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Diimidazolium phthalate monohydrate

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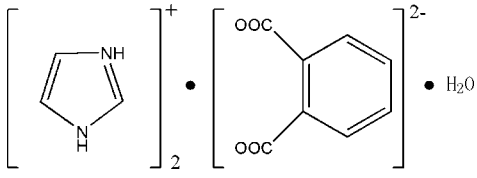
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.058; wR factor = 0.141; data-to-parameter ratio = 17.1.

In the title compound, $2C_3H_5N_2^+\cdot C_8H_4O_4^{2-}\cdot H_2O$, the cations, anion and water molecule are connected by N—H···O and O—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

The title compound was synthesized during a search for ferroelectric materials. For background to ferroelectric organic materials with framework structures, see: Zhang *et al.* (2009, 2010); Zhang & Xiong (2012). For related structures, see: Yu & Zhu (2012); Zhu & Yu (2011).



Experimental

Crystal data

$2C_3H_5N_2^+\cdot C_8H_4O_4^{2-}\cdot H_2O$
M_r = 320.31
Monoclinic, $P2_1/n$
a = 9.1250 (18) Å
b = 12.979 (3) Å
c = 13.549 (3) Å
β = 103.85 (3)°
V = 1558.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.32 × 0.28 × 0.26 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.967, T_{max} = 0.973
15753 measured reflections
3567 independent reflections
2758 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
3 standard reflections every 180 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.141
S = 1.10
3567 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.96	1.71	2.665 (2)	170
N2—H2A···O2^{i}	0.91	1.75	2.655 (2)	172
N3—H3A···O3	0.95	1.80	2.747 (2)	177
N3—H3A···O4	0.95	2.56	3.191 (2)	124
N4—H4A···O3^{ii}	0.86	1.93	2.774 (2)	168
O1W—H1WA···O4	0.86	1.98	2.834 (2)	170
O1W—H1WB···O2^{iii}	0.90	1.94	2.8326 (19)	172

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

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: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

The author thanks the Ordered Matter Science Research Center, Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2058).

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supporting information

Acta Cryst. (2012). E**68**, o2295 [https://doi.org/10.1107/S1600536812028619]

Diimidazolium phthalate monohydrate

Chua-Hua Yu

S1. Comment

In our search for potential ferroelectric phase change materials, the title compound was synthesized. This search is carried out by measurement of the dielectric constant of compounds on the basis of temperature, for eaxmple, (Zhang, Chen *et al.*, 2009; Zhang, Ye *et al.*, 2010; Zhang & Xiong, 2012), this has been carried out for $C_3H_5N_2^+.C_2HO_4^-$ (Yu & Zhu, 2012) and $C_5H_9N_2^+.C_8H_5O_4^-$ (Zhu & Yu, 2011). In the case of the title compound no dielectric anomaly was observed ranging from 130 K to 375 K.

The crystal structure of the title compound contains two protonated imidazolium cations, one phthalate anion, losing two H atoms, and one molecule of water molecule is shown in Fig. 1. The asymmetric unit was selected with the cations, anion and water molecule connected by the intramolecular hydrogen bonds, N1–1A···O1, N3–H3A···O3, N3–H3A···O4 and O1W–H1WA···O4 all of which connect the cations and the water molecule to the anion, Table 1. These units are connected by the intermolecular hydrogen bonds, N2–H2A···O2(-1+x,y,z), N4-H4A···O3(-x+1/2,y-1/2,-z+1/2) and O1W–H1WB···O2(-x+2,-y+1,-z+1), to form a three-dimensional network Table2 and Figure 1.

S2. Experimental

0.83 g (5 mmol) of phthalic acid was added to in 10 ml water which was heated. A few drops of ethanol and 0.68 g (10 mmol) imidazole were added to the solution. The mixture was stirreduntil it reached ambient temperature, the liquid was filtered to give a clear solution. Colourless crystals suitable for X-ray structure analysis were obtained by the slow evaporation of the solution after several days at the ambient temperature.

S3. Refinement

H atoms attached to C were placed in calculated positions (C—H = 0.93 Å for C_{sp2} atoms) while those attached to N and O were found in positions derived from a difference electron density map, with U_{iso}(H) = 1.2 U_{iso}(C, N), U_{iso}(H) = 1.5 U_{iso}(O). The -2 0 2 reflection was omitted since its measured value appear to be anomalous.

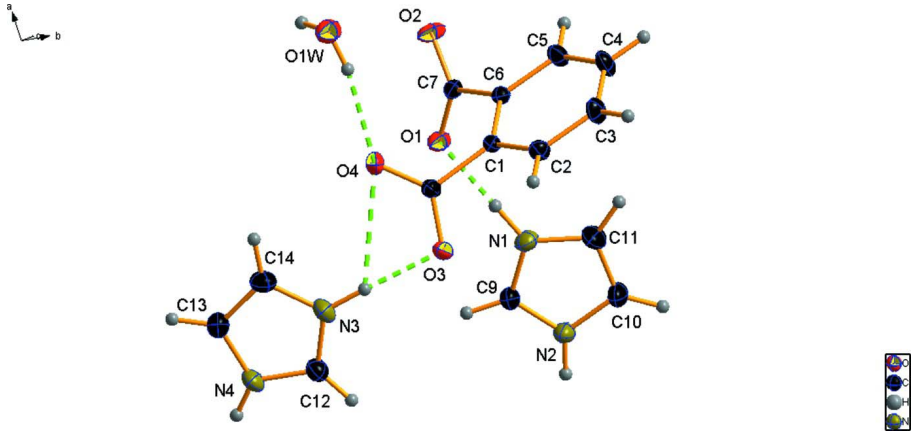

Figure 1

A diagram of the title compound, with displacement ellipsoids drawn at the 30% probability level. The atomic numbering scheme is shown.

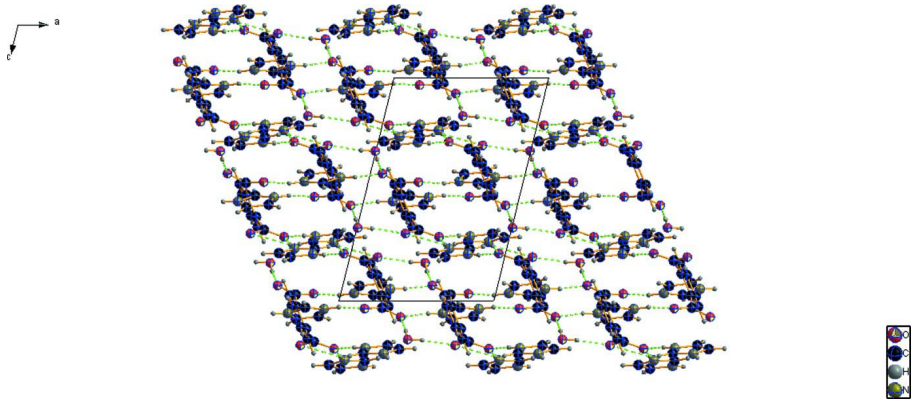

Figure 2

A view of the packing diagram of the title compound, stacking along the *b* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$2C_3H_5N_2^+·C_8H_4O_4^{2-}·H_2O$
M_r = 320.31
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.1250 (18) Å
b = 12.979 (3) Å
c = 13.549 (3) Å
β = 103.85 (3)°
V = 1558.0 (6) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3567 reflections
θ = 3.1–27.5°
μ = 0.11 mm^{-1}
T = 293 K
Block, colourless
0.32 × 0.28 × 0.26 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.967, T_{max} = 0.973
15753 measured reflections
3567 independent reflections
2758 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −11→11
k = −16→16
l = −17→17
3 standard reflections every 180 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.141
S = 1.10
3567 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.3812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 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}
O1	0.67631 (14)	0.59458 (9)	0.53305 (10)	0.0375 (3)
O2	0.92261 (14)	0.61977 (10)	0.57079 (11)	0.0423 (3)
O3	0.45616 (13)	0.66464 (9)	0.28714 (10)	0.0386 (3)
O4	0.67947 (15)	0.59102 (10)	0.30286 (12)	0.0466 (4)
C1	0.66990 (17)	0.75577 (12)	0.37728 (12)	0.0242 (3)
C2	0.64193 (19)	0.85209 (13)	0.33196 (13)	0.0310 (4)
H2	0.5809	0.8575	0.2665	0.037*
C3	0.7038 (2)	0.93997 (13)	0.38309 (14)	0.0385 (4)
H3	0.6833	1.0041	0.3523	0.046*
C4	0.7959 (2)	0.93247 (13)	0.47976 (15)	0.0417 (5)
H4	0.8372	0.9915	0.5144	0.050*
C5	0.8267 (2)	0.83681 (14)	0.52503 (13)	0.0353 (4)
H5	0.8898	0.8319	0.5899	0.042*
C6	0.76452 (17)	0.74796 (12)	0.47483 (12)	0.0254 (3)
C7	0.79001 (18)	0.64612 (13)	0.52933 (12)	0.0266 (4)

C8	0.59838 (19)	0.66201 (12)	0.31870 (12)	0.0275 (4)
N1	0.41475 (17)	0.68481 (12)	0.53530 (12)	0.0390 (4)
H1A	0.5080	0.6559	0.5268	0.047*
N2	0.18863 (17)	0.69028 (12)	0.55394 (12)	0.0375 (4)
H2A	0.0940	0.6703	0.5552	0.045*
C9	0.2907 (2)	0.63160 (14)	0.52810 (15)	0.0385 (4)
H9	0.2770	0.5631	0.5079	0.046*
C10	0.2510 (2)	0.78435 (16)	0.58006 (18)	0.0499 (5)
H10	0.2043	0.8406	0.6021	0.060*
C11	0.3926 (2)	0.78088 (16)	0.5680 (2)	0.0548 (6)
H11	0.4625	0.8344	0.5799	0.066*
N3	0.37146 (18)	0.46491 (12)	0.23556 (11)	0.0375 (4)
H3A	0.4030	0.5339	0.2521	0.045*
N4	0.24322 (18)	0.32527 (12)	0.20991 (12)	0.0403 (4)
H4A	0.1710	0.2820	0.2070	0.048*
C12	0.2391 (2)	0.42411 (14)	0.23263 (15)	0.0398 (4)
H12	0.1560	0.4590	0.2446	0.048*
C13	0.3841 (2)	0.30217 (16)	0.19825 (16)	0.0446 (5)
H13	0.4179	0.2383	0.1820	0.053*
C14	0.4641 (2)	0.39014 (16)	0.21490 (16)	0.0452 (5)
H14	0.5645	0.3983	0.2127	0.054*
O1W	0.99551 (16)	0.57033 (11)	0.32839 (11)	0.0479 (4)
H1WA	0.9020	0.5841	0.3250	0.072*
H1WB	1.0210	0.5131	0.3660	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0274 (6)	0.0290 (6)	0.0568 (8)	−0.0006 (5)	0.0116 (6)	0.0092 (6)
O2	0.0250 (6)	0.0430 (8)	0.0569 (8)	0.0060 (6)	0.0058 (6)	0.0199 (6)
O3	0.0273 (7)	0.0295 (7)	0.0526 (8)	−0.0025 (5)	−0.0032 (6)	−0.0047 (6)
O4	0.0372 (8)	0.0359 (7)	0.0664 (9)	0.0010 (6)	0.0118 (7)	−0.0221 (6)
C1	0.0216 (7)	0.0219 (8)	0.0291 (8)	−0.0012 (6)	0.0060 (6)	−0.0019 (6)
C2	0.0316 (9)	0.0288 (9)	0.0295 (9)	0.0003 (7)	0.0015 (7)	0.0029 (7)
C3	0.0500 (12)	0.0205 (8)	0.0423 (10)	−0.0005 (8)	0.0057 (8)	0.0041 (7)
C4	0.0529 (12)	0.0236 (9)	0.0434 (11)	−0.0073 (8)	0.0016 (9)	−0.0066 (7)
C5	0.0380 (10)	0.0323 (9)	0.0307 (9)	−0.0044 (8)	−0.0013 (7)	−0.0039 (7)
C6	0.0219 (8)	0.0242 (8)	0.0299 (8)	0.0000 (6)	0.0056 (6)	0.0003 (6)
C7	0.0257 (8)	0.0252 (8)	0.0293 (8)	0.0029 (7)	0.0075 (6)	0.0009 (6)
C8	0.0265 (8)	0.0255 (8)	0.0288 (8)	−0.0020 (7)	0.0037 (6)	−0.0015 (6)
N1	0.0283 (8)	0.0405 (9)	0.0500 (9)	0.0023 (7)	0.0129 (7)	0.0026 (7)
N2	0.0290 (8)	0.0320 (8)	0.0522 (9)	−0.0025 (6)	0.0113 (7)	−0.0003 (7)
C9	0.0351 (10)	0.0295 (9)	0.0501 (11)	0.0001 (8)	0.0083 (8)	−0.0045 (8)
C10	0.0416 (12)	0.0293 (10)	0.0811 (16)	−0.0004 (9)	0.0194 (11)	−0.0093 (10)
C11	0.0416 (12)	0.0357 (11)	0.0872 (17)	−0.0119 (9)	0.0155 (11)	−0.0056 (11)
N3	0.0434 (9)	0.0313 (8)	0.0373 (8)	−0.0099 (7)	0.0088 (7)	−0.0018 (6)
N4	0.0394 (9)	0.0317 (8)	0.0490 (10)	−0.0117 (7)	0.0088 (7)	−0.0040 (7)
C12	0.0403 (11)	0.0346 (10)	0.0458 (11)	−0.0065 (8)	0.0126 (9)	−0.0049 (8)

C13	0.0446 (11)	0.0375 (10)	0.0508 (12)	0.0022 (9)	0.0100 (9)	−0.0068 (9)
C14	0.0340 (10)	0.0504 (12)	0.0527 (12)	−0.0067 (9)	0.0131 (9)	−0.0013 (10)
O1W	0.0393 (8)	0.0419 (8)	0.0624 (10)	0.0004 (6)	0.0116 (7)	0.0079 (7)

Geometric parameters (Å, º)

O1—C7	1.246 (2)	N2—C9	1.314 (2)
O2—C7	1.253 (2)	N2—C10	1.358 (2)
O3—C8	1.266 (2)	N2—H2A	0.9055
O4—C8	1.232 (2)	C9—H9	0.9300
C1—C2	1.389 (2)	C10—C11	1.342 (3)
C1—C6	1.399 (2)	C10—H10	0.9300
C1—C8	1.513 (2)	C11—H11	0.9300
C2—C3	1.383 (2)	N3—C12	1.310 (2)
C2—H2	0.9300	N3—C14	1.360 (3)
C3—C4	1.380 (3)	N3—H3A	0.9507
C3—H3	0.9300	N4—C12	1.322 (2)
C4—C5	1.384 (3)	N4—C13	1.365 (3)
C4—H4	0.9300	N4—H4A	0.8591
C5—C6	1.389 (2)	C12—H12	0.9300
C5—H5	0.9300	C13—C14	1.345 (3)
C6—C7	1.505 (2)	C13—H13	0.9300
N1—C9	1.309 (2)	C14—H14	0.9300
N1—C11	1.355 (3)	O1W—H1WA	0.8623
N1—H1A	0.9615	O1W—H1WB	0.8997
C2—C1—C6	119.30 (14)	C9—N2—H2A	125.4
C2—C1—C8	118.78 (14)	C10—N2—H2A	126.5
C6—C1—C8	121.91 (14)	N1—C9—N2	109.25 (16)
C3—C2—C1	120.73 (15)	N1—C9—H9	125.4
C3—C2—H2	119.6	N2—C9—H9	125.4
C1—C2—H2	119.6	C11—C10—N2	107.09 (18)
C4—C3—C2	120.01 (16)	C11—C10—H10	126.5
C4—C3—H3	120.0	N2—C10—H10	126.5
C2—C3—H3	120.0	C10—C11—N1	107.11 (18)
C3—C4—C5	119.80 (16)	C10—C11—H11	126.4
C3—C4—H4	120.1	N1—C11—H11	126.4
C5—C4—H4	120.1	C12—N3—C14	108.62 (16)
C4—C5—C6	120.81 (16)	C12—N3—H3A	127.8
C4—C5—H5	119.6	C14—N3—H3A	123.5
C6—C5—H5	119.6	C12—N4—C13	108.66 (16)
C5—C6—C1	119.34 (15)	C12—N4—H4A	125.5
C5—C6—C7	119.47 (14)	C13—N4—H4A	125.7
C1—C6—C7	121.02 (14)	N3—C12—N4	108.79 (18)
O1—C7—O2	124.02 (15)	N3—C12—H12	125.6
O1—C7—C6	117.34 (14)	N4—C12—H12	125.6
O2—C7—C6	118.61 (14)	C14—C13—N4	106.43 (18)
O4—C8—O3	124.83 (15)	C14—C13—H13	126.8

O4—C8—C1	119.42 (15)	N4—C13—H13	126.8
O3—C8—C1	115.72 (14)	C13—C14—N3	107.50 (18)
C9—N1—C11	108.42 (17)	C13—C14—H14	126.2
C9—N1—H1A	124.0	N3—C14—H14	126.2
C11—N1—H1A	127.1	H1WA—O1W—H1WB	108.6
C9—N2—C10	108.12 (16)		

C6—C1—C2—C3	−1.4 (3)	C2—C1—C8—O4	122.83 (18)
C8—C1—C2—C3	179.38 (16)	C6—C1—C8—O4	−56.3 (2)
C1—C2—C3—C4	0.8 (3)	C2—C1—C8—O3	−55.5 (2)
C2—C3—C4—C5	0.3 (3)	C6—C1—C8—O3	125.37 (17)
C3—C4—C5—C6	−0.7 (3)	C11—N1—C9—N2	0.7 (2)
C4—C5—C6—C1	0.1 (3)	C10—N2—C9—N1	−0.9 (2)
C4—C5—C6—C7	−175.09 (17)	C9—N2—C10—C11	0.7 (3)
C2—C1—C6—C5	1.0 (2)	N2—C10—C11—N1	−0.3 (3)
C8—C1—C6—C5	−179.85 (16)	C9—N1—C11—C10	−0.2 (3)
C2—C1—C6—C7	176.10 (15)	C14—N3—C12—N4	−0.5 (2)
C8—C1—C6—C7	−4.7 (2)	C13—N4—C12—N3	0.2 (2)
C5—C6—C7—O1	125.27 (18)	C12—N4—C13—C14	0.1 (2)
C1—C6—C7—O1	−49.8 (2)	N4—C13—C14—N3	−0.4 (2)
C5—C6—C7—O2	−52.5 (2)	C12—N3—C14—C13	0.6 (2)
C1—C6—C7—O2	132.45 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1	0.96	1.71	2.665 (2)	170
N2—H2*A*···O2^{i}	0.91	1.75	2.655 (2)	172
N3—H3*A*···O3	0.95	1.80	2.747 (2)	177
N3—H3*A*···O4	0.95	2.56	3.191 (2)	124
N4—H4*A*···O3^{ii}	0.86	1.93	2.774 (2)	168
O1*W*—H1*WA*···O4	0.86	1.98	2.834 (2)	170
O1*W*—H1*WB*···O2^{iii}	0.90	1.94	2.8326 (19)	172

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