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Ethyl 2-amino-4-(4-chlorophenyl)-6-(4-methoxyphenyl)pyridine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.115
Data-to-parameter ratio = 16.7

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

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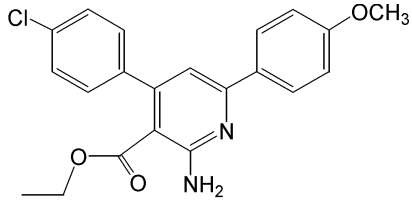
The title compound, $C_{21}H_{19}ClN_2O_3$, was synthesized by the reaction of 4-chlorobenzaldehyde, 4-methoxyacetophenone, ethyl cyanoacetate and ammonium acetate under microwave irradiation. X-ray analysis reveals that the benzene rings are orthogonal to each other and form dihedral angles of 29.34 (6) and 79.45 (6)° with the pyridine ring. In the solid state, the molecules exist as centrosymmetric N—H···O hydrogen-bonded dimers.

Comment

Small organic molecules such as nitrogen-containing heterocyclic compounds have received a large amount of attention in the literature, not only for their theoretical interest but also because they exhibit a variety of biological activity. The pyridine ring system represents an important class of compounds because of its biological activities (Temple *et al.*, 1992; Kambe *et al.*, 1980; Shintani *et al.*, 2003). Recently, we have synthesized some pyridine derivatives under microwave irradiation, without any solvent. Now, we report the crystal structure of the title compound, (I).



(I)

In (I), the pyridine ring is essentially planar, with a maximum deviation of 0.011 (1) Å for atom C4. The 4-methoxyphenyl ring [*A* (C15–C20)] and 4-chlorophenyl [*B* (C9–C14)] ring are perpendicular to one another [dihedral angle 89.73 (6)°]. The pyridine ring forms dihedral angles of 29.34 (6) and 79.45 (6)°, respectively, with rings *A* and *B*. The ethoxycarbonyl moiety is planar, with an r.m.s deviation of 0.034 Å, and is nearly coplanar with the pyridine ring [dihedral angle 4.6 (1)°]. An intramolecular N2—H2*B*···O1 hydrogen bond is observed in the molecular structure. In the crystal structure, the molecules form centrosymmetric N—H···O hydrogen-bonded dimers (Table 1 and Fig. 2).

Experimental

Compound (I) was prepared by the reaction of 4-chlorobenzaldehyde (2 mmol), 4-methoxyacetophenone (2 mmol) ethyl cyanoacetate (2 mmol) and ammonium acetate (2 mmol) under microwave irradiation (yield 43%; m.p. 442–443 K). Single crystals of (I) suitable for

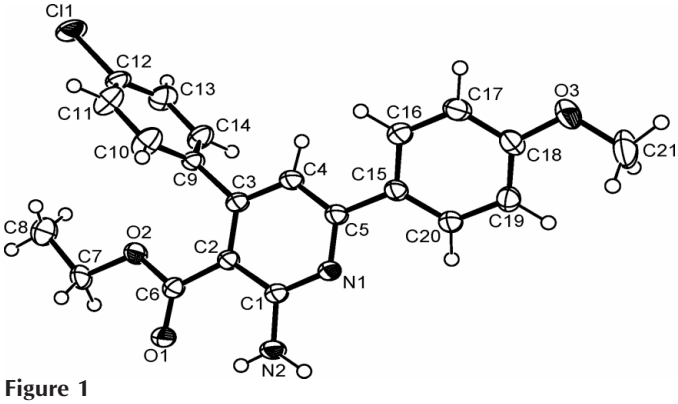


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{21}H_{19}ClN_2O_3$	$Z = 2$
$M_r = 382.83$	$D_x = 1.341$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.5398$ (14) Å	Cell parameters from 4130 reflections
$b = 10.1117$ (13) Å	$\theta = 3.1$–$27.5°$
$c = 12.729$ (2) Å	$\mu = 0.23$ mm^{-1}
$\alpha = 72.153$ (14)°	$T = 193$ (2) K
$\beta = 81.338$ (16)°	Block, colourless
$\gamma = 65.053$ (13)°	0.70 × 0.41 × 0.34 mm
$V = 948.3$ (3) Å^3	

Data collection

Rigaku Mercury diffractometer	3813 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.024$
Absorption correction: multi-scan (Jacobson, 1998)	$\theta_{max} = 27.5°$
$T_{min} = 0.858$, $T_{max} = 0.927$	$h = -11 \rightarrow 9$
10603 measured reflections	$k = -13 \rightarrow 13$
4254 independent reflections	$l = -16 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.3701P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.115$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.28$ e Å^{-3}
4254 reflections	
254 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1^{i}	0.88 (2)	2.26 (2)	3.1288 (19)	169 (2)
N2—H2B···O1	0.85 (2)	1.97 (2)	2.6529 (19)	136 (2)
C4—H4···Cl1^{ii}	0.95	2.77	3.7125 (18)	175

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

H atoms of the amino group were located in a difference Fourier map and were refined isotropically [N—H = 0.85 (2) and 0.88 (2) Å].

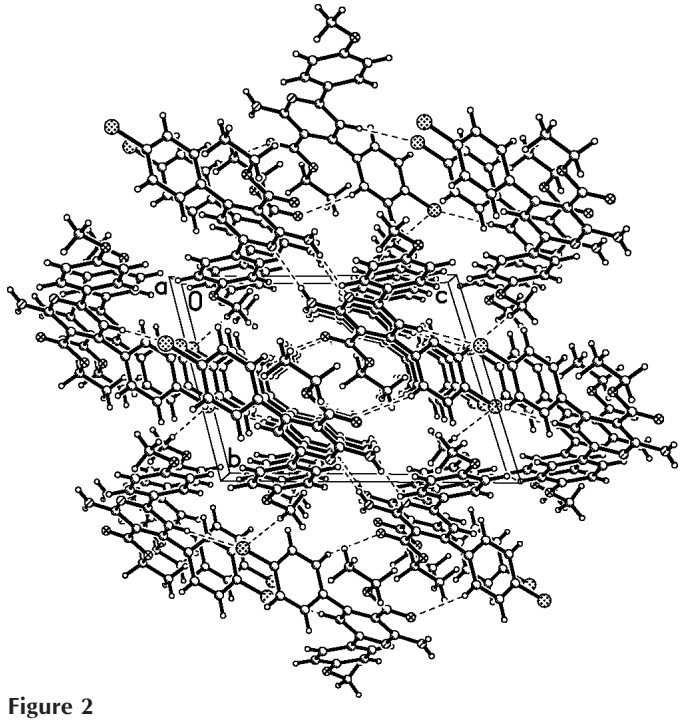


Figure 2
Molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

All other H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.95–0.99 Å, and U_{iso} (H) = 1.5U_{eq}(C) for the methyl H atoms and 1.2 U_{eq}(C) for the aromatic and methylene H atoms.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000–2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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