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2-Phenyl-1*H*-imidazol-3-ium hydrogen fumarate–fumaric acid (2/1)

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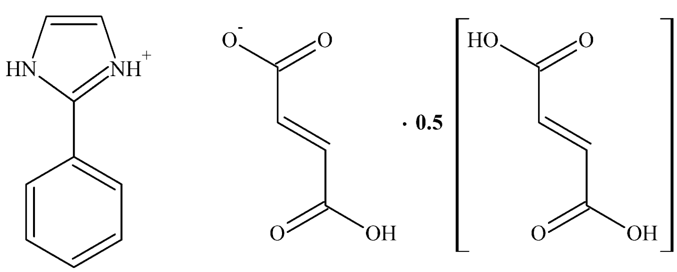
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.091; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound, $C_9H_9N_2^+\cdot C_4H_3O_4^-\cdot 0.5C_4H_4O_4$, consists of one 2-phenylimidazolium cation, one hydrogen fumarate anion and half a fumaric acid molecule, which lies on an inversion center. N—H···O and O—H···O hydrogen bonds connect the cations, anions and fumaric acid molecules into sheets parallel to the (102) plane.

Related literature

For similar structures, see: Jiang (2009); Song (2011); Xia & Yao (2010).



Experimental

Crystal data

$C_9H_9N_2^+\cdot C_4H_3O_4^-\cdot 0.5C_4H_4O_4$
M_r = 318.28
Monoclinic, $P2_1/c$
a = 9.572 (3) Å
b = 19.276 (4) Å
c = 8.289 (5) Å
β = 106.480 (3)°
V = 1466.6 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.18 × 0.15 × 0.14 mm

Data collection

Oxford Diffraction Gemini R Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.980, T_{max} = 0.984
11042 measured reflections
2675 independent reflections
1963 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.091
S = 1.00
2675 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.82	1.75	2.5721 (14)	176
O5—H5A···O6^{i}	0.82	1.83	2.6360 (14)	169
N1—H1A···O2	0.86	1.98	2.7903 (15)	157
N2—H2A···O1^{ii}	0.86	1.86	2.7106 (14)	168

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2037).

References

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supporting information

Acta Cryst. (2012). E**68**, o1021 [https://doi.org/10.1107/S1600536812005557]

2-Phenyl-1*H*-imidazol-3-ium hydrogen fumarate–fumaric acid (2/1)

Hong Guo, Xin Zhang and Jian-Xia Li

S1. Comment

Noncovalent hydrogen-bonding interactions have attracted great interest for chemists because of their physicochemical properties (Jiang, 2009). 2-Phenylimidazole can be used to form various supramolecular architectures with anions such as acetate (Xia & Yao, 2010) and hydrogen oxalate (Song, 2011). In this work, we report the crystal structure of the title compound prepared from 2-phenylimidazole and fumaric acid.

The asymmetric unit of the title compound contains one 2-phenylimidazolium cation, one hydrogen fumarate anion and a half of fumaric acid (Fig. 1). The fumaric acid molecule lies on an inversion center. N—H···O and O—H···O hydrogen bonds connect the cations, anions and fumaric acid molecules into sheets parallel to the (1 0 2) plane (Fig. 2, Table 1).

S2. Experimental

2-Phenylimidazole (2 mmol), fumaric acid (1 mmol) and ethanol (15 ml) were mixed. Colorless crystals were obtained by slow evaporation of the solution at room temperature after three days.

S3. Refinement

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93, N—H = 0.86 and O—H = 0.82 Å and with $U_{iso}(H) = 1.2U_{eq}(C, N)$ or $1.5U_{eq}(O)$.

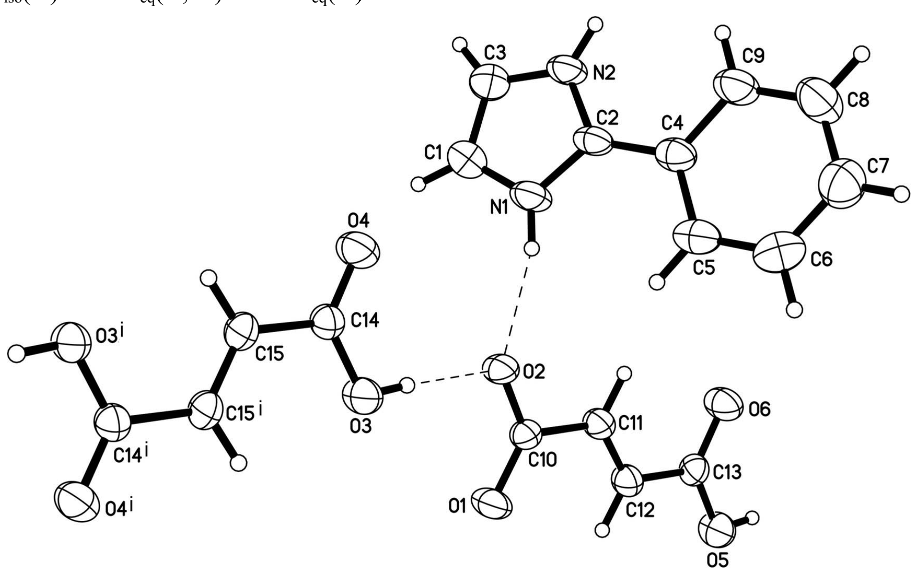

Figure 1

The asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level. Dashed lines denote hydrogen bonds. [Symmetry code: (i) -*x*, 1-*y*, 1-*z*.]

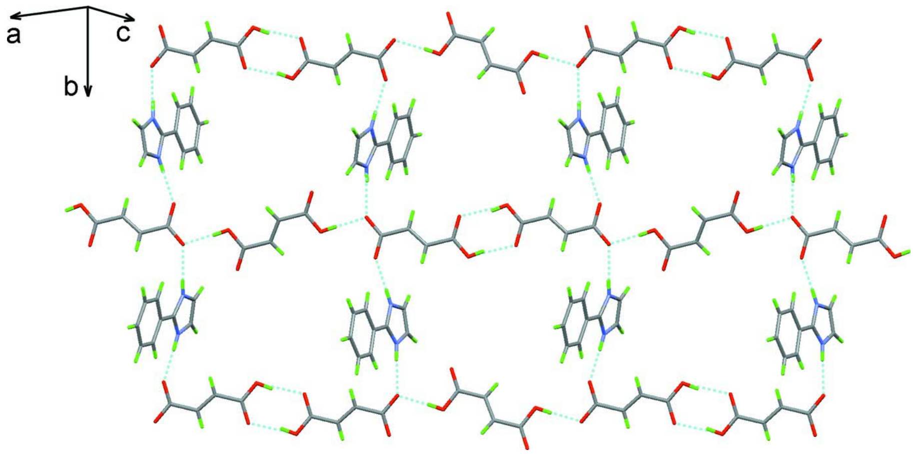

Figure 2

The two-dimensional network connected by hydrogen bonds (dashed lines).

2-Phenyl-1*H*-imidazol-3-ium 3-carboxyprop-2-enoate–fumaric acid (2/1)

Crystal data

$C_9H_9N_2^+\cdot C_4H_3O_4^-\cdot 0.5C_4H_4O_4$
M_r = 318.28
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.572 (3) Å
b = 19.276 (4) Å
c = 8.289 (5) Å
β = 106.480 (3)°
V = 1466.6 (10) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2675 reflections
θ = 2.2–25.3°
μ = 0.11 mm^{-1}
T = 293 K
Block, colorless
0.18 × 0.15 × 0.14 mm

Data collection

Oxford Diffraction Gemini R Ultra 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} = 0.980, T_{max} = 0.984
11042 measured reflections
2675 independent reflections
1963 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 25.3°, θ_{min} = 2.2°
h = −11→11
k = 0→23
l = 0→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.091
S = 1.00
2675 reflections
209 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
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0068 (12)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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	0.30636 (17)	0.70313 (7)	0.15392 (19)	0.0466 (4)
H1	0.2295	0.6753	0.0948	0.056*
C2	0.51445 (15)	0.73599 (6)	0.33290 (17)	0.0339 (3)
C3	0.31826 (17)	0.77144 (7)	0.1375 (2)	0.0458 (4)
H3	0.2516	0.8002	0.0642	0.055*
C4	0.65236 (15)	0.73529 (6)	0.46476 (17)	0.0361 (3)
C5	0.71012 (18)	0.67306 (7)	0.53922 (19)	0.0441 (4)
H5	0.6609	0.6317	0.5041	0.053*
C6	0.83936 (19)	0.67245 (8)	0.6642 (2)	0.0531 (4)
H6	0.8772	0.6306	0.7138	0.064*
C7	0.91375 (19)	0.73335 (8)	0.7170 (2)	0.0559 (4)
H7	1.0020	0.7325	0.8011	0.067*
C8	0.85745 (19)	0.79517 (8)	0.6455 (2)	0.0554 (4)
H8	0.9075	0.8363	0.6817	0.067*
C9	0.72735 (18)	0.79677 (7)	0.52033 (19)	0.0464 (4)
H9	0.6894	0.8389	0.4728	0.056*
C10	0.50312 (14)	0.48691 (6)	0.25178 (15)	0.0284 (3)
C11	0.63137 (14)	0.50545 (6)	0.19048 (15)	0.0316 (3)
H11	0.6584	0.5519	0.1930	0.038*
C12	0.70748 (14)	0.46016 (7)	0.13372 (15)	0.0329 (3)
H12	0.6801	0.4138	0.1308	0.039*
C13	0.83360 (14)	0.47805 (7)	0.07425 (15)	0.0299 (3)
C14	0.13846 (14)	0.54984 (6)	0.41335 (15)	0.0306 (3)
C15	0.01299 (14)	0.53111 (7)	0.47557 (15)	0.0325 (3)
H15	−0.0525	0.5661	0.4804	0.039*
N1	0.42847 (13)	0.68174 (5)	0.27399 (15)	0.0414 (3)
H1A	0.4472	0.6396	0.3068	0.050*
N2	0.44703 (13)	0.79138 (5)	0.24890 (14)	0.0389 (3)
H2A	0.4796	0.8332	0.2627	0.047*
O1	0.46797 (11)	0.42536 (4)	0.25506 (12)	0.0448 (3)
O2	0.43619 (9)	0.53724 (4)	0.29443 (11)	0.0356 (2)
O3	0.21778 (10)	0.49744 (5)	0.39383 (12)	0.0462 (3)
H3A	0.2850	0.5114	0.3593	0.055*
O4	0.16258 (11)	0.60952 (5)	0.38435 (13)	0.0482 (3)
O5	0.90474 (11)	0.42773 (5)	0.03852 (12)	0.0447 (3)

H5A	0.9724	0.4427	0.0065	0.054*
O6	0.86586 (11)	0.54051 (5)	0.06280 (12)	0.0453 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0451 (10)	0.0360 (8)	0.0582 (9)	−0.0057 (7)	0.0139 (8)	−0.0014 (7)
C2	0.0424 (9)	0.0209 (6)	0.0464 (8)	−0.0019 (6)	0.0256 (7)	0.0009 (6)
C3	0.0454 (10)	0.0354 (8)	0.0562 (9)	0.0006 (6)	0.0137 (8)	0.0061 (7)
C4	0.0423 (9)	0.0277 (7)	0.0453 (8)	−0.0013 (6)	0.0241 (7)	0.0002 (6)
C5	0.0561 (11)	0.0288 (7)	0.0516 (9)	0.0015 (6)	0.0221 (8)	0.0008 (6)
C6	0.0614 (12)	0.0431 (9)	0.0549 (10)	0.0113 (8)	0.0165 (9)	0.0079 (7)
C7	0.0523 (11)	0.0583 (11)	0.0544 (10)	0.0015 (8)	0.0106 (8)	0.0029 (8)
C8	0.0592 (12)	0.0439 (9)	0.0607 (10)	−0.0144 (8)	0.0131 (9)	−0.0017 (8)
C9	0.0550 (11)	0.0289 (8)	0.0565 (9)	−0.0039 (7)	0.0176 (8)	0.0036 (7)
C10	0.0279 (7)	0.0247 (7)	0.0349 (7)	0.0016 (5)	0.0125 (6)	0.0001 (5)
C11	0.0305 (8)	0.0263 (6)	0.0411 (8)	−0.0017 (5)	0.0150 (6)	−0.0001 (5)
C12	0.0300 (8)	0.0292 (7)	0.0428 (8)	−0.0025 (5)	0.0156 (6)	−0.0018 (6)
C13	0.0266 (7)	0.0314 (7)	0.0332 (7)	0.0007 (6)	0.0109 (6)	−0.0024 (5)
C14	0.0297 (7)	0.0290 (7)	0.0351 (7)	−0.0016 (6)	0.0122 (6)	−0.0021 (5)
C15	0.0279 (7)	0.0335 (6)	0.0394 (7)	0.0011 (6)	0.0150 (6)	−0.0023 (6)
N1	0.0488 (8)	0.0220 (6)	0.0566 (8)	−0.0036 (5)	0.0202 (6)	0.0024 (5)
N2	0.0456 (8)	0.0211 (5)	0.0543 (7)	−0.0024 (5)	0.0211 (6)	0.0033 (5)
O1	0.0463 (6)	0.0222 (5)	0.0781 (7)	−0.0021 (4)	0.0375 (6)	−0.0028 (4)
O2	0.0352 (6)	0.0235 (5)	0.0566 (6)	0.0021 (4)	0.0271 (5)	−0.0006 (4)
O3	0.0433 (6)	0.0322 (5)	0.0782 (7)	0.0002 (4)	0.0418 (6)	0.0038 (5)
O4	0.0498 (7)	0.0303 (6)	0.0741 (7)	−0.0033 (4)	0.0329 (6)	0.0027 (5)
O5	0.0381 (6)	0.0384 (6)	0.0694 (7)	0.0005 (4)	0.0343 (5)	−0.0041 (5)
O6	0.0446 (6)	0.0320 (6)	0.0692 (7)	−0.0033 (4)	0.0324 (5)	−0.0017 (5)

Geometric parameters (Å, º)

C1—C3	1.332 (2)	C10—O1	1.2357 (14)
C1—N1	1.3663 (19)	C10—O2	1.2669 (15)
C1—H1	0.9300	C10—C11	1.4989 (18)
C2—N1	1.3346 (16)	C11—C12	1.3072 (18)
C2—N2	1.3366 (17)	C11—H11	0.9300
C2—C4	1.456 (2)	C12—C13	1.4686 (18)
C3—N2	1.3687 (19)	C12—H12	0.9300
C3—H3	0.9300	C13—O6	1.2532 (15)
C4—C5	1.3899 (19)	C13—O5	1.2676 (15)
C4—C9	1.3944 (19)	C14—O4	1.2108 (15)
C5—C6	1.370 (2)	C14—O3	1.3009 (15)
C5—H5	0.9300	C14—C15	1.4799 (18)
C6—C7	1.378 (2)	C15—C15^{i}	1.312 (3)
C6—H6	0.9300	C15—H15	0.9300
C7—C8	1.372 (2)	N1—H1A	0.8592
C7—H7	0.9300	N2—H2A	0.8602

C8—C9	1.377 (2)	O3—H3A	0.8203
C8—H8	0.9300	O5—H5A	0.8193
C9—H9	0.9300		
C3—C1—N1	107.00 (13)	O1—C10—O2	124.52 (12)
C3—C1—H1	126.5	O1—C10—C11	119.39 (11)
N1—C1—H1	126.5	O2—C10—C11	116.08 (11)
N1—C2—N2	106.19 (12)	C12—C11—C10	123.81 (12)
N1—C2—C4	126.96 (11)	C12—C11—H11	118.1
N2—C2—C4	126.84 (11)	C10—C11—H11	118.1
C1—C3—N2	107.20 (13)	C11—C12—C13	124.00 (12)
C1—C3—H3	126.4	C11—C12—H12	118.0
N2—C3—H3	126.4	C13—C12—H12	118.0
C5—C4—C9	118.96 (14)	O6—C13—O5	123.87 (12)
C5—C4—C2	120.21 (12)	O6—C13—C12	119.64 (11)
C9—C4—C2	120.82 (12)	O5—C13—C12	116.49 (11)
C6—C5—C4	120.22 (13)	O4—C14—O3	124.20 (12)
C6—C5—H5	119.9	O4—C14—C15	121.35 (12)
C4—C5—H5	119.9	O3—C14—C15	114.45 (11)
C5—C6—C7	120.47 (14)	C15^{i}—C15—C14	125.05 (16)
C5—C6—H6	119.8	C15^{i}—C15—H15	117.5
C7—C6—H6	119.8	C14—C15—H15	117.5
C8—C7—C6	119.90 (16)	C2—N1—C1	109.96 (11)
C8—C7—H7	120.0	C2—N1—H1A	125.0
C6—C7—H7	120.0	C1—N1—H1A	125.1
C7—C8—C9	120.39 (14)	C2—N2—C3	109.65 (11)
C7—C8—H8	119.8	C2—N2—H2A	125.2
C9—C8—H8	119.8	C3—N2—H2A	125.2
C8—C9—C4	120.05 (13)	C14—O3—H3A	109.4
C8—C9—H9	120.0	C13—O5—H5A	109.5
C4—C9—H9	120.0		
N1—C1—C3—N2	−0.55 (17)	O1—C10—C11—C12	2.4 (2)
N1—C2—C4—C5	2.4 (2)	O2—C10—C11—C12	−176.38 (12)
N2—C2—C4—C5	−179.07 (12)	C10—C11—C12—C13	−179.71 (11)
N1—C2—C4—C9	−176.51 (13)	C11—C12—C13—O6	−6.1 (2)
N2—C2—C4—C9	2.0 (2)	C11—C12—C13—O5	173.31 (12)
C9—C4—C5—C6	−0.5 (2)	O4—C14—C15—C15^{i}	−173.42 (16)
C2—C4—C5—C6	−179.49 (13)	O3—C14—C15—C15^{i}	6.5 (2)
C4—C5—C6—C7	−0.3 (2)	N2—C2—N1—C1	−0.52 (15)
C5—C6—C7—C8	0.7 (2)	C4—C2—N1—C1	178.24 (13)
C6—C7—C8—C9	−0.4 (3)	C3—C1—N1—C2	0.67 (17)
C7—C8—C9—C4	−0.4 (2)	N1—C2—N2—C3	0.17 (15)
C5—C4—C9—C8	0.9 (2)	C4—C2—N2—C3	−178.59 (12)
C2—C4—C9—C8	179.82 (13)	C1—C3—N2—C2	0.24 (16)

Symmetry code: (i) $-x$, $-y+1$, $-z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.82	1.75	2.5721 (14)	176
O5—H5*A*···O6^{ii}	0.82	1.83	2.6360 (14)	169
N1—H1*A*···O2	0.86	1.98	2.7903 (15)	157
N2—H2*A*···O1^{iii}	0.86	1.86	2.7106 (14)	168

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