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N′-[(1*E*)-2,5-Dimethoxybenzylidene]pyridine-2-carbohydrazide

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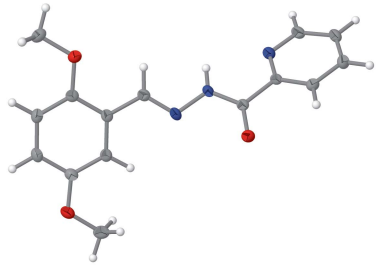
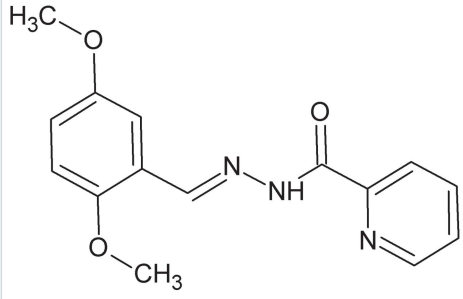
Structural data: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, $C_{15}H_{15}N_3O_3$, is twisted, with the dihedral angle between the pyridyl and benzene rings being 58.34 (6)°. In the crystal, amide-N—H···O(amide) and imine-C—H···O(amide) hydrogen bonds lead to zigzag (glide symmetry) chains extending along the *c* axis which are joined into layers parallel to the [100] direction by offset π–π stacking interactions between inversion-related benzene rings [centroid–centroid distance = 3.7468 (7) Å] and by C—H···π(pyridyl) interactions. Pyridyl rings protrude from the surfaces of the layers and partially intercalate with those of adjacent layers.

3D view



Chemical scheme



Structure description

Picolinic acid (PA) is a naturally occurring product of the degradation of tryptophan which is known to up-regulate host immune responses, especially macrophage cell functions (Shanshan *et al.*, 2006). The antimicrobial activity of PA against several strains of microorganisms has been reported (Maria *et al.*, 2008). In addition, PA and its derivatives have other biological activities, such as their use as dietary supplements (Komorowski *et al.*, 2008) and anti-oxidants (Kırkıl *et al.*, 2008), and they are metabolites of fungi (Dowd, 1999). Picolinic acid hydrazones have been found to possess significant antifungal activity against a wide range of soil borne pathogens (Aditi & Supradi, 2014). As a continuation of our efforts on the synthesis of biologically active compounds containing hydrazones, we report herein the crystal structure of *N*′-[(1*E*)-2,5-dimethoxybenzylidene]pyridine-2-carbohydrazide.

In the title compound (Fig. 1), the dihedral angle between the pyridyl and benzene rings is 58.34 (6)°. In the crystal, N2—H2···O3 hydrogen bonds, assisted by C9—

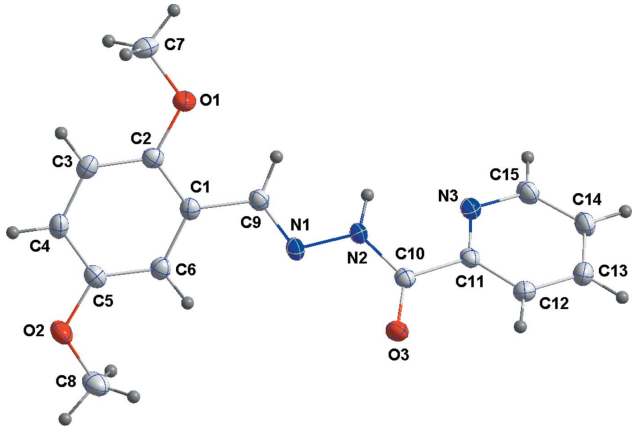


Figure 1
The title molecule of the title compound, with the atom-labelling scheme and 50% probability displacement ellipsoids.

H9···O3 hydrogen bonds, form chains extending along the *c* axis (Table 1 and Fig. 2). The chains are connected into layers parallel to [100] by offset π–π stacking interactions between inversion-related benzene rings [centroid–centroid = 3.7468 (7) Å; interplanar spacing = 3.3311 (5) Å] and by C4—H4···*Cg*1 interactions (*Cg*1 is the centroid of the N3/C11–C15 pyridine ring.; Table 1 and Fig. 2). The pyridine rings protrude from the surfaces of the layers and partially intercalate with those of adjacent layers.

Synthesis and crystallization

The title compound was synthesized according to our previously reported procedure (Mohamed *et al.*, 2013).

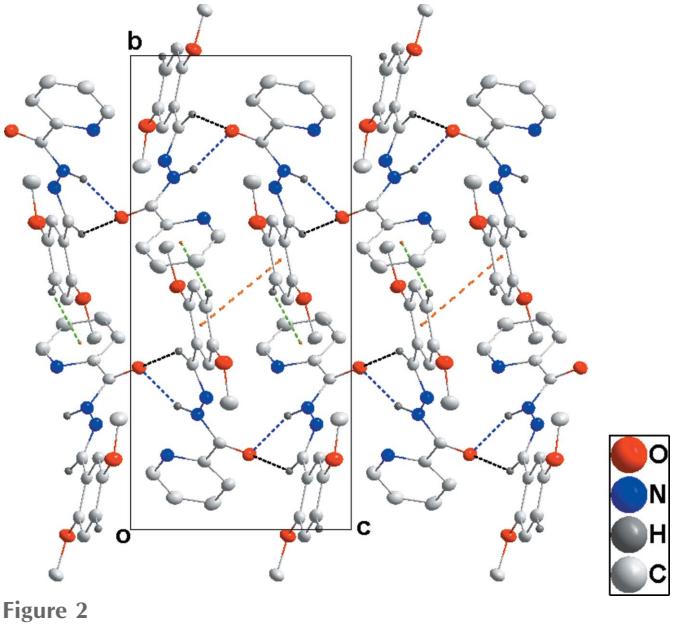


Figure 2
A view in projection along the *a* axis of the unit-cell contents, showing their association through offset π–π stacking (orange dashed lines) and C—H···π(ring) interactions (green dashed lines). The N—N···O and C—H···O hydrogen bonds are shown, respectively, as blue and black dashed lines.

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

*Cg*1 is the centroid of the N3/C11–C15 pyridine ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.879 (18)	2.103 (18)	2.9403 (13)	159.0 (15)
C9—H9···O3^{i}	0.967 (16)	2.525 (15)	3.3202 (14)	139.5 (12)
C4—H4···*Cg*1^{ii}	0.979 (15)	2.839 (16)	3.7240 (13)	150.6 (12)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{15}N_3O_3$
M_r	285.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	10.7311 (3), 16.4986 (4), 8.2033 (2)
β (°)	110.458 (1)
V (Å^3)	1360.78 (6)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.82
Crystal size (mm)	0.22 × 0.16 × 0.04
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.88, 0.97
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10229, 2647, 2375
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.035, 0.090, 1.06
No. of reflections	2647
No. of parameters	250
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.25

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x180128 [https://doi.org/10.1107/S2414314618001281]

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Crystal data

$C_{15}H_{15}N_3O_3$
M_r = 285.30
Monoclinic, $P2_1/c$
a = 10.7311 (3) Å
b = 16.4986 (4) Å
c = 8.2033 (2) Å
β = 110.458 (1)°
V = 1360.78 (6) Å^3
Z = 4
$F(000)$ = 600
D_x = 1.393 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 8226 reflections
θ = 4.4–72.4°
μ = 0.82 mm^{-1}
T = 150 K
Plate, colourless
0.22 × 0.16 × 0.04 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.88, T_{max} = 0.97
10229 measured reflections
2647 independent reflections
2375 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 72.4°, θ_{min} = 4.4°
h = −12→13
k = −20→19
l = −9→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.06
2647 reflections
250 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.3581P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58069 (8)	0.51301 (5)	0.21382 (11)	0.0264 (2)
O2	0.21048 (9)	0.35193 (5)	0.42335 (12)	0.0296 (2)
O3	0.81174 (8)	0.15852 (5)	0.53933 (11)	0.0249 (2)
H3	0.3676 (14)	0.5724 (9)	0.2692 (19)	0.027 (4)*
N1	0.65193 (9)	0.27831 (6)	0.34398 (13)	0.0216 (2)
N2	0.75529 (9)	0.24318 (6)	0.30438 (13)	0.0208 (2)
H2	0.7731 (16)	0.2602 (10)	0.213 (2)	0.038 (4)*
N3	0.90627 (10)	0.15664 (6)	0.16736 (13)	0.0223 (2)
C1	0.50657 (11)	0.39155 (7)	0.30184 (14)	0.0194 (2)
C2	0.48793 (11)	0.47459 (7)	0.26627 (14)	0.0204 (2)
C3	0.37985 (11)	0.51426 (7)	0.28827 (15)	0.0228 (2)
C4	0.29014 (11)	0.47126 (7)	0.34139 (15)	0.0234 (2)
H4	0.2133 (15)	0.4986 (9)	0.355 (2)	0.031 (4)*
C5	0.30648 (11)	0.38848 (7)	0.37352 (15)	0.0217 (2)
C6	0.41474 (11)	0.34879 (7)	0.35589 (15)	0.0206 (2)
H6	0.4310 (14)	0.2915 (9)	0.3807 (18)	0.025 (3)*
C7	0.55518 (14)	0.59604 (7)	0.16353 (17)	0.0284 (3)
H7A	0.5597 (16)	0.6316 (10)	0.263 (2)	0.037 (4)*
H7B	0.6227 (16)	0.6116 (10)	0.115 (2)	0.038 (4)*
H7C	0.4644 (17)	0.6035 (10)	0.077 (2)	0.041 (4)*
C8	0.21909 (15)	0.26674 (8)	0.4476 (2)	0.0333 (3)
H8A	0.3058 (18)	0.2514 (10)	0.537 (2)	0.039 (4)*
H8B	0.1455 (19)	0.2525 (11)	0.486 (2)	0.051 (5)*
H8C	0.2072 (17)	0.2381 (10)	0.333 (2)	0.042 (4)*
C9	0.62063 (11)	0.34959 (7)	0.28163 (14)	0.0205 (2)
H9	0.6707 (15)	0.3764 (10)	0.220 (2)	0.031 (4)*
C10	0.82496 (11)	0.18240 (6)	0.40395 (14)	0.0190 (2)
C11	0.92327 (11)	0.14317 (6)	0.33502 (15)	0.0191 (2)
C12	1.02277 (12)	0.09433 (7)	0.44403 (16)	0.0238 (3)
H12	1.0303 (15)	0.0879 (9)	0.566 (2)	0.029 (4)*
C13	1.10766 (12)	0.05531 (7)	0.37400 (18)	0.0287 (3)
H13	1.1779 (17)	0.0204 (11)	0.449 (2)	0.040 (4)*
C14	1.09010 (12)	0.06731 (7)	0.20079 (17)	0.0277 (3)
H14	1.1471 (15)	0.0400 (9)	0.1478 (19)	0.032 (4)*
C15	0.98899 (12)	0.11861 (8)	0.10286 (16)	0.0256 (3)
H15	0.9737 (15)	0.1272 (9)	−0.021 (2)	0.031 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0258 (4)	0.0227 (4)	0.0338 (5)	0.0000 (3)	0.0143 (4)	0.0049 (3)
O2	0.0264 (4)	0.0270 (5)	0.0426 (5)	−0.0011 (3)	0.0209 (4)	−0.0004 (4)
O3	0.0311 (4)	0.0231 (4)	0.0263 (4)	0.0027 (3)	0.0173 (4)	0.0026 (3)
N1	0.0202 (5)	0.0232 (5)	0.0247 (5)	0.0033 (4)	0.0118 (4)	−0.0015 (4)
N2	0.0209 (5)	0.0222 (5)	0.0235 (5)	0.0042 (4)	0.0128 (4)	0.0015 (4)
N3	0.0217 (5)	0.0231 (5)	0.0243 (5)	0.0010 (4)	0.0107 (4)	−0.0001 (4)
C1	0.0191 (5)	0.0215 (5)	0.0172 (5)	0.0013 (4)	0.0058 (4)	−0.0016 (4)
C2	0.0200 (5)	0.0226 (6)	0.0181 (5)	−0.0008 (4)	0.0059 (4)	0.0002 (4)
C3	0.0237 (6)	0.0208 (6)	0.0222 (6)	0.0036 (4)	0.0061 (5)	0.0005 (4)
C4	0.0210 (5)	0.0253 (6)	0.0241 (6)	0.0050 (4)	0.0081 (5)	−0.0010 (4)
C5	0.0200 (5)	0.0252 (6)	0.0212 (6)	−0.0017 (4)	0.0089 (5)	−0.0020 (4)
C6	0.0219 (5)	0.0199 (6)	0.0205 (6)	0.0013 (4)	0.0079 (5)	−0.0015 (4)
C7	0.0331 (7)	0.0223 (6)	0.0299 (7)	−0.0013 (5)	0.0109 (6)	0.0055 (5)
C8	0.0352 (7)	0.0276 (7)	0.0435 (8)	−0.0068 (5)	0.0219 (7)	−0.0019 (5)
C9	0.0197 (5)	0.0239 (6)	0.0187 (5)	0.0007 (4)	0.0077 (4)	0.0001 (4)
C10	0.0191 (5)	0.0167 (5)	0.0225 (5)	−0.0023 (4)	0.0089 (4)	−0.0020 (4)
C11	0.0184 (5)	0.0170 (5)	0.0240 (6)	−0.0022 (4)	0.0101 (4)	−0.0011 (4)
C12	0.0239 (6)	0.0219 (6)	0.0272 (6)	0.0009 (4)	0.0110 (5)	0.0039 (4)
C13	0.0242 (6)	0.0261 (6)	0.0380 (7)	0.0063 (5)	0.0137 (5)	0.0071 (5)
C14	0.0263 (6)	0.0248 (6)	0.0383 (7)	0.0028 (5)	0.0191 (5)	−0.0005 (5)
C15	0.0264 (6)	0.0276 (6)	0.0276 (6)	0.0005 (5)	0.0154 (5)	−0.0017 (5)

Geometric parameters (Å, º)

O1—C2	1.3702 (13)	C5—C6	1.3845 (16)
O1—C7	1.4293 (14)	C6—H6	0.969 (15)
O2—C5	1.3734 (14)	C7—H7A	0.994 (17)
O2—C8	1.4180 (16)	C7—H7B	0.977 (17)
O3—C10	1.2324 (14)	C7—H7C	0.993 (17)
N1—C9	1.2795 (15)	C8—H8A	0.997 (18)
N1—N2	1.3869 (13)	C8—H8B	0.975 (19)
N2—C10	1.3435 (15)	C8—H8C	1.019 (18)
N2—H2	0.879 (18)	C9—H9	0.967 (16)
N3—C15	1.3380 (15)	C10—C11	1.5062 (14)
N3—C11	1.3412 (15)	C11—C12	1.3862 (16)
C1—C2	1.4005 (16)	C12—C13	1.3929 (17)
C1—C6	1.4039 (15)	C12—H12	0.984 (15)
C1—C9	1.4652 (15)	C13—C14	1.3807 (19)
C2—C3	1.3973 (16)	C13—H13	0.977 (17)
C3—C4	1.3831 (17)	C14—C15	1.3891 (18)
C3—H3	0.973 (15)	C14—H14	0.976 (16)
C4—C5	1.3905 (17)	C15—H15	0.983 (16)
C4—H4	0.979 (15)		
C2—O1—C7	116.56 (9)	H7A—C7—H7C	105.6 (14)

C5—O2—C8	117.26 (9)	H7B—C7—H7C	111.0 (13)
C9—N1—N2	114.22 (9)	O2—C8—H8A	110.7 (9)
C10—N2—N1	119.36 (9)	O2—C8—H8B	105.3 (11)
C10—N2—H2	121.0 (11)	H8A—C8—H8B	110.7 (14)
N1—N2—H2	119.6 (11)	O2—C8—H8C	110.4 (9)
C15—N3—C11	117.17 (10)	H8A—C8—H8C	110.4 (13)
C2—C1—C6	119.51 (10)	H8B—C8—H8C	109.3 (14)
C2—C1—C9	120.18 (10)	N1—C9—C1	120.13 (10)
C6—C1—C9	120.31 (10)	N1—C9—H9	121.0 (9)
O1—C2—C3	123.43 (10)	C1—C9—H9	118.9 (9)
O1—C2—C1	116.83 (10)	O3—C10—N2	124.86 (10)
C3—C2—C1	119.73 (10)	O3—C10—C11	121.15 (10)
C4—C3—C2	120.07 (11)	N2—C10—C11	113.98 (9)
C4—C3—H3	119.1 (9)	N3—C11—C12	123.77 (10)
C2—C3—H3	120.9 (9)	N3—C11—C10	116.85 (10)
C3—C4—C5	120.52 (10)	C12—C11—C10	119.36 (10)
C3—C4—H4	120.3 (9)	C11—C12—C13	118.03 (11)
C5—C4—H4	119.1 (9)	C11—C12—H12	119.4 (9)
O2—C5—C6	124.57 (11)	C13—C12—H12	122.6 (9)
O2—C5—C4	115.44 (10)	C14—C13—C12	118.98 (11)
C6—C5—C4	119.98 (10)	C14—C13—H13	122.0 (10)
C5—C6—C1	120.17 (10)	C12—C13—H13	119.1 (10)
C5—C6—H6	122.3 (8)	C13—C14—C15	118.72 (11)
C1—C6—H6	117.5 (8)	C13—C14—H14	120.7 (9)
O1—C7—H7A	112.3 (9)	C15—C14—H14	120.5 (9)
O1—C7—H7B	106.0 (10)	N3—C15—C14	123.31 (11)
H7A—C7—H7B	110.6 (14)	N3—C15—H15	116.6 (9)
O1—C7—H7C	111.5 (10)	C14—C15—H15	120.0 (9)

C9—N1—N2—C10	158.68 (11)	N2—N1—C9—C1	175.33 (10)
C7—O1—C2—C3	−6.40 (16)	C2—C1—C9—N1	166.98 (11)
C7—O1—C2—C1	174.85 (10)	C6—C1—C9—N1	−13.43 (17)
C6—C1—C2—O1	−179.91 (10)	N1—N2—C10—O3	−6.16 (17)
C9—C1—C2—O1	−0.31 (15)	N1—N2—C10—C11	173.16 (9)
C6—C1—C2—C3	1.28 (16)	C15—N3—C11—C12	1.74 (16)
C9—C1—C2—C3	−179.12 (10)	C15—N3—C11—C10	−176.62 (10)
O1—C2—C3—C4	179.90 (10)	O3—C10—C11—N3	162.00 (10)
C1—C2—C3—C4	−1.38 (17)	N2—C10—C11—N3	−17.34 (14)
C2—C3—C4—C5	0.06 (17)	O3—C10—C11—C12	−16.44 (16)
C8—O2—C5—C6	−4.14 (17)	N2—C10—C11—C12	164.22 (10)
C8—O2—C5—C4	176.43 (11)	N3—C11—C12—C13	−1.80 (17)
C3—C4—C5—O2	−179.18 (10)	C10—C11—C12—C13	176.52 (10)
C3—C4—C5—C6	1.35 (17)	C11—C12—C13—C14	0.54 (18)
O2—C5—C6—C1	179.15 (10)	C12—C13—C14—C15	0.63 (18)
C4—C5—C6—C1	−1.43 (17)	C11—N3—C15—C14	−0.44 (17)
C2—C1—C6—C5	0.12 (16)	C13—C14—C15—N3	−0.71 (19)
C9—C1—C6—C5	−179.48 (10)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the N3/C11–C15 pyridine ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.879 (18)	2.103 (18)	2.9403 (13)	159.0 (15)
C9—H9···O3^{i}	0.967 (16)	2.525 (15)	3.3202 (14)	139.5 (12)
C4—H4···*Cg*1^{ii}	0.979 (15)	2.839 (16)	3.7240 (13)	150.6 (12)

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