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## Structure Reports

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## 2-Phenylimidazolium hemi(benzene-1,3-dicarboxylate) monohydrate

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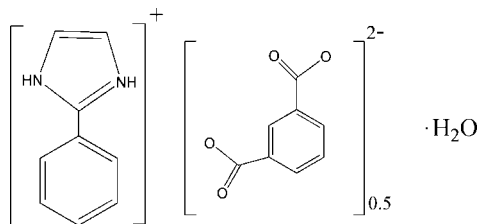
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}–\text{C}) = 0.004$  Å;  $R$  factor = 0.067;  $wR$  factor = 0.194; data-to-parameter ratio = 14.0.

The asymmetric unit of the title compound,  $\text{C}_9\text{H}_9\text{N}_2^+ \cdot 0.5\text{C}_8\text{H}_4\text{O}_4^- \cdot \text{H}_2\text{O}$ , contains one 2-phenylimidazolium cation, half a benzene-1,3-dicarboxylate anion and one water molecule. In the crystal, components are connected by  $\text{N}–\text{H} \cdots \text{O}$  and  $\text{O}–\text{H} \cdots \text{O}$  hydrogen-bonding interactions into a three-dimensional network.

### Related literature

For related 2-phenylimidazolium structures, see: Xia *et al.* (2009); Zhang *et al.* (2007).



### Experimental

#### Crystal data

 $\text{C}_9\text{H}_9\text{N}_2^+ \cdot 0.5\text{C}_8\text{H}_4\text{O}_4^- \cdot \text{H}_2\text{O}$ 
 $M_r = 245.25$ 

 Monoclinic,  $C2/c$ 
 $a = 17.092$  (5) Å

 $b = 7.152$  (4) Å

 $c = 20.322$  (5) Å

 $\beta = 108.449$  (3)°

 $V = 2356.5$  (16) Å<sup>3</sup>
 $Z = 8$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.10$  mm<sup>-1</sup>
 $T = 293$  K

 $0.23 \times 0.19 \times 0.18$  mm

#### Data collection

Oxford Diffraction Gemini R Ultra diffractometer

Absorption correction: multi-scan

 (*CrysAlis RED*; Oxford

Diffraction, 2006)

 $T_{\min} = 0.57$ ,  $T_{\max} = 0.74$ 

4898 measured reflections

2400 independent reflections

 1762 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.052$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.067$ 
 $wR(F^2) = 0.194$ 
 $S = 1.10$ 

2400 reflections

172 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.42$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.38$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D–H \cdots A$	$D–H$	$H \cdots A$	$D \cdots A$	$D–H \cdots A$
$\text{N1}–\text{H1} \cdots \text{O1W}$	0.86	1.88	2.725 (3)	169
$\text{N2}–\text{H2} \cdots \text{O2}^{\text{i}}$	0.86	1.89	2.731 (3)	167
$\text{O1W}–\text{HW11} \cdots \text{O1}$	0.85 (1)	1.92 (1)	2.718 (3)	156 (3)
$\text{O1W}–\text{HW12} \cdots \text{O2}^{\text{ii}}$	0.85 (1)	2.01 (1)	2.858 (2)	176 (3)

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

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); software used to prepare material for publication: *SHELXTL*.

We thank the China–Japan Union Hospital for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5571).

### References

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## supporting information

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## 2-Phenylimidazolium hemi(benzene-1,3-dicarboxylate) monohydrate

Wen-Yu Zhang, Zhi-Hua Zhu and Jian-Shi Du

### S1. Comment

The 2-phenylimidazole easily forms supramolecular structures with anions. The 2-phenylimidazolium nitrate structure has been reported as a hydrate (Xia *et al.*, 2009) and a hemihydrate (Zhang *et al.*, 2007). In this work, we report the synthesis and structure of the hemi-benzene-1,3-dicarboxylate hydrate, namely  $C_9H_9N_2^+ \cdot 0.5(C_8O_4H_4) \cdot H_2O$ .

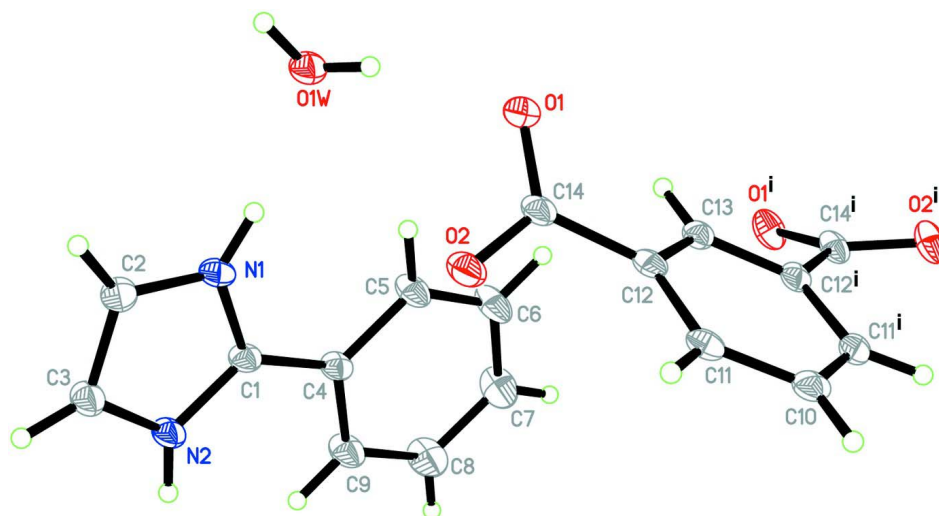
The asymmetric unit of the title compound consists of one 2-phenylimidazolium cation and half a benzene-1,3-dicarboxylate, and one water molecule (Fig. 1). In the structure, there exist N—H $\cdots$ O and O—H $\cdots$ O hydrogen-bonding interactions (Table I).

### S2. Experimental

A mixture of 2-phenylimidazole (0.5 mmol), benzene-1,3-dicarboxylic acid (0.5 mmol) and H<sub>2</sub>O (5 ml) was mixed. After seven days, colorless crystals were obtained at room temperature.

### S3. Refinement

All H atoms on C and N atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93 Å) and refined as riding, with  $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ . H atoms of the water molecules were located in a difference Fourier map and refined as riding with the O—H and H $\cdots$ H distance restraints of  $0.85 \pm 0.01$  and  $1.35 \pm 0.01$  Å, respectively.



**Figure 1**

The structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (i)  $-x, y, 0.5 - z$ .

## 2-Phenylimidazolium hemi(benzene-1,3-dicarboxylate) monohydrate

## Crystal data

C<sub>9</sub>H<sub>9</sub>N<sub>2</sub><sup>+</sup>·0.5C<sub>8</sub>H<sub>4</sub>O<sub>4</sub><sup>-</sup>·H<sub>2</sub>O $M_r = 245.25$ Monoclinic,  $C2/c$ Hall symbol:  $-C\ 2yc$  $a = 17.092\ (5)\ \text{\AA}$  $b = 7.152\ (4)\ \text{\AA}$  $c = 20.322\ (5)\ \text{\AA}$  $\beta = 108.449\ (3)^\circ$  $V = 2356.5\ (16)\ \text{\AA}^3$  $Z = 8$  $F(000) = 1032$  $D_x = 1.383\ \text{Mg m}^{-3}$ Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$ 

Cell parameters from 2400 reflections

 $\theta = 2.1\text{--}26.4^\circ$  $\mu = 0.10\ \text{mm}^{-1}$  $T = 293\ \text{K}$ 

Block, colorless

 $0.23 \times 0.19 \times 0.18\ \text{mm}$ 

## Data collection

Oxford Diffraction Gemini R Ultra  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $10.0\ \text{pixels mm}^{-1}$  $\omega$  scans

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2006)

 $T_{\min} = 0.57$ ,  $T_{\max} = 0.74$ 

4898 measured reflections

2400 independent reflections

1762 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.052$  $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.1^\circ$  $h = -20 \rightarrow 21$  $k = -7 \rightarrow 8$  $l = -25 \rightarrow 18$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.067$  $wR(F^2) = 0.194$  $S = 1.10$ 

2400 reflections

172 parameters

3 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0998P)^2 + 0.3011P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.42\ \text{e \AA}^{-3}$  $\Delta\rho_{\min} = -0.38\ \text{e \AA}^{-3}$ 

## 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.24852 (14)	0.4967 (3)	0.50929 (13)	0.0382 (5)
C2	0.20650 (15)	0.4816 (4)	0.60149 (13)	0.0467 (6)
H2A	0.1734	0.4629	0.6294	0.056*

C3	0.28571 (15)	0.5424 (4)	0.62248 (13)	0.0486 (7)
H3	0.3173	0.5726	0.6676	0.058*
C4	0.24990 (14)	0.4906 (3)	0.43856 (12)	0.0382 (6)
C5	0.17948 (15)	0.4488 (4)	0.38343 (13)	0.0492 (7)
H5	0.1305	0.4193	0.3919	0.059*
C6	0.18210 (17)	0.4509 (4)	0.31638 (14)	0.0604 (8)
H6	0.1348	0.4238	0.2797	0.072*
C7	0.2544 (2)	0.4929 (5)	0.30332 (16)	0.0674 (9)
H7	0.2561	0.4949	0.2580	0.081*
C8	0.32373 (19)	0.5315 (5)	0.35745 (16)	0.0677 (9)
H8	0.3726	0.5589	0.3485	0.081*
C9	0.32284 (16)	0.5307 (4)	0.42428 (14)	0.0538 (7)
H9	0.3708	0.5569	0.4604	0.065*
C10	0.0000	1.1129 (5)	0.2500	0.0448 (8)
H10	0.0000	1.2429	0.2500	0.054*
C11	0.00630 (13)	1.0179 (3)	0.31049 (12)	0.0408 (6)
H11	0.0122	1.0838	0.3512	0.049*
C12	0.00388 (12)	0.8237 (3)	0.31058 (11)	0.0363 (5)
C13	0.0000	0.7293 (4)	0.2500	0.0368 (7)
H13	0.0000	0.5993	0.2500	0.044*
C14	0.00529 (13)	0.7151 (4)	0.37391 (12)	0.0421 (6)
N1	0.18476 (11)	0.4532 (3)	0.53205 (10)	0.0417 (5)
H1	0.1374	0.4135	0.5064	0.050*
N2	0.31015 (12)	0.5509 (3)	0.56483 (10)	0.0427 (5)
H2	0.3581	0.5859	0.5643	0.051*
O1	-0.03238 (11)	0.5623 (2)	0.36545 (9)	0.0539 (5)
O2	0.04493 (10)	0.7828 (3)	0.43224 (8)	0.0541 (5)
O1W	0.03320 (10)	0.3051 (3)	0.46633 (9)	0.0501 (5)
HW11	0.0013 (19)	0.383 (4)	0.4395 (15)	0.114 (15)*
HW12	0.0109 (18)	0.273 (4)	0.4965 (12)	0.079 (10)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0242 (11)	0.0425 (12)	0.0480 (13)	-0.0003 (9)	0.0114 (10)	0.0017 (10)
C2	0.0323 (13)	0.0656 (16)	0.0466 (14)	-0.0023 (11)	0.0187 (11)	0.0042 (12)
C3	0.0337 (13)	0.0716 (18)	0.0409 (13)	-0.0074 (11)	0.0125 (11)	-0.0009 (12)
C4	0.0233 (11)	0.0438 (12)	0.0472 (13)	0.0031 (9)	0.0109 (10)	0.0003 (10)
C5	0.0250 (12)	0.0703 (17)	0.0496 (14)	0.0002 (11)	0.0082 (10)	-0.0061 (12)
C6	0.0316 (14)	0.093 (2)	0.0512 (15)	0.0030 (13)	0.0050 (12)	-0.0105 (14)
C7	0.0512 (18)	0.104 (2)	0.0505 (16)	0.0047 (16)	0.0209 (14)	-0.0061 (15)
C8	0.0388 (15)	0.111 (2)	0.0629 (18)	-0.0018 (15)	0.0302 (15)	-0.0041 (16)
C9	0.0282 (13)	0.0802 (19)	0.0537 (15)	-0.0025 (12)	0.0140 (12)	-0.0036 (13)
C10	0.0234 (15)	0.0411 (17)	0.066 (2)	0.000	0.0092 (15)	0.000
C11	0.0221 (11)	0.0498 (14)	0.0488 (14)	-0.0019 (9)	0.0086 (10)	-0.0085 (10)
C12	0.0191 (10)	0.0475 (13)	0.0419 (13)	-0.0031 (9)	0.0090 (9)	-0.0037 (10)
C13	0.0241 (15)	0.0393 (17)	0.0448 (17)	0.000	0.0076 (13)	0.000
C14	0.0221 (10)	0.0626 (15)	0.0414 (13)	-0.0059 (10)	0.0097 (9)	-0.0002 (11)

N1	0.0225 (10)	0.0525 (12)	0.0501 (12)	-0.0045 (8)	0.0117 (9)	0.0002 (9)
N2	0.0270 (10)	0.0589 (13)	0.0417 (11)	-0.0072 (9)	0.0101 (9)	-0.0014 (9)
O1	0.0406 (10)	0.0668 (12)	0.0494 (10)	-0.0176 (9)	0.0074 (8)	0.0070 (8)
O2	0.0379 (10)	0.0851 (13)	0.0381 (10)	-0.0188 (9)	0.0105 (8)	-0.0028 (8)
O1W	0.0341 (9)	0.0674 (13)	0.0502 (11)	-0.0059 (9)	0.0152 (9)	0.0019 (9)

*Geometric parameters (Å, °)*

C1—N2	1.335 (3)	C8—H8	0.9300
C1—N1	1.348 (3)	C9—H9	0.9300
C1—C4	1.446 (3)	C10—C11 <sup>i</sup>	1.378 (3)
C2—N1	1.356 (3)	C10—C11	1.378 (3)
C2—C3	1.356 (3)	C10—H10	0.9300
C2—H2A	0.9300	C11—C12	1.389 (3)
C3—N2	1.364 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.387 (3)
C4—C5	1.393 (3)	C12—C14	1.497 (3)
C4—C9	1.396 (3)	C13—C12 <sup>i</sup>	1.387 (3)
C5—C6	1.378 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—O1	1.252 (3)
C6—C7	1.377 (4)	C14—O2	1.261 (3)
C6—H6	0.9300	N1—H1	0.8600
C7—C8	1.365 (4)	N2—H2	0.8600
C7—H7	0.9300	O1W—HW11	0.848 (10)
C8—C9	1.363 (4)	O1W—HW12	0.848 (10)
N2—C1—N1	106.5 (2)	C8—C9—H9	120.0
N2—C1—C4	126.35 (19)	C4—C9—H9	120.0
N1—C1—C4	127.1 (2)	C11 <sup>i</sup> —C10—C11	121.0 (3)
N1—C2—C3	107.0 (2)	C11 <sup>i</sup> —C10—H10	119.5
N1—C2—H2A	126.5	C11—C10—H10	119.5
C3—C2—H2A	126.5	C10—C11—C12	120.0 (2)
C2—C3—N2	106.9 (2)	C10—C11—H11	120.0
C2—C3—H3	126.5	C12—C11—H11	120.0
N2—C3—H3	126.5	C13—C12—C11	118.6 (2)
C5—C4—C9	118.6 (2)	C13—C12—C14	119.6 (2)
C5—C4—C1	121.6 (2)	C11—C12—C14	121.9 (2)
C9—C4—C1	119.8 (2)	C12—C13—C12 <sup>i</sup>	121.7 (3)
C6—C5—C4	120.2 (2)	C12—C13—H13	119.1
C6—C5—H5	119.9	C12 <sup>i</sup> —C13—H13	119.1
C4—C5—H5	119.9	O1—C14—O2	124.4 (2)
C7—C6—C5	120.3 (3)	O1—C14—C12	117.9 (2)
C7—C6—H6	119.8	O2—C14—C12	117.8 (2)
C5—C6—H6	119.8	C1—N1—C2	109.7 (2)
C8—C7—C6	119.4 (3)	C1—N1—H1	125.1
C8—C7—H7	120.3	C2—N1—H1	125.1
C6—C7—H7	120.3	C1—N2—C3	109.82 (19)
C9—C8—C7	121.4 (3)	C1—N2—H2	125.1

C9—C8—H8	119.3	C3—N2—H2	125.1
C7—C8—H8	119.3	HW11—O1W—HW12	107.3 (16)
C8—C9—C4	120.0 (3)		

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

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...O1W	0.86	1.88	2.725 (3)	169
N2—H2...O2 <sup>ii</sup>	0.86	1.89	2.731 (3)	167
O1W—HW11...O1	0.85 (1)	1.92 (1)	2.718 (3)	156 (3)
O1W—HW12...O2 <sup>iii</sup>	0.85 (1)	2.01 (1)	2.858 (2)	176 (3)

Symmetry codes: (ii)  $-x+1/2, -y+3/2, -z+1$ ; (iii)  $-x, -y+1, -z+1$ .