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Crystal structure of 3-(3-(4-carboxyphenyl)ureido)pyridin-1-ium perchlorate, $C_{26}H_{24}Cl_2N_6O_{14}$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.24 × 0.23 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.31 mm ⁻¹
Diffractometer, scan mode:	CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18314, 5101, 0.060
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3165
$N(\text{param})_{\text{refined}}$:	433
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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Abstract

$C_{26}H_{24}Cl_2N_6O_{14}$, monoclinic, $P2_1/c$ (no. 14), $a = 15.7471(18)$ Å, $b = 14.1486(15)$ Å, $c = 13.7894(15)$ Å, $\beta = 109.588(3)^\circ$, $V = 2894.5(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0744$, $wR_{\text{ref}}(F^2) = 0.2246$, $T = 293(2)$ K.

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The molecular 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

A solution of pyridine-3-isocyanate (4 mmol) in ethanol (15 mL) was added to a stirred ethanol solution (15 mL) of

p-aminobenzoic acid (4 mmol) in a three-necked flask. The reaction mixture was refluxed for 4 h, after that the solution was cooled to room temperature. The white precipitated residue was washed with ethanol and ether 3 times. The white solids (0.1 mmol) were dissolved in 15 mL ethanol. Five drops of HClO₄ were added. Single crystals were obtained from an ethanol by slow evaporation at room temperature.

Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically. The use of a different weighting scheme may reduce the wR^2 factor to 15%.

Comment

As a double hydrogen bond donor, ureido derivatives may form double hydrogen bonds to a Y-type hydrogen bond binding site such as AcO⁻ or tetrahedral anions (such as SO₄²⁻) [5]. It is more likely that a hydrogen bond may form between ureido and ureido, or between ureido and solvent molecules, etc., which may assemble into supramolecular compounds. This phenomenon has caused extensive attention [6].

In the crystal structure of the title compound, the asymmetric unit consists of two organic cations and two ClO₄⁻ anions. The structure of the two independent molecules are slightly different; the dihedral angle of the benzene ring and pyridine ring of one molecule is 172°, and the dihedral angle of the benzene ring and pyridine ring of the other molecule is 161°. The bond lengths and bond angles are in the normal ranges. There are hydrogen bonds between molecules.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	−0.3160(4)	0.0886(6)	0.6687(4)	0.100(2)
H1	−0.2648	0.0786	0.6509	0.120*
C2	−0.3105(4)	0.0823(7)	0.7680(4)	0.117(3)
H2	−0.2551	0.0687	0.8175	0.140*
C3	−0.3815(5)	0.0949(6)	0.7971(5)	0.097(2)
H3	−0.3763	0.0927	0.8663	0.116*
C4	−0.4704(4)	0.1178(5)	0.6211(4)	0.0808(18)
H4	−0.5268	0.1279	0.5721	0.097*
C5	−0.3977(3)	0.1098(3)	0.5920(4)	0.0546(12)
C6	−0.4711(3)	0.1253(3)	0.4040(3)	0.0522(12)
C7	−0.4978(3)	0.1364(4)	0.2154(3)	0.0531(12)
C8	−0.4524(3)	0.1265(4)	0.1447(4)	0.0689(16)
H8	−0.3901	0.1195	0.1682	0.083*
C9	−0.4985(4)	0.1271(4)	0.0415(4)	0.0683(15)
H9	−0.4675	0.1210	−0.0049	0.082*
C10	−0.5932(3)	0.1370(3)	0.0047(3)	0.0534(12)
C11	−0.6375(3)	0.1462(3)	0.0751(4)	0.0553(13)
H11	−0.6999	0.1531	0.0515	0.066*
C12	−0.5915(3)	0.1455(4)	0.1797(4)	0.0581(13)
H12	−0.6227	0.1510	0.2260	0.070*
C13	−0.6400(4)	0.1389(4)	−0.1062(4)	0.0580(13)
C14	0.0123(3)	0.3871(3)	0.3999(3)	0.0452(11)
C15	−0.0603(3)	0.3625(3)	0.3133(3)	0.0545(12)
H15	−0.1108	0.3337	0.3209	0.065*
C16	−0.0574(4)	0.3809(4)	0.2163(4)	0.0606(14)
H16	−0.1055	0.3636	0.1585	0.073*
C17	0.0157(3)	0.4244(3)	0.2046(3)	0.0545(12)
H17	0.0180	0.4378	0.1395	0.065*
C18	0.0857(3)	0.4299(3)	0.3849(3)	0.0489(11)
H18	0.1358	0.4464	0.4408	0.059*
C19	0.0743(3)	0.3810(3)	0.5899(3)	0.0449(11)
C20	0.0991(3)	0.3575(3)	0.7761(3)	0.0456(11)
C21	0.1923(3)	0.3687(4)	0.8142(4)	0.0576(13)
H21	0.2247	0.3762	0.7694	0.069*
C22	0.2358(3)	0.3685(4)	0.9190(4)	0.0612(14)
H22	0.2980	0.3763	0.9445	0.073*
C23	0.1896(3)	0.3571(3)	0.9872(3)	0.0467(11)
C24	0.0976(3)	0.3458(3)	0.9497(3)	0.0487(11)
H24	0.0658	0.3376	0.9950	0.058*
C25	0.0524(3)	0.3467(3)	0.8447(3)	0.0502(12)
H25	−0.0099	0.3400	0.8199	0.060*
C26	0.2370(3)	0.3570(3)	1.0999(3)	0.0514(12)
N1	−0.3973(3)	0.1159(3)	0.4926(3)	0.0594(11)
H1A	−0.3454	0.1136	0.4848	0.071*
N2	−0.4443(3)	0.1353(3)	0.3199(3)	0.0670(13)
H2A	−0.3873	0.1418	0.3326	0.080*
N3	0.0476(2)	0.3563(3)	0.6713(3)	0.0492(10)
H3A	−0.0073	0.3379	0.6562	0.059*
N4	0.0076(3)	0.3677(3)	0.4969(3)	0.0519(10)
H4A	−0.0425	0.3449	0.4987	0.062*
N5	0.0835(3)	0.4471(3)	0.2884(3)	0.0523(10)
H5	0.1293	0.4749	0.2805	0.063*
N6	−0.4615(5)	0.1109(5)	0.7239(5)	0.111(2)
H6	−0.5086	0.1172	0.7417	0.133*
O1	−0.5977(3)	0.1258(4)	−0.1692(3)	0.0928(15)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O2	−0.7256(2)	0.1528(2)	−0.1377(2)	0.0633(10)
H2B	−0.7450	0.1527	−0.2008	0.095*
O3	0.3201(2)	0.3818(3)	1.1319(3)	0.0830(13)
H3B	0.3398	0.3795	1.1950	0.125*
O4	0.1962(2)	0.3326(3)	1.1604(2)	0.0641(10)
O5	−0.5484(2)	0.1241(3)	0.4026(3)	0.0736(11)
O6	0.1483(2)	0.4106(3)	0.5983(2)	0.0685(10)
O7	−0.1470(3)	0.3210(3)	0.6502(3)	0.0963(14)
O8	−0.2920(3)	0.2929(3)	0.5344(4)	0.0991(15)
O9	−0.2521(3)	0.4403(3)	0.6048(3)	0.0824(12)
O10	−0.1874(3)	0.3820(3)	0.4873(3)	0.0839(12)
O11	−0.2486(3)	0.1750(3)	0.3333(3)	0.0867(13)
O12	−0.1522(3)	0.0446(3)	0.3595(3)	0.0911(13)
O13	−0.1057(2)	0.1798(3)	0.4577(3)	0.0844(12)
O14	−0.2164(3)	0.0860(3)	0.4832(3)	0.0772(11)
Cl1	−0.18074(8)	0.12159(8)	0.40724(8)	0.0512(4)
Cl2	−0.22089(8)	0.35742(8)	0.56956(9)	0.0512(4)

Carboxy groups within the molecules meet *via* the hydrogen bonds, such as O2—H2B···O4 and O3—H3B···O1, resulting into a dimer. By extending the hydrogen bonds between molecules, such as N6—H6···O1, N6—H6···O3, the dimer form a one-dimensional structure. And by forming a hydrogen bond with ClO[−], such as the one-dimensional structure expands into a three-dimensional structure.

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