Yang Liu, Xiao-Yuan Zheng, Liu-Lei Qin, Chun-Li Zhu and Zunqi Liu*

Crystal structure of citric acid–acetonitrile (1/1), $C_8H_{11}NO_7$

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Abstract

$C_8H_{11}NO_7$, triclinic, $P\bar{1}$ (no. 2), $a = 5.5944(13)$ Å, $b = 10.402(3)$ Å, $c = 10.666(3)$ Å, $\alpha = 110.297(2)^\circ$, $\beta = 101.342(3)^\circ$, $\gamma = 104.701(2)^\circ$, $V = 534.6(2)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0318$, $wR_{ref}(F^2) = 0.0833$, $T = 293(2)$ K.

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The asymmetric unit of the title 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 materials

The title compound was prepared upon reacting citric acid (0.55 g, 2.86 mmol) with acetonitrile in a mixture of aqueous solution and acetonitrile. Single crystals were obtained upon evaporation of the solvent at room temperature conditions. Yield: 46% (based on citric acid); m.p: 160 °C, Elemental analysis: found (%) C, 40.81; H, 4.36; N, 5.84; calcd (%) C, 40.92; H, 4.42; N, 5.98; for $C_8H_{11}NO_7$: C, 41.21; H, 4.75; N, 6.01. IR (KBr pellet (cm⁻¹)): 3495(s), 3292(s), 1755(vs), 1712(vs), 1415(m), 1370(m), 1242(s), 1217(s), 1199(s), 1180(s), 1141(s),

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.22 × 0.21 × 0.2 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.13 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
θ_{max} , completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	4273, 1900, 0.014
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1791
$N(param)_{refined}$:	151
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [3]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	0.6344(3)	−0.17007(13)	0.11160(12)	0.0530(3)
O2	0.5859(2)	−0.26459(13)	0.26514(13)	0.0508(3)
H2	0.681723	−0.305487	0.233037	0.076*
O3	0.71657(17)	0.10159(10)	0.41818(9)	0.0316(2)
H3	0.815639	0.180677	0.429412	0.047*
O4	0.3138(2)	0.08143(14)	0.10177(11)	0.0477(3)
H4	0.346675	0.102617	0.038311	0.072*
O5	0.7469(2)	0.16162(12)	0.19289(12)	0.0485(3)
O6	0.5906(2)	0.36407(10)	0.43320(13)	0.0488(3)
O7	0.22138(18)	0.34600(10)	0.48584(11)	0.0383(3)
H7	0.278316	0.435099	0.512809	0.057*
N1	−0.1228(4)	0.55783(19)	0.1767(2)	0.0725(5)
C1	0.5393(3)	−0.18198(14)	0.20076(13)	0.0311(3)
C2	0.3577(3)	−0.10645(13)	0.25212(14)	0.0315(3)
H2A	0.309794	−0.133701	0.324403	0.038*
H2B	0.200046	−0.139718	0.174617	0.038*
C3	0.4801(2)	0.06086(13)	0.31220(13)	0.0249(3)
C4	0.5335(3)	0.10849(14)	0.19648(13)	0.0299(3)
C5	0.2902(2)	0.12614(13)	0.37389(13)	0.0274(3)
H5A	0.124444	0.087547	0.300564	0.033*
H5B	0.261514	0.095227	0.447330	0.033*
C6	0.3845(2)	0.28966(14)	0.43387(13)	0.0289(3)
C7	−0.0003(3)	0.48598(18)	0.16316(18)	0.0479(4)
C8	0.1587(3)	0.39477(18)	0.1450(2)	0.0525(4)
H8A	0.211889	0.387703	0.063319	0.079*
H8B	0.059802	0.298853	0.133036	0.079*
H8C	0.309802	0.437297	0.226611	0.079*

*Corresponding author: Zunqi Liu, Chemical Engineering College, Xinjiang Agricultural University, Urumqi 830052, P.R. China, e-mail: zunqi85@163.com

Yang Liu, Xiao-Yuan Zheng, Liu-Lei Qin and Chun-Li Zhu: Chemical Engineering College, Xinjiang Agricultural University, Urumqi 830052, P.R. China

1090(w), 1050(w), 950(m) 841(w), 821(w), 783(w), 783(m), 701(w), 652(w), 602(w), 545(w), 498(w).

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package. All H atoms were located geometrically ($d(\text{C}_{\text{methyl}}\text{-H}) = 0.96 \text{ \AA}$, $d(\text{C}_{\text{methylene}}\text{-H}) = 0.97 \text{ \AA}$ and $d(\text{O-H}) = 0.90 \text{ \AA}$) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}_{\text{methyl}}$ and $\text{C}_{\text{methylene}})$ and $1.7 U_{\text{eq}}(\text{O})$.

Comment

Similar compounds may have wide applications in many fields of materials research [5, 6]. Recently, this kind of organic materials has been identified to be of considerable importance owing to their synthetic flexibility to design and easily degrades [7, 8]. Furthermore, they can demonstrate interesting supramolecular interaction through the non-covalent interactions such as hydrogen bonds, ionic interactions, hydrophobic bonding and van der Waals [9]. Among them, the proton transfer in hydrogen-bonding interactions of the crystal packing has aroused great interest because of its fascinating physical properties.

Citric acid has three position of different proton-donor centers. Therefore, it can be designed for crystal materials, which meet with the requirements for hydrogen-bonding type ferroelectrics. Herein, a report on the synthesis and crystal structure of the title compound $[\text{C}_6\text{H}_8\text{O}_7](\text{CH}_3\text{CN})$ is presented. The asymmetric unit content contains one citric acid and one acetonitrile molecule, which are constructed by hydrogen-bonding interaction between neighboring molecules. The bond lengths represent single bonds for $\text{C}_1\text{-O}_1$ (1.316 Å), $\text{C}_3\text{-O}_3$ (1.415 Å), $\text{C}_4\text{-O}_4$ (1.323 Å), $\text{C}_6\text{-O}_7$ (1.315 Å), and double bonds for $\text{C}_1\text{-O}_1$ (1.204 Å), $\text{C}_4\text{-O}_5$ (1.196 Å), $\text{C}_6\text{-O}_6$ (1.218 Å), and triple bonds for $\text{C}_7\text{-N}_1$ (1.128 Å), which agree with the literature [10, 11].

The crystal exhibits weak intermolecular $\text{O-H}\cdots\text{O}$ and $\text{O-H}\cdots\text{N}$ hydrogen bonds. Adjacent molecules are connected through $\text{O}_3\text{-H}_3\text{WA}\cdots\text{O}_6$, $\text{O}_3\text{-H}_3\text{WA}\cdots\text{O}_7$, $\text{O}_4\text{-H}_4\text{WA}\cdots\text{O}_1$, $\text{O}_7\text{-H}_7\text{WA}\cdots\text{O}_6$ and $\text{O}_2\text{-H}_2\text{WA}\cdots\text{N}_1$ hydrogen bonds: $d(\text{O}_3\cdots\text{O}_6) = 2.95 \text{ \AA}$, bond angle ($\text{O}_3\text{-H}_3\text{WA}\cdots\text{O}_6$) = 112.8°; $d(\text{O}_3\cdots\text{O}_7) = 3.04 \text{ \AA}$, bond angle ($\text{O}_3\text{-H}_3\text{WA}\cdots\text{O}_7$) = 152.7°; $d(\text{O}_4\cdots\text{O}_1) = 2.77 \text{ \AA}$, bond angle ($\text{O}_4\text{-H}_4\text{WA}\cdots\text{O}_1$) = 167.9°; $d(\text{O}_7\cdots\text{O}_6) = 2.68 \text{ \AA}$, bond angle

($\text{O}_7\text{-H}_7\text{WA}\cdots\text{O}_6$) = 177.6°; $d(\text{O}_2\cdots\text{N}_1) = 2.81 \text{ \AA}$, bond angle ($\text{O}_2\text{-H}_2\text{WA}\cdots\text{N}_1$) = 164.6°. Intramolecular $\text{O-H}\cdots\text{O}$ and $\text{O-H}\cdots\text{N}$ hydrogen bonds may influence the molecular conformation. In the crystal, the molecules are linked by $\text{O-H}\cdots\text{O}$ hydrogen bonds into two-dimensional layered structure.

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