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(±)-5-Ethyl-2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydro-1H-imidazol-2-yl)nicotinic acid

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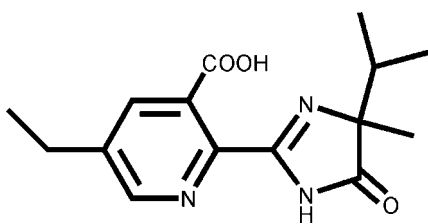
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.137; data-to-parameter ratio = 17.2.

In the title compound, $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_3$, owing to an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond, the pyridine and imidazole rings are nearly coplanar and are twisted from each other by a dihedral angle of only 0.92 (9°). The molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding, forming an infinite chain parallel to the b axis.

Related literature

For usages of nicotinic acid and imidazole in coordination chemistry and medicinal chemistry, see: Liu *et al.* (2005); Zhao *et al.* (2007); He *et al.* (2005); Boovanahalli *et al.* (2007); Song *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_3$
 $M_r = 289.33$
 Monoclinic, $P2_1/c$
 $a = 12.6916$ (15) Å

 $b = 16.0748$ (17) Å
 $c = 7.3801$ (8) Å
 $\beta = 100.213$ (7) $^\circ$
 $V = 1481.8$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 $0.25 \times 0.25 \times 0.20$ mm

Data collection

 Rigaku Mercury2 diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.978$, $T_{\max} = 0.988$

 15016 measured reflections
 3357 independent reflections
 2413 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.136$
 $S = 1.03$
 3357 reflections

 195 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³
Table 1

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N2}$	0.82	1.68	2.4984 (18)	178
$\text{N3}-\text{H3}\cdots\text{O2}^i$	0.86	2.10	2.9330 (19)	162

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This work was supported by a Start-up Grant from Southeast University to Professor Ren-Gen Xiong.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2325).

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supplementary materials

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(±)-5-Ethyl-2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydro-1*H*-imidazol-2-yl)nicotinic acid

W. Dai and D.-W. Fu

Comment

The nicotinic acid and the imidazole group have found a wide range of applications in coordination chemistry as ligands, in medicinal chemistry and materials science (Liu *et al.* 2005; Zhao *et al.* 2007; He *et al.* 2005; Boovannahalli *et al.* 2007; Song *et al.* 2006). We report here the crystal structure of the title compound, C₁₅H₁₉N₃O₃.

Owing to an intramolecular O1-H1...N2 hydrogen, the pyridine and the imidazole rings are nearly planar, they are only twisted to each other by a dihedral angle of 0.91 (9)°. In the imidazole ring, the C6=N2 bond distance of 1.282 (4) Å conforms to the value for a double bond, while the C11—N2 bond length of 1.472 (4) Å conforms to the value for a single bond. To the carboxyl group, the C9=O2 bond distance of 1.212 (4) Å conforms to the value for a double bond, while the C9—O1 bond length of 1.298 (4) Å conforms to the value for a single bond.

The molecules are linked through intermolecular N3-H3...O2 hydrogen bond forming an infinite chain parallel to the b axis. (Table 1 and Fig. 2).

Experimental

5-ethyl-2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydro-1*H*-imidazol-2-yl)nicotinic acid (3 mmol) was dissolved in ethanol (20 ml) and evaporated in the air affording colorless block crystals of this compound suitable for X-ray analysis were obtained.

Refinement

All H atoms attached to C, N and O atoms were fixed geometrically and treated as riding with C—H = 0.98 Å (methine), 0.97 Å (methylene), 0.96 Å (methyl) and N—H = 0.86 Å or O—H = 0.82 Å with U_{iso}(H) = 1.2U_{eq}(C, N) or U_{iso}(H) = 1.5U_{eq}(O, methyl).

Figures

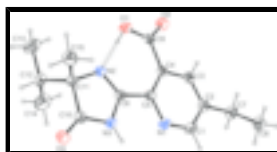


Fig. 1. Molecular view of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. H bond is shown as dashed line

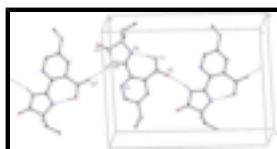


Fig. 2. Partial packing view of the title compound showing the formation of the chain parallel to the b axis. H bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) 1-x, 1/2+y, 1/2-z]

(±)-5-Ethyl-2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydro-1H-imidazol-2-yl)nicotinic acid

Crystal data

$C_{15}H_{19}N_3O_3$	$F_{000} = 616$
$M_r = 289.33$	$D_x = 1.297 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 12.6916 (15) \text{ \AA}$	Cell parameters from 2685 reflections
$b = 16.0748 (17) \text{ \AA}$	$\theta = 3.0\text{--}27.5^\circ$
$c = 7.3801 (8) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 100.213 (7)^\circ$	$T = 293 (2) \text{ K}$
$V = 1481.8 (3) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.25 \times 0.25 \times 0.20 \text{ mm}$

Data collection

Rigaku Mercury2 (2x2 bin mode) diffractometer	3357 independent reflections
Radiation source: fine-focus sealed tube	2413 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.045$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.4^\circ$
$T = 293(2) \text{ K}$	$\theta_{\text{min}} = 3.0^\circ$
ω scans	$h = -16 \rightarrow 16$
Absorption correction: Multi-scan (CrystalClear; Rigaku, 2005)	$k = -20 \rightarrow 20$
$T_{\text{min}} = 0.978$, $T_{\text{max}} = 0.988$	$l = -9 \rightarrow 9$
15016 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.136$	$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.3777P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3357 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
195 parameters	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.56210 (12)	0.92867 (8)	0.3216 (2)	0.0442 (4)
N2	0.31126 (10)	0.86524 (8)	0.06785 (19)	0.0358 (3)
N3	0.38168 (11)	0.99041 (8)	0.1425 (2)	0.0419 (4)
H3	0.4293	1.0259	0.1901	0.050*
O1	0.36104 (10)	0.71477 (7)	0.09236 (19)	0.0484 (4)
H1	0.3450	0.7642	0.0817	0.073*
O2	0.48765 (11)	0.63866 (7)	0.2486 (2)	0.0590 (4)
O3	0.24362 (11)	1.07812 (8)	0.0169 (2)	0.0626 (4)
C1	0.65661 (14)	0.90689 (11)	0.4182 (3)	0.0465 (5)
H1A	0.7023	0.9491	0.4702	0.056*
C2	0.69167 (13)	0.82543 (11)	0.4465 (2)	0.0385 (4)
C3	0.62079 (13)	0.76450 (10)	0.3712 (2)	0.0354 (4)
H3A	0.6409	0.7090	0.3883	0.042*
C4	0.51990 (12)	0.78314 (9)	0.2704 (2)	0.0304 (3)
C5	0.49432 (12)	0.86849 (9)	0.2486 (2)	0.0328 (4)
C6	0.39348 (13)	0.90513 (9)	0.1502 (2)	0.0337 (4)
C7	0.80073 (15)	0.80620 (13)	0.5550 (3)	0.0508 (5)
H7A	0.8117	0.7465	0.5559	0.061*
H7B	0.8035	0.8240	0.6813	0.061*
C8	0.89069 (16)	0.84795 (14)	0.4789 (3)	0.0624 (6)
H8A	0.8871	0.8321	0.3525	0.094*
H8B	0.9583	0.8309	0.5492	0.094*
H8C	0.8837	0.9072	0.4868	0.094*
C9	0.45278 (13)	0.70696 (10)	0.2013 (2)	0.0366 (4)
C10	0.28196 (14)	1.00956 (10)	0.0468 (3)	0.0429 (4)
C11	0.22886 (13)	0.92565 (10)	-0.0124 (2)	0.0373 (4)
C12	0.12537 (15)	0.91484 (12)	0.0676 (3)	0.0492 (5)
H12	0.0754	0.9583	0.0133	0.059*
C13	0.0711 (2)	0.83164 (17)	0.0180 (4)	0.0788 (8)
H13A	0.1161	0.7876	0.0757	0.118*
H13B	0.0592	0.8244	-0.1131	0.118*
H13C	0.0038	0.8303	0.0602	0.118*
C14	0.1441 (2)	0.92685 (19)	0.2749 (3)	0.0857 (9)

supplementary materials

H14A	0.0772	0.9224	0.3177	0.128*
H14B	0.1745	0.9809	0.3050	0.128*
H14C	0.1924	0.8849	0.3329	0.128*
C15	0.21042 (17)	0.91982 (12)	-0.2218 (3)	0.0527 (5)
H15A	0.2770	0.9283	-0.2638	0.079*
H15B	0.1600	0.9617	-0.2737	0.079*
H15C	0.1827	0.8658	-0.2596	0.079*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0384 (8)	0.0285 (7)	0.0620 (10)	-0.0038 (6)	-0.0019 (7)	-0.0006 (7)
N2	0.0334 (8)	0.0269 (7)	0.0458 (8)	0.0002 (5)	0.0031 (6)	-0.0001 (6)
N3	0.0331 (8)	0.0247 (7)	0.0644 (10)	-0.0015 (5)	-0.0008 (7)	-0.0006 (6)
O1	0.0437 (7)	0.0250 (6)	0.0718 (9)	-0.0017 (5)	-0.0033 (6)	-0.0029 (6)
O2	0.0513 (8)	0.0237 (6)	0.0970 (11)	0.0032 (5)	-0.0003 (7)	0.0047 (6)
O3	0.0492 (8)	0.0285 (7)	0.1029 (12)	0.0087 (6)	-0.0057 (8)	0.0011 (7)
C1	0.0397 (10)	0.0354 (9)	0.0593 (12)	-0.0075 (7)	-0.0049 (8)	0.0004 (8)
C2	0.0355 (9)	0.0396 (9)	0.0395 (9)	-0.0003 (7)	0.0046 (7)	0.0054 (7)
C3	0.0386 (9)	0.0292 (8)	0.0400 (9)	0.0040 (7)	0.0113 (7)	0.0051 (7)
C4	0.0324 (8)	0.0261 (7)	0.0344 (8)	0.0009 (6)	0.0105 (6)	0.0024 (6)
C5	0.0317 (8)	0.0266 (8)	0.0403 (9)	-0.0008 (6)	0.0072 (7)	0.0011 (7)
C6	0.0349 (9)	0.0251 (8)	0.0421 (9)	-0.0004 (6)	0.0091 (7)	0.0006 (7)
C7	0.0420 (10)	0.0496 (11)	0.0555 (12)	0.0018 (8)	-0.0059 (9)	0.0090 (9)
C8	0.0375 (11)	0.0660 (14)	0.0808 (16)	0.0043 (9)	0.0022 (10)	-0.0005 (11)
C9	0.0375 (9)	0.0255 (8)	0.0482 (10)	0.0001 (7)	0.0112 (8)	-0.0016 (7)
C10	0.0386 (10)	0.0300 (9)	0.0593 (11)	0.0029 (7)	0.0064 (8)	0.0006 (8)
C11	0.0334 (9)	0.0291 (8)	0.0476 (10)	0.0020 (6)	0.0021 (7)	0.0004 (7)
C12	0.0350 (10)	0.0503 (11)	0.0615 (12)	-0.0035 (8)	0.0063 (8)	-0.0040 (9)
C13	0.0650 (15)	0.0823 (18)	0.0907 (19)	-0.0356 (13)	0.0180 (13)	-0.0154 (14)
C14	0.0713 (17)	0.122 (2)	0.0717 (17)	-0.0306 (15)	0.0344 (13)	-0.0326 (16)
C15	0.0595 (13)	0.0493 (11)	0.0468 (11)	0.0068 (9)	0.0029 (9)	0.0032 (9)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.329 (2)	C7—H7A	0.9700
N1—C5	1.342 (2)	C7—H7B	0.9700
N2—C6	1.282 (2)	C8—H8A	0.9600
N2—C11	1.472 (2)	C8—H8B	0.9600
N3—C10	1.370 (2)	C8—H8C	0.9600
N3—C6	1.379 (2)	C10—C11	1.536 (2)
N3—H3	0.8600	C11—C15	1.524 (3)
O1—C9	1.298 (2)	C11—C12	1.543 (3)
O1—H1	0.8200	C12—C14	1.518 (3)
O2—C9	1.2118 (19)	C12—C13	1.519 (3)
O3—C10	1.209 (2)	C12—H12	0.9800
C1—C2	1.387 (2)	C13—H13A	0.9600
C1—H1A	0.9300	C13—H13B	0.9600
C2—C3	1.378 (2)	C13—H13C	0.9600

C2—C7	1.503 (2)	C14—H14A	0.9600
C3—C4	1.395 (2)	C14—H14B	0.9600
C3—H3A	0.9300	C14—H14C	0.9600
C4—C5	1.412 (2)	C15—H15A	0.9600
C4—C9	1.527 (2)	C15—H15B	0.9600
C5—C6	1.477 (2)	C15—H15C	0.9600
C7—C8	1.515 (3)		
C1—N1—C5	118.60 (14)	O2—C9—O1	120.50 (15)
C6—N2—C11	108.73 (13)	O2—C9—C4	118.47 (15)
C10—N3—C6	109.15 (14)	O1—C9—C4	121.02 (13)
C10—N3—H3	125.4	O3—C10—N3	127.14 (17)
C6—N3—H3	125.4	O3—C10—C11	127.32 (16)
C9—O1—H1	109.5	N3—C10—C11	105.54 (13)
N1—C1—C2	124.36 (16)	N2—C11—C15	109.74 (14)
N1—C1—H1A	117.8	N2—C11—C10	102.72 (13)
C2—C1—H1A	117.8	C15—C11—C10	108.85 (15)
C3—C2—C1	116.18 (15)	N2—C11—C12	111.34 (14)
C3—C2—C7	122.83 (16)	C15—C11—C12	113.15 (15)
C1—C2—C7	120.99 (16)	C10—C11—C12	110.51 (14)
C2—C3—C4	122.28 (15)	C14—C12—C13	109.8 (2)
C2—C3—H3A	118.9	C14—C12—C11	112.33 (16)
C4—C3—H3A	118.9	C13—C12—C11	112.79 (17)
C3—C4—C5	116.13 (14)	C14—C12—H12	107.2
C3—C4—C9	114.27 (14)	C13—C12—H12	107.2
C5—C4—C9	129.60 (14)	C11—C12—H12	107.2
N1—C5—C4	122.43 (15)	C12—C13—H13A	109.5
N1—C5—C6	110.34 (13)	C12—C13—H13B	109.5
C4—C5—C6	127.23 (14)	H13A—C13—H13B	109.5
N2—C6—N3	113.84 (14)	C12—C13—H13C	109.5
N2—C6—C5	126.50 (14)	H13A—C13—H13C	109.5
N3—C6—C5	119.66 (14)	H13B—C13—H13C	109.5
C2—C7—C8	113.25 (16)	C12—C14—H14A	109.5
C2—C7—H7A	108.9	C12—C14—H14B	109.5
C8—C7—H7A	108.9	H14A—C14—H14B	109.5
C2—C7—H7B	108.9	C12—C14—H14C	109.5
C8—C7—H7B	108.9	H14A—C14—H14C	109.5
H7A—C7—H7B	107.7	H14B—C14—H14C	109.5
C7—C8—H8A	109.5	C11—C15—H15A	109.5
C7—C8—H8B	109.5	C11—C15—H15B	109.5
H8A—C8—H8B	109.5	H15A—C15—H15B	109.5
C7—C8—H8C	109.5	C11—C15—H15C	109.5
H8A—C8—H8C	109.5	H15A—C15—H15C	109.5
H8B—C8—H8C	109.5	H15B—C15—H15C	109.5

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N2	0.82	1.68	2.4984 (18)	178
N3—H3 \cdots O2 ⁱ	0.86	2.10	2.9330 (19)	162

supplementary materials

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

Fig. 1

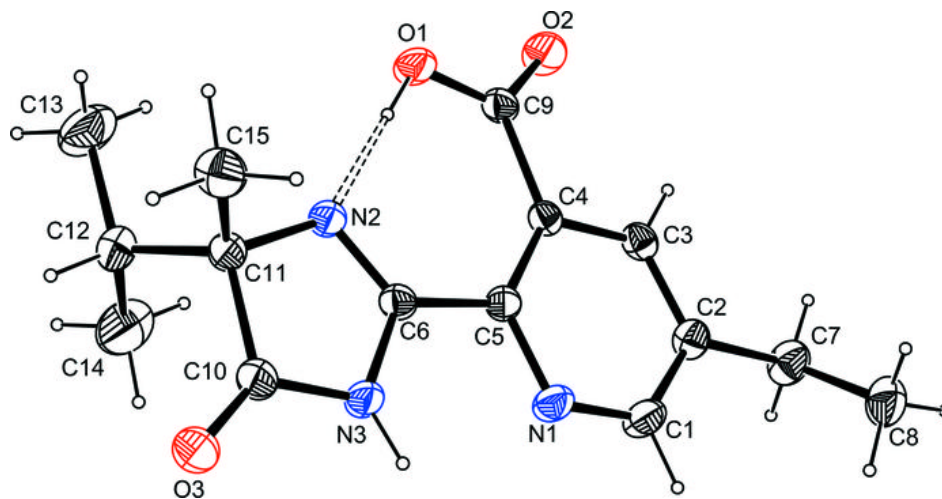


Fig. 2

