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1-(3-Carboxybenzyl)-1*H*-benzo[*d*]-imidazol-3-ium nitrate monohydrate

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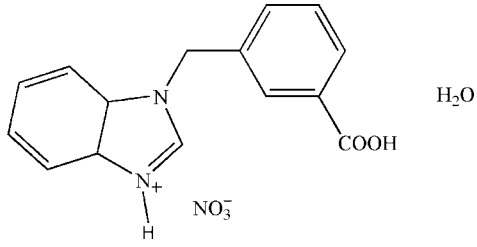
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.066; wR factor = 0.166; data-to-parameter ratio = 12.2.

In the crystal structure of the title compound, $C_{15}H_{13}N_2O_2^+\cdot NO_3^-\cdot H_2O$, O—H···O and N—H···O hydrogen bonds link the cations, the nitrate anions and the water molecules.



Experimental

Crystal data

$C_{15}H_{13}N_2O_2^+\cdot NO_3^-\cdot H_2O$
M_r = 333.30
Triclinic, $P\overline{1}$
a = 7.473 (2) Å
b = 7.974 (2) Å
c = 13.092 (4) Å
α = 80.201 (4)°
β = 87.226 (4)°
γ = 89.146 (4)°
V = 767.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.39 × 0.17 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
3255 measured reflections
2669 independent reflections
1892 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.166
S = 1.07
2669 reflections
218 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.86	1.91	2.735 (4)	159
N2—H2A···O5^{i}	0.86	2.44	3.168 (4)	143
N2—H2A···N3^{i}	0.86	2.51	3.352 (4)	168
O2—H2···O6	0.82	1.78	2.599 (3)	173
O6—H6A···O3^{ii}	0.86	1.91	2.775 (3)	175
O6—H6A···N3^{ii}	0.86	2.68	3.454 (4)	150
O6—H6B···O1^{ii}	0.85	1.94	2.773 (3)	167

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2033).

References

Bruker (2000). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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1-(3-Carboxybenzyl)-1*H*-benzo[*d*]imidazol-3-ium nitrate monohydrate

X. X. Zhao, J. P. Ma, R. Q. Huang and Y. B. Dong

Comment

The title compound was obtained by accident in the reaction of 2,5-bis(benzo[*d*]imidazol methylphenyl)-1,3,4-oxadiazol with nitric acid and its crystal structure was confirmed by X-ray diffraction. In the crystal structure the organic cations are connected *via* O—H···O hydrogen bonding between the carboxyl hydrogen atoms and the water molecules. The water molecules act also as donor for O—H···O hydrogen bonds to the carbonyl oxygen atoms and to the nitrate anions (Fig. 1 and Table 1). The N—H H atoms forms weak bifurcated hydrogen bond to the nitrate anions.

Experimental

2,5-bis(benzo[*d*]imidazol methylphenyl)-1,3,4-oxadiazol (48.2 mg, 0.10 mmol), 0.04 ml of concentrated nitric acid and 5 ml of water were seeled in a teflon-lined stainless steel autoclave, heated at 150 degree for 40 h and cooled down to room

Refinement

The C—H, N—H and carboxyl H atoms were placed in geometrically idealized positions (carboxyl allowed to rotate but not to tip) and were refined isotropic (U_{iso}(H) = 1.2U_{eq}(C, N)) or 1.5U_{eq}(O)) using a riding model with C—H 0.93 Å for aromatic, 0.970Å for methylene, 0.86Å for N—H and 0.82Å for O—H H atoms. The water H atoms were located in difference map and afterwards they were refined isotropic using a riding model.

Figures

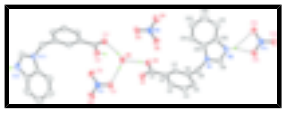

Fig. 1. Crystal structure of I with labelling and displacement ellipsoids drawn at the 30% probability level. Intermolecular hydrogen bonding is shown as dashed lines. Symmetry codes: (i):1 − *x*,-*y*,2 − *z*; (ii): −*x*,1 − *y*,1 − *z*)

1-(3-Carboxybenzyl)-1*H*-benzo[*d*]imidazol-3-ium nitrate monohydrate

Crystal data

$C_{15}H_{13}N_2O_2^+ \cdot NO_3^- \cdot H_2O$	$Z = 2$
$M_r = 333.30$	$F_{000} = 348$
Triclinic, $P\overline{1}$	$D_x = 1.442$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.473$ (2) Å	Cell parameters from 884 reflections
$b = 7.974$ (2) Å	$\theta = 2.6$–23.5°
$c = 13.092$ (4) Å	$\mu = 0.11$ mm^{-1}

α = 80.201 (4)°	T = 298 (2) K
β = 87.226 (4)°	Block, yellow
γ = 89.146 (4)°	0.39 × 0.17 × 0.16 mm
V = 767.8 (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1892 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.016
Monochromator: graphite	θ_{max} = 25.0°
T = 298(2) K	θ_{min} = 2.6°
φ and ω scans	$h = -6 \rightarrow 8$
Absorption correction: none	$k = -9 \rightarrow 9$
3255 measured reflections	$l = -14 \rightarrow 15$
2669 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.066	H-atom parameters constrained
$wR(F^2)$ = 0.166	$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.234P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
2669 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
218 parameters	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O5	0.0870 (4)	0.2889 (4)	0.7702 (2)	0.0833 (8)

O3	−0.0372 (4)	0.5364 (3)	0.7297 (2)	0.0791 (8)
C1	0.2502 (4)	0.2061 (4)	0.5525 (2)	0.0478 (8)
C2	0.3531 (4)	0.0568 (4)	0.6032 (2)	0.0418 (7)
C3	0.2819 (4)	−0.1032 (4)	0.6114 (2)	0.0489 (8)
H3	0.1680	−0.1158	0.5881	0.059*
C4	0.3772 (5)	−0.2447 (4)	0.6537 (3)	0.0571 (9)
H4	0.3288	−0.3528	0.6591	0.068*
C5	0.5468 (4)	−0.2241 (4)	0.6883 (2)	0.0511 (8)
H5	0.6122	−0.3194	0.7166	0.061*
C6	0.6202 (4)	−0.0643 (4)	0.6816 (2)	0.0415 (7)
C7	0.5228 (4)	0.0756 (4)	0.6377 (2)	0.0428 (7)
H7	0.5716	0.1837	0.6313	0.051*
C8	0.8047 (4)	−0.0442 (4)	0.7193 (2)	0.0495 (8)
H8A	0.8845	−0.1291	0.6966	0.059*
H8B	0.8506	0.0673	0.6889	0.059*
C9	0.8492 (4)	−0.2011 (4)	0.8964 (3)	0.0602 (9)
H9	0.8939	−0.2998	0.8753	0.072*
C10	0.7576 (4)	−0.0185 (5)	0.9952 (3)	0.0560 (9)
C11	0.7128 (5)	0.0675 (6)	1.0765 (3)	0.0736 (11)
H11	0.7212	0.0165	1.1455	0.088*
C12	0.6555 (5)	0.2319 (6)	1.0489 (3)	0.0853 (13)
H12	0.6264	0.2951	1.1010	0.102*
C13	0.6389 (5)	0.3082 (5)	0.9462 (3)	0.0795 (12)
H13	0.5981	0.4201	0.9313	0.095*
C14	0.6818 (5)	0.2214 (4)	0.8661 (3)	0.0613 (9)
H14	0.6705	0.2710	0.7971	0.074*
C15	0.7422 (4)	0.0575 (4)	0.8936 (2)	0.0461 (7)
N1	0.8026 (3)	−0.0630 (3)	0.83307 (19)	0.0473 (6)
N2	0.8239 (4)	−0.1808 (4)	0.9934 (2)	0.0656 (8)
H2A	0.8451	−0.2559	1.0469	0.079*
O1	0.1054 (3)	0.1933 (3)	0.51702 (19)	0.0648 (7)
O2	0.3318 (3)	0.3517 (3)	0.54908 (18)	0.0615 (6)
H2	0.2747	0.4280	0.5147	0.092*
O6	0.1746 (3)	0.6002 (3)	0.43115 (18)	0.0626 (6)
H6A	0.1330	0.5518	0.3831	0.094*
H6B	0.0926	0.6570	0.4572	0.094*
N3	0.0482 (4)	0.4312 (4)	0.7898 (2)	0.0619 (8)
O4	0.0916 (4)	0.4709 (3)	0.8734 (2)	0.0901 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.099 (2)	0.0782 (18)	0.0726 (19)	0.0321 (16)	−0.0032 (14)	−0.0163 (14)
O3	0.106 (2)	0.0669 (16)	0.0624 (17)	0.0138 (15)	−0.0301 (15)	−0.0006 (13)
C1	0.0491 (19)	0.058 (2)	0.0396 (18)	0.0042 (16)	−0.0025 (14)	−0.0176 (14)
C2	0.0449 (17)	0.0519 (18)	0.0310 (16)	0.0026 (14)	0.0022 (12)	−0.0153 (13)
C3	0.0479 (18)	0.059 (2)	0.0425 (18)	−0.0013 (16)	0.0006 (13)	−0.0169 (15)
C4	0.066 (2)	0.053 (2)	0.053 (2)	−0.0115 (17)	0.0020 (16)	−0.0144 (16)

C5	0.061 (2)	0.0510 (19)	0.0411 (18)	0.0056 (16)	−0.0007 (14)	−0.0087 (14)
C6	0.0446 (17)	0.0498 (17)	0.0298 (16)	0.0023 (14)	0.0020 (12)	−0.0075 (13)
C7	0.0450 (17)	0.0503 (17)	0.0342 (16)	−0.0015 (14)	0.0015 (12)	−0.0111 (13)
C8	0.0470 (18)	0.0559 (18)	0.0448 (19)	0.0058 (15)	0.0006 (13)	−0.0080 (14)
C9	0.056 (2)	0.061 (2)	0.061 (2)	0.0052 (17)	−0.0157 (16)	0.0001 (18)
C10	0.0477 (19)	0.075 (2)	0.045 (2)	−0.0176 (17)	−0.0078 (14)	−0.0072 (17)
C11	0.063 (2)	0.110 (3)	0.048 (2)	−0.026 (2)	−0.0002 (17)	−0.013 (2)
C12	0.080 (3)	0.114 (4)	0.071 (3)	−0.028 (3)	0.021 (2)	−0.047 (3)
C13	0.086 (3)	0.076 (3)	0.080 (3)	−0.008 (2)	0.020 (2)	−0.031 (2)
C14	0.066 (2)	0.061 (2)	0.056 (2)	−0.0068 (18)	0.0057 (16)	−0.0129 (17)
C15	0.0408 (17)	0.0532 (19)	0.0440 (18)	−0.0103 (14)	−0.0014 (13)	−0.0065 (14)
N1	0.0440 (15)	0.0502 (15)	0.0466 (16)	0.0010 (12)	−0.0090 (11)	−0.0031 (12)
N2	0.0603 (18)	0.074 (2)	0.056 (2)	−0.0014 (16)	−0.0205 (14)	0.0121 (15)
O1	0.0537 (14)	0.0676 (15)	0.0794 (17)	0.0101 (12)	−0.0249 (12)	−0.0254 (12)
O2	0.0622 (15)	0.0528 (14)	0.0696 (17)	0.0026 (12)	−0.0157 (11)	−0.0076 (12)
O6	0.0566 (13)	0.0565 (13)	0.0749 (17)	0.0027 (11)	−0.0119 (11)	−0.0098 (11)
N3	0.0667 (19)	0.065 (2)	0.0504 (19)	0.0015 (16)	−0.0042 (14)	−0.0002 (15)
O4	0.133 (2)	0.0772 (18)	0.0630 (18)	0.0008 (17)	−0.0435 (16)	−0.0091 (14)

Geometric parameters (Å, °)

O5—N3	1.232 (4)	C9—N1	1.314 (4)
O3—N3	1.240 (3)	C9—H9	0.9300
C1—O1	1.211 (3)	C10—C15	1.374 (4)
C1—O2	1.313 (3)	C10—N2	1.382 (4)
C1—C2	1.484 (4)	C10—C11	1.386 (5)
C2—C3	1.375 (4)	C11—C12	1.367 (5)
C2—C7	1.386 (4)	C11—H11	0.9300
C3—C4	1.375 (4)	C12—C13	1.389 (6)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.387 (4)	C13—C14	1.375 (5)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.382 (4)	C14—C15	1.371 (4)
C5—H5	0.9300	C14—H14	0.9300
C6—C7	1.379 (4)	C15—N1	1.402 (4)
C6—C8	1.507 (4)	N2—H2A	0.8600
C7—H7	0.9300	O2—H2	0.8200
C8—N1	1.471 (4)	O6—H6A	0.8642
C8—H8A	0.9700	O6—H6B	0.8499
C8—H8B	0.9700	N3—O4	1.249 (4)
C9—N2	1.312 (4)		
O1—C1—O2	123.6 (3)	C15—C10—N2	106.6 (3)
O1—C1—C2	122.7 (3)	C15—C10—C11	121.5 (4)
O2—C1—C2	113.7 (3)	N2—C10—C11	131.9 (3)
C3—C2—C7	119.7 (3)	C12—C11—C10	115.8 (4)
C3—C2—C1	119.2 (3)	C12—C11—H11	122.1
C7—C2—C1	121.0 (3)	C10—C11—H11	122.1
C4—C3—C2	120.6 (3)	C11—C12—C13	122.6 (4)
C4—C3—H3	119.7	C11—C12—H12	118.7

C2—C3—H3	119.7	C13—C12—H12	118.7
C3—C4—C5	119.1 (3)	C14—C13—C12	121.2 (4)
C3—C4—H4	120.5	C14—C13—H13	119.4
C5—C4—H4	120.5	C12—C13—H13	119.4
C6—C5—C4	121.2 (3)	C15—C14—C13	116.3 (4)
C6—C5—H5	119.4	C15—C14—H14	121.9
C4—C5—H5	119.4	C13—C14—H14	121.9
C7—C6—C5	118.7 (3)	C14—C15—C10	122.6 (3)
C7—C6—C8	120.8 (3)	C14—C15—N1	131.2 (3)
C5—C6—C8	120.5 (3)	C10—C15—N1	106.2 (3)
C6—C7—C2	120.7 (3)	C9—N1—C15	107.8 (3)
C6—C7—H7	119.6	C9—N1—C8	125.4 (3)
C2—C7—H7	119.6	C15—N1—C8	126.8 (2)
N1—C8—C6	111.5 (2)	C9—N2—C10	108.6 (3)
N1—C8—H8A	109.3	C9—N2—H2A	125.7
C6—C8—H8A	109.3	C10—N2—H2A	125.7
N1—C8—H8B	109.3	C1—O2—H2	109.5
C6—C8—H8B	109.3	H6A—O6—H6B	110.3
H8A—C8—H8B	108.0	O5—N3—O3	121.9 (3)
N2—C9—N1	110.8 (3)	O5—N3—O4	119.7 (3)
N2—C9—H9	124.6	O3—N3—O4	118.3 (3)
N1—C9—H9	124.6		
O1—C1—C2—C3	−0.5 (4)	C11—C12—C13—C14	−0.7 (6)
O2—C1—C2—C3	−179.9 (2)	C12—C13—C14—C15	−0.4 (5)
O1—C1—C2—C7	176.4 (3)	C13—C14—C15—C10	0.8 (5)
O2—C1—C2—C7	−3.0 (4)	C13—C14—C15—N1	−177.4 (3)
C7—C2—C3—C4	0.1 (4)	N2—C10—C15—C14	−179.3 (3)
C1—C2—C3—C4	177.1 (3)	C11—C10—C15—C14	−0.1 (5)
C2—C3—C4—C5	0.1 (4)	N2—C10—C15—N1	−0.6 (3)
C3—C4—C5—C6	0.5 (5)	C11—C10—C15—N1	178.5 (3)
C4—C5—C6—C7	−1.2 (4)	N2—C9—N1—C15	0.1 (3)
C4—C5—C6—C8	−179.9 (3)	N2—C9—N1—C8	−176.8 (3)
C5—C6—C7—C2	1.4 (4)	C14—C15—N1—C9	178.8 (3)
C8—C6—C7—C2	−180.0 (2)	C10—C15—N1—C9	0.3 (3)
C3—C2—C7—C6	−0.8 (4)	C14—C15—N1—C8	−4.4 (5)
C1—C2—C7—C6	−177.8 (2)	C10—C15—N1—C8	177.1 (3)
C7—C6—C8—N1	103.7 (3)	C6—C8—N1—C9	100.1 (3)
C5—C6—C8—N1	−77.7 (3)	C6—C8—N1—C15	−76.1 (3)
C15—C10—C11—C12	−0.9 (5)	N1—C9—N2—C10	−0.5 (4)
N2—C10—C11—C12	177.9 (3)	C15—C10—N2—C9	0.7 (3)
C10—C11—C12—C13	1.3 (6)	C11—C10—N2—C9	−178.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O4^{i}	0.86	1.91	2.735 (4)	159
N2—H2A···O5^{i}	0.86	2.44	3.168 (4)	143
N2—H2A···N3^{i}	0.86	2.51	3.352 (4)	168

O2—H2···O6	0.82	1.78	2.599 (3)	173
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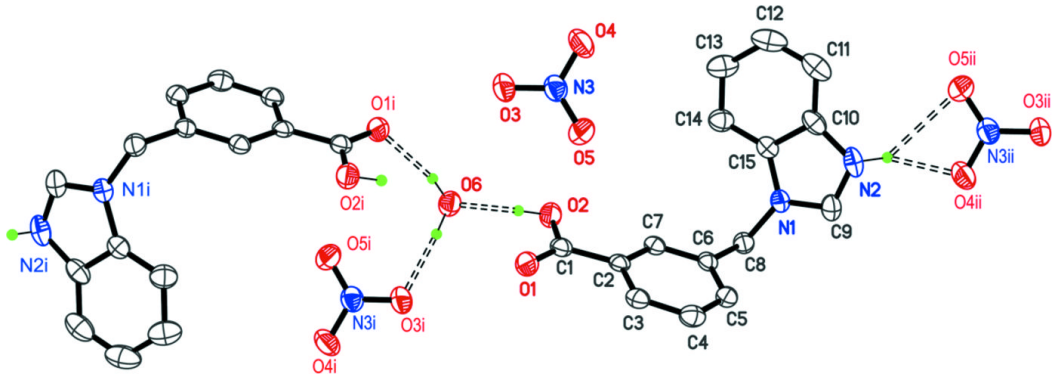
Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1


C12
C13
C11
O4
N3
O3
O5
O5ii
O3ii
O1i
C14
C10
N3ii
C15
N2
O4ii
N1i
O2i
O6
O2
C7
C6
N1
C9
N2i
O5i
C1
C2
C8
O1
N3i
O3i
C3
C5
O4i
C4