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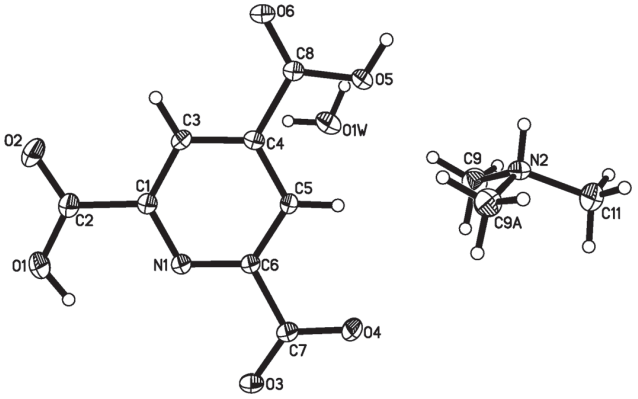
Crystal structure of trimethyammonium 2,6-dicarboxyisonicotinate monohydrate, $C_{11}H_{16}N_2O_7$



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Abstract

$C_{11}H_{16}N_2O_7$, orthorhombic, *Pnma* (no. 62), $a = 13.753(3)$ Å, $b = 6.9471(15)$ Å, $c = 13.797(3)$ Å, $V = 1318.2(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0386$, $wR_{ref}(F^2) = 0.1650$, $T = 296$ K.

CCDC no.: 1577314

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.

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.310 × 0.20 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.12 mm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω-scans
$2\theta_{max}$, completeness:	25°, 92.5%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2337, 1176, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 952
$N(param)_{refined}$:	129
Programs:	SHELX [1]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	−0.00104(13)	0.7500	−0.24357(14)	0.0512(6)
O1W	0.16822(18)	0.2500	0.1588(2)	0.1134(16)
H1WA	0.2249(19)	0.2500	0.184(4)	0.170*
H1WB[a]	0.171(4)	0.204(12)	0.101(2)	0.170*
H1	−0.050(2)	0.7500	−0.205(2)	0.077*
O2	0.15759(15)	0.7500	−0.24728(16)	0.0771(9)
O3	−0.17174(12)	0.7500	0.04999(15)	0.0445(6)
O4	−0.09335(12)	0.7500	0.19156(16)	0.0584(7)
O5	0.25857(12)	0.7500	0.20436(14)	0.0527(7)
O6	0.34730(12)	0.7500	0.06927(15)	0.0526(6)
N1	0.00062(13)	0.7500	−0.04626(15)	0.0293(5)
N2	0.13312(14)	0.7500	0.42134(15)	0.0358(6)
H2	0.1964	0.7500	0.4264	0.043*
C1	0.08627(15)	0.7500	−0.0920(2)	0.0311(6)
C2	0.08378(17)	0.7500	−0.2003(2)	0.0427(7)
C3	0.17511(16)	0.7500	−0.04319(19)	0.0328(6)
H3A	0.2334	0.7500	−0.0774	0.039*
C4	0.17479(16)	0.7500	0.05624(19)	0.0310(6)
C5	0.08616(14)	0.7500	0.1041(2)	0.0319(6)
H5A	0.0838	0.7500	0.1714	0.038*
H5	0.3125(16)	0.7500	0.237(2)	0.079*
C6	0.00097(15)	0.7500	0.04994(19)	0.0286(6)
C7	−0.09705(15)	0.7500	0.1010(2)	0.0333(6)
C8	0.26997(16)	0.7500	0.1110(2)	0.0354(6)
C9	0.10307(14)	0.5741(3)	0.36902(16)	0.0470(6)
H9A	0.1377	0.5660	0.3087	0.070*
H9B	0.0345	0.5790	0.3565	0.070*
H9C	0.1176	0.4631	0.4078	0.070*
C11	0.0954(2)	0.7500	0.5219(2)	0.0535(9)
H11A	0.1178	0.6373	0.5553	0.064*
H11B	0.0257	0.7500	0.5201	0.064*

[a]Occupancy: 0.5.

Source of material

Pyridine-2,4,6-tricarboxylic acid was synthesized with 2,4,6-trimethyl pyridine (99%, A. R.), which is commercially afforded by the Alfa Aesar company, following the procedures in the literature [2]. The acid was dissolved in small amount of trimethylamine (30% aqueous solution) with the molar ratio of 1:3. After stirring for half an hour, the clean solution was set aside to yield colorless crystals after about 7 days.

Experimental details

Carbon-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the

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riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). U_{iso}(H) = 1.5U_{eq}(C).

Discussion

Pyridine-2,4,6-tricarboxylic acid is in a perfect triangular configuration with three carboxyl groups and one nitrogen atom located in the six-membered ring. Obviously, the acid is a good hydrogen bond donor and acceptor, which is very similar with 1,3,5-benzenetricarboxylic acid except the only nitrogen atom of the ring. Searching in CSD database [3], it can be found that pyridine-2,4,6-tricarboxylic acid can interact with tetraalkylammonium cations to form varied crystal structures [4]. However, the crystal structure of pyridine-2,4,6-tricarboxylic acid and trialkylammonium has not been reported before. Herein the crystal structure of the title compound is described to enrich the related crystal structures of pyridine-2,4,6-tricarboxylic acid.

In the asymmetric unit of the title compound, the anion and the trimethylammonium cation are both as well as the water molecule located on mirror planes. One proton of pyridine-2,4,6-tricarboxylic acid was deprotonated and accepted by trimethylamine to generate the related cation. With the help of the water molecule, the anion connects with each other to form the hydrogen-bonded layers by O—H···O and O—H···N interactions. The cation is contained between the aforementioned layers to construct the final stable crystal structure with the existence of N—H···O hydrogen bonds and weak C—H···O contacts. Bond lengths and angles, espcially those of the anion, are in the expected ranges [5].

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