Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

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Electronic paper
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Tetraammonium benzene-1,2,4,5-tetracarboxylate tetrahydrate

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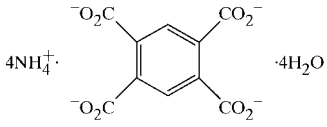
Received 18 September 2000
Accepted 30 October 2000

Data validation number: IUC0000317

The anions in the title compound, $4NH_4^+\cdot C_{10}H_2O_8^{4-}\cdot 4H_2O$, are held together by regular hydrogen bonds from the carboxylate



(I)

O atoms to the ammonium cations and water molecules, forming a three-dimensional network.

Experimental

The title compound was prepared by reacting benzene-1,2,4,5-tetracarboxylic acid (2.0 g, 8 mmol, Aldrich) and concentrated aqueous ammonia (250 ml, 14.8 *N*, Aldrich) in a 250 ml beaker. The mixture was stirred and the volume was concentrated to 10 ml by the application of heat. Crystals were obtained by allowing the complete evaporation of liquid from this mixture at room temperature over a period of several weeks.

Crystal data

$4NH_4^+\cdot C_{10}H_2O_8^{4-}\cdot 4H_2O$
M_r = 394.34
Monoclinic, $P2_1/n$
a = 6.5590 (11) Å
b = 23.134 (5) Å
c = 6.7460 (12) Å
β = 119.071 (15)°
V = 894.7 (3) Å^3
Z = 2
D_x = 1.464 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.134 mm^{-1}
T = 293 (2) K
Prism, pale yellow
0.40 × 0.25 × 0.18 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Non-profiled ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.899, T_{max} = 0.980
1695 measured reflections
1560 independent reflections
1169 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 24.97°
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 27$
$l = -8 \rightarrow 7$
3 standard reflections
frequency: 166 min
intensity decay: 7%

Refinement

Refinement on F^2
$R(F)$ = 0.039
$wR(F^2)$ = 0.104
S = 1.074
1560 reflections
170 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.2097P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O51	0.89 (3)	2.65 (3)	3.241 (2)	124 (3)
O1—H1···O52	0.89 (3)	1.92 (4)	2.816 (2)	175 (3)
O1—H2···O51^{i}	0.88 (3)	1.91 (3)	2.732 (2)	155 (3)
O2—H4···O1^{ii}	0.91 (5)	2.14 (5)	2.948 (3)	147 (4)
O2—H3···O11^{iii}	0.85 (4)	2.02 (4)	2.870 (3)	176 (4)
O2—H4···O52^{iv}	0.91 (5)	2.44 (5)	3.039 (3)	124 (4)
N1—H13···O1	0.84 (5)	3.15 (4)	3.554 (3)	113 (3)
N1—H11···O2^{i}	0.98 (4)	1.95 (4)	2.919 (3)	169 (4)
N1—H12···O11^{v}	1.00 (4)	1.87 (4)	2.850 (3)	165 (4)
N1—H13···O1^{i}	0.84 (5)	2.13 (5)	2.941 (3)	163 (4)
N1—H14···O2	0.93 (5)	2.15 (5)	2.999 (4)	152 (4)
N2—H22···O2	0.93 (3)	2.99 (2)	3.448 (3)	111.9 (16)
N2—H23···O12	0.91 (3)	1.93 (3)	2.816 (2)	164 (2)
N2—H21···O51^{vi}	0.95 (3)	1.84 (3)	2.788 (3)	178 (2)
N2—H22···O52^{iv}	0.93 (3)	1.98 (3)	2.905 (3)	175 (2)
N2—H24···O11^{vii}	0.98 (3)	2.62 (3)	3.293 (3)	126 (2)
N2—H24···O12^{vii}	0.98 (3)	1.81 (3)	2.787 (2)	177 (3)

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

The space group was chosen based on the fact that a data set collected in an orthorhombic setting did not produce a reasonable solution. The C—H, O—H, N—H and U_{iso} values for the refined H atoms are 0.95 (2), 0.85 (4)–0.91 (5), 0.84 (5)–1.00 (4) Å and 0.020 (5)–0.140 (19) Å^2, respectively.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

RLL thanks Michigan Technological University for support.

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