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The crystal structure of 3,5-dicarboxybenzenaminium perchlorate monohydrate, $C_8H_8ClNO_9$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.25 \times 0.15 \times 0.09$ mm
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.39 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.2° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17482, 2024, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1827
$N(\text{param})_{\text{refined}}$:	180
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2078(3)	0.0638(2)	0.45455(11)	0.0131(4)
C2	0.3756(3)	0.1048(2)	0.42138(11)	0.0125(4)
C3	0.3941(3)	0.0312(2)	0.35643(11)	0.0131(4)
H3	0.3002(3)	−0.0405(2)	0.33421(11)	0.0157(5)*
C4	0.5515(3)	0.0644(2)	0.32497(11)	0.0126(4)
C5	0.6899(3)	0.1691(2)	0.35606(11)	0.0137(4)
H5	0.7972(3)	0.1906(2)	0.33371(11)	0.0164(5)*
C6	0.6686(3)	0.2425(2)	0.42085(11)	0.0137(4)
C7	0.5121(3)	0.2112(2)	0.45407(11)	0.0130(4)
H7	0.4985(3)	0.2614(2)	0.49820(11)	0.0156(5)*
C8	0.8149(3)	0.3568(3)	0.45430(11)	0.0157(4)
Cl1	0.07953(7)	0.06810(6)	0.18260(3)	0.01678(18)
H1	0.099(5)	0.099(4)	0.5339(19)	0.051(10)*
H3a	0.908(8)	0.483(7)	0.541(3)	0.13(2)*
N1	0.5731(2)	−0.0127(2)	0.25680(9)	0.0158(4)
H1a	0.6058(2)	0.0595(2)	0.22535(9)	0.0189(5)*
H1b	0.6658(2)	−0.0867(2)	0.26573(9)	0.0189(5)*
H1c	0.4612(2)	−0.0586(2)	0.23703(9)	0.0189(5)*
O1	0.2000(2)	0.13317(19)	0.51658(8)	0.0185(4)
O2	0.0874(2)	−0.03085(19)	0.42635(8)	0.0185(4)
O3	0.8048(3)	0.4030(2)	0.51798(9)	0.0278(4)
O4	0.9415(2)	0.3993(2)	0.41818(9)	0.0233(4)
O5	−0.0293(3)	0.0159(3)	0.23594(12)	0.0559(7)
O6	0.1932(3)	0.1986(2)	0.20877(12)	0.0516(7)
O7	0.2019(2)	−0.0569(2)	0.16849(10)	0.0325(5)
O8	−0.0494(2)	0.1079(2)	0.11703(9)	0.0248(4)
O1W	0.6548(2)	0.25237(19)	0.17656(8)	0.0210(4)
H1Wa	0.5537(2)	0.29702(19)	0.15639(8)	0.0315(6)*
H1Wb	0.7109(2)	0.22378(19)	0.14205(8)	0.0315(6)*

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Abstract

$C_8H_8ClNO_9$, monoclinic, $P2_1/n$ (no. 14), $a = 7.1284(3) \text{ \AA}$, $b = 8.5538(5) \text{ \AA}$, $c = 18.7089(10) \text{ \AA}$, $\beta = 99.760(2)^\circ$, $V = 1124.26(10) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0381$, $wR_{\text{ref}}(F^2) = 0.1031$, $T = 150 \text{ K}$.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All starting materials are commercially available and used without further purification. The title compound was synthesized in water: solid 5-aminoisophthalic acid (1.81 g,

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0.01 mol) was dissolved in 6 M HClO₄ (10 mL) in a flask equipped with a stirrer and stirred under room temperature for 10 min. Then the solution was filtered and let evaporate at room temperature in air. Colorless single crystals were obtained after five days, yield: 86.8% (based on 5-aminoisophthalic acid).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C atoms and N atoms were positioned with idealized geometry and refined isotropic ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.2U_{\text{eq}}(\text{N})$ for all H atoms) using a riding model with C–H = 0.950 Å and N–H = 0.91 Å. The water H-atom positions were fixed as found (O–H = 0.846–1.043 Å, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$).

Comment

In general, 5-aminoisophthalic acid and its derivatives are the raw materials of contrast agents and dyes. 5-Aminoisophthalic acid is adapt to crystallize in the form of zwitterion [5–8]. To the best of our knowledge, only two examples of 3,5-dicarboxybenzenaminium salts have been characterized structurally: one is the sulfate salt [9], and a nitrate salt [10]. The crystal structure of the perchlorate salt of the ligand has not been reported. Thus, we herein report the crystal structure of 3,5-dicarboxybenzenaminium perchlorate monohydrate.

As shown in the figure, the title compound, the molecule formula of C₈H₈ClNO₉, crystallizes in the monoclinic space group $P2_1/n$ with one crystal water molecules. The title compound is a perchlorate salt of 5-aminoisophthalic acid. The amino group is protonated to form the organic cation, which is countered by one perchlorate anion. There are three kinds of hydrogen bonds: O–H...O(carboxyl), O–H...O(perchlorate), N–H...O(water) and N–H...O(perchlorate), which link the

moieties to a three-dimensional supramolecular structure. All bond lengths and angles of the title compound are comparable to its analogues [9, 10].

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