Yongxia Cui, Xiaohui Chen and Tingting Zhang*

Crystal structure of tetrabutylammonium 4,4'-oxydibenzoate – boric acid – water (1/2/6)

$C_{46}H_{98}B_2N_2O_{17}$

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Abstract

$C_{46}H_{98}B_2N_2O_{17}$, orthorhombic, *Pbca* (no. 61), $a = 16.485(3)$ Å, $b = 17.441(3)$ Å, $c = 41.100(7)$ Å, $V = 11817(3)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0516$, $wR_{ref}(F^2) = 0.1557$, $T = 296(2)$ K.

CCDC no.: 1911935

The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.08 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	22.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	57604, 7252, 0.043
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5024
$N(param)_{refined}$:	679
Programs:	SHELX [1, 2], Bruker [3]

contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

4,4'-Oxydibenzoic acid (0.25 mmol), boric acid (0.5 mmol) and tetrabutylammonium hydroxide (0.5 mmol) were

*Corresponding author: Tingting Zhang, Henan University of Chinese Medicine, Zhengzhou 450046, China, e-mail: ztt07979@163.com

Yongxia Cui and Xiaohui Chen: Henan University of Chinese Medicine, Zhengzhou 450046, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.7073(2)	0.94426(19)	0.58029(6)	0.0674(8)
N1	0.56831(16)	0.2718(2)	0.71713(7)	0.0947(9)
O1	0.68855(16)	0.90474(13)	0.60877(4)	0.0945(8)
O1W	0.67539(18)	0.81013(14)	0.76281(5)	0.0957(8)
H1WA	0.676(3)	0.796(2)	0.7830(4)	0.144*
H1WB	0.697(3)	0.8548(12)	0.7624(9)	0.144*
B1	0.7244(2)	0.0694(2)	0.82804(8)	0.0694(10)
C2	0.75057(19)	0.90427(18)	0.55779(7)	0.0675(8)
H2A	0.7713	0.8561	0.5628	0.081*
N2	0.44334(15)	0.24926(18)	0.49780(7)	0.0900(9)
O2	0.77343(14)	0.99363(12)	0.46490(4)	0.0801(7)
O2W	0.6626(3)	0.7008(2)	0.71327(7)	0.1385(11)
H2WB	0.663(4)	0.736(2)	0.7279(10)	0.208*
H2WA	0.672(4)	0.6578(15)	0.7228(12)	0.208*
B2	0.7734(2)	0.1021(2)	0.39576(8)	0.0646(9)
C3	0.76332(17)	0.93588(17)	0.52743(6)	0.0614(8)
H3A	0.7917	0.9080	0.5119	0.074*
O3	0.73524(13)	1.10889(12)	0.48088(4)	0.0711(6)
O3W	0.84965(17)	0.84987(15)	0.46045(5)	0.0938(7)
H3WA	0.850(2)	0.832(2)	0.4409(5)	0.141*
H3WB	0.817(2)	0.8887(16)	0.4602(9)	0.141*
C4	0.73502(16)	1.00753(16)	0.51969(6)	0.0530(7)
O4	0.7178(2)	0.96119(14)	0.75915(5)	0.1174(10)
O4W	0.68880(14)	0.24535(14)	0.51208(5)	0.0792(6)
H4WA	0.679(2)	0.2744(16)	0.4955(6)	0.119*
H4WB	0.703(2)	0.2015(10)	0.5037(7)	0.119*
C5	0.69168(19)	1.04770(18)	0.54309(7)	0.0696(8)
H5A	0.6725	1.0967	0.5385	0.084*
O5	0.75355(16)	1.07632(14)	0.74328(5)	0.0868(7)
O5W	0.15947(16)	0.21477(15)	0.59974(6)	0.0954(7)
H5WA	0.174(2)	0.187(2)	0.6159(7)	0.143*
H5WB	0.1981(18)	0.247(2)	0.5964(10)	0.143*
O6	0.72238(15)	−0.00608(12)	0.82112(4)	0.0813(7)
H6	0.723(2)	−0.013(2)	0.8004(3)	0.122*
C6	0.6769(2)	1.0149(2)	0.57328(7)	0.0796(10)
H6A	0.6463	1.0411	0.5887	0.095*
O6W	0.83641(15)	0.24923(15)	0.32581(6)	0.0915(7)
H6WB	0.818(2)	0.2184(18)	0.3405(8)	0.137*
H6WA	0.7992(18)	0.2827(18)	0.3223(10)	0.137*
O7	0.71654(17)	0.09290(13)	0.85936(5)	0.0900(7)
H7	0.712(3)	0.0553(17)	0.8727(8)	0.135*
C7	0.74867(17)	1.03946(18)	0.48607(6)	0.0579(7)
O8	0.73449(17)	0.12493(13)	0.80545(5)	0.0911(7)
H8	0.742(3)	0.108(2)	0.7861(5)	0.137*
C8	0.69960(19)	0.93656(18)	0.63925(6)	0.0642(8)
O9	0.77805(15)	0.02709(12)	0.40284(4)	0.0814(7)
H9	0.778(2)	0.019(2)	0.4236(3)	0.122*
C9	0.7436(2)	1.00152(18)	0.64558(6)	0.0733(9)
H9A	0.7671	1.0291	0.6287	0.088*
O10	0.77946(17)	0.12572(13)	0.36440(5)	0.0879(7)
H10	0.783(2)	0.0913(18)	0.3494(7)	0.132*
C10	0.75249(19)	1.02568(17)	0.67753(6)	0.0661(8)
H10A	0.7812	1.0704	0.6818	0.079*
O11	0.76303(15)	0.15763(12)	0.41854(5)	0.0801(6)
H11	0.751(2)	0.144(2)	0.4382(4)	0.120*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C11	0.72000(17)	0.98513(17)	0.70285(6)	0.0570(7)
C12	0.67686(19)	0.91959(18)	0.69559(7)	0.0719(9)
H12A	0.6543	0.8911	0.7125	0.086*
C13	0.66635(19)	0.89525(18)	0.66403(7)	0.0729(9)
H13A	0.6368	0.8510	0.6596	0.088*
C14	0.7316(2)	1.0100(2)	0.73743(7)	0.0724(9)
C17	0.6339(2)	0.2588(3)	0.69179(10)	0.1176(15)
H17A	0.6699	0.3027	0.6918	0.141*
H17B	0.6655	0.2144	0.6982	0.141*
C18	0.6031(2)	0.2463(3)	0.65738(10)	0.1195(15)
H18A	0.5679	0.2018	0.6570	0.143*
H18B	0.5713	0.2905	0.6508	0.143*
C19	0.6706(3)	0.2348(3)	0.63382(11)	0.1299(16)
H19A	0.7057	0.2794	0.6343	0.156*
H19B	0.7024	0.1908	0.6406	0.156*
C20	0.6424(3)	0.2224(3)	0.59986(11)	0.1321(16)
H20A	0.6884	0.2154	0.5859	0.198*
H20B	0.6086	0.1776	0.5990	0.198*
H20C	0.6119	0.2663	0.5928	0.198*
C21	0.5167(2)	0.3404(2)	0.70840(9)	0.0969(12)
H21A	0.4760	0.3466	0.7253	0.116*
H21B	0.4882	0.3288	0.6883	0.116*
C22	0.5592(3)	0.4165(3)	0.70406(13)	0.1348(17)
H22A	0.5825	0.4324	0.7246	0.162*
H22B	0.6030	0.4107	0.6885	0.162*
C23 ^a	0.5015(5)	0.4771(4)	0.69226(18)	0.170(2)
H23A ^a	0.4708	0.4514	0.6753	0.204*
H23B ^a	0.4638	0.4832	0.7102	0.204*
C23 ^b	0.5015(5)	0.4771(4)	0.69226(18)	0.170(2)
H23C ^b	0.4487	0.4705	0.7023	0.204*
H23D ^b	0.5217	0.5277	0.6977	0.204*
C24 ^a	0.5114(8)	0.5528(10)	0.6802(4)	0.172(8)
H24A ^a	0.4592	0.5740	0.6750	0.258*
H24B ^a	0.5372	0.5839	0.6964	0.258*
H24C ^a	0.5444	0.5516	0.6609	0.258*
C24 ^b	0.4955(6)	0.4686(7)	0.6582(3)	0.167(5)
H24D ^b	0.4589	0.5065	0.6497	0.250*
H24E ^b	0.5481	0.4753	0.6485	0.250*
H24F ^b	0.4754	0.4184	0.6531	0.250*
C25	0.6122(2)	0.2846(3)	0.74914(10)	0.1223(16)
H25A	0.6446	0.2395	0.7538	0.147*
H25B	0.6492	0.3273	0.7465	0.147*
C26	0.5591(2)	0.3005(3)	0.77822(10)	0.1251(16)
H26A	0.5271	0.3462	0.7743	0.150*
H26B	0.5222	0.2579	0.7815	0.150*
C27	0.6100(3)	0.3116(4)	0.80814(11)	0.148(2)
H27A	0.6429	0.2662	0.8115	0.178*
H27B	0.6464	0.3545	0.8046	0.178*
C28	0.5623(3)	0.3259(4)	0.83747(12)	0.175(2)
H28A	0.5981	0.3325	0.8557	0.263*
H28B	0.5305	0.3715	0.8346	0.263*
H28C	0.5271	0.2831	0.8414	0.263*
C29	0.5108(2)	0.2046(2)	0.71880(9)	0.0985(12)
H29A	0.4831	0.2005	0.6980	0.118*
H29B	0.4700	0.2156	0.7352	0.118*
C30	0.5484(3)	0.1270(3)	0.72660(13)	0.1379(17)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H30A	0.5931	0.1174	0.7118	0.166*
H30B	0.5699	0.1279	0.7486	0.166*
C31	0.4868(5)	0.0630(4)	0.72363(16)	0.173(2)
H31A	0.5106	0.0164	0.7323	0.207*
H31B	0.4403	0.0756	0.7370	0.207*
C32	0.4596(5)	0.0483(4)	0.6916(2)	0.230(4)
H32A	0.4209	0.0071	0.6920	0.345*
H32B	0.4344	0.0935	0.6830	0.345*
H32C	0.5048	0.0342	0.6782	0.345*
C33	0.3889(2)	0.2773(3)	0.47060(10)	0.1067(13)
H33A	0.3548	0.2351	0.4636	0.128*
H33B	0.3536	0.3169	0.4792	0.128*
C34	0.4317(3)	0.3087(4)	0.44148(12)	0.148(2)
H34A	0.4588	0.3560	0.4475	0.177*
H34B	0.4729	0.2725	0.4346	0.177*
C35	0.3760(3)	0.3240(5)	0.41362(16)	0.194(3)
H35A	0.3414	0.2795	0.4109	0.233*
H35B	0.3412	0.3666	0.4196	0.233*
C36	0.4104(4)	0.3405(5)	0.38385(17)	0.218(3)
H36A	0.3683	0.3492	0.3681	0.327*
H36B	0.4433	0.3857	0.3857	0.327*
H36C	0.4435	0.2982	0.3770	0.327*
C37	0.4949(2)	0.3144(2)	0.51148(10)	0.0937(11)
H37A	0.5293	0.2939	0.5285	0.112*
H37B	0.5300	0.3333	0.4943	0.112*
C38	0.4477(2)	0.3813(3)	0.52521(13)	0.1338(17)
H38A	0.4079	0.3626	0.5407	0.161*
H38B	0.4189	0.4070	0.5078	0.161*
C39 ^c	0.5039(4)	0.4381(4)	0.54203(18)	0.167(3)
H39A ^c	0.5545	0.4401	0.5300	0.200*
H39B ^c	0.4794	0.4886	0.5409	0.200*
C39 ^d	0.5039(4)	0.4381(4)	0.54203(18)	0.167(3)
H39C ^d	0.5477	0.4078	0.5511	0.200*
H39D ^d	0.5277	0.4687	0.5248	0.200*
C40 ^c	0.5203(7)	0.4228(9)	0.5726(3)	0.178(6)
H40A ^c	0.5559	0.4615	0.5812	0.267*
H40B ^c	0.4709	0.4223	0.5850	0.267*
H40C ^c	0.5461	0.3736	0.5741	0.267*
C40 ^d	0.4826(13)	0.4881(13)	0.5655(6)	0.149(10)
H40D ^d	0.5292	0.5173	0.5720	0.224*
H40E ^d	0.4415	0.5221	0.5573	0.224*
H40F ^d	0.4617	0.4605	0.5839	0.224*
C41	0.3884(2)	0.2152(2)	0.52365(10)	0.1009(12)
H41A	0.3492	0.2537	0.5301	0.121*
H41B	0.3587	0.1728	0.5141	0.121*
C42	0.4309(2)	0.1867(3)	0.55371(11)	0.1121(13)
H42A	0.4748	0.1529	0.5474	0.134*
H42B	0.4542	0.2300	0.5652	0.134*
C43	0.3745(3)	0.1447(3)	0.57593(12)	0.1248(15)
H43A	0.3532	0.1002	0.5646	0.150*
H43B	0.3292	0.1778	0.5813	0.150*
C44	0.4145(3)	0.1187(3)	0.60700(12)	0.1481(19)
H44A	0.3756	0.0922	0.6202	0.222*
H44B	0.4587	0.0849	0.6020	0.222*
H44C	0.4347	0.1625	0.6186	0.222*
C45	0.50316(19)	0.1898(2)	0.48544(10)	0.0945(11)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H45A	0.5408	0.1777	0.5028	0.113*
H45B	0.5342	0.2122	0.4678	0.113*
C46	0.4651(2)	0.1155(3)	0.47340(11)	0.1143(14)
H46A	0.4220	0.1272	0.4581	0.137*
H46B	0.4413	0.0883	0.4916	0.137*
C47	0.5277(3)	0.0643(3)	0.45690(12)	0.1226(15)
H47A	0.5761	0.0619	0.4703	0.147*
H47B	0.5061	0.0128	0.4550	0.147*
C48	0.5495(3)	0.0920(3)	0.42484(15)	0.162(2)
H48A	0.5890	0.0583	0.4153	0.243*
H48B	0.5020	0.0935	0.4113	0.243*
H48C	0.5720	0.1426	0.4266	0.243*

^aOccupancy: 0.418(12), ^bOccupancy: 0.582(12), ^cOccupancy: 0.712(17), ^dOccupancy: 0.288(17).

dissolved in a small amount of a solution of water-ethanol (50/50 v/v). The mixture was stirred for half an hour and set aside to crystallize. After 14 days colorless prism-shaped crystals suitable for single crystal X-ray diffraction were obtained.

Experimental details

All H atoms bonded to C were idealized and refined using riding model with the SHELXL program [1]. All H bonded to O were refined with the O—H distance restrained to 0.86 Å. The U_{iso} values of the H atoms bonded to O were set to $1.5U_{eq}(O)$.

Discussion

4,4'-Oxydibenzoic acid, which is generally employed to construct different MOFs [4, 5], exists as a V-configuration molecule with terminal carboxyl groups acting as good hydrogen-bond acceptors and donors. Additionally, boric acid is a threefold planar molecule that can form various hydrogen bonds. It is reasonable to conclude that 4,4'-oxydibenzoic acid and boric acid should interact with each other to generate interesting host lattice with the existence of the related guest template. Based on this idea, the title compound constructed with tetrabutylammonium 4,4'-oxydibenzoate and boric acid was synthesized, and six water molecules were also found to take part in the construction of the final crystal structure.

In the asymmetric unit of the title compound, there are one independent dianion of 4,4'-oxydibenzoate, two boric acids, six water molecules and two tetrabutylammonium cations. Two boric acid firstly link with each other to form the dimer of $(H_3BO_3)_2$, then the dianion interacts with the dimer to generate a long hydrogen-bonded chain along to the *c* axis. Between the long chains, six water molecules cooperately yield various O—H...O hydrogen bonds with the

long chains to generate the layered structure parallel to (100) plane. Finally, tetrabutylammonium cations are contained between the layers to get the final stable sandwich-like crystal structure. Geometric parameters are all in the expected ranges [6].

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