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1-Butylbenzimidazolium nitrate

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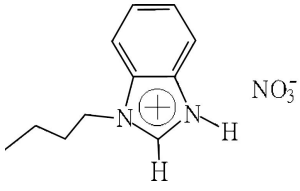
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.115; data-to-parameter ratio = 14.0.

The title salt, $C_{11}H_{15}N_2^+\cdot NO_3^-$, was prepared from benzimidazole by alkylation with *n*-butane iodide followed by reaction with nitric acid. In the crystal structure, N—H···O hydrogen bonds between the 1-butylbenzimidazolium cations and nitrate anions are observed.

Related literature

For related literature, see: Juan & Lee (1999); Kurdziel & Glowiak (2000); Losier & Zaworotko (1996); Ockwig *et al.* (2005); Sundberg & Martin (1974).



Experimental

Crystal data

$C_{11}H_{15}N_2^+\cdot NO_3^-$
M_r = 237.26
Monoclinic, $P2_1/c$
a = 5.4051 (19) Å
b = 16.885 (6) Å
c = 13.626 (5) Å
β = 100.439 (4)°
V = 1223.0 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.24 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.784, T_{max} = 1.000
6471 measured reflections
2168 independent reflections
1667 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.03
2168 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86	1.87	2.7177 (19)	168

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: WK2066).

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supplementary materials

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1-Butylbenzimidazolium nitrate

J.-C. Feng, Y.-L. Bian, Q.-X. Liu, X.-M. Wu and J.-H. Guo

Comment

In crystal engineering the most important driving forces are coordination, (Losier & Zaworotko, 1996 and Ockwig *et al.*, 2005.) however, some weak interactions, such as hydrogen bonding (Juan & Lee, 1999.) often affect the structures of complexes, and they can further link discrete subunits or low-dimentional entities into high-dimensional supramolecular networks. Imidazole groups play important roles in biological systems and constitute most of the metal binding sites of metalloenzymes (Sundberg & Martin, 1974). We are interested in the benzimidazole compounds with hydrogen bonds. Herein, we report the synthesis and crystal structure of 1-butylbenzimidazolium nitrate (2). Benzimidazole was reacted with n-butane iodide to form 1-butylbenzimidazole (1), and 1-butylbenzimidazole was further reacted with nitric acid to afford 1-butylbenzimidazolium nitrate (2). The crystals of (2) suitable for X-ray diffraction were obtained by evaporating slowly a CH_3OH solution at room temperature. In the molecular structure of 2 (Figure 1), the N(1)—C(1), N(1)—C(7), N(2)—C(6) and N(2)—C(7) bond distances are 1.3846 (19), 1.315 (2), 1.3897 (19) and 1.323 (2) Å, respectively, which are similar to those observed in 1-allylimidazole (Kurdziel *et al.*, 2000.), and there exists N1—H1···O3 hydrogen bonds between 1-butylbenzimidazolium ion and nitrate (Table 1).

Experimental

A 1,4-dioxane solution (50 ml) of benzimidazole (3.662 g, 31.000 mmol) was added to a suspension of oil-free sodium hydride (1.364 g, 34.000 mmol) in 1, 4-dioxane (50 ml) and stirred for 1 h at 90°C. Then a 1, 4-dioxane (50 ml) solution of n-butane iodide (5.152 g, 28.000 mmol) was added dropwise to above the solution. The mixture was stirred for 22 h at 90°C, and a brown solution was obtained. The solvent was removed with a rotary evaporator and H_2O (100 ml) was added to the residue. Then the solution was extracted with CH_2Cl_2 (100 ml), and the extracting solution was dried with anhydrous $MgSO_4$. After removing CH_2Cl_2, a yellow liquid of 1-butylbenzimidazole was obtained. Yield: 4.591 g (85%). 1-butylbenzimidazole (2.000 g, 9.603 mmol) was reacted with nitric acid to afford 1-butylbenzimidazolium nitrate (2) as a pale yellow solid. Yield: 2.614 g (96%); mp: 258–260°C. Anal. Calcd for $C_{11}H_{15}N_3O_3$: C 55.69, H 6.37, N 17.71%; found: C 55.48, H 6.22, N 17.65%.

Refinement

All H atoms were initially located in a difference Fourier map. All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

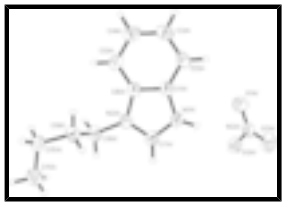

Fig. 1. Perspective view of 2 and anisotropic displacement parameters depicting 30% probability.

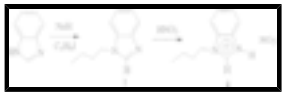

Fig. 2. The formation of the title salt.

1-Butylbenzimidazolium nitrate

Crystal data

$C_{11}H_{15}N_2^+ \cdot NO_3^-$	F_{000} = 504
M_r = 237.26	D_x = 1.289 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 5.4051 (19) Å	Cell parameters from 2013 reflections
b = 16.885 (6) Å	θ = 2.4–26.8º
c = 13.626 (5) Å	μ = 0.10 mm^{-1}
β = 100.439 (4)º	T = 293 (2) K
V = 1223.0 (7) Å^3	Block, colourless
Z = 4	0.24 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	2168 independent reflections
Radiation source: fine-focus sealed tube	1667 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.015
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −6→6
T_{min} = 0.784, T_{max} = 1.000	k = −20→15
6471 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.1736P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2168 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
155 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
O1	0.1856 (3)	0.36499 (9)	0.29577 (11)	0.1054 (6)
O2	−0.0556 (3)	0.30020 (9)	0.37516 (12)	0.1024 (5)
O3	0.1610 (2)	0.39744 (7)	0.44602 (8)	0.0701 (4)
N1	0.3501 (2)	0.53711 (8)	0.39341 (9)	0.0571 (3)
H1	0.3124	0.4905	0.4113	0.068*
N2	0.3537 (2)	0.66503 (7)	0.38025 (9)	0.0538 (3)
N3	0.0971 (3)	0.35307 (8)	0.37095 (11)	0.0645 (4)
C1	0.5190 (3)	0.55428 (9)	0.33119 (10)	0.0517 (4)
C2	0.6668 (3)	0.50653 (11)	0.28269 (12)	0.0650 (5)
H2	0.6646	0.4516	0.2878	0.078*
C3	0.8173 (3)	0.54485 (13)	0.22651 (12)	0.0732 (5)
H3A	0.9206	0.5150	0.1932	0.088*
C4	0.8196 (3)	0.62714 (13)	0.21798 (12)	0.0706 (5)
H4	0.9242	0.6504	0.1791	0.085*
C5	0.6726 (3)	0.67470 (11)	0.26513 (11)	0.0612 (4)
H5	0.6735	0.7295	0.2589	0.073*
C6	0.5219 (3)	0.63639 (9)	0.32282 (10)	0.0509 (4)
C7	0.2568 (3)	0.60384 (10)	0.42069 (12)	0.0584 (4)
H7	0.1392	0.6075	0.4626	0.070*
C8	0.2926 (3)	0.74849 (9)	0.39348 (13)	0.0637 (4)
H8A	0.1384	0.7517	0.4203	0.076*
H8B	0.2636	0.7746	0.3290	0.076*
C9	0.4994 (3)	0.79099 (10)	0.46253 (13)	0.0652 (4)
H9A	0.6576	0.7822	0.4402	0.078*
H9B	0.5142	0.7690	0.5291	0.078*
C10	0.4517 (3)	0.87944 (10)	0.46674 (13)	0.0683 (5)

H10A	0.5992	0.9045	0.5056	0.082*
H10B	0.4293	0.9007	0.3996	0.082*
C11	0.2286 (4)	0.90059 (13)	0.5109 (2)	0.1015 (7)
H11A	0.0784	0.8840	0.4668	0.152*
H11B	0.2242	0.9569	0.5203	0.152*
H11C	0.2390	0.8745	0.5741	0.152*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1618 (15)	0.0780 (10)	0.0898 (10)	−0.0088 (10)	0.0584 (10)	−0.0149 (8)
O2	0.1119 (11)	0.0737 (10)	0.1213 (12)	−0.0362 (9)	0.0200 (9)	−0.0161 (8)
O3	0.0881 (8)	0.0599 (7)	0.0618 (7)	−0.0150 (6)	0.0126 (6)	−0.0055 (6)
N1	0.0680 (8)	0.0483 (7)	0.0534 (7)	−0.0099 (6)	0.0071 (6)	0.0019 (6)
N2	0.0558 (7)	0.0485 (7)	0.0546 (7)	−0.0020 (6)	0.0032 (6)	−0.0037 (6)
N3	0.0762 (9)	0.0448 (8)	0.0725 (9)	0.0043 (7)	0.0137 (7)	−0.0009 (7)
C1	0.0577 (8)	0.0498 (9)	0.0440 (8)	−0.0039 (7)	−0.0004 (6)	−0.0023 (6)
C2	0.0713 (10)	0.0591 (10)	0.0595 (9)	0.0054 (8)	−0.0016 (8)	−0.0123 (8)
C3	0.0664 (11)	0.0934 (15)	0.0593 (10)	0.0034 (10)	0.0101 (8)	−0.0177 (9)
C4	0.0690 (11)	0.0902 (14)	0.0527 (9)	−0.0150 (10)	0.0114 (8)	−0.0031 (9)
C5	0.0687 (10)	0.0612 (10)	0.0509 (8)	−0.0116 (8)	0.0030 (8)	0.0030 (7)
C6	0.0540 (8)	0.0516 (9)	0.0441 (8)	−0.0031 (7)	0.0003 (6)	−0.0013 (6)
C7	0.0594 (9)	0.0603 (10)	0.0549 (9)	−0.0078 (8)	0.0086 (7)	−0.0034 (7)
C8	0.0625 (9)	0.0507 (9)	0.0733 (10)	0.0062 (7)	−0.0002 (8)	−0.0058 (8)
C9	0.0614 (9)	0.0581 (10)	0.0718 (10)	0.0037 (8)	0.0003 (8)	−0.0085 (8)
C10	0.0772 (11)	0.0544 (10)	0.0723 (11)	−0.0053 (8)	0.0110 (9)	−0.0009 (8)
C11	0.0955 (15)	0.0696 (14)	0.147 (2)	0.0099 (11)	0.0421 (15)	−0.0130 (13)

Geometric parameters (Å, °)

O1—N3	1.2233 (19)	C4—H4	0.9300
O2—N3	1.2241 (18)	C5—C6	1.390 (2)
O3—N3	1.2645 (18)	C5—H5	0.9300
N1—C7	1.315 (2)	C7—H7	0.9300
N1—C1	1.3846 (19)	C8—C9	1.506 (2)
N1—H1	0.8600	C8—H8A	0.9700
N2—C7	1.323 (2)	C8—H8B	0.9700
N2—C6	1.3897 (19)	C9—C10	1.518 (2)
N2—C8	1.466 (2)	C9—H9A	0.9700
C1—C2	1.384 (2)	C9—H9B	0.9700
C1—C6	1.391 (2)	C10—C11	1.486 (3)
C2—C3	1.375 (2)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.394 (3)	C11—H11A	0.9600
C3—H3A	0.9300	C11—H11B	0.9600
C4—C5	1.369 (2)	C11—H11C	0.9600
C7—N1—C1	108.83 (13)	N1—C7—N2	110.52 (14)
C7—N1—H1	125.6	N1—C7—H7	124.7

C1—N1—H1	125.6	N2—C7—H7	124.7
C7—N2—C6	108.15 (13)	N2—C8—C9	112.12 (13)
C7—N2—C8	125.78 (14)	N2—C8—H8A	109.2
C6—N2—C8	126.07 (13)	C9—C8—H8A	109.2
O1—N3—O2	121.91 (16)	N2—C8—H8B	109.2
O1—N3—O3	119.27 (15)	C9—C8—H8B	109.2
O2—N3—O3	118.81 (15)	H8A—C8—H8B	107.9
C2—C1—N1	132.22 (15)	C8—C9—C10	112.37 (14)
C2—C1—C6	121.68 (15)	C8—C9—H9A	109.1
N1—C1—C6	106.10 (13)	C10—C9—H9A	109.1
C3—C2—C1	116.24 (17)	C8—C9—H9B	109.1
C3—C2—H2	121.9	C10—C9—H9B	109.1
C1—C2—H2	121.9	H9A—C9—H9B	107.9
C2—C3—C4	122.03 (17)	C11—C10—C9	114.04 (16)
C2—C3—H3A	119.0	C11—C10—H10A	108.7
C4—C3—H3A	119.0	C9—C10—H10A	108.7
C5—C4—C3	122.05 (16)	C11—C10—H10B	108.7
C5—C4—H4	119.0	C9—C10—H10B	108.7
C3—C4—H4	119.0	H10A—C10—H10B	107.6
C4—C5—C6	116.22 (16)	C10—C11—H11A	109.5
C4—C5—H5	121.9	C10—C11—H11B	109.5
C6—C5—H5	121.9	H11A—C11—H11B	109.5
C5—C6—N2	131.83 (15)	C10—C11—H11C	109.5
C5—C6—C1	121.78 (14)	H11A—C11—H11C	109.5
N2—C6—C1	106.39 (13)	H11B—C11—H11C	109.5
C7—N1—C1—C2	179.94 (16)	C8—N2—C6—C1	−179.51 (13)
C7—N1—C1—C6	0.11 (16)	C2—C1—C6—C5	0.2 (2)
N1—C1—C2—C3	−179.41 (15)	N1—C1—C6—C5	−179.93 (12)
C6—C1—C2—C3	0.4 (2)	C2—C1—C6—N2	179.97 (13)
C1—C2—C3—C4	−0.6 (2)	N1—C1—C6—N2	−0.18 (15)
C2—C3—C4—C5	0.1 (3)	C1—N1—C7—N2	0.00 (17)
C3—C4—C5—C6	0.5 (2)	C6—N2—C7—N1	−0.12 (17)
C4—C5—C6—N2	179.65 (15)	C8—N2—C7—N1	179.58 (13)
C4—C5—C6—C1	−0.7 (2)	C7—N2—C8—C9	105.17 (18)
C7—N2—C6—C5	179.90 (15)	C6—N2—C8—C9	−75.18 (19)
C8—N2—C6—C5	0.2 (2)	N2—C8—C9—C10	173.05 (14)
C7—N2—C6—C1	0.19 (15)	C8—C9—C10—C11	65.3 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	1.87	2.7177 (19)	168

Fig. 1

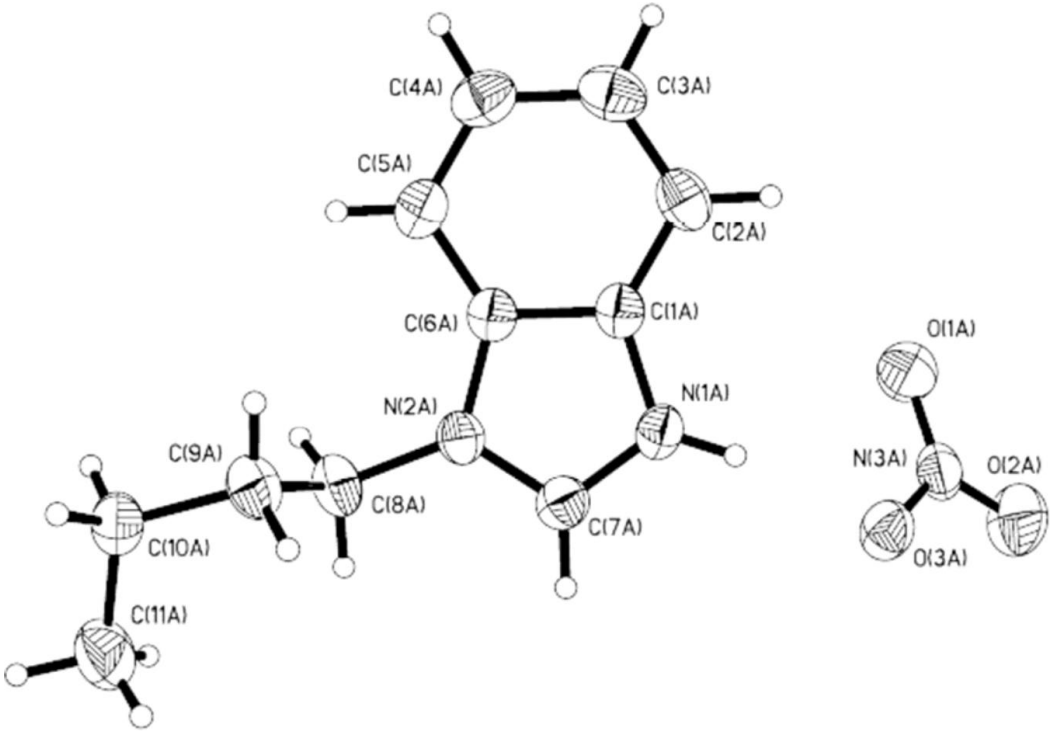
C(4A)
C(3A)
C(5A)
C(2A)
C(6A)
C(1A)
O(1A)
N(2A)
N(1A)
C(9A)
N(3A)
O(2A)
C(8A)
C(7A)
C(10A)
O(3A)
C(11A)

Fig. 2