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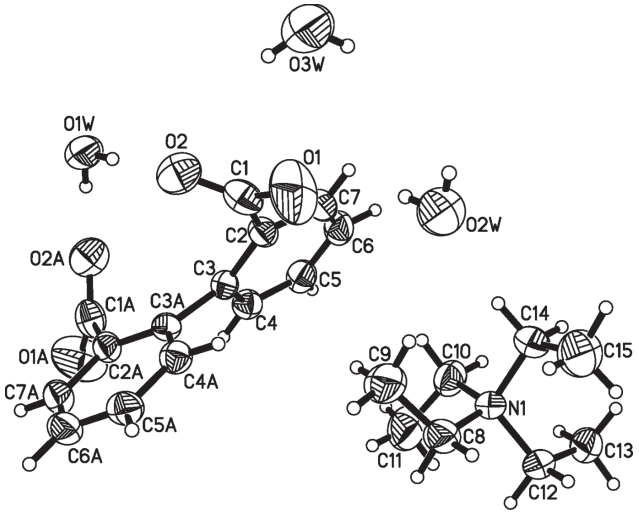
Crystal structure of bis(tetraethylammonium) [1,1′-biphenyl]-2,2′-dicarboxylate trihydrate, $C_{30}H_{54}N_2O_7$



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Abstract

$C_{30}H_{54}N_2O_7$, orthorhombic, $P2_12_12$ (no. 18), $a = 11.6240(9)$ Å, $b = 12.9628(11)$ Å, $c = 10.5166(9)$ Å, $V = 1584.6(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.038$, $wR_{ref}(F^2) = 0.115$, $T = 296$ K.

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The crystal structure is shown in the figure. Table 1 and 2 contain details on crystal structure and measurement conditions as well as a list of atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was prepared by accident. Firstly, a synthesized aromatic acid, 2,2′-biphenyldicarboxylic acid (A.R.) and tetraethylammonium (25% aqueous solution, C. R.) were

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.4 × 0.2 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω-scans
$2\theta_{max}$, completeness:	56.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4621, 2277, 0.012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2013
$N(param)_{refined}$:	193
Programs:	SHELXL [1], Bruker programs [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.21858(16)	−0.00349(16)	0.70986(19)	0.0814(6)
O1	0.31512(12)	−0.01609(19)	0.6633(2)	0.1581(8)
N1	0.22988(11)	0.16342(10)	0.15011(11)	0.0618(4)
O1W	0.0000	0.0000	0.95116(19)	0.1295(9)
H1WA	0.0579(9)	−0.016(2)	0.9057(13)	0.194*
C2	0.13925(13)	0.07349(12)	0.64484(15)	0.0595(4)
O2	0.18306(12)	−0.04730(11)	0.80602(13)	0.0973(5)
O2W	0.5000	0.0000	0.4943(2)	0.1391(10)
H2WA[a]	0.4488(9)	0.0199(19)	0.5472(12)	0.209*
H2WB[a]	0.5606(8)	−0.014(2)	0.5371(15)	0.209*
C3	0.02521(12)	0.05285(11)	0.60990(13)	0.0522(4)
O3W	0.5000	0.0000	0.8577(3)	0.1596(13)
H3WA[a]	0.503(2)	0.0471(8)	0.8008(11)	0.239*
H3WB[a]	0.4301(7)	−0.0200(15)	0.862(3)	0.239*
C4	−0.03850(14)	0.13321(12)	0.55664(14)	0.0613(4)
H4A	−0.1142	0.1206	0.5327	0.074*
C5	0.00624(16)	0.23024(13)	0.53806(15)	0.0706(5)
H5A	−0.0391	0.2824	0.5038	0.085*
C6	0.11895(18)	0.24928(13)	0.57066(17)	0.0781(6)
H6A	0.1508	0.3142	0.5574	0.094*
C7	0.18365(16)	0.17201(14)	0.62277(17)	0.0759(5)
H7A	0.2598	0.1855	0.6442	0.091*
C8	0.2110(2)	0.04930(14)	0.16940(18)	0.0848(6)
H8A	0.1373	0.0308	0.1326	0.102*
H8B	0.2700	0.0121	0.1230	0.102*
C9	0.2129(3)	0.01390(19)	0.30577(19)	0.1109(8)
H9A	0.2003	−0.0592	0.3091	0.166*
H9B	0.2863	0.0298	0.3428	0.166*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H9C	0.1533	0.0485	0.3524	0.166*
C10	0.14116(16)	0.22702(17)	0.21944(18)	0.0829(6)
H10A	0.1504	0.2154	0.3100	0.100*
H10B	0.1570	0.2993	0.2034	0.100*
C11	0.01779(19)	0.2061(3)	0.1850(2)	0.1288(10)
H11C	−0.0316	0.2502	0.2340	0.193*
H11B	0.0064	0.2194	0.0961	0.193*
H11A	−0.0002	0.1353	0.2030	0.193*
C12	0.22350(17)	0.18183(15)	0.00775(15)	0.0751(5)
H12A	0.2835	0.1417	−0.0331	0.090*
H12B	0.1502	0.1562	−0.0229	0.090*
C13	0.23594(18)	0.29290(16)	−0.03295(18)	0.0859(6)
H13A	0.2309	0.2973	−0.1239	0.129*
H13B	0.1756	0.3333	0.0047	0.129*
H13C	0.3092	0.3188	−0.0055	0.129*
C14	0.34386(14)	0.19695(16)	0.20370(17)	0.0776(5)
H14A	0.3537	0.2699	0.1864	0.093*
H14B	0.3415	0.1885	0.2953	0.093*
C15	0.4466(2)	0.1409(2)	0.1541(3)	0.1273(10)
H15B	0.5148	0.1676	0.1937	0.191*
H15A	0.4394	0.0687	0.1729	0.191*
H15C	0.4516	0.1503	0.0637	0.191*

[a]Occupancy: 0.5.

dissolved in small amount of H_2O/EtOH (v/v, 2:1) with the molar ratio of 1:1:4 to evaporate under room temperature to prepare an inclusion compound. About after 14 days, colorless block crystals of the title compound appeared in the clean solution.

Experimental details

Positions of hydrogen atoms connected to carbon atoms were modelled at the ideal positions according to the riding model, whereas those of water molecules were refined with constrained bond distances and angles. Hydrogen U_{iso} values were set to 1.2 or 1.5 times U_{eq} of the parent atoms.

Discussion

2,2′-Biphenyldicarboxylic acid is a good hydrogen-bond unit because of its two carboxyl groups. Searching the related literatures, it can be seen that the corresponding dicarboxylates can be regarded as important motif in constructing MOFs [3–6]. Here the crystal structure of the title structure is reported to enrich the related organic structures.

In the asymmetric unit of the title compound, there are one half of a 2,2′-biphenyldicarboxylate positioned around a twofold rotation axis parallel to the *c* axis, one and one half water molecules at the special positions and one tetraethylammonium cation. Observing the configuration of the anion, it can be seen that the interplanar angle between two phenyl rings is 48.1° and the dihedral angle between the carboxyl group and the related ring is 52.1°. The cation located in a general position shows the typical geometric parameters, which means that the nitrogen atom is tetrahedrally surrounded. With the help of water molecules, the anion can form 2D hydrogen bonded layers by different O—H···O hydrogen bonds. The tetraethylammonium cations are accomodated between the layers to obtain the typical sandwich-like crystal structure.

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