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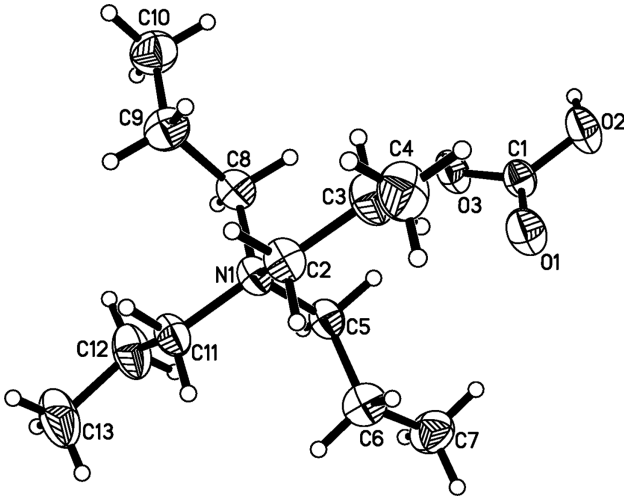
Crystal structure of tetrapropylammonium hydrogen carbonate, $C_{13}H_{29}NO_3$



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Abstract

$C_{13}H_{29}NO_3$, monoclinic, $P2_1/n$ (no. 14), $a = 8.9493(3)$ Å, $b = 15.2470(7)$ Å, $c = 11.3846(5)$ Å, $\beta = 93.824(3)°$, $V = 1549.97(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0501$, $wR_{ref}(F^2) = 0.1849$, $T = 296$ K.

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

Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.63 × 0.23 × 0.16 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer	CCD area detector
$2\theta_{max}$, completeness:	58°, 97.3%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9797, 3952, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2349
$N(param)_{refined}$:	157
Programs:	Bruker programs [5], SHELXL2014/7 [6]

Source of materials

The title compound was prepared by accident. Firstly, a synthesized aromatic acid and tetrapropylammonium (25% aqueous solution, C. R.) were dissolved in small amount of H_2O/EtOH (v/v, 2:1) with the molar ratio of 1:3 to evaporate at room temperature to prepare the cocrystal of the related acid. About after 7 days, colorless block crystals appeared in the solution. Determined by single crystal X-ray diffraction, the crystal was proved to be the title compound. It can be concluded that the anions should be from the vigorous stirring during preparation of the solution.

Discussion

Bicarbonate is a triangular anion with three O atoms and one H atom. Due to the existence of H and O atoms, it can act as hydrogen bond donor and acceptor. In many crystal structures, bicarbonate was regarded as the ancillary molecule to help different host molecules form various hydrogen-bonded host frameworks under the guidance of tetraalkylammonium template [1–3]. However, the crystal structures that only involving bicarbonate and tetraalkylammonium are rarely reported. Only two structures of tetraethylammonium bicarbonate and tetrabutylammonium bicarbonate have been reported [4, 5].

Hydrogen carbonate can act as hydrogen bond donor and acceptor. In many crystal structures, hydrogen carbonate was regarded as the ancillary anion to form various hydrogen-bonded frameworks under the guidance of the tetraalkylammonium template [1–3]. However, the crystal structures that only involve hydrogen carbonate and tetraalkylammonium are rarely reported.

There is one crystallography independent hydrogen carbonate anion and one tetrapropylammonium cation in the asymmetric unit of the title compound. In the structure, the

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.3411(7)	0.26406(10)	−0.1012(4)	0.1293(17)
H1A	−0.4716	0.2549	−0.1178	0.194*
H1B	−0.3171	0.2728	−0.1831	0.194*
H1C	−0.3359	0.2791	−0.0336	0.194*
C2	−0.2196(4)	0.22459(6)	0.0489(3)	0.0588(7)
C3	−0.0928(4)	0.20057(6)	0.0757(3)	0.0633(7)
H3	0.0066	0.1975	0.0254	0.076*
C4	−0.1137(4)	0.18125(6)	0.1767(3)	0.0610(7)
H4	−0.0282	0.1650	0.1943	0.073*
C5	−0.2587(4)	0.18557(5)	0.2517(2)	0.0477(6)
C6	−0.3827(5)	0.20947(7)	0.2264(3)	0.0724(9)
H6	−0.4807	0.2126	0.2777	0.087*
C7	−0.3637(5)	0.22914(7)	0.1245(3)	0.0789(9)
H7	−0.4489	0.2454	0.1076	0.095*
C8	−0.3962(4)	0.14275(6)	0.3527(2)	0.0507(6)
C9	−0.5151(4)	0.13514(6)	0.2117(3)	0.0504(6)
C10	−0.1313(4)	0.17106(5)	0.4890(2)	0.0496(6)
C11	0.0482(4)	0.15607(6)	0.5151(2)	0.0576(7)
H11	0.0779	0.1424	0.4531	0.069*
C12	0.1853(4)	0.16125(6)	0.6335(3)	0.0629(7)
H12	0.3077	0.1512	0.6510	0.075*
C13	0.1395(4)	0.18131(6)	0.7255(3)	0.0596(7)
C14	−0.0412(5)	0.19644(6)	0.6983(3)	0.0633(7)
H14	−0.0713	0.2101	0.7601	0.076*
C15	−0.1772(4)	0.19128(6)	0.5798(3)	0.0595(7)
H15	−0.2992	0.2014	0.5616	0.071*
C16	0.4445(5)	0.17124(8)	0.8805(3)	0.0927(11)
H16A	0.4118	0.1506	0.8745	0.139*
H16B	0.5101	0.1759	0.9711	0.139*
H16C	0.5328	0.1758	0.8198	0.139*
C17	1.5746(4)	0.04762(7)	1.5043(3)	0.0717(8)
H17A	1.5792	0.0303	1.4501	0.108*
H17B	1.6307	0.0431	1.5969	0.108*
H17C	1.6512	0.0632	1.4738	0.108*
C18	1.2664(4)	0.06381(5)	1.3672(2)	0.0468(6)

planar anion is connected to another symmetry related anion to form an anionic dimer by classical O—H···O hydrogen bonds. These dimers and tetrapropylammonium cations are arranged to form the final stable salt structure. Obviously, the structure of the title compound is different from the structure of tetraethylammonium hydrogen carbonate [3], in which the related anions and water molecules yield hydrogen-bonded layers and the cations were contained between the layers to form the related structure. Meanwhile, in the structure of tetrabutylammonium bicarbonate, the compound only exist separate cations and anions due to the absence of water molecule [4], which is very similar with the title compound.

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