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An orthorhombic polymorph of *N*,*N*′-bis(2-pyridylmethyl)pyrazine-2,3-dicarboxamide

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The title compound, $C_{18}H_{16}N_6O_2$, can be crystallized in two polymorphic forms, *viz*. orthorhombic, described here, and triclinic. In the orthorhombic polymorph, both 2-pyridylmethyl moieties lie out of the plane of the pyrazine ring by *ca* 80°. One amide moiety lies in the plane of the pyrazine ring, while the other is almost perpendicular to the pyrazine ring. This structure contrasts with that of the triclinic polymorph, which is L-shaped, with one of the (2-pyridylmethyl)amide substituents lying almost in the plane of the pyrazine ring because of the presence of a bifurcated (N—H···N,N′) intramolecular hydrogen bond.

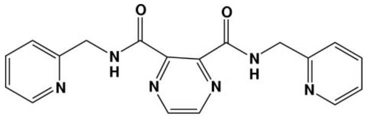
Key indicators

Single-crystal X-ray study
T = 153 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.079
Data-to-parameter ratio = 10.1

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

Comment

The title compound, (I), has been synthesized in order to study its coordination behaviour with first-row transition metals. Recently, the structures of both copper(II) (Hausmann *et al.*, 2003; Cati *et al.*, 2004) and nickel(II) (Cati *et al.*, 2004) [2 × 2] grids have been described. Cati *et al.* (2004) also described the synthesis of (I) and the structure of its triclinic polymorph.



(I)

The molecular structure of the orthorhombic polymorph of (I) is illustrated in Fig. 1. The bond distances and angles are normal for such compounds (see Table 1). In the triclinic polymorph (Cati *et al.*, 2004), the molecule is L-shaped, with a bifurcated intramolecular hydrogen bond maintaining one pyridine ring relatively coplanar with the amide group and the pyrazine ring (Fig. 2). In the orthorhombic polymorph, there is no such intramolecular hydrogen bond and the molecule is more V-shaped, as shown in Fig. 1. One amide moiety (N3 and

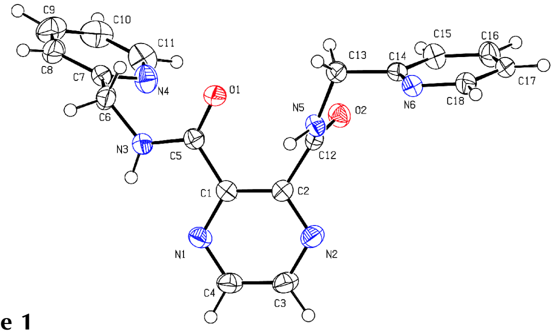


Figure 1
View of the orthorhombic polymorph of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

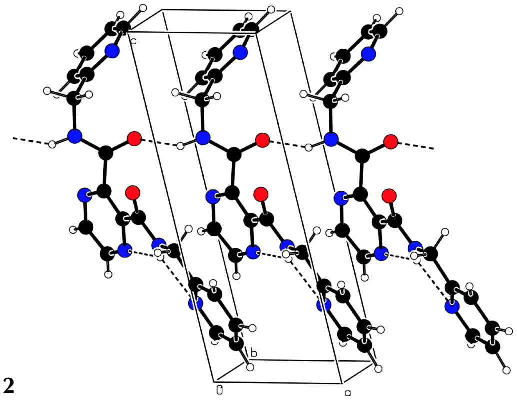


Figure 2
View of the intra- and intermolecular hydrogen bonding (dashed lines) in the triclinic polymorph of the title compound (Cati *et al.*, 2004).

O1) lies almost in the plane of the pyrazine ring [7.0 (1)°], with a short N3—H3···N1 distance (see Table 2), while the other (N5 and O2) is almost perpendicular [84.5 (1)°] to the pyrazine ring. Selected dihedral angles involving the pyrazine and pyridine rings in the two polymorphs are listed in Table 3.

In the crystal structure, the molecules are linked by a number of N—H···N and C—H···O hydrogen bonds to form a three-dimensional network (Table 2 and Fig. 3). This structure contrasts with that of the triclinic polymorph, where a relatively strong N—H···O hydrogen bond links molecules to form a hydrogen-bonded polymer (Fig. 2).

Experimental

The synthesis of and analytical details concerning the title compound have been described elsewhere (Cati *et al.*, 2004). Large colourless crystals of the orthorhombic polymorph were obtained by slow evaporation of an acetonitrile solution. Crystals of the triclinic polymorph were obtained from a solution of ethyl acetate and dichloromethane (50:3).

Crystal data

$C_{18}H_{16}N_6O_2$
$M_r = 348.37$
Orthorhombic, *Pbca*
$a = 8.5131\ (6)$ Å
$b = 31.862\ (2)$ Å
$c = 12.1757\ (6)$ Å
$V = 3302.6\ (4)$ Å^3
$Z = 8$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15 934 reflections
$\theta = 1.3–25.6°$
$\mu = 0.10$ mm^{-1}
$T = 153$ K
Block, colourless
0.50 × 0.50 × 0.40 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: none
18 389 measured reflections
3034 independent reflections
2456 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\rm max} = 25.5°$
$h = -10 \rightarrow 7$
$k = -38 \rightarrow 38$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.079$
$S = 1.03$
3034 reflections
300 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.1168P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0087 (9)

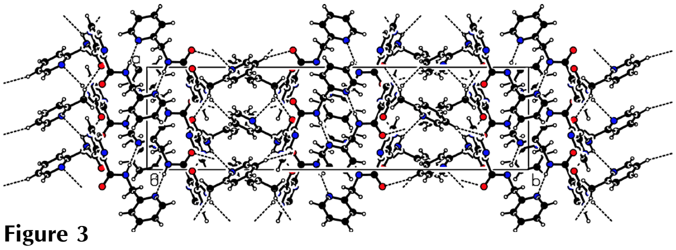


Figure 3
Molecular packing of the orthorhombic polymorph, viewed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.2299 (15)	N3—C6	1.4466 (17)
O2—C12	1.2265 (16)	N4—C7	1.3337 (18)
N1—C1	1.3408 (16)	N4—C11	1.3409 (17)
N1—C4	1.3298 (18)	N5—C12	1.3363 (16)
N2—C2	1.3407 (17)	N5—C13	1.4647 (17)
N2—C3	1.3358 (19)	N6—C14	1.3389 (14)
N3—C5	1.3297 (16)	N6—C18	1.3453 (17)
C1—N1—C4	116.68 (11)	O1—C5—C1	120.52 (11)
C2—N2—C3	116.20 (11)	N3—C5—C1	115.09 (11)
C5—N3—C6	122.59 (11)	N3—C6—C7	112.94 (11)
C7—N4—C11	117.46 (13)	N4—C7—C8	122.33 (12)
C12—N5—C13	119.41 (11)	N4—C7—C6	117.40 (12)
C14—N6—C18	117.17 (11)	N4—C11—C10	123.87 (16)
N1—C1—C5	117.61 (11)	O2—C12—C2	120.46 (11)
N1—C1—C2	121.28 (11)	N5—C12—C2	114.68 (11)
N2—C2—C12	114.71 (11)	O2—C12—N5	124.74 (12)
N2—C2—C1	121.76 (11)	N5—C13—C14	113.65 (11)
N2—C3—C4	122.04 (13)	N6—C14—C13	115.99 (11)
N1—C4—C3	121.99 (13)	N6—C14—C15	122.34 (11)
O1—C5—N3	124.38 (12)	N6—C18—C17	123.91 (12)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···N1	0.896 (17)	2.267 (17)	2.6921 (16)	108.7 (13)
N3—H3N···N4$^{\rm i}$	0.896 (17)	2.547 (17)	3.2785 (16)	139.2 (14)
N5—H5N···N6$^{\rm ii}$	0.928 (16)	2.122 (16)	3.0493 (16)	178.3 (13)
C3—H3···O2$^{\rm iii}$	0.980 (19)	2.378 (19)	3.3178 (18)	160.5 (14)
C6—H6*A*···O1$^{\rm i}$	0.972 (17)	2.505 (16)	3.1855 (17)	127.0 (11)
C6—H6*B*···O1	1.021 (15)	2.437 (15)	2.8114 (16)	100.7 (9)
C10—H10···O2$^{\rm iv}$	1.013 (16)	2.509 (16)	3.4749 (17)	159.2 (13)

Symmetry codes: (i) $x - \frac{1}{2}, y, \frac{3}{2} - z$; (ii) $1 - x, -y, 1 - z$; (iii) $x - \frac{1}{2}, y, \frac{1}{2} - z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Table 3
Dihedral angles (°) between various aromatic rings in the orthorhombic and triclinic polymorphs of (I); plane *A* is the pyrazine ring, plane *B* is the pyridine ring containing N4 and plane *C* is the pyridine ring containing N6.

Orthorhombic polymorph		Triclinic polymorph	
Plane–plane	Angle	Plane–plane	Angle
A–*B*	81.5 (1)	*A*–*B*	17.8 (2)
A–*C*	80.7 (1)	*A*–*C*	60.5 (2)
B–*C*	25.1 (1)	*B*–*C*	74.1 (2)

H atoms were located in Fourier difference maps and refined freely [C—H = 0.942 (18)–1.021 (15) Å and N—H = 0.896 (17) and 0.928 (16) Å].

Data collection: *X-AREA* (Stoe & Cie, 2003); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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