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Crystal structure of 4-dimethylamino-pyridin-1-ium uracil-1-acetate, $C_{13}H_{16}N_4O_4$

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Abstract

$C_{13}H_{16}N_4O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 4.7965(12)$ Å, $b = 10.053(2)$ Å, $c = 27.570(6)$ Å, $\beta = 90.292(8)^\circ$, $V = 1329.3(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0426$, $wR_{\text{ref}}(F^2) = 0.1115$, $T = 292(3)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Uracil-1-acetic acid was obtained by the literature method [4]. Uracil-1-acetic acid (0.054 g, 0.3 mmol) and AgBF_4 (0.058 g, 0.3 mmol) were dissolved in DMF (4 mL) under ultrasonication. After adding 0.042 mL Et_3N , N,N -dimethylamino pyridine (0.037 g, 0.3 mmol) was added to the mixture under ultrasonication. The mixture was sealed and heated to 70 °C for 20 h. The solution was cooled to room temperature and filtered; slow evaporation afforded colorless block crystals with moderate yield in one week.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.18 \times 0.15 \times 0.12$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-III CMOS, φ and ω
θ_{max} , completeness:	27.5° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13851, 3027, 0.056
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2528
$N(\text{param})_{\text{refined}}$:	192
Programs:	SHELX [1], Diamond [2], Bruker [3]

Experimental details

All hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms.

Comment

Cocrystal compounds have been widely used in pharmaceutical [5], materials [6, 7] and many other respects [8, 9]. The nucleobases [10] have distinctly unique advantages to form cocrystals because they are outstanding H donors and acceptors. In order to combine some functional groups like nucleobases with coinage metal complexes [11–14], we reacted uracil-1-acetic acid with AgBF_4 under the assistance of N,N -dimethylamino pyridine. The title salt compound (systematic name: 4-(dimethylamino)pyridin-1-ium 2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)acetate) was obtained as an unexpected product. As we know, uracil (U) is one of the four nucleobases in the nucleic acid of RNA and forming base-pairs with adenine during DNA transcription [15].

The title compound has four cations and four anions in the unit cell. The dimeric structure (two cations and two anions) is shown in the figure. The bond lengths and angles are in the normal ranges (Uracil (U) [4] or 4-dimethylamino-pyridin-1-ium groups [16]). Similar to bis(4-dimethylamino-pyridin-1-ium) tetrafluorosuccinate [16], the uracil-1-acetate and 4-dimethylamino-pyridin-1-ium form $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds (cyan colored dashed lines in the figure). The Uracil ring is perpendicular to the pyridine ring with the dihedral angle between each other being 89.7° . Interesting observation is that the adjacent uracil groups form mismatched basepairs via two $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds (green colored dashed

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}
N1	−0.0278(3)	0.98003(13)	0.23986(4)	0.0315(3)
H1N	−0.1806	0.9922	0.2634	0.047 [*]
C1	0.0568(3)	0.85556(16)	0.23020(6)	0.0344(3)
H1	−0.0238	0.7839	0.2477	0.041 [*]
C2	0.2540(3)	0.82840(14)	0.19623(5)	0.0313(3)
H2	0.3047	0.7388	0.1898	0.038 [*]
C3	0.3846(3)	0.93367(13)	0.17037(5)	0.0246(3)
C4	0.2928(3)	1.06402(14)	0.18215(5)	0.0282(3)
H4	0.3738	1.1389	0.1666	0.034 [*]
C5	0.0887(3)	1.08183(15)	0.21578(5)	0.0309(3)
H5	0.0265	1.1696	0.2224	0.037 [*]
N2	0.5799(3)	0.91006(11)	0.13705(4)	0.0274(3)
C6	0.6488(4)	0.77353(15)	0.12318(6)	0.0357(4)
H6A	0.7046	0.7232	0.1521	0.054 [*]
H6B	0.8027	0.7745	0.0999	0.054 [*]
H6C	0.4853	0.7315	0.1082	0.054 [*]
C7	0.6932(3)	1.01437(15)	0.10629(5)	0.0310(3)
H7A	0.5650	1.0311	0.0792	0.047 [*]
H7B	0.8745	0.9862	0.0937	0.047 [*]
H7C	0.7165	1.0960	0.1253	0.047 [*]
N11	1.0336(2)	0.39145(11)	0.07326(4)	0.0225(2)
C12	0.8145(3)	0.43027(13)	0.04428(5)	0.0219(3)
N13	0.7531(2)	0.34779(11)	0.00602(4)	0.0241(3)
H13	0.6049	0.3708	−0.0122	0.036 [*]
C14	0.8807(3)	0.22661(14)	−0.00454(5)	0.0277(3)
C15	1.1060(3)	0.19284(14)	0.02775(5)	0.0296(3)
H15	1.2082	0.1130	0.0230	0.035 [*]
C16	1.1717(3)	0.27427(14)	0.06468(5)	0.0269(3)
H16	1.3203	0.2497	0.0858	0.032 [*]
C17	1.0908(3)	0.46853(14)	0.11725(5)	0.0246(3)
H17A	1.0780	0.5645	0.1095	0.030 [*]
H17B	1.2839	0.4501	0.1282	0.030 [*]
C18	0.8894(3)	0.43705(13)	0.15891(5)	0.0248(3)
O1	0.9125(2)	0.51628(11)	0.19437(4)	0.0353(3)
O2	0.7286(2)	0.34086(10)	0.15531(4)	0.0349(3)
O3	0.6828(2)	0.53293(9)	0.05194(4)	0.0298(2)
O4	0.7967(3)	0.15952(11)	−0.03887(4)	0.0416(3)

lines in the figure) in the crystal packing. Symmetry Code: #1 1 − *x*, 1 − *y*, −*z*; #2 0.5 + *x*, 1.5 − *y*, −0.5 + *z*; #3 0.5 − *x*, −0.5 + *y*, 0.5 − *z*.

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