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Dimethylammonium 5-carboxy-2-(1-oxo-1λ^5-pyridin-2-yl)-1*H*-imidazole-4-carboxylate

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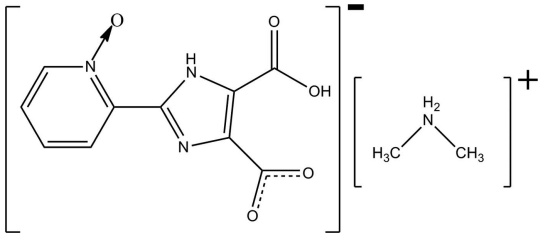
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.036; wR factor = 0.085; data-to-parameter ratio = 7.4.

In the title salt, $C_2H_8N^+\cdot C_{10}H_6N_3O_5^-$, the imidazolecarboxylate anion is essentially planar [maximum deviation from the least-squares plane = 0.046 (5) Å], with a dihedral angle between the rings of 2.7 (2)°. This conformation is maintained by the presence of both intramolecular carboxy–carboxylate O—H···O and imidazole–oxide N—H···O hydrogen bonds. Iin the crystal, cation–carboxylate N—H···O and cation–imidazole N—H···N hydrogen bonds result in chains along the *b* axis.

Related literature

For the structures of compounds with similar ligands, see: Chen (2008; Chen *et al.* (2011); Sun *et al.* (2005). For the synthesis of the ligand, see: Sun *et al.* (2006).



Experimental

Crystal data

$C_2H_8N^+\cdot C_{10}H_6N_3O_5^-$
M_r = 294.27
Monoclinic, *Cc*
a = 10.9690 (18) Å
b = 17.305 (3) Å
c = 8.0160 (13) Å
β = 120.901 (2)°
V = 1305.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.32 × 0.28 × 0.26 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.970
3782 measured reflections
1419 independent reflections
1204 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.085
S = 1.05
1419 reflections
193 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4B···N1^{i}	0.90	2.49	3.166 (3)	132
N4—H4B···O1^{i}	0.90	2.11	2.933 (3)	151
N4—H4A···O1^{ii}	0.90	1.95	2.806 (3)	159
O3—H3···O2	0.82	1.64	2.455 (3)	170
N2—H2···O5	0.86	2.06	2.603 (3)	120

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The work was supported by Zhongshan Polytechnic.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2224).

References

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supporting information

Acta Cryst. (2012). E**68**, o2600 [https://doi.org/10.1107/S1600536812033557]

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Chuntao Dai, Jianhua Nie, Yuehua Lin and Jun Wang

S1. Comment

Imidazole-4,5-dicarboxylic acid and its derivatives have a variety of coordination modes as ligands in the formation of metal complexes (Chen, 2008; Sun *et al.*, 2005), which include those with the lanthanide metals (Chen *et al.*, 2011). In the title salt, C_2 H_8N^+ $C_{10}H_6N_3O_5^-$, (Fig. 1), which consists of a dimethylammonium cation and a 5-carboxy-2-(2-pyridyl-*N*-oxide)-1*H*-imidazole-4-carboxylate anion, the anion is essentially planar [maximum deviation from the l.s. plane = 0.046 (5) Å], with the dihedral angle between the rings of 2.7 (2)Å. This conformation is maintained by the presence of both intramolecular carboxyl O—H···O and imidazole N—H···O_{oxide} hydrogen bonds while intermolecular cation N—H···$O_{carboxyl}$ and N—H···$N_{imidazole}$ hydrogen bonds (Table 1) give a one-dimensional chain structure.

S2. Experimental

The ligand,(4,5-dicarboxy-1*H*-imidazol-2-yl)pyridine-1-oxide was prepared by the method reported in the literature (Sun *et al.*, 2006). A diluted dimethylamine aqueous solution was added dropwise to an ethanolic solution of the ligand until the pH reached 7.4. Crystals of the title compound suitable for X-ray analysis were obtained after a few days of slow evaporation of the solvent.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C—H = 0.95–0.99 Å, N—H = 0.90 Å and O—H = 0.82 Å) and were treated as riding on their parent atoms, with U_{iso}(H) = 1.2–1.5U_{eq}(C, N, O). In the absence of a suitable heavy atom, Friedel pairs were averaged in the refinement.

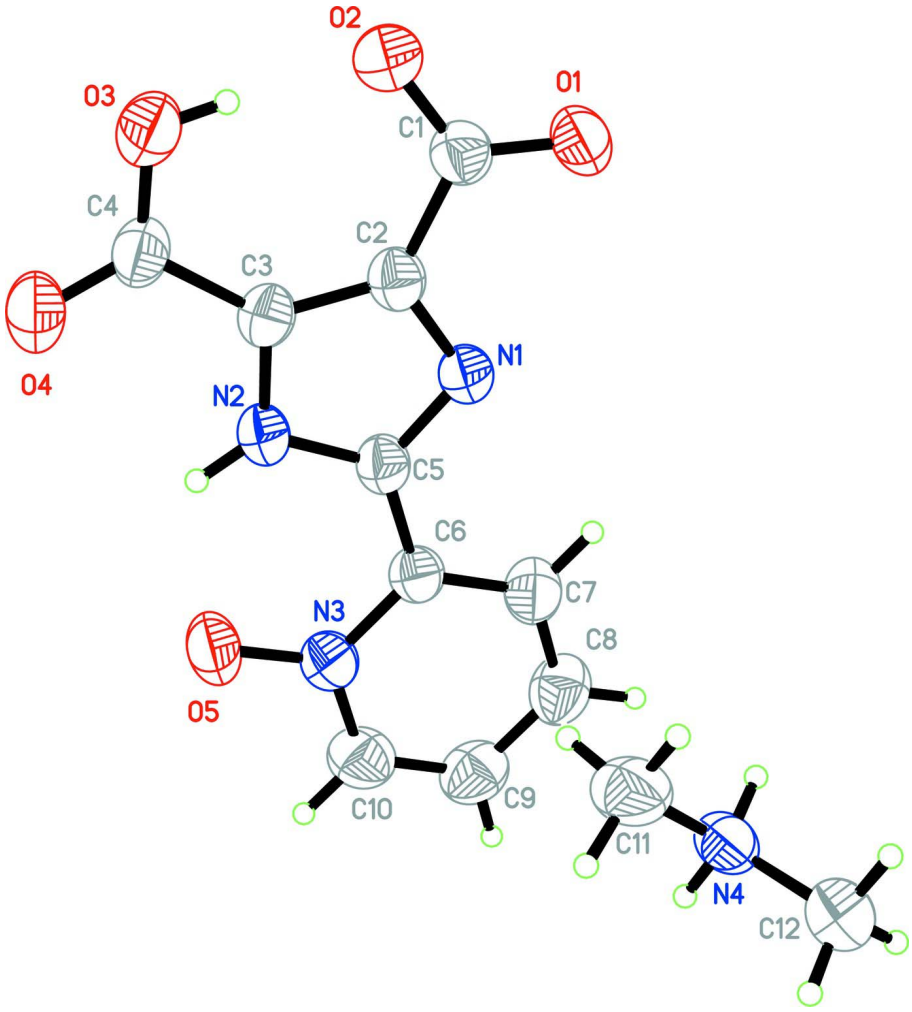

Figure 1

Molecular conformation and atom-numbering scheme for the title compound, with displacement ellipsoids drawn at the 50% probability level.

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Crystal data

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Hall symbol: C -2yc
$a = 10.9690\ (18)$ Å
$b = 17.305\ (3)$ Å
$c = 8.0160\ (13)$ Å
$\beta = 120.901\ (2)°$
$V = 1305.6\ (4)$ Å^3
$Z = 4$
$F(000) = 616$
$D_x = 1.497$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3600 reflections
$\theta = 1.3–28.0°$
$\mu = 0.12$ mm^{-1}
$T = 298$ K
Block, colourless
0.32 × 0.28 × 0.26 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.970
3782 measured reflections
1419 independent reflections
1204 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.0°, θ_{min} = 2.4°
h = −14→12
k = −19→21
l = −8→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.085
S = 1.05
1419 reflections
193 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.8048 (3)	0.88560 (14)	0.6235 (4)	0.0435 (6)
C2	0.6875 (3)	0.92371 (14)	0.4485 (4)	0.0399 (6)
C3	0.6630 (3)	1.00178 (15)	0.4067 (4)	0.0405 (6)
C4	0.7352 (3)	1.07430 (16)	0.5114 (4)	0.0462 (7)
C5	0.4999 (3)	0.93154 (13)	0.1662 (4)	0.0392 (6)
C6	0.3776 (3)	0.90945 (15)	−0.0207 (4)	0.0408 (6)
C7	0.3453 (3)	0.83297 (16)	−0.0743 (4)	0.0504 (7)
H7	0.4041	0.7945	0.0095	0.060*
C8	0.2281 (4)	0.81248 (17)	−0.2486 (5)	0.0581 (8)
H8	0.2074	0.7607	−0.2825	0.070*
C9	0.1414 (3)	0.86974 (19)	−0.3729 (4)	0.0578 (8)
H9	0.0610	0.8570	−0.4910	0.069*
C10	0.1752 (4)	0.94533 (19)	−0.3202 (5)	0.0584 (8)
H10	0.1173	0.9838	−0.4047	0.070*
C11	0.1058 (4)	0.83037 (18)	0.1270 (5)	0.0663 (9)
H11A	0.0357	0.8668	0.1150	0.099*

H11B	0.1698	0.8554	0.0960	0.099*
H11C	0.1580	0.8113	0.2580	0.099*
C12	−0.0512 (4)	0.71875 (19)	0.0443 (6)	0.0688 (9)
H12A	0.0075	0.6984	0.1731	0.103*
H12B	−0.0929	0.6769	−0.0464	0.103*
H12C	−0.1250	0.7501	0.0399	0.103*
N1	0.5853 (3)	0.88073 (11)	0.2989 (4)	0.0414 (5)
N2	0.5433 (2)	1.00487 (12)	0.2268 (3)	0.0418 (5)
H2	0.5024	1.0462	0.1629	0.050*
N3	0.2913 (2)	0.96589 (13)	−0.1477 (3)	0.0468 (6)
N4	0.0354 (3)	0.76580 (12)	−0.0071 (4)	0.0483 (5)
H4A	−0.0202	0.7846	−0.1280	0.058*
H4B	0.1019	0.7356	−0.0080	0.058*
O1	0.8070 (2)	0.81390 (9)	0.6329 (3)	0.0522 (5)
O2	0.8994 (2)	0.92884 (11)	0.7573 (3)	0.0584 (6)
O3	0.8499 (2)	1.06669 (12)	0.6792 (3)	0.0583 (6)
H3	0.8722	1.0209	0.6990	0.087*
O4	0.6859 (3)	1.13664 (11)	0.4385 (4)	0.0669 (6)
O5	0.3198 (3)	1.03909 (11)	−0.1066 (3)	0.0666 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0445 (15)	0.0422 (14)	0.0441 (15)	0.0006 (13)	0.0229 (13)	0.0027 (13)
C2	0.0444 (15)	0.0369 (13)	0.0429 (15)	0.0007 (13)	0.0256 (13)	0.0014 (13)
C3	0.0437 (16)	0.0379 (13)	0.0433 (16)	−0.0040 (11)	0.0249 (14)	−0.0003 (11)
C4	0.0520 (19)	0.0372 (16)	0.0480 (18)	−0.0081 (12)	0.0248 (16)	−0.0012 (12)
C5	0.0427 (16)	0.0351 (14)	0.0415 (16)	−0.0004 (11)	0.0227 (14)	0.0032 (12)
C6	0.0405 (15)	0.0405 (13)	0.0404 (15)	−0.0010 (12)	0.0199 (13)	0.0046 (12)
C7	0.0578 (19)	0.0426 (14)	0.0482 (18)	−0.0033 (13)	0.0253 (17)	0.0002 (13)
C8	0.067 (2)	0.0522 (18)	0.0520 (18)	−0.0110 (16)	0.0279 (17)	−0.0071 (15)
C9	0.053 (2)	0.067 (2)	0.0443 (18)	−0.0098 (16)	0.0188 (16)	−0.0052 (15)
C10	0.0516 (19)	0.065 (2)	0.0466 (17)	0.0030 (16)	0.0167 (15)	0.0110 (15)
C11	0.0513 (19)	0.0587 (19)	0.067 (2)	0.0029 (15)	0.0145 (17)	−0.0142 (17)
C12	0.057 (2)	0.059 (2)	0.078 (2)	0.0050 (15)	0.0259 (19)	0.0192 (17)
N1	0.0419 (12)	0.0355 (11)	0.0410 (12)	−0.0004 (9)	0.0170 (10)	0.0029 (10)
N2	0.0450 (13)	0.0328 (11)	0.0447 (13)	0.0004 (9)	0.0211 (11)	0.0060 (9)
N3	0.0454 (14)	0.0442 (13)	0.0453 (14)	0.0005 (11)	0.0194 (13)	0.0065 (11)
N4	0.0450 (13)	0.0410 (12)	0.0476 (12)	0.0066 (10)	0.0157 (11)	0.0004 (11)
O1	0.0579 (12)	0.0383 (10)	0.0505 (12)	0.0056 (10)	0.0208 (11)	0.0057 (9)
O2	0.0515 (14)	0.0500 (13)	0.0542 (14)	−0.0042 (10)	0.0131 (12)	0.0017 (10)
O3	0.0610 (14)	0.0452 (12)	0.0562 (13)	−0.0110 (10)	0.0212 (12)	−0.0028 (10)
O4	0.0746 (16)	0.0372 (10)	0.0702 (15)	−0.0051 (12)	0.0238 (13)	0.0021 (11)
O5	0.0696 (15)	0.0361 (11)	0.0670 (15)	0.0016 (10)	0.0157 (13)	0.0075 (10)

Geometric parameters (Å, º)

C1—O1	1.243 (3)	C9—C10	1.366 (4)
C1—O2	1.282 (3)	C9—H9	0.9300
C1—C2	1.485 (4)	C10—N3	1.361 (4)
C2—N1	1.367 (4)	C10—H10	0.9300
C2—C3	1.385 (4)	C11—N4	1.465 (4)
C3—N2	1.364 (3)	C11—H11A	0.9600
C3—C4	1.491 (4)	C11—H11B	0.9600
C4—O4	1.214 (3)	C11—H11C	0.9600
C4—O3	1.293 (4)	C12—N4	1.462 (4)
C5—N1	1.326 (3)	C12—H12A	0.9600
C5—N2	1.355 (3)	C12—H12B	0.9600
C5—C6	1.458 (4)	C12—H12C	0.9600
C6—N3	1.377 (3)	N2—H2	0.8600
C6—C7	1.380 (4)	N3—O5	1.306 (3)
C7—C8	1.373 (5)	N4—H4A	0.9000
C7—H7	0.9300	N4—H4B	0.9000
C8—C9	1.380 (4)	O3—H3	0.8200
C8—H8	0.9300		

O1—C1—O2	123.4 (3)	N3—C10—H10	119.1
O1—C1—C2	118.8 (3)	C9—C10—H10	119.1
O2—C1—C2	117.9 (2)	N4—C11—H11A	109.5
N1—C2—C3	110.4 (3)	N4—C11—H11B	109.5
N1—C2—C1	120.7 (2)	H11A—C11—H11B	109.5
C3—C2—C1	128.9 (3)	N4—C11—H11C	109.5
N2—C3—C2	104.8 (2)	H11A—C11—H11C	109.5
N2—C3—C4	120.4 (2)	H11B—C11—H11C	109.5
C2—C3—C4	134.7 (3)	N4—C12—H12A	109.5
O4—C4—O3	123.1 (3)	N4—C12—H12B	109.5
O4—C4—C3	120.0 (3)	H12A—C12—H12B	109.5
O3—C4—C3	116.8 (3)	N4—C12—H12C	109.5
N1—C5—N2	111.1 (2)	H12A—C12—H12C	109.5
N1—C5—C6	123.3 (2)	H12B—C12—H12C	109.5
N2—C5—C6	125.6 (2)	C5—N1—C2	105.44 (19)
N3—C6—C7	118.8 (3)	C5—N2—C3	108.2 (2)
N3—C6—C5	119.6 (2)	C5—N2—H2	125.9
C7—C6—C5	121.6 (3)	C3—N2—H2	125.9
C8—C7—C6	121.4 (3)	O5—N3—C10	119.2 (2)
C8—C7—H7	119.3	O5—N3—C6	121.1 (2)
C6—C7—H7	119.3	C10—N3—C6	119.7 (2)
C7—C8—C9	119.1 (3)	C12—N4—C11	113.1 (3)
C7—C8—H8	120.4	C12—N4—H4A	109.0
C9—C8—H8	120.4	C11—N4—H4A	109.0
C10—C9—C8	119.2 (3)	C12—N4—H4B	109.0
C10—C9—H9	120.4	C11—N4—H4B	109.0
C8—C9—H9	120.4	H4A—N4—H4B	107.8

N3—C10—C9	121.9 (3)	C4—O3—H3	109.5
O1—C1—C2—N1	−1.3 (4)	C6—C7—C8—C9	0.3 (4)
O2—C1—C2—N1	178.4 (2)	C7—C8—C9—C10	0.7 (5)
O1—C1—C2—C3	179.8 (3)	C8—C9—C10—N3	−0.6 (5)
O2—C1—C2—C3	−0.6 (4)	N2—C5—N1—C2	−0.6 (3)
N1—C2—C3—N2	−0.3 (3)	C6—C5—N1—C2	178.4 (2)
C1—C2—C3—N2	178.8 (2)	C3—C2—N1—C5	0.6 (3)
N1—C2—C3—C4	179.4 (3)	C1—C2—N1—C5	−178.6 (2)
C1—C2—C3—C4	−1.5 (5)	N1—C5—N2—C3	0.5 (3)
N2—C3—C4—O4	1.6 (4)	C6—C5—N2—C3	−178.6 (2)
C2—C3—C4—O4	−178.0 (3)	C2—C3—N2—C5	−0.1 (2)
N2—C3—C4—O3	−178.4 (2)	C4—C3—N2—C5	−179.8 (2)
C2—C3—C4—O3	2.0 (4)	C9—C10—N3—O5	178.8 (3)
N1—C5—C6—N3	177.7 (2)	C9—C10—N3—C6	−0.4 (4)
N2—C5—C6—N3	−3.4 (4)	C7—C6—N3—O5	−177.8 (2)
N1—C5—C6—C7	−2.2 (4)	C5—C6—N3—O5	2.3 (3)
N2—C5—C6—C7	176.7 (3)	C7—C6—N3—C10	1.4 (4)
N3—C6—C7—C8	−1.4 (4)	C5—C6—N3—C10	−178.5 (2)
C5—C6—C7—C8	178.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*B*···N1^{i}	0.90	2.49	3.166 (3)	132
N4—H4*B*···O1^{i}	0.90	2.11	2.933 (3)	151
N4—H4*A*···O1^{ii}	0.90	1.95	2.806 (3)	159
O3—H3···O2	0.82	1.64	2.455 (3)	170
N2—H2···O5	0.86	2.06	2.603 (3)	120

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) *x*−1, *y*, *z*−1.