4^{ii}	0.96	2.64	3.395 (7)	136

Symmetry codes: (i) $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$; (ii) $x, y - 1, z$.

H atoms are geometrically positioned and refined using a riding model (secondary CH_2 and aromatic CH groups) and additional rotating-group refinement (CH_3 groups) by maximizing the sum of the electron density at the three calculated H-atom positions.

Data collection: *XSCANS* (Siemens, 1996). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST95* (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1337). Services for accessing these data are described at the back of the journal.

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4-Benzoyl-2-chloro-6-phenylpyrimidine

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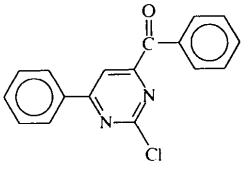
Abstract

In the title compound, $C_{17}H_{11}ClN_2O$, the molecule contains an essentially planar pyrimidine ring with the 4- and 6-positions substituted by benzoyl and phenyl groups, respectively. The phenyl ring and the planar part of the benzoyl moiety form dihedral angles of 12.3 (1) and 62.1 (1)°, respectively, with the pyrimidine ring. The two phenyl rings are inclined at an angle of 130.0 (1)° with respect to one another.

Comment

The pyrimidines form a class of biologically active organic compounds important for their possible pharma-

cological applications (Brown, 1985). Several 6-substituted pyrimidines and related uracil derivatives display specific inhibitory effects on the HIV-1 virus (Massa *et al.*, 1995; Miyasaka *et al.*, 1989) and significant cytotoxicity against CCRF–CEM human lymphoblastoid cell (Das *et al.*, 1996). As part of our ongoing studies on the synthesis and characterization of new nitrogen-containing heterocyclic systems of biological importance (Khan & Kundu, 1999*a*,*b*), the structure determination of 4-benzoyl-2-chloro-6-phenylpyrimidine, (I), was undertaken.



(I)

A view of molecule (I) with the atom-numbering and ring-labelling schemes is given in Fig 1. The molecules of (I) consist of an essentially planar pyrimidine ring *A* (r.m.s. deviation 0.007 Å), with the 2-, 4- and 6-positions substituted by chloro, benzoyl and phenyl groups, respectively. The N—C bond lengths in the pyrimidine ring [1.310 (4)–1.349 (4) Å] and the Cl—C distance [1.750 (3) Å] are comparable with those reported for similar structures (Song *et al.*, 1998; Lai *et al.*, 1997; Larson *et al.*, 1989). The chloro substitution at the 2-position of the pyrimidine ring introduces a considerable in-plane distortion, which is reflected in the widening of the N1—C11—N2 bond angle to 130.1 (3)° compared with the other ring angles (Table 1). The conformation of the molecule is described by the torsion angles C6—C7—C8—C9 and C9—C10—C12—C17 of 157.3 (3) and −167.6 (3)°, respectively, with the planar part of the benzoyl moiety twisted out of the plane of the pyrimidine ring by 62.1 (1)°; the two phenyl rings (ring *B* consists of atoms C12–C17 and ring *C* of atoms C1–C6) are inclined at an angle of 130.0 (1)° with respect to one another. The 12.3 (1)° angle between the least-squares planes of the pyrimidine and phenyl-*B* rings could allow substantial conjugation, yet the degree to which electrons are attracted by the pyrimidine ring from phenyl ring *B* seems very limited. The C10—C12 bond length of 1.488 (4) Å is slightly longer than the average Csp^2—C_{ar} bond length of 1.476 (14) Å in conjugated C_{ar}—C=N—C systems (Allen *et al.*, 1987).

The crystal packing is essentially stabilized by van der Waals interactions in order to optimize the intermolecular contacts between the phenyl groups.

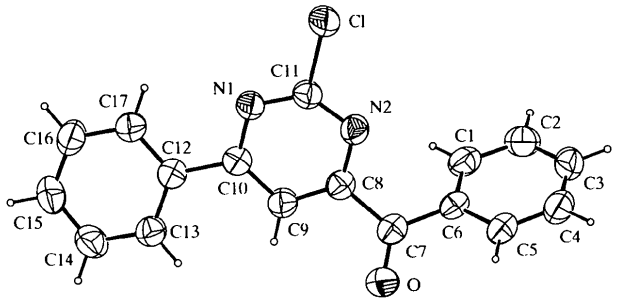


Fig. 1. *ZORTEP* view (Zsolnai, 1995; 50% probability level) of (I).

Experimental

Compound (I) [m.p. 402 (1) K] was synthesized by a Friedel–Crafts reaction. 2,6-Dichloropyrimidine-4-carbonyl chloride (1 mmol) was added dropwise to a mixture of ice-cold benzene (10 ml) and anhydrous aluminium chloride (3 mmol). The resulting mixture was stirred for 2 h at 298 K followed by usual work-up and purification by chromatography (silica-gel 60–120 mesh). Single crystals suitable for X-ray analysis were obtained from CCl_4.

Crystal data

$C_{17}H_{11}ClN_2O$
M_r = 294.73
Triclinic
$P\bar{1}$
a = 10.336 (4) Å
b = 11.650 (4) Å
c = 5.846 (3) Å
α = 97.45 (4)°
β = 96.11 (4)°
γ = 83.50 (3)°
V = 690.5 (5) Å^3
Z = 2
D_x = 1.418 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71071 Å
Cell parameters from 23 reflections
θ = 10.1–17.1°
μ = 0.276 mm^{-1}
T = 296.2 K
Prism
0.50 × 0.50 × 0.25 mm
Yellow

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: empirical (North *et al.*, 1968)
T_{min} = 0.897, T_{max} = 0.933
2129 measured reflections
2076 independent reflections
1633 reflections with $I > 2\sigma(I)$
R_{int} = 0.098
θ_{max} = 25.04°
$h = 0 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -6 \rightarrow 6$
3 standard reflections every 150 reflections
intensity decay: <1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.123
S = 1.109
2076 reflections
190 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.0701P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.220 e Å^{-3}
$\Delta\rho_{min}$ = −0.228 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C11	1.310 (4)	N2—C8	1.345 (4)
N1—C10	1.349 (4)	Cl—C11	1.750 (3)
N2—C11	1.325 (4)		
C11—N1—C10	115.7 (2)	C6—C7—C8	118.3 (3)
C11—N2—C8	113.6 (2)	N2—C8—C9	122.6 (3)
O—C7—C6	122.7 (3)	N1—C10—C9	120.0 (3)
O—C7—C8	119.0 (3)	N1—C11—N2	130.1 (3)
C1—C6—C7—O	135.7 (3)	O—C7—C8—N2	153.3 (3)
C1—C6—C7—C8	−45.8 (4)	C6—C7—C8—C9	157.3 (3)
C5—C6—C7—C8	135.8 (3)	C9—C10—C12—C17	−167.6 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1294). Services for accessing these data are described at the back of the journal.

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A cyclam-like macrocycle side-bridged by a propyl chain

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Abstract

The preparation and crystal structure of *meso*-5,5,7,12,12,14-hexamethyl-1,8-diazonia-4,11-diazabicyclo-[9.3.3]heptadecane chloride iodide 0.5-diethyl ether solvate, $C_{19}H_{42}N_4^{2+}\cdot Cl^-\cdot I^-\cdot 0.5C_4H_{10}O$, are described. The 14-membered macrocycle has an extended conformation with the $-(CH_2)_3-$ bridge to one side. It contains two N—H···Cl hydrogen bonds.

Comment

The compound *meso*-5,5,7,12,12,14-hexamethyl-1,8-diazonia-4,11-diazabicyclo[9.3.3]heptadecane chloride iodide 0.5-diethyl ether solvate, (1), was synthesized unexpectedly during our study of the transition metal complexes of cross-bridged tetraazamacrocycles (Hubin *et al.*, 1998). Generally, ethylene bridges across non-adjacent N atoms of tetraazamacrocycles are obtained by the condensation of NH with glyoxal, selective alkylation at two non-adjacent N atoms, and finally, ring-opening reduction of the diquat intermediate (Weisman *et al.*, 1996). However, substituted macrocycles, like *meso*-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane, (2), are more difficult to bridge by this route because of the added steric bulk of the six C-methyl groups (Hubin *et al.*, 1999). We have explored another route to the cross-bridged analogue of (2) based on the successful *trans*-dialkylation of (2), in which the geminal dimethyl groups apparently sterically protect two of the four secondary amines of the macrocycle from reaction (Hay *et al.*, 1996). We chose 1,3-diiodopropane as the bis-electrophile in our bridging reaction because 1,2-dihaloethanes have been shown to preferentially bridge adjacent N atoms of tetraazamacrocycles giving the stable piperazine derivatives, which we wanted to avoid (Wainwright, 1980; Ramasubbu & Wainwright, 1982). However, rather than the expected cross-bridged product, we isolated (1), in which the trimethylene group has bridged adjacent N atoms N1 and N11, giving an eight-membered ring as part of the bicyclic product. The structure determination also revealed one H atom on N8,