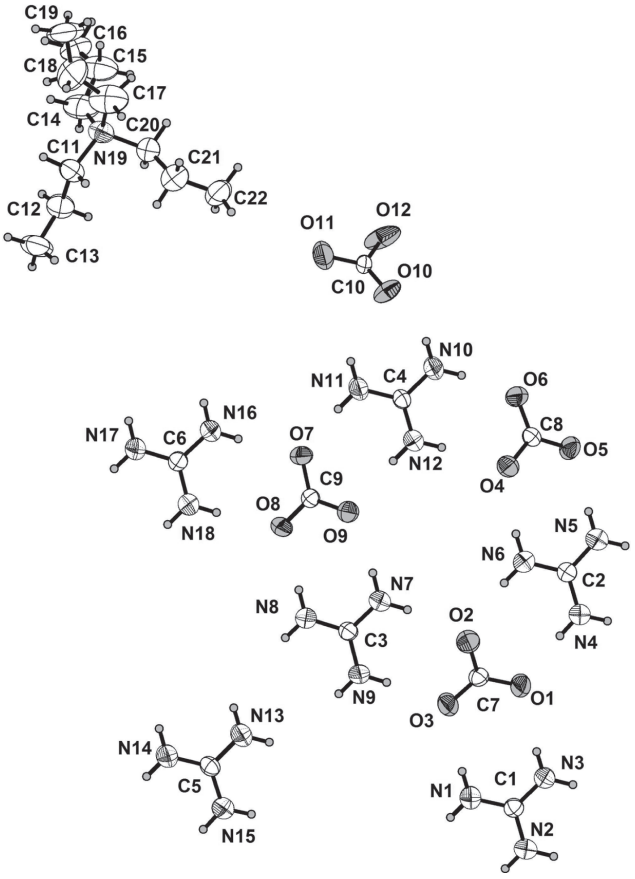
Open Access

Xin Zhu* and Xianghua Liu

Crystal structure of dodecaguanidinium bis(tetrapropylammonium) heptacarbonate, $C_{43}H_{128}N_{38}O_{21}$



DOI 10.1515/ncrs-2017-0086
Received March 31, 2017; accepted August 15, 2017; available online September 13, 2017

Abstract

$C_{43}H_{128}N_{38}O_{21}$, orthorhombic, *Pbca* (no. 61), $a = 19.556(4)$ Å, $b = 20.105(4)$ Å, $c = 20.573(4)$ Å, $V = 8089(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0592$, $wR_{ref}(F^2) = 0.1028$, $T = 296$ K.

***Corresponding author: Xin Zhu,** Henan University of Traditional Chinese Medicine, Zhengzhou, 450046, China, e-mail: xerz2@126.com
Xianghua Liu: Henan University of Traditional Chinese Medicine, Zhengzhou, 450046, China

CCDC no.: 1569060

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.24 \times 0.22 \times 0.22$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19503, 7418, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2739
$N(param)_{refined}$:	475
Programs:	Bruker programs [1], SHELX [2]

Source of materials

2,2-Bis(4-hydroxyphenyl)propane (A. R.), guanidine hydrochloride (A. R.) and tetrapropylammonium hydroxide (30% aqueous solution) were dissolved in small amount of water/ethanol (v/v, 1:2) to yield a clean solution. After vigorous stirring for an hour, the solution was set aside to generate colorless block crystals of the title compound after about 2 weeks. 2,2-Bis(4-hydroxyphenyl)propane was not present in the final structure. It can be concluded that carbonate should be from carbon dioxide during the stirring process.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 23, 33 or 93 option) [2].

Comment

Guanidinium is a hydrogen bond donor with a perfect threefold geometry due to its NH_2 groups, while carbonate is a qualified hydrogen bond acceptor with almost the similar

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8453(2)	0.66729(17)	−0.17526(18)	0.0481(10)
N1	0.79265(15)	0.63715(11)	−0.14829(13)	0.0603(9)
H1A	0.7796	0.6482	−0.1099	0.072*
H1B	0.7713	0.6064	−0.1691	0.072*
O1	0.81195(13)	0.77986(10)	−0.04001(10)	0.0617(7)
C2	0.8312(2)	0.91655(18)	0.07572(19)	0.0544(11)
N2	0.86640(15)	0.64699(11)	−0.23323(13)	0.0605(9)
H2A	0.8458	0.6148	−0.2525	0.073*
H2B	0.9007	0.6661	−0.2516	0.073*
O2	0.75993(12)	0.75288(9)	0.05309(10)	0.0570(7)
C3	0.5945(2)	0.67475(17)	0.08011(17)	0.0465(10)
N3	0.87863(15)	0.71450(12)	−0.14396(13)	0.0617(9)
H3A	0.8660	0.7259	−0.1055	0.074*
H3B	0.9130	0.7338	−0.1620	0.074*
O3	0.73193(13)	0.70107(10)	−0.03914(11)	0.0646(8)
C4	0.5988(2)	0.90809(16)	0.34587(18)	0.0454(10)
N4	0.86137(16)	0.88999(12)	0.02488(14)	0.0700(10)
H4A	0.8928	0.9116	0.0047	0.084*
H4B	0.8496	0.8510	0.0118	0.084*
O4	0.74822(12)	0.93989(9)	0.23780(10)	0.0520(7)
C5	0.7946(2)	0.18193(17)	0.32001(18)	0.0505(10)
N5	0.84912(15)	0.97632(13)	0.09589(14)	0.0749(11)
H5A	0.8806	0.9980	0.0758	0.090*
H5B	0.8293	0.9936	0.1292	0.090*
O5	0.77788(12)	1.03926(9)	0.19805(11)	0.0564(7)
C6	0.1531(2)	0.16662(17)	0.33705(18)	0.0477(10)
N6	0.78328(15)	0.88367(12)	0.10761(13)	0.0617(9)
H6A	0.7715	0.8445	0.0951	0.074*
H6B	0.7638	0.9015	0.1408	0.074*
O6	0.70469(13)	1.03301(9)	0.28084(11)	0.0600(7)
C7	0.7680(2)	0.74449(17)	−0.00831(17)	0.0495(10)
N7	0.62687(15)	0.72444(11)	0.10946(12)	0.0575(9)
H7A	0.6090	0.7431	0.1430	0.069*
H7B	0.6657	0.7379	0.0949	0.069*
O7	0.48164(12)	0.78628(10)	0.29411(11)	0.0577(7)
C8	0.7440(2)	1.00414(17)	0.23898(17)	0.0447(10)
N8	0.53504(16)	0.65396(12)	0.10222(13)	0.0655(9)
H8A	0.5142	0.6217	0.0832	0.079*
H8B	0.5170	0.6727	0.1357	0.079*
O8	0.47794(12)	0.71029(10)	0.21560(10)	0.0524(7)
C9	0.5125(2)	0.75220(17)	0.24963(17)	0.0442(9)
N9	0.62255(15)	0.64639(11)	0.02881(12)	0.0575(9)
H9A	0.6020	0.6141	0.0096	0.069*
H9B	0.6614	0.6602	0.0146	0.069*
O9	0.57663(12)	0.76053(10)	0.23924(10)	0.0518(7)
C10	1.0000	0.0000	0.0000	0.0480(15)
N10	0.62893(14)	0.96037(12)	0.37356(13)	0.0609(9)
H10A	0.6155	0.9740	0.4110	0.073*
H10B	0.6618	0.9804	0.3539	0.073*
O10[a]	0.9391(3)	0.0231(4)	−0.0112(3)	0.0609(16)
C11	0.0416(3)	0.89429(19)	0.6083(2)	0.123(2)
H11A	0.0865	0.8755	0.6161	0.148*
H11B	0.0115	0.8752	0.6408	0.148*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N11	0.54823(15)	0.87679(11)	0.37547(13)	0.0570(9)
H11C	0.5299	0.8424	0.3578	0.068*
H11D	0.5336	0.8908	0.4124	0.068*
O11[a]	1.0375(4)	0.0382(4)	0.0377(3)	0.0764(18)
C12	0.0206(3)	0.8693(2)	0.5503(2)	0.131(2)
H12A	0.0457	0.8931	0.5170	0.157*
H12B	−0.0270	0.8818	0.5454	0.157*
N12	0.62127(15)	0.88650(11)	0.28897(13)	0.0548(9)
H12C	0.6029	0.8521	0.2713	0.066*
H12D	0.6542	0.9069	0.2697	0.066*
O12[a]	1.0113(5)	−0.0586(3)	−0.0024(3)	0.088(2)
C13	0.0251(3)	0.79883(17)	0.5337(2)	0.120(2)
H13A	0.0076	0.7920	0.4906	0.179*
H13B	0.0719	0.7848	0.5354	0.179*
H13C	−0.0014	0.7733	0.5641	0.179*
N13	0.82226(15)	0.23545(12)	0.34624(13)	0.0645(9)
H13D	0.8074	0.2501	0.3829	0.077*
H13E	0.8551	0.2556	0.3267	0.077*
C14	−0.0213(3)	1.0008(2)	0.6190(2)	0.117(2)
H14A	−0.0356	0.9952	0.5742	0.141*
H14B	−0.0527	0.9748	0.6451	0.141*
N14	0.74428(16)	0.15059(12)	0.34932(13)	0.0622(9)
H14C	0.7291	0.1649	0.3860	0.075*
H14D	0.7266	0.1158	0.3319	0.075*
C15	−0.0341(3)	1.0661(2)	0.6341(2)	0.123(2)
H15A	−0.0030	1.0934	0.6089	0.148*
H15B	−0.0227	1.0726	0.6796	0.148*
N15	0.82094(15)	0.15921(12)	0.26500(13)	0.0636(9)
H15C	0.8052	0.1233	0.2479	0.076*
H15D	0.8538	0.1803	0.2465	0.076*
C16	−0.1047(2)	1.0923(2)	0.6237(2)	0.0969(16)
H16A	−0.1065	1.1382	0.6362	0.145*
H16B	−0.1167	1.0881	0.5786	0.145*
H16C	−0.1364	1.0672	0.6496	0.145*
N16	0.12556(15)	0.22207(12)	0.36024(13)	0.0632(9)
H16D	0.1411	0.2394	0.3955	0.076*
H16E	0.0921	0.2406	0.3400	0.076*
C17	0.0751(3)	0.9770(2)	0.6923(2)	0.134(2)
H17A	0.1192	0.9548	0.6941	0.161*
H17B	0.0839	1.0242	0.6970	0.161*
N17	0.20425(15)	0.13815(11)	0.36782(13)	0.0597(9)
H17C	0.2199	0.1555	0.4030	0.072*
H17D	0.2221	0.1022	0.3527	0.072*
C18	0.0380(3)	0.9567(3)	0.7478(3)	0.159(3)
H18A	−0.0045	0.9402	0.7298	0.191*
H18B	0.0625	0.9174	0.7622	0.191*
N18	0.12643(15)	0.13854(11)	0.28445(14)	0.0587(9)
H18C	0.1426	0.1015	0.2703	0.070*
H18D	0.0930	0.1574	0.2645	0.070*
N19	0.0472(2)	0.96651(14)	0.62450(14)	0.0580(9)
C19	0.0196(3)	0.9837(2)	0.8005(3)	0.192(3)
H19A	−0.0055	0.9522	0.8262	0.288*
H19B	0.0592	0.9981	0.8242	0.288*
H19C	−0.0089	1.0214	0.7912	0.288*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C20	0.0981(3)	1.0002(2)	0.5783(3)	0.1127(19)
H20A	0.0750	1.0031	0.5367	0.135*
H20B	0.1027	1.0456	0.5939	0.135*
C21	0.1627(3)	0.9795(2)	0.5647(3)	0.137(2)
H21A	0.1589	0.9342	0.5490	0.165*
H21B	0.1868	0.9774	0.6058	0.165*
C22	0.2068(3)	1.0140(2)	0.5200(2)	0.1246(19)
H22A	0.2499	0.9913	0.5174	0.187*
H22B	0.1859	1.0151	0.4778	0.187*
H22C	0.2141	1.0587	0.5350	0.187*

[a]Occupancy: 0.5.

planar shape. A searching in the CSD database [3] verified our presumption that these moieties are usually used as ancillary molecules to construct crystal structures of tetraalkylammonium salts [4–7]. However, a crystal structure containing tetraalkylammonium, guanidinium and carbonate all together has not been reported. The asymmetric unit contains one tetrapropylammonium cation six guanidinium cations, three carbonate anions at general positions and one carbonate anion at an inversion center. In the title compound, guanidinium cations and carbonate anions form hydrogen bonds to yield the 3-dimensional network. The tetrapropylammonium cations fill the cavities of the network to form the final stable crystal structure. Clearly, the two similar trigonal planar molecules are very useful to form the hydrogen bonding motif of the title structure.

Acknowledgements: This work is financially supported by Henan provincial key scientific research projects 17 A350009.

References

1. Bruker. *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA, 2012.
2. Sheldrick, G. M.: Crystal structure refinement with *SHELXL*. Acta Crystallogr. **C71** (2015) 3–8.
3. Allen, F. H.: The Cambridge Structural Database: a quarter of a million crystal structures and rising. Acta Crystallogr. **B58** (2002) 380–388.
4. Mak, T. C.; Xue, F.: Supramolecular rosette ribbon constructed from guanidinium and hydrogen carbonate ions in the crystal engineering of hydrogen-bonded networks. J. Am. Chem. Soc. **122** (2000) 9860–9861.
5. Han, J.; Yau, C. W.; Chan, C. W.; Mak, T. C.: Anionic Host Layers Constructed with Guanidinium–Hydrogen Carbonate Dimer 2: 1 Rosette-Ribbons and Bridging Carboxylate Connectors. Cryst. Growth Des. **12** (2012) 4457–4465.
6. Han, J.; Yau, C. W.; Lam, C. K.; Mak, T. C.: Designed supramolecular assembly of hydrogen-bonded anionic rosette layers. J. Am.Chem. Soc. **130** (2008) 10315–10326.
7. Lam, C. K.; Xue, F.; Zhang, J. P.; Chen, X. M.; Mak, T. C.: Hydrogen-bonded anionic rosette networks assembled with guanidinium and C_3 symmetric oxoanion building blocks. J. Am. Chem. Soc. **127** (2005) 11536–11537.