

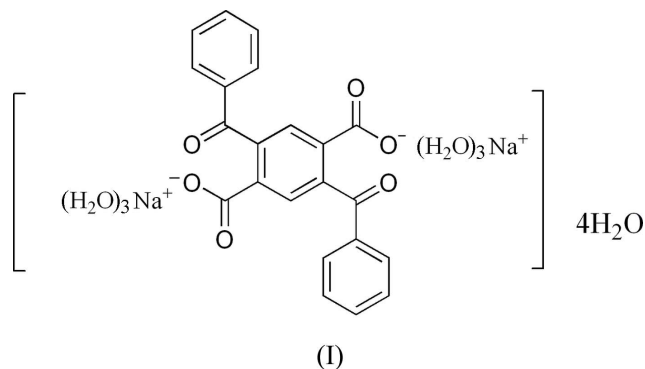
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.056
 wR factor = 0.205
Data-to-parameter ratio = 12.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(triaquasodium) 2,5-dibenzoylterephthalate
tetrahydrateThe centrosymmetric title compound, $[\text{Na}(\text{H}_2\text{O})_3]_2 \cdot (\text{C}_{22}\text{H}_{12}\text{O}_6) \cdot 4\text{H}_2\text{O}$, was prepared by slow evaporation of an aqueous solution of sodium dibenzoylterephthalate. The uncoordinated water molecules are connected by intermolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.Received 11 October 2005
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Comment

2,5-Dibenzoylterephthalic acid is a significant material in the synthesis of extended lattice compounds with its centrosymmetric structure. The synthesis of 2,5-dibenzoylterephthalic acid has been reported (Imai *et al.*, 1975), as has the crystal structure (Zhu *et al.*, 2005). Surprisingly, no related structures containing group 1 metal cations have been reported. This paper presents the results of the single-crystal X-ray diffraction analysis of bis(triaquasodium) 2,5-dibenzoylterephthalate tetrahydrate, (I).The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The 2,5-dibenzoylterephthalate dianion is disposed about a crystallographic center of symmetry. The coordination around each Na^+ ion is octahedral. Neighbouring sodium ions are linked by face sharing of three water molecules on opposite faces of each octahedron to give an infinite array (Fig. 2).In the crystal structure, uncoordinated water molecules are linked by $\text{O}-\text{H} \cdots \text{O}$ intermolecular hydrogen bonds. The anions are linked by intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 2), forming a two-dimensional layer structure (Fig. 3).

Experimental

2,5-Dibenzoylterephthalic acid was prepared by the reaction of pyromellitic acid dianhydride (6.9 g, 32 mmol), powdered AlCl_3 (15 g, 120 mmol) and benzene (100 ml) with stirring at 338–343 K for 4 h. Analysis calculated (%): C 44.15, H 5.40; found (%): C 44.06, H

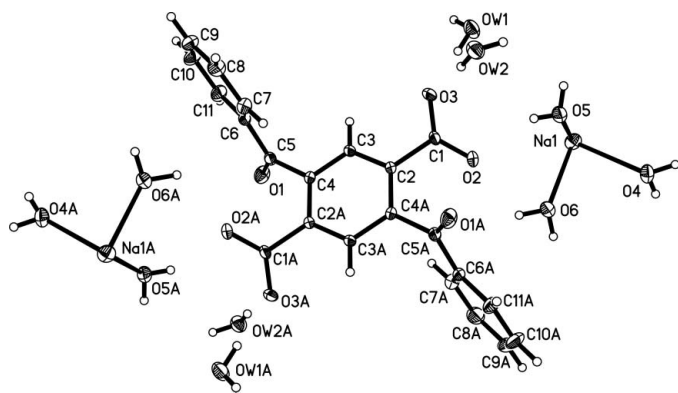


Figure 1
The structures of component ions and molecules of (I). Displacement ellipsoids are drawn at the 30% probability level. Atoms marked with an A are at the symmetry position $(-x, -y, -z - 1)$.

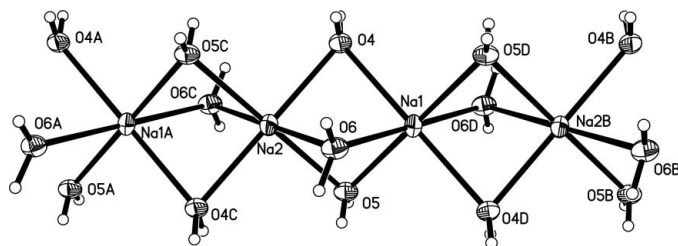


Figure 2
The coordination polyhedra of Na^+ , showing a trigonally distorted octahedral configuration. Atoms marked with A, B, C and D are at the symmetry positions $(x + 1, y, z)$, $(x - 1, y, z)$, $(-x + 1, -y + 1, -z)$ and $(-x, -y + 1, -z)$, respectively.

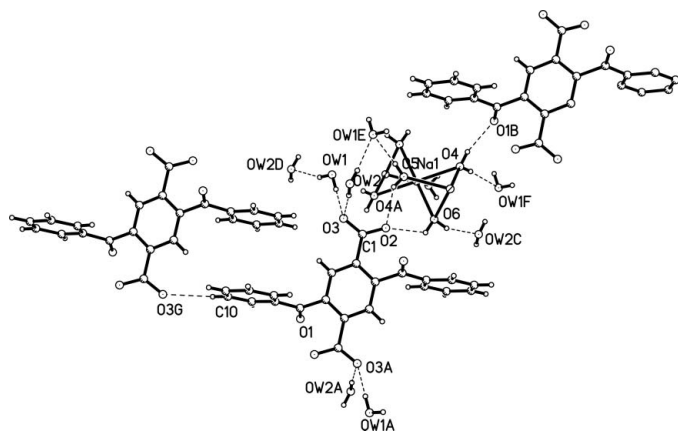


Figure 3
A segment of the crystal structure of (I). Dashed lines indicate $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\text{O}$ interactions. Atoms marked with A, B, C, D, E, F and G are at the symmetry positions $(-x, -y, -z - 1)$, $(x + 1, y + 1, z + 1)$, $(x, y + 1, z)$, $(x - 1, y, z)$, $(-x, -y, -z)$, $(-x, -y + 1, z)$ and $(-x - 1, -y - 1, -z - 1)$, respectively.

5.47. IR (KBr, ν cm^{-1}): 3450, 3005, 2912, 2835, 2355, 1691, 1595, 1465, 1341. Crystals of (I) suitable for diffraction measurements were obtained by slow evaporation of an aqueous solution of sodium hydroxide and 2,5-dibenzoyltterephthalic acid (2:1) at room temperature.

Crystal data

$[\text{Na}(\text{H}_2\text{O})_3]_2(\text{C}_{22}\text{H}_{12}\text{O}_6)\cdot 4\text{H}_2\text{O}$
 $M_r = 598.46$
 Triclinic, $P\bar{1}$
 $a = 6.2800$ (13) \AA
 $b = 8.6650$ (17) \AA
 $c = 13.709$ (3) \AA
 $\alpha = 105.41$ (3) $^\circ$
 $\beta = 99.37$ (3) $^\circ$
 $\gamma = 92.70$ (3) $^\circ$
 $V = 706.4$ (3) \AA^3

$Z = 1$
 $D_x = 1.407$ Mg m^{-3}
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 10\text{--}12^\circ$
 $\mu = 0.14$ mm^{-1}
 $T = 293$ (2) K
 Block, colorless
 $0.4 \times 0.3 \times 0.2$ mm^3

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2002)
 $T_{\min} = 0.949$, $T_{\max} = 0.972$
 3040 measured reflections
 2775 independent reflections
 2262 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.111$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = 0 \rightarrow 7$
 $k = -10 \rightarrow 10$
 $l = -16 \rightarrow 16$
 3 standard reflections every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.205$
 $S = 1.00$
 2775 reflections
 224 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.15P)^2 + 0.41P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.031$
 $\Delta\rho_{\text{max}} = 0.41$ e \AA^{-3}
 $\Delta\rho_{\text{min}} = -0.54$ e \AA^{-3}

Table 1

Selected geometric parameters (\AA , $^\circ$).

Na1—O5	2.372 (2)	O3—C1	1.250 (3)
Na1—O6	2.390 (2)	Na2—O4 ⁱ	2.387 (2)
Na1—O4	2.442 (2)	Na2—O6 ⁱ	2.387 (2)
O2—C1	1.243 (3)	Na2—O5 ⁱ	2.440 (2)
O5—Na1—O5 ⁱⁱ	180	O6—Na1—O4 ⁱⁱ	101.32 (8)
O5—Na1—O6	77.24 (8)	O5—Na1—O4	89.12 (8)
O5 ⁱⁱ —Na1—O6	102.76 (8)	O6—Na1—O4	78.68 (8)
O6—Na1—O6 ⁱⁱ	180	O4 ⁱⁱ —Na1—O4	180
O5—Na1—O4 ⁱⁱ	90.88 (8)		

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

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O6—H6B2 \cdots OW2 ⁱⁱⁱ	0.88 (5)	1.97 (5)	2.799 (4)	158 (4)
O4—H4B1 \cdots O1 ^{iv}	0.85 (7)	2.12 (6)	2.953 (3)	167 (5)
OW2—HW2B \cdots O3	0.92 (5)	1.83 (5)	2.747 (3)	175 (5)
OW1—HW1A \cdots O3	0.86 (5)	1.91 (5)	2.762 (3)	169 (4)
OW2—HW2A \cdots OW1 ^v	0.82 (5)	2.03 (5)	2.839 (3)	174 (4)
OW1—HW1C \cdots OW2 ^{vi}	0.88 (5)	1.99 (5)	2.829 (4)	159 (4)
O5—H5B1 \cdots O2	0.76 (6)	2.10 (6)	2.755 (3)	145 (6)
O5—H5B3 \cdots OW1 ^v	0.86 (4)	2.16 (4)	3.016 (3)	172 (4)
O6—H6B1 \cdots O2	0.96 (5)	2.18 (6)	2.812 (3)	122 (4)
O4—H4B2 \cdots OW1 ⁱⁱ	0.71 (6)	2.30 (6)	2.998 (4)	167 (7)
C10—H10A \cdots O3 ^{vii}	0.93	2.52	3.317 (4)	144

Symmetry codes: (ii) $-x, -y + 1, -z$; (iii) $x, y + 1, z$; (iv) $x + 1, y + 1, z + 1$; (v) $-x, -y, -z$; (vi) $x - 1, y, z$; (vii) $-x - 1, -y - 1, -z - 1$.

H atoms attached to O atoms were located in a difference Fourier map and their positions were refined, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. H

atoms bonded to C were placed in calculated positions (C–H = 0.93 Å) and refined as riding, with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 1995); 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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References

- Bruker (2000). *XSCANS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. (1995). *XCAD4*. University of Marburg, Germany.
- Imai, Y., Johnson, E. F., Katto, T., Kurihara, M. & Stille, J. K. (1975). *J. Polym. Sci.* **13**, 2233–2249.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2002). *SADABS*. University of Göttingen, Germany.
- Zhu, H.-J., Wang, D.-D. & Song, G.-L. (2005). *Acta Cryst.* **E61**, o2237–o2238.