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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.041

wR factor = 0.116

Data-to-parameter ratio = 12.2

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

Methyl 5-nitro-2-(phenylamino)pyridine-4-carboxylate

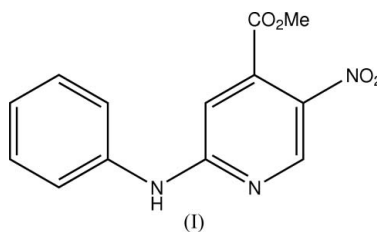
In the crystal structure of the title compound, $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_4$, there are two independent molecules in the asymmetric unit. In each case, the methylcarboxylate group is twisted by about 90° relative to the rest of the molecule. The molecules are packed in the crystal structure through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Many pharmaceutical products consist of oxygen and/or nitrogen heterocycles, and the title compound, (I), is the result of a study to produce bis-heterocyclic compounds from pyridyl isocyanates. It crystallizes in the space group $P\bar{1}$ with two molecules in the asymmetric unit (Fig. 1). The phenyl and pyridine rings make a dihedral angle of $11.9(2)^\circ$ in one molecule and $11.9(3)^\circ$ in the other. In both molecules, the nitro group is almost coplanar with the pyridine ring, while the methylcarboxylate group is approximately perpendicular to this ring, due to steric hindrance; this is demonstrated by the selected torsion angles in Table 1. Bond lengths are all within the normal ranges (Allen *et al.*, 1987) and equivalent bond lengths in the two independent molecules are equal within their standard deviations. The molecules are packed in the crystal structure through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2).



Experimental

Methyl 5-nitro-2-(phenylamino)-4-pyridinecarboxylate, (I), was prepared by oxidative nucleophilic substitution (Finch *et al.*, 1980) of methyl 3-nitro-4-pyridinecarboxylate (Holt *et al.*, 2005) by sodioformanilide and subsequent hydrolysis (Tjosås & Fiksdahl, 2006). Crystals were grown by vapour diffusion of pentane into a solution in acetone in a refrigerator.

Crystal data

 $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_4$ $M_r = 273.25$ Triclinic, $P\bar{1}$ $a = 9.990(2) \text{ \AA}$ $b = 10.913(3) \text{ \AA}$ $c = 12.6758(19) \text{ \AA}$ $\alpha = 81.055(17)^\circ$ $\beta = 78.137(16)^\circ$ $\gamma = 70.86(2)^\circ$ $V = 1271.8(5) \text{ \AA}^3$

Z = 4

 $D_x = 1.427 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation $\mu = 0.11 \text{ mm}^{-1}$

T = 293(2) K

Block, light yellow

 $0.50 \times 0.40 \times 0.30 \text{ mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer
 ω – 2θ scans
Absorption correction: ψ scan
(*ABSCALC* in *OSCAIL*;
McArdle & Daly, 1999; North *et al.*, 1968)
 $T_{\min} = 0.948$, $T_{\max} = 0.968$

4900 measured reflections
4452 independent reflections
2078 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\text{max}} = 25.0^\circ$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.116$
 $S = 0.95$
4452 reflections
364 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.021$
 $\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0074 (14)

Table 1

Selected torsion angles ($^\circ$).

O14–N13–C19–C110	178.7 (2)	C111–C110–C112–O12	90.4 (3)
C211–C210–C212–O22	–94.7 (3)	O24–N23–C29–C210	–172.3 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N21–H21 \cdots O11	0.86	2.25	3.060 (3)	158
N21–H21 \cdots O11 ⁱ	0.86	2.62	3.107 (3)	117
N11–H11 \cdots O21 ⁱⁱ	0.86	2.23	3.094 (3)	179

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

H atoms were positioned geometrically and refined using a riding model, with aromatic C–H = 0.93 \AA , methyl C–H = 0.96 \AA , N–H = 0.86 \AA and $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C}, \text{N})$ or $1.4U_{\text{eq}}(\text{methyl C})$.

Data collection: *CAD-4-PC* Software (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC* Software; data reduction: *XCAD4* (McArdle & Higgins, 1995); program(s) used to solve structure: *OSCAIL* (McArdle, 2005) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1993) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *OSCAIL*.

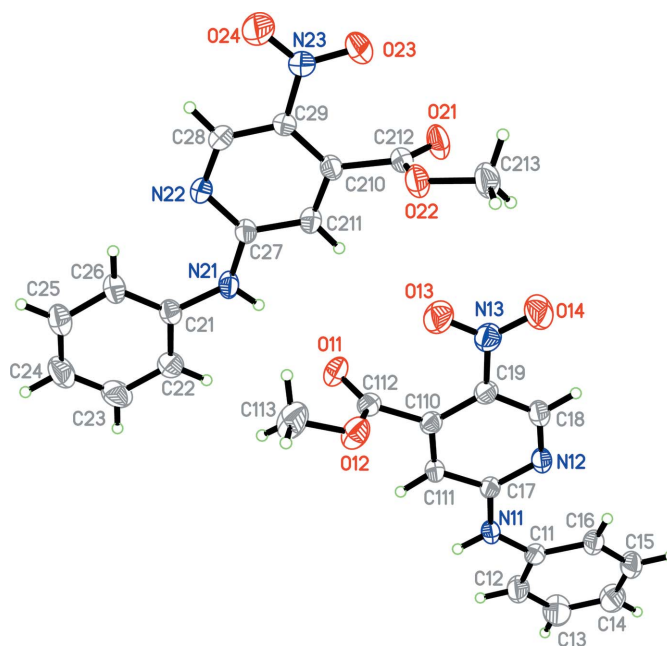


Figure 1

The asymmetric unit of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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