Yalin Liu* and Qiufang Li

Crystal structure of guanidinium tetrabutylammonium 5-hydroxyisophthalate dihydrate, $C_{25}H_{50}N_4O_7$

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	$0.46 \times 0.30 \times 0.220$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17687, 6678, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5046
$N(\text{param})_{\text{refined}}$:	344
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.26094(9)	−0.00743(11)	0.22203(9)	0.0338(3)
N1	0.16590(8)	0.59465(10)	0.23172(8)	0.0427(3)
H1A	0.1917	0.5341	0.2207	0.051*
H1B	0.1951	0.6565	0.2329	0.051*
O1	0.26808(7)	−0.19869(8)	0.25701(7)	0.0456(3)
O1W	0.54965(8)	0.28281(10)	0.16030(9)	0.0605(3)
H1WA	0.6046(8)	0.2910(18)	0.1884(12)	0.091*
H1WB	0.5151(12)	0.3324(15)	0.1763(13)	0.091*
C2	0.21444(9)	0.09255(11)	0.22192(10)	0.0389(3)
H2A	0.1566	0.0937	0.2362	0.047*
N2	0.03777(9)	0.49681(10)	0.24492(10)	0.0554(4)
H2B	−0.0172	0.4942	0.2549	0.066*
H2C	0.0644	0.4370	0.2336	0.066*
O2 ^a	0.13582(7)	−0.11019(8)	0.25325(8)	0.0503(3)
O2W ^a	0.4241(12)	0.4449(16)	0.2042(9)	0.086(3)
H2WA ^a	0.3710(14)	0.421(2)	0.2109(16)	0.128*
H2WB ^a	0.4487(18)	0.479(2)	0.2485(14)	0.128*
O2W ^a	0.4363(12)	0.4244(16)	0.2295(10)	0.086(3)
H2WC ^a	0.3710(14)	0.421(2)	0.2109(16)	0.128*
H2WD ^a	0.4487(18)	0.479(2)	0.2485(14)	0.128*
C3	0.25268(9)	0.19064(11)	0.20100(10)	0.0389(3)
N3	0.04311(9)	0.68655(10)	0.26408(9)	0.0485(3)
H3A	−0.0118	0.6865	0.2743	0.058*
H3B	0.0732	0.7478	0.2650	0.058*
O3	0.12022(8)	0.29529(9)	0.21203(11)	0.0764(5)
C4	0.33794(10)	0.18803(11)	0.17825(10)	0.0405(3)
H4A	0.3634	0.2531	0.1625	0.049*
N4	0.22780(10)	0.73428(11)	0.00827(8)	0.0496(3)
O4	0.24601(7)	0.38755(8)	0.20041(8)	0.0552(3)
C5	0.38556(9)	0.08909(12)	0.17884(9)	0.0384(3)
O5	0.47085(7)	0.08314(9)	0.15899(8)	0.0531(3)
H5	0.4926(14)	0.1494(10)	0.1594(13)	0.080*
C6	0.34698(9)	−0.00835(11)	0.20052(9)	0.0362(3)

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Abstract

$C_{25}H_{50}N_4O_7$, monoclinic, $P2_1/c$ (no. 14), $a = 14.7637(11)$ Å, $b = 12.0608(9)$ Å, $c = 16.7391(14)$ Å, $\beta = 101.086(1)^\circ$, $V = 2925.0(4)$, $Z = 4$, $R_{\text{gt}}(F) = 0.0489$, $wR_{\text{ref}}(F^2) = 0.1076$, $T = 296(2)$ K.

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

Source of material

5-Hydroxyisophthalic acid (A. R.), guanidine hydrochloride (A. R.) and tetrabutylammonium hydroxide (25% aqueous solution) were dissolved in small amount of water/ethanol (1:2, v/v) with the molar ratio of 1:2:2. After stirring for half an hour, the clean solution was set aside at room temperature to yield colorless crystals after about 7 days.

*Corresponding author: Yalin Liu, School of Information Technology, Henan University of Chinese Medicine, Zhengzhou 450046, China, e-mail: liuyalin_1984@163.com

Qiufang Li: Henan Province Hospital of Traditional Chinese Medicine, Zhengzhou 450002, China

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H6A	0.3790	−0.0748	0.2006	0.043*
C7	0.21861(9)	−0.11310(11)	0.24634(9)	0.0356(3)
C8	0.20253(10)	0.29971(12)	0.20461(11)	0.0466(4)
C9	0.08131(9)	0.59258(11)	0.24698(9)	0.0372(3)
C10	0.31133(12)	0.71109(15)	0.07447(10)	0.0545(4)
H10A	0.3316	0.7806	0.1012	0.065*
H10B	0.2922	0.6632	0.1147	0.065*
C11	0.39265(13)	0.65813(17)	0.04713(12)	0.0629(5)
H11A	0.4032	0.6941	−0.0021	0.076*
H11B	0.3796	0.5805	0.0349	0.076*
C13	0.56061(15)	0.6092(2)	0.09177(15)	0.0848(7)
H13A	0.6127	0.6183	0.1355	0.127*
H13B	0.5470	0.5317	0.0837	0.127*
H13C	0.5747	0.6402	0.0428	0.127*
C14	0.25274(13)	0.81361(14)	−0.05490(10)	0.0538(4)
H14A	0.2909	0.7741	−0.0866	0.065*
H14B	0.1964	0.8347	−0.0918	0.065*
C15	0.30301(13)	0.91829(15)	−0.02192(11)	0.0587(5)
H15A	0.2685	0.9556	0.0140	0.070*
H15B	0.3633	0.8992	0.0095	0.070*
C16	0.31419(16)	0.99502(17)	−0.09036(13)	0.0706(6)
H16A	0.3469	0.9562	−0.1269	0.085*
H16B	0.2536	1.0143	−0.1209	0.085*
C17	0.36551(19)	1.0997(2)	−0.06192(17)	0.0965(8)
H17A	0.3704	1.1449	−0.1081	0.145*
H17B	0.3328	1.1397	−0.0268	0.145*
H17C	0.4262	1.0815	−0.0327	0.145*
C18	0.15525(12)	0.78483(15)	0.04978(10)	0.0526(4)
H18A	0.1797	0.8535	0.0753	0.063*
H18B	0.1456	0.7350	0.0928	0.063*
C19	0.06225(13)	0.80944(16)	−0.00318(11)	0.0605(5)
H19A	0.0688	0.8678	−0.0416	0.073*
H19B	0.0392	0.7437	−0.0339	0.073*
C20	−0.00554(13)	0.84524(17)	0.04885(13)	0.0674(5)
H20A	−0.0125	0.7858	0.0862	0.081*
H20B	0.0194	0.9091	0.0810	0.081*
C21	−0.09895(15)	0.8746(2)	0.00014(17)	0.0896(7)
H21A	−0.1389	0.8966	0.0363	0.134*
H21B	−0.0929	0.9346	−0.0361	0.134*
H21C	−0.1247	0.8113	−0.0309	0.134*
C22	0.19283(14)	0.62865(15)	−0.03645(11)	0.0572(4)
H22A	0.2425	0.5965	−0.0592	0.069*
H22B	0.1435	0.6478	−0.0815	0.069*
C23	0.15725(16)	0.54142(16)	0.01521(13)	0.0702(6)
H23A	0.1075	0.5725	0.0384	0.084*
H23B	0.2065	0.5199	0.0596	0.084*
C25	0.12238(17)	0.43951(18)	−0.03525(16)	0.0818(7)
H25A	0.0911	0.3910	−0.0031	0.098*
H25B	0.0776	0.4626	−0.0826	0.098*
C26	0.1968(2)	0.3770(2)	−0.0625(2)	0.1223(12)
H26A	0.1710	0.3139	−0.0939	0.183*
H26B	0.2272	0.4240	−0.0955	0.183*
H26C	0.2407	0.3523	−0.0160	0.183*
C27	0.47778(14)	0.66828(18)	0.11282(13)	0.0695(5)
H27A	0.4648	0.6377	0.1630	0.083*
H27B	0.4926	0.7462	0.1221	0.083*

^aOccupancy: 0.5.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and ride on their parent atoms.

Discussion

In the asymmetric unit of the title compound, there are one crystallographically independent 5-hydroxyisophthalate, one guanidinium, one tetrabutylammonium and two water molecules (*cf.* the figure). It is obvious that 5-hydroxyisophthalic acid is deprotonated to form the related dianion that can interact with the guanidinium to yield the hydrogen-bonded ribbons by several N—H···O hydrogen bonds. Two water molecules further link these ribbons to extend the 2-D hydrogen-bonded layers with varied O—H···O hydrogen bonds. Finally, the tetrabutylammonium cation is contained between the layers to obtain the 3-D sandwich-like crystal structure [4–6].

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