Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetra-*n*-propylammonium acetate–boric acid (1/1)

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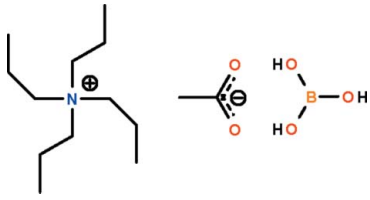
Received 20 October 2009; accepted 23 October 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.183; data-to-parameter ratio = 22.5.

In the crystal structure of the ammonium carboxylate–boric acid cocrystal, $(C_3H_7)_4N^+\cdot CH_3CO_2^-\cdot H_3BO_3$, the boric acid forms two O—H···O hydrogen bonds to the acetate anion. The acetate–boric acid species is hydrogen bonded to another acetate–boric acid species through the third OH unit of the boric acid about a twofold rotation axis.

Related literature

For the crystal structure of tetra-*n*-propyl pentaborate–boric acid co-crystal, see: Freyhardt *et al.* (1994).



Experimental

Crystal data

$C_{12}H_{28}N^+\cdot C_2H_3O_2^-\cdot BH_3O_3$
M_r = 307.23
Orthorhombic, *Pccn*
a = 16.4594 (3) Å
b = 16.7680 (3) Å
c = 14.4526 (3) Å
V = 3988.79 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 K
0.24 × 0.24 × 0.22 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
19907 measured reflections
4581 independent reflections
2803 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.183
S = 1.02
4581 reflections
204 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4	0.86 (1)	1.75 (1)	2.600 (2)	175 (2)
O2—H2···O5	0.85 (1)	1.79 (1)	2.638 (2)	172 (3)
O3—H3···O1^{i}	0.85 (1)	1.88 (1)	2.729 (2)	174 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank Beijing Normal University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2651).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

Acta Cryst. (2009). E**65**, o2958 [https://doi.org/10.1107/S1600536809044225]

Tetra-*n*-propylammonium acetate–boric acid (1/1)

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S1. Experimental

1,3,5-Tris(4-carboxyphenyl)benzene (0.055 g, 0.125 mmol) and boric acid (0.25 mmol, 0.015 g) were dissolved in a water-ethanol (50/100 *v*/*v*) mixture. An aqueous solution of 30% tetra-*n*-propylammonium hydroxide was added in an acid:base ratio of 1.3. Several drops of acetic acid were added and the clear solution set aside for the formation of crystals after several weeks.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C). The hydroxy H-atoms were located in a difference Fourier map and were refined with a distance restraint of 0.85±0.01 Å; their temperature factors were freely refined.

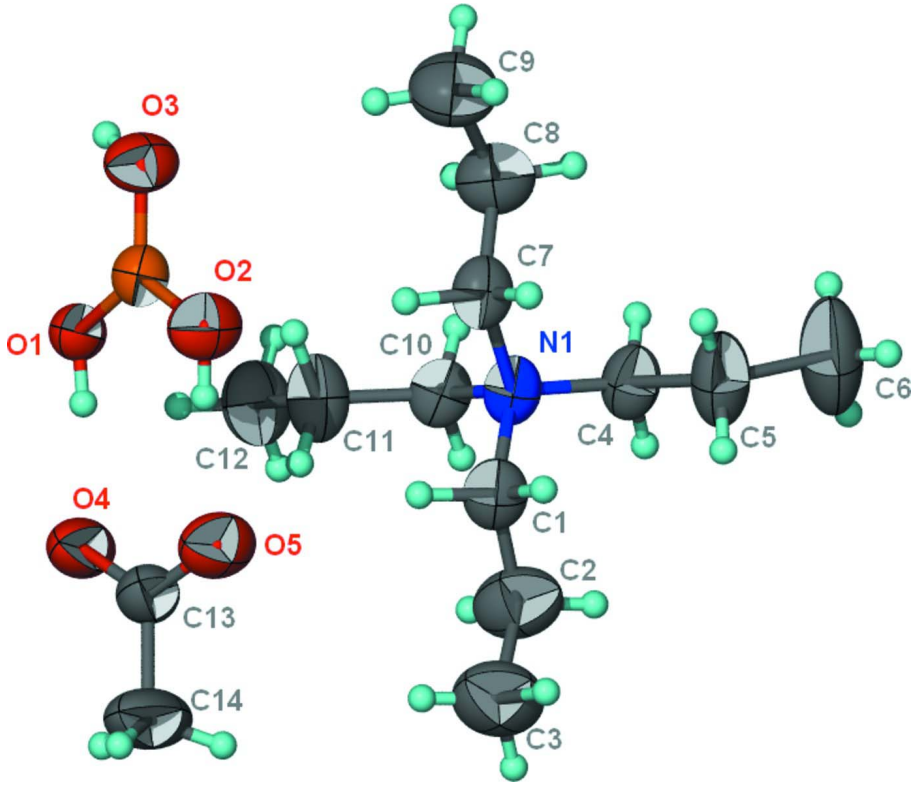

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[N(C_3H_7)_4]$ $[CH_3CO_2]\cdot H_3BO_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Tetra-*n*-propylammonium acetate–boric acid (1/1)

Crystal data

$C_{12}H_{28}N^+·C_2H_3O_2^-·BH_3O_3$
$M_r = 307.23$
Orthorhombic, *Pccn*
Hall symbol: -P 2ab 2ac
$a = 16.4594$ (3) Å
$b = 16.7680$ (3) Å
$c = 14.4526$ (3) Å
$V = 3988.79$ (13) Å^3
$Z = 8$
$F(000) = 1360$
$D_x = 1.023$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5166 reflections
$\theta = 2.2$–$25.7°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Block, colorless
0.24 × 0.24 × 0.22 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and ω scans
19907 measured reflections
4581 independent reflections
2803 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
$h = -20 \rightarrow 21$
$k = -21 \rightarrow 21$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.183$
$S = 1.02$
4581 reflections
204 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.6196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (8)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.65031 (7)	0.26154 (7)	0.38659 (8)	0.0628 (3)
H1	0.5997 (7)	0.2504 (13)	0.3918 (13)	0.083 (6)*
O2	0.60919 (9)	0.39766 (8)	0.38631 (11)	0.0822 (4)
H2	0.5628 (10)	0.3765 (17)	0.396 (2)	0.131 (11)*
O3	0.74569 (8)	0.36496 (8)	0.37814 (10)	0.0783 (4)
H3	0.7800 (12)	0.3273 (10)	0.3834 (16)	0.104 (8)*
O4	0.49799 (8)	0.22127 (8)	0.39542 (12)	0.0942 (5)
O5	0.46002 (8)	0.34620 (9)	0.41629 (12)	0.0923 (5)
N1	0.57206 (9)	0.42369 (8)	0.68832 (9)	0.0628 (4)
C1	0.48754 (11)	0.42868 (11)	0.64651 (13)	0.0732 (5)
H1A	0.4724	0.4845	0.6421	0.088*
H1B	0.4898	0.4076	0.5840	0.088*
C2	0.42181 (13)	0.38518 (18)	0.69837 (19)	0.1086 (8)

H2A	0.4354	0.3290	0.7024	0.130*
H2B	0.4179	0.4061	0.7608	0.130*
C3	0.34159 (15)	0.39487 (19)	0.6499 (2)	0.1243 (10)
H3A	0.3001	0.3675	0.6841	0.186*
H3B	0.3452	0.3728	0.5888	0.186*
H3C	0.3283	0.4505	0.6459	0.186*
C4	0.57384 (13)	0.45737 (12)	0.78584 (12)	0.0750 (5)
H4A	0.5387	0.4250	0.8245	0.090*
H4B	0.6287	0.4522	0.8097	0.090*
C5	0.54799 (17)	0.54338 (15)	0.79533 (16)	0.1045 (8)
H5A	0.5782	0.5761	0.7520	0.125*
H5B	0.4907	0.5484	0.7809	0.125*
C6	0.5634 (2)	0.5719 (2)	0.8927 (2)	0.1579 (16)
H6A	0.5471	0.6266	0.8982	0.237*
H6B	0.6203	0.5672	0.9066	0.237*
H6C	0.5327	0.5399	0.9353	0.237*
C7	0.62782 (12)	0.46947 (10)	0.62404 (12)	0.0693 (5)
H7A	0.6252	0.4450	0.5633	0.083*
H7B	0.6072	0.5234	0.6182	0.083*
C8	0.71601 (13)	0.47402 (14)	0.65287 (17)	0.0922 (6)
H8A	0.7204	0.5036	0.7103	0.111*
H8B	0.7367	0.4206	0.6635	0.111*
C9	0.76609 (17)	0.51429 (15)	0.5795 (2)	0.1162 (9)
H9A	0.8216	0.5173	0.5995	0.174*
H9B	0.7456	0.5671	0.5690	0.174*
H9C	0.7630	0.4842	0.5231	0.174*
C10	0.59924 (11)	0.33770 (10)	0.69796 (12)	0.0647 (4)
H10A	0.6521	0.3372	0.7276	0.078*
H10B	0.5616	0.3106	0.7390	0.078*
C11	0.60502 (16)	0.29084 (12)	0.61010 (14)	0.0875 (7)
H11A	0.6441	0.3158	0.5691	0.105*
H11B	0.5527	0.2907	0.5793	0.105*
C12	0.63080 (17)	0.20624 (12)	0.62980 (17)	0.0967 (7)
H12A	0.6343	0.1771	0.5727	0.145*
H12B	0.5916	0.1813	0.6696	0.145*
H12C	0.6829	0.2064	0.6596	0.145*
C13	0.44575 (11)	0.27397 (13)	0.40662 (15)	0.0781 (5)
C14	0.35937 (14)	0.24694 (18)	0.4140 (3)	0.1411 (12)
H14A	0.3351	0.2698	0.4682	0.212*
H14B	0.3577	0.1898	0.4183	0.212*
H14C	0.3299	0.2639	0.3601	0.212*
B1	0.66733 (12)	0.34081 (11)	0.38298 (13)	0.0591 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0555 (7)	0.0561 (7)	0.0769 (8)	0.0019 (5)	0.0024 (5)	−0.0096 (5)
O2	0.0748 (9)	0.0586 (7)	0.1131 (12)	0.0115 (6)	0.0009 (8)	0.0088 (7)

O3	0.0670 (8)	0.0588 (7)	0.1091 (11)	−0.0027 (6)	0.0061 (7)	0.0111 (7)
O4	0.0616 (8)	0.0783 (9)	0.1426 (14)	−0.0003 (6)	0.0061 (8)	−0.0330 (8)
O5	0.0673 (8)	0.0771 (9)	0.1325 (13)	0.0137 (7)	0.0019 (8)	−0.0022 (8)
N1	0.0729 (8)	0.0629 (8)	0.0527 (8)	0.0194 (6)	−0.0066 (6)	0.0040 (6)
C1	0.0755 (12)	0.0766 (12)	0.0675 (11)	0.0210 (9)	−0.0140 (9)	0.0027 (9)
C2	0.0780 (14)	0.137 (2)	0.1105 (19)	0.0113 (14)	−0.0109 (13)	0.0199 (16)
C3	0.0839 (16)	0.134 (2)	0.155 (3)	0.0127 (15)	−0.0238 (16)	−0.0001 (19)
C4	0.0852 (12)	0.0848 (12)	0.0548 (10)	0.0224 (10)	−0.0081 (9)	−0.0031 (9)
C5	0.133 (2)	0.0994 (16)	0.0814 (14)	0.0463 (15)	−0.0204 (13)	−0.0260 (12)
C6	0.199 (4)	0.165 (3)	0.109 (2)	0.071 (3)	−0.046 (2)	−0.063 (2)
C7	0.0914 (12)	0.0551 (9)	0.0613 (10)	0.0137 (9)	−0.0014 (9)	0.0079 (7)
C8	0.0873 (14)	0.0955 (15)	0.0937 (15)	0.0014 (11)	−0.0033 (11)	0.0164 (12)
C9	0.114 (2)	0.1079 (18)	0.127 (2)	−0.0218 (15)	0.0101 (16)	0.0175 (16)
C10	0.0757 (11)	0.0598 (9)	0.0584 (10)	0.0148 (8)	−0.0033 (8)	0.0128 (7)
C11	0.1289 (19)	0.0639 (11)	0.0698 (12)	0.0226 (11)	−0.0088 (12)	0.0032 (9)
C12	0.1239 (18)	0.0607 (11)	0.1054 (17)	0.0151 (11)	−0.0040 (14)	−0.0001 (11)
C13	0.0555 (10)	0.0852 (14)	0.0936 (14)	0.0044 (9)	−0.0024 (9)	−0.0198 (11)
C14	0.0603 (13)	0.125 (2)	0.238 (4)	−0.0056 (14)	0.0121 (17)	−0.054 (2)
B1	0.0658 (11)	0.0570 (10)	0.0545 (10)	0.0053 (8)	0.0001 (8)	0.0027 (8)

Geometric parameters (Å, º)

O1—B1	1.359 (2)	C5—H5B	0.9700
O1—H1	0.856 (9)	C6—H6A	0.9600
O2—B1	1.352 (2)	C6—H6B	0.9600
O2—H2	0.853 (10)	C6—H6C	0.9600
O3—B1	1.354 (2)	C7—C8	1.512 (3)
O3—H3	0.851 (9)	C7—H7A	0.9700
O4—C13	1.244 (2)	C7—H7B	0.9700
O5—C13	1.242 (2)	C8—C9	1.503 (3)
N1—C7	1.515 (2)	C8—H8A	0.9700
N1—C10	1.516 (2)	C8—H8B	0.9700
N1—C4	1.519 (2)	C9—H9A	0.9600
N1—C1	1.519 (2)	C9—H9B	0.9600
C1—C2	1.505 (3)	C9—H9C	0.9600
C1—H1A	0.9700	C10—C11	1.496 (3)
C1—H1B	0.9700	C10—H10A	0.9700
C2—C3	1.503 (3)	C10—H10B	0.9700
C2—H2A	0.9700	C11—C12	1.508 (3)
C2—H2B	0.9700	C11—H11A	0.9700
C3—H3A	0.9600	C11—H11B	0.9700
C3—H3B	0.9600	C12—H12A	0.9600
C3—H3C	0.9600	C12—H12B	0.9600
C4—C5	1.510 (3)	C12—H12C	0.9600
C4—H4A	0.9700	C13—C14	1.496 (3)
C4—H4B	0.9700	C14—H14A	0.9600
C5—C6	1.508 (3)	C14—H14B	0.9600
C5—H5A	0.9700	C14—H14C	0.9600

B1—O1—H1	114.6 (15)	N1—C7—H7A	108.3
B1—O2—H2	110 (2)	C8—C7—H7B	108.3
B1—O3—H3	113.9 (16)	N1—C7—H7B	108.3
C7—N1—C10	111.08 (13)	H7A—C7—H7B	107.4
C7—N1—C4	111.66 (15)	C9—C8—C7	110.8 (2)
C10—N1—C4	105.23 (12)	C9—C8—H8A	109.5
C7—N1—C1	106.44 (13)	C7—C8—H8A	109.5
C10—N1—C1	111.05 (14)	C9—C8—H8B	109.5
C4—N1—C1	111.49 (13)	C7—C8—H8B	109.5
C2—C1—N1	115.70 (16)	H8A—C8—H8B	108.1
C2—C1—H1A	108.4	C8—C9—H9A	109.5
N1—C1—H1A	108.4	C8—C9—H9B	109.5
C2—C1—H1B	108.4	H9A—C9—H9B	109.5
N1—C1—H1B	108.4	C8—C9—H9C	109.5
H1A—C1—H1B	107.4	H9A—C9—H9C	109.5
C3—C2—C1	110.3 (2)	H9B—C9—H9C	109.5
C3—C2—H2A	109.6	C11—C10—N1	116.12 (13)
C1—C2—H2A	109.6	C11—C10—H10A	108.3
C3—C2—H2B	109.6	N1—C10—H10A	108.3
C1—C2—H2B	109.6	C11—C10—H10B	108.3
H2A—C2—H2B	108.1	N1—C10—H10B	108.3
C2—C3—H3A	109.5	H10A—C10—H10B	107.4
C2—C3—H3B	109.5	C10—C11—C12	110.60 (16)
H3A—C3—H3B	109.5	C10—C11—H11A	109.5
C2—C3—H3C	109.5	C12—C11—H11A	109.5
H3A—C3—H3C	109.5	C10—C11—H11B	109.5
H3B—C3—H3C	109.5	C12—C11—H11B	109.5
C5—C4—N1	115.72 (15)	H11A—C11—H11B	108.1
C5—C4—H4A	108.4	C11—C12—H12A	109.5
N1—C4—H4A	108.4	C11—C12—H12B	109.5
C5—C4—H4B	108.4	H12A—C12—H12B	109.5
N1—C4—H4B	108.4	C11—C12—H12C	109.5
H4A—C4—H4B	107.4	H12A—C12—H12C	109.5
C6—C5—C4	109.9 (2)	H12B—C12—H12C	109.5
C6—C5—H5A	109.7	O5—C13—O4	125.24 (18)
C4—C5—H5A	109.7	O5—C13—C14	117.86 (19)
C6—C5—H5B	109.7	O4—C13—C14	116.8 (2)
C4—C5—H5B	109.7	C13—C14—H14A	109.5
H5A—C5—H5B	108.2	C13—C14—H14B	109.5
C5—C6—H6A	109.5	H14A—C14—H14B	109.5
C5—C6—H6B	109.5	C13—C14—H14C	109.5
H6A—C6—H6B	109.5	H14A—C14—H14C	109.5
C5—C6—H6C	109.5	H14B—C14—H14C	109.5
H6A—C6—H6C	109.5	O2—B1—O3	117.74 (16)
H6B—C6—H6C	109.5	O2—B1—O1	122.85 (17)
C8—C7—N1	115.99 (15)	O3—B1—O1	119.39 (15)
C8—C7—H7A	108.3		

C7—N1—C1—C2	178.63 (18)	C10—N1—C7—C8	−60.1 (2)
C10—N1—C1—C2	57.6 (2)	C4—N1—C7—C8	57.0 (2)
C4—N1—C1—C2	−59.4 (2)	C1—N1—C7—C8	178.90 (16)
N1—C1—C2—C3	180.0 (2)	N1—C7—C8—C9	174.83 (18)
C7—N1—C4—C5	60.1 (2)	C7—N1—C10—C11	−57.4 (2)
C10—N1—C4—C5	−179.33 (19)	C4—N1—C10—C11	−178.41 (18)
C1—N1—C4—C5	−58.9 (2)	C1—N1—C10—C11	60.8 (2)
N1—C4—C5—C6	−172.4 (2)	N1—C10—C11—C12	−178.76 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4	0.86 (1)	1.75 (1)	2.600 (2)	175 (2)
O2—H2···O5	0.85 (1)	1.79 (1)	2.638 (2)	172 (3)
O3—H3···O1^{i}	0.85 (1)	1.88 (1)	2.729 (2)	174 (2)

Symmetry code: (i) −*x*+3/2, −*y*+1/2, *z*.