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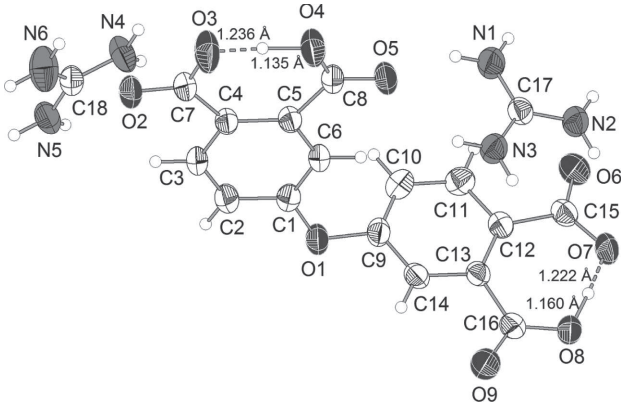
Crystal structure of bis(guanidinium) 3,3′-oxybis(6-carboxybenzoate), $C_{18}H_{20}N_6O_9$



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Abstract

$C_{18}H_{20}N_6O_9$, triclinic, $P\bar{1}$ (no. 2), $a = 7.0287(13)$ Å, $b = 10.188(2)$ Å, $c = 15.349(3)$ Å, $\alpha = 86.513(3)°$, $\beta = 88.854(3)°$, $\gamma = 75.047(3)°$, $V = 1060.0(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0427$, $wR_{ref}(F^2) = 0.1409$, $T = 296(2)$ K.

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The figure shows the asymmetric unit of the title structure. Tables 1–3 contain details of the methods used and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3,3′-Oxybis(6-carboxybenzoic acid) (99%, A. R.), guanidium hydrochloride (99%, A. R.) and tetrabutylammonium hydroxide (10% aqueous solution) were solved in small amount of water/ethanol (1:2, v/v) with a molar ratio of 1:2:2. The mixture was stirred for half an hour and set aside at room temperature. After about 7 days, colorless crystals were yielded without the existence of tetrabutylammonium.

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Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.20×0.25×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.19 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5344, 3688
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3088
$N(param)_{refined}$:	305
Programs:	SHELX [3], Bruker programs [4]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.2438	1.0586	0.1156	0.065
H(1B)	2*i*	0.3611	0.9880	0.1925	0.065
H(2A)	2*i*	0.1857	0.5600	0.5272	0.053
H(2B)	2*i*	0.4129	0.8181	−0.0214	0.054
H(2C)	2*i*	0.2780	0.9564	−0.0147	0.054
H(3)	2*i*	0.736(4)	0.896(3)	0.484(2)	0.101
H(3A)	2*i*	0.3683	0.6568	0.6136	0.049
H(3B)	2*i*	0.5893	0.7105	0.0981	0.056
H(3C)	2*i*	0.5683	0.7793	0.1819	0.056
H(4A)	2*i*	−0.0711	0.9229	0.6385	0.064
H(4B)	2*i*	0.1110	0.9686	0.6506	0.064
H(5A)	2*i*	−0.1511	0.7687	0.7366	0.064
H(5B)	2*i*	−0.0211	0.7137	0.8129	0.064
H(6A)	2*i*	0.3864	0.7338	0.3163	0.046
H(6B)	2*i*	0.3072	0.8880	0.7686	0.081
H(6C)	2*i*	0.2566	0.7859	0.8323	0.081
H(7)	2*i*	0.195(4)	0.537(3)	−0.039(2)	0.075
H(10A)	2*i*	−0.0234	0.7923	0.2871	0.054
H(11A)	2*i*	−0.0713	0.8448	0.1406	0.051
H(14A)	2*i*	0.3223	0.4289	0.2343	0.045

Experimental details

The nitrogen and carbon bonded hydrogen atoms were set on calculated positions using the AFIX 93 or AFIX 43 option of the SHELXL program [3]. The oxygen bonded hydrogen atoms were refined freely.

Discussion

3,3′-Oxybis(6-carboxybenzoic acid) is a popular constructing unit in MOFs due to its flexiblity [1, 2]. Considering

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*	0.1742(2)	0.5658(1)	0.36280(8)	0.0738(9)	0.0579(8)	0.0275(7)	−0.0370(7)	−0.0031(6)	0.0017(6)
C(1)	2*i*	0.2723(3)	0.6348(2)	0.4127(1)	0.046(1)	0.0411(9)	0.0299(9)	−0.0140(8)	0.0001(7)	−0.0031(7)
N(1)	2*i*	0.3288(3)	0.9894(2)	0.1386(1)	0.068(1)	0.0444(9)	0.0387(9)	0.0071(8)	−0.0011(8)	−0.0002(7)
O(2)	2*i*	0.5821(2)	0.7755(1)	0.66521(8)	0.0706(9)	0.0586(8)	0.0275(7)	−0.0232(7)	−0.0072(6)	0.0036(6)
C(2)	2*i*	0.2638(3)	0.6133(2)	0.5021(1)	0.058(1)	0.046(1)	0.032(1)	−0.0212(9)	0.0041(8)	0.0014(8)
N(2)	2*i*	0.3613(2)	0.8868(2)	0.0089(1)	0.0512(9)	0.0449(9)	0.0349(9)	−0.0052(7)	−0.0059(7)	0.0008(7)
O(3)	2*i*	0.7309(2)	0.8704(2)	0.56362(9)	0.081(1)	0.107(1)	0.0326(8)	−0.061(1)	−0.0102(7)	0.0068(8)
C(3)	2*i*	0.3732(3)	0.6724(2)	0.5533(1)	0.056(1)	0.044(1)	0.0244(9)	−0.0161(8)	0.0032(8)	0.0008(7)
N(3)	2*i*	0.5372(2)	0.7795(2)	0.1280(1)	0.052(1)	0.0439(9)	0.0375(9)	0.0011(7)	−0.0052(7)	−0.0026(7)
O(4)	2*i*	0.7310(2)	0.9147(2)	0.41036(9)	0.072(1)	0.085(1)	0.0334(7)	−0.0491(8)	−0.0042(7)	0.0066(7)
C(4)	2*i*	0.4912(2)	0.7546(2)	0.5194(1)	0.0407(9)	0.0340(8)	0.0271(8)	−0.0056(7)	0.0001(7)	0.0003(7)
N(4)	2*i*	0.0329(3)	0.9198(2)	0.6680(1)	0.060(1)	0.068(1)	0.0396(9)	−0.0346(9)	−0.0101(8)	0.0180(8)
O(5)	2*i*	0.5675(2)	0.8927(1)	0.29569(8)	0.0671(9)	0.0582(8)	0.0290(7)	−0.0265(7)	−0.0020(6)	0.0060(6)
C(5)	2*i*	0.4942(2)	0.7796(2)	0.4276(1)	0.0393(9)	0.0342(9)	0.0293(9)	−0.0070(7)	−0.0004(7)	0.0010(7)
N(5)	2*i*	−0.0471(3)	0.7657(2)	0.7660(1)	0.074(1)	0.053(1)	0.0371(9)	−0.0266(9)	−0.0059(8)	0.0105(7)
O(6)	2*i*	−0.0857(2)	0.8352(1)	−0.00811(9)	0.0662(9)	0.0442(8)	0.0524(9)	0.0052(7)	−0.0145(7)	0.0084(6)
C(6)	2*i*	0.3846(3)	0.7183(2)	0.3766(1)	0.050(1)	0.0408(9)	0.0241(9)	−0.0136(8)	0.0006(7)	0.0004(7)
N(6)	2*i*	0.2305(3)	0.8378(2)	0.7855(1)	0.053(1)	0.107(2)	0.044(1)	−0.024(1)	−0.0093(8)	0.009(1)
O(7)	2*i*	0.0994(2)	0.6516(1)	−0.06156(8)	0.0654(9)	0.0466(7)	0.0303(7)	−0.0016(6)	−0.0063(6)	0.0071(6)
C(7)	2*i*	0.6058(3)	0.8022(2)	0.5875(1)	0.049(1)	0.0410(9)	0.0279(9)	−0.0102(8)	−0.0033(8)	0.0031(7)
O(8)	2*i*	0.3080(2)	0.4381(1)	−0.01351(8)	0.0680(9)	0.0403(7)	0.0315(7)	−0.0012(6)	−0.0004(6)	−0.0013(5)
C(8)	2*i*	0.6020(3)	0.8687(2)	0.3739(1)	0.045(1)	0.0429(9)	0.0269(9)	−0.0128(8)	0.0012(7)	−0.0003(7)
O(9)	2*i*	0.4247(2)	0.3220(1)	0.10651(9)	0.078(1)	0.0425(8)	0.0454(8)	0.0106(7)	−0.0115(7)	0.0003(6)
C(9)	2*i*	0.1500(3)	0.6048(2)	0.2735(1)	0.048(1)	0.047(1)	0.0274(9)	−0.0233(8)	−0.0012(7)	−0.0015(7)
C(10)	2*i*	0.0348(3)	0.7293(2)	0.2468(1)	0.048(1)	0.048(1)	0.040(1)	−0.0112(8)	0.0016(8)	−0.0109(8)
C(11)	2*i*	0.0068(3)	0.7596(2)	0.1589(1)	0.0381(9)	0.0400(9)	0.046(1)	−0.0040(8)	−0.0043(8)	−0.0048(8)
C(12)	2*i*	0.0897(2)	0.6686(2)	0.0955(1)	0.0336(8)	0.0364(9)	0.0334(9)	−0.0123(7)	−0.0031(7)	0.0017(7)
C(13)	2*i*	0.2126(2)	0.5408(2)	0.1246(1)	0.0377(9)	0.0324(8)	0.0307(9)	−0.0141(7)	−0.0031(7)	0.0015(7)
C(14)	2*i*	0.2409(3)	0.5120(2)	0.2143(1)	0.046(1)	0.0337(9)	0.0338(9)	−0.0151(7)	−0.0068(8)	0.0050(7)
C(15)	2*i*	0.0293(3)	0.7235(2)	0.0034(1)	0.041(1)	0.0353(9)	0.038(1)	−0.0087(8)	−0.0060(8)	0.0056(8)
C(16)	2*i*	0.3235(3)	0.4258(2)	0.0698(1)	0.042(1)	0.0328(9)	0.036(1)	−0.0076(7)	−0.0041(8)	0.0026(7)
C(17)	2*i*	0.4098(3)	0.8855(2)	0.0913(1)	0.0377(9)	0.0372(9)	0.035(1)	−0.0089(7)	0.0032(7)	0.0016(7)
C(18)	2*i*	0.0724(3)	0.8405(2)	0.7394(1)	0.044(1)	0.046(1)	0.0294(9)	−0.0094(8)	0.0014(8)	0.0003(7)

the ability to form different hydrogen bonds, 3,3′-oxybis(6-carboxybenzoic acid) is a hydrogen bond donor and acceptor. The flexible aromatic acid should interact with tetraalkylammonium and guanidium to explore the related crystal structure, but only the title salt was obtained.

In the asymmetric unit of title compound, there are one independent 3,3′-oxybis(6-carboxybenzoate) and two guanidium ions. The anion can stabilize its T-like configuration with two strong intramolecular hydrogen bonds of O3—H···O4 and O7—H···O8, in which the two hydrogen atoms are almost positioned on the center of the related two oxygen atoms. The two rings are distorted against each other because of the steric effects and the central bridging O atom by an angle of 75.8° and the corresponding C1—O1—C9 angle is 118.1°. These T-like dianions are linked by two planar guanidium ions to form a 3-dimensional hydrogen-bonded network.

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