Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Phenyl-1*H*-imidazol-3-ium hydrogen oxalate

Jin-Na Song

School of Biological and Agricultural Engineering, Jilin University, Changchun 130022, People's Republic of China
Correspondence e-mail: songjn2010@jlu.edu.cn

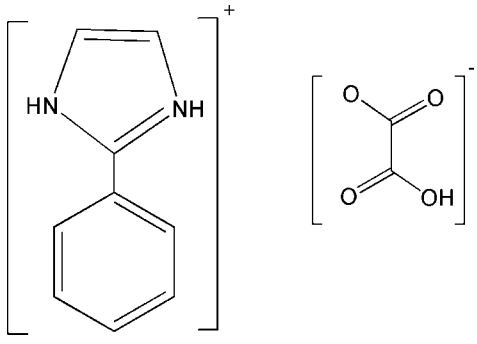
Received 12 June 2011; accepted 15 June 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.104; data-to-parameter ratio = 16.3.

In the title molecular salt, $C_9H_9N_2^+\cdot C_2HO_4^-$, the dihedral angle between the aromatic rings of the cation is 17.5 (3)° and the dihedral angle between the –CO_2H and –CO_2 groups of the anion is 38.6 (2)°. In the crystal, the components interact by way of O—H···O and N—H···O hydrogen bonds.

Related literature

For backgrond to 2-phenylimidazole as a ligand, see: Liu *et al.* (2008). For a related 2-phenylimidazolium nitrate structure, see: Zhang *et al.* (2007).



Experimental

Crystal data

$C_9H_9N_2^+\cdot C_2HO_4^-$
M_r = 234.21
Triclinic, $P\overline{1}$
a = 5.571 (4) Å
b = 9.216 (5) Å
c = 11.918 (6) Å
α = 70.262 (5)°
β = 80.460 (1)°
γ = 74.871 (5)°
V = 554.0 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.15 mm

Data collection

Oxford Diffraction Gemini R Ultra CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.38, T_{max} = 0.57
4030 measured reflections
2505 independent reflections
1629 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.104
S = 0.93
2505 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O4^{i}	0.82	1.76	2.5793 (19)	172
N2—H2···O3^{ii}	0.86	1.90	2.732 (2)	164
N1—H1···O4^{i}	0.86	1.93	2.777 (2)	169

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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*.

This work was supported by the Specialized Research Fund for the Doctoral Program of Higher Education (20100061120071), the Young Scientific Research Foundation of Jilin Province (201101068) and the Natural Science Foundation of Jilin University (201003051).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5908).

References

Liu, Y.-Y., Ma, J.-F., Yang, J., Ma, J.-C. & Ping, G.-J. (2008). *CrystEngComm*, **10**, 565–572.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, L.-P., Ma, J.-F. & Ping, G.-J. (2007). *Acta Cryst.* E**63**, o2438–o2439.

supporting information

Acta Cryst. (2011). E**67**, o1773 [doi:10.1107/S1600536811023300]

2-Phenyl-1*H*-imidazol-3-ium hydrogen oxalate

Jin-Na Song

S1. Comment

2-Phenylimidazole has been extensively used to build supramolecular architectures because of its excellent coordinating abilities and fruitful aromatic systems, (Liu *et al.*, 2008). The 2-phenylimidazolium nitrate structure has been reported as a hemihydrate (Zhang *et al.*, 2007). In this work, we will report the synthesis and crystal structure of the 2-phenyl-imidazolium hydrogen oxalate, namely, $C_{11}H_{10}N_2O_4$.

The asymmetric unit of the title compound contains one 2-phenylimidazolium cation and one hydrogen oxalate anion (Fig. 1). There are O—H···O and N—H···O hydrogen-bonding interactions in the structure (Table I).

S2. Experimental

A mixture of 2-phenylimidazole (0.3 mmol), oxalic acid (0.3 mmol) and H_2O (8 ml) was mixed. After one week, colorless blocks of the title compound were obtained at room temperature.

S3. Refinement

All H atoms on C and N atoms were positioned geometrically (N—H = 0.86 Å, O—H = 0.82 Å and C—H = 0.93 Å) and refined as riding, with U_{iso}(H)=1.2U_{eq}(carrier).

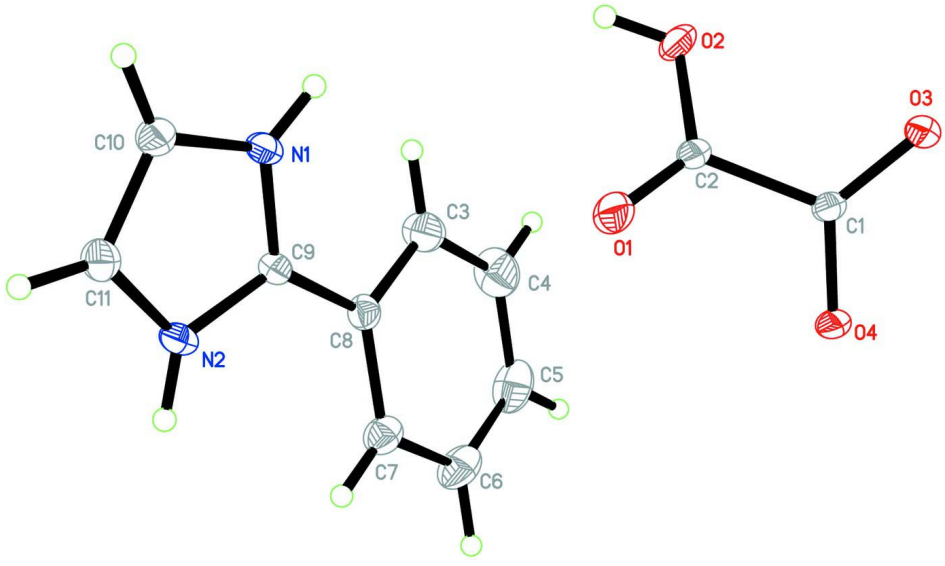


Figure 1

The structure of (I), showing displacement ellipsoids drawn at the 30% probability level.

2-Phenyl-1*H*-imidazol-3-ium hydrogen oxalate

Crystal data

$C_9H_9N_2^+·C_2HO_4^-$
M_r = 234.21
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.571 (4) Å
b = 9.216 (5) Å
c = 11.918 (6) Å
α = 70.262 (5)°
β = 80.460 (1)°
γ = 74.871 (5)°
V = 554.0 (6) Å^3
Z = 2
$F(000)$ = 244
D_x = 1.404 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2505 reflections
θ = 1.8–29.1°
μ = 0.11 mm^{-1}
T = 293 K
Block, colorless
0.22 × 0.20 × 0.15 mm

Data collection

Oxford Diffraction Gemini R Ultra CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.38, T_{max} = 0.57
4030 measured reflections
2505 independent reflections
1629 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 29.1°, θ_{min} = 1.8°
h = −5→7
k = −9→11
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.104
S = 0.93
2505 reflections
154 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.1534 (2)	0.24120 (16)	0.80993 (12)	0.0329 (3)
C2	0.0697 (2)	0.30067 (16)	0.82279 (12)	0.0351 (3)
C3	0.2653 (3)	0.6592 (2)	0.61457 (15)	0.0559 (4)

H3	0.4206	0.5901	0.6257	0.067*
C4	0.1280 (4)	0.6664 (2)	0.52534 (17)	0.0712 (6)
H4	0.1910	0.6018	0.4770	0.085*
C5	−0.1001 (4)	0.7681 (2)	0.50819 (16)	0.0685 (5)
H5	−0.1925	0.7726	0.4484	0.082*
C6	−0.1917 (3)	0.8628 (2)	0.57895 (16)	0.0605 (5)
H6	−0.3468	0.9319	0.5669	0.073*
C7	−0.0578 (3)	0.85767 (18)	0.66808 (14)	0.0463 (4)
H7	−0.1221	0.9234	0.7154	0.056*
C8	0.1728 (3)	0.75450 (16)	0.68720 (12)	0.0364 (3)
C9	0.3166 (2)	0.74777 (15)	0.78093 (12)	0.0329 (3)
C11	0.4589 (3)	0.81235 (18)	0.91542 (13)	0.0451 (4)
H11	0.4762	0.8682	0.9645	0.054*
C10	0.5995 (3)	0.67346 (17)	0.91074 (13)	0.0414 (4)
H10	0.7336	0.6140	0.9556	0.050*
N1	0.5092 (2)	0.63481 (12)	0.82744 (10)	0.0363 (3)
H1	0.5683	0.5498	0.8079	0.044*
N2	0.2843 (2)	0.85814 (13)	0.83489 (11)	0.0409 (3)
H2	0.1713	0.9444	0.8211	0.049*
O1	0.04670 (19)	0.40133 (14)	0.86821 (12)	0.0591 (3)
O2	0.27912 (17)	0.22797 (13)	0.78035 (11)	0.0565 (3)
H2A	0.3941	0.2626	0.7889	0.085*
O3	−0.12709 (18)	0.09884 (11)	0.82754 (11)	0.0547 (3)
O4	−0.35046 (16)	0.34460 (11)	0.78500 (9)	0.0420 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0231 (6)	0.0340 (8)	0.0460 (8)	−0.0023 (6)	−0.0051 (6)	−0.0198 (6)
C2	0.0249 (7)	0.0331 (8)	0.0510 (9)	−0.0015 (6)	−0.0098 (6)	−0.0183 (7)
C3	0.0585 (11)	0.0547 (10)	0.0585 (10)	−0.0027 (8)	−0.0167 (8)	−0.0242 (8)
C4	0.0890 (15)	0.0786 (14)	0.0595 (11)	−0.0185 (12)	−0.0179 (11)	−0.0330 (10)
C5	0.0738 (14)	0.0881 (15)	0.0521 (11)	−0.0333 (12)	−0.0248 (10)	−0.0119 (10)
C6	0.0459 (10)	0.0726 (12)	0.0573 (10)	−0.0132 (9)	−0.0182 (8)	−0.0063 (9)
C7	0.0400 (8)	0.0490 (9)	0.0468 (9)	−0.0089 (7)	−0.0087 (7)	−0.0094 (7)
C8	0.0383 (8)	0.0332 (8)	0.0379 (8)	−0.0103 (6)	−0.0067 (6)	−0.0080 (6)
C9	0.0322 (7)	0.0257 (7)	0.0399 (8)	−0.0042 (6)	−0.0030 (6)	−0.0106 (6)
C11	0.0470 (9)	0.0474 (9)	0.0493 (9)	−0.0059 (7)	−0.0126 (7)	−0.0252 (7)
C10	0.0403 (8)	0.0394 (8)	0.0453 (8)	−0.0024 (7)	−0.0139 (7)	−0.0142 (7)
N1	0.0371 (6)	0.0279 (6)	0.0441 (7)	0.0010 (5)	−0.0094 (5)	−0.0148 (5)
N2	0.0380 (7)	0.0308 (6)	0.0551 (8)	0.0034 (5)	−0.0114 (6)	−0.0195 (6)
O1	0.0379 (6)	0.0655 (8)	0.0974 (9)	−0.0077 (5)	−0.0096 (6)	−0.0560 (7)
O2	0.0216 (5)	0.0618 (7)	0.1047 (9)	−0.0057 (5)	−0.0051 (5)	−0.0522 (7)
O3	0.0314 (5)	0.0331 (6)	0.1060 (9)	−0.0020 (4)	−0.0108 (6)	−0.0311 (6)
O4	0.0235 (5)	0.0358 (6)	0.0717 (7)	0.0009 (4)	−0.0142 (5)	−0.0241 (5)

Geometric parameters (Å, º)

C1—O3	1.2291 (17)	C7—C8	1.385 (2)
C1—O4	1.2541 (16)	C7—H7	0.9300
C1—C2	1.531 (2)	C8—C9	1.455 (2)
C2—O1	1.1934 (17)	C9—N1	1.3293 (18)
C2—O2	1.3003 (16)	C9—N2	1.3374 (17)
C3—C8	1.384 (2)	C11—C10	1.329 (2)
C3—C4	1.385 (2)	C11—N2	1.366 (2)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.368 (3)	C10—N1	1.3649 (19)
C4—H4	0.9300	C10—H10	0.9300
C5—C6	1.362 (3)	N1—H1	0.8600
C5—H5	0.9300	N2—H2	0.8600
C6—C7	1.378 (2)	O2—H2A	0.8200
C6—H6	0.9300		
O3—C1—O4	126.05 (12)	C8—C7—H7	120.0
O3—C1—C2	118.29 (11)	C3—C8—C7	118.80 (14)
O4—C1—C2	115.64 (12)	C3—C8—C9	120.56 (14)
O1—C2—O2	125.70 (13)	C7—C8—C9	120.64 (13)
O1—C2—C1	122.10 (12)	N1—C9—N2	106.32 (12)
O2—C2—C1	112.19 (12)	N1—C9—C8	127.46 (12)
C8—C3—C4	120.32 (17)	N2—C9—C8	126.19 (12)
C8—C3—H3	119.8	C10—C11—N2	107.50 (13)
C4—C3—H3	119.8	C10—C11—H11	126.3
C5—C4—C3	120.15 (18)	N2—C11—H11	126.3
C5—C4—H4	119.9	C11—C10—N1	106.80 (13)
C3—C4—H4	119.9	C11—C10—H10	126.6
C6—C5—C4	119.83 (17)	N1—C10—H10	126.6
C6—C5—H5	120.1	C9—N1—C10	110.07 (11)
C4—C5—H5	120.1	C9—N1—H1	125.0
C5—C6—C7	120.91 (17)	C10—N1—H1	125.0
C5—C6—H6	119.5	C9—N2—C11	109.30 (12)
C7—C6—H6	119.5	C9—N2—H2	125.3
C6—C7—C8	120.00 (16)	C11—N2—H2	125.3
C6—C7—H7	120.0	C2—O2—H2A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O4^{i}	0.82	1.76	2.5793 (19)	172
N2—H2···O3^{ii}	0.86	1.90	2.732 (2)	164
N1—H1···O4^{i}	0.86	1.93	2.777 (2)	169

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