

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

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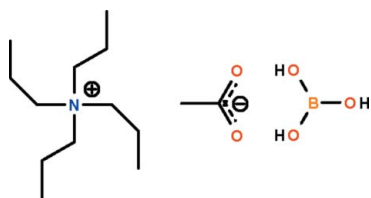
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.183; data-to-parameter ratio = 22.5.

In the crystal structure of the ammonium carboxylate–boric acid cocrystal, $(\text{C}_3\text{H}_7)_4\text{N}^+\cdot\text{CH}_3\text{CO}_2^-\cdot\text{H}_3\text{BO}_3$, the boric acid forms two $\text{O}-\text{H}\cdots\text{O}$ 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

$\text{C}_{12}\text{H}_{28}\text{N}^+\cdot\text{C}_2\text{H}_3\text{O}_2^-\cdot\text{BH}_3\text{O}_3$
 $M_r = 307.23$

Orthorhombic, $Pccn$
 $a = 16.4594$ (3) Å

$b = 16.7680$ (3) Å
 $c = 14.4526$ (3) Å
 $V = 3988.79$ (13) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 293$ K
 $0.24 \times 0.24 \times 0.22$ mm

Data collection

Bruker APEXII diffractometer
 Absorption correction: none
 19907 measured reflections

4581 independent reflections
 2803 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

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
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O4}$	0.86 (1)	1.75 (1)	2.600 (2)	175 (2)
$\text{O2}-\text{H2}\cdots\text{O5}$	0.85 (1)	1.79 (1)	2.638 (2)	172 (3)
$\text{O3}-\text{H3}\cdots\text{O1}^{\dagger}$	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: *pubCIF* (Westrip, 2009).

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

References

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2009). *pubCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, o2958 [doi:10.1107/S1600536809044225]

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

Y.-X. Yang, Q. Li and S. W. Ng

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.

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 $U(\text{H})$ set to 1.2–1.5 $U(\text{C})$. 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.

Figures

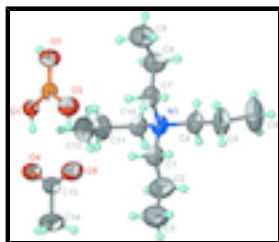


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $[\text{N}(\text{C}_3\text{H}_7)_4][\text{CH}_3\text{CO}_2]\cdot\text{H}_3\text{BO}_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

$\text{C}_{12}\text{H}_{28}\text{N}^+\cdot\text{C}_2\text{H}_3\text{O}_2^-\cdot\text{BH}_3\text{O}_3$

$M_r = 307.23$

Orthorhombic, *Pccn*

Hall symbol: -P 2ab 2ac

$a = 16.4594(3) \text{ \AA}$

$b = 16.7680(3) \text{ \AA}$

$c = 14.4526(3) \text{ \AA}$

$V = 3988.79(13) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1360$

$D_x = 1.023 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5166 reflections

$\theta = 2.2\text{--}25.7^\circ$

$\mu = 0.07 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.24 \times 0.24 \times 0.22 \text{ mm}$

supplementary materials

Data collection

Bruker APEXII diffractometer	2803 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.022$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 293$ K	$\theta_{\text{min}} = 2.2^\circ$
ω and ϕ scans	$h = -20 \rightarrow 21$
Absorption correction: None	$k = -21 \rightarrow 21$
19907 measured reflections	$l = -18 \rightarrow 17$
4581 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
$R[F^2 > 2\sigma(F^2)] = 0.051$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.183$	$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.6196P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
4581 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
204 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{\AA}^{-3}$
3 restraints	$\Delta\rho_{\text{min}} = -0.13 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0029 (8)

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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)

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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, °)

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)

supplementary materials

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, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots O4	0.86 (1)	1.75 (1)	2.600 (2)	175 (2)
O2—H2 \cdots O5	0.85 (1)	1.79 (1)	2.638 (2)	172 (3)
O3—H3 \cdots O1 ⁱ	0.85 (1)	1.88 (1)	2.729 (2)	174 (2)

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

Fig. 1

