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Crystal structure of dimethylammonium 5-fluorouracil-1-acetate, $C_8H_{12}N_3O_4F$

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Table 1: Data collection and handling.

Crystal:	Colorless needle
Size:	0.17 × 0.15 × 0.11 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	1.11 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-III, ϕ and ω
$\theta_{\max}$, completeness:	73.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3999, 2076, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1637
$N(\text{param})_{\text{refined}}$:	147
Programs:	SHELX [1], Diamond [2], Bruker [3]

Abstract

$C_8H_{12}N_3O_4F$, monoclinic, $P2_1/c$ (no. 14), $a = 4.8090(3)$ Å, $b = 9.9340(6)$ Å, $c = 22.2437(13)$ Å, $\beta = 90.138(8)^\circ$, $V = 1062.64(11)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0452$, $wR_{\text{ref}}(F^2) = 0.1227$, $T = 293(2)$ 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

5-Fluorouracil-1-acetic acid was obtained by the literature method [4]. 5-Fluorouracil-1-acetic acid (0.054 g, 0.3 mmol) and $AgBF_4$ (0.058 g, 0.3 mmol) were dissolved in CH_3OH (4 mL) under ultrasonication. After adding 0.3 mmol phenanthroline, DMF (2 mL) was added to the mixture under ultrasonication. The mixture was sealed and heated to 70 °C for 20 h. Then the solution was cooled to room temperature and

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
F13	0.7841(3)	0.09962(12)	0.49448(5)	0.0267(3)
O1	0.3712(3)	0.45061(14)	0.25356(6)	0.0203(3)
O2	0.1524(3)	0.31808(13)	0.32033(6)	0.0194(3)
O11	0.1585(3)	0.52371(13)	0.43199(6)	0.0183(3)
O12	0.3714(3)	0.20050(14)	0.57022(6)	0.0223(3)
N5	0.5141(3)	0.38024(16)	0.41055(7)	0.0160(4)
N7	0.2662(3)	0.35912(16)	0.49992(6)	0.0157(3)
H7	0.1139	0.3837	0.5241	0.019*
C3	0.3380(4)	0.39767(19)	0.30545(8)	0.0162(4)
C4	0.5545(4)	0.44330(19)	0.35168(8)	0.0168(4)
H4A	0.7387	0.4212	0.3370	0.020*
H4B	0.5442	0.5403	0.3561	0.020*
C6	0.3035(4)	0.42648(18)	0.44629(8)	0.0150(4)
C8	0.4171(4)	0.24957(19)	0.52092(8)	0.0170(4)
C9	0.6262(4)	0.20551(19)	0.47827(8)	0.0181(4)
C10	0.6699(4)	0.26921(19)	0.42643(8)	0.0177(4)
H10	0.8076	0.2383	0.4006	0.021*
N21	0.1248(3)	0.90736(15)	0.30591(7)	0.0178(4)
H21A	−0.0297	0.9247	0.2853	0.021*
H21B	0.2690	0.9292	0.2828	0.021*
C22	0.1366(5)	0.7613(2)	0.31927(9)	0.0270(5)
H22A	0.3031	0.7417	0.3415	0.041*
H22B	0.1371	0.7114	0.2823	0.041*
H22C	−0.0226	0.7361	0.3427	0.041*
C23	0.1292(5)	0.9925(2)	0.36074(9)	0.0223(4)
H23A	−0.0321	0.9735	0.3846	0.033*
H23B	0.1285	1.0857	0.3494	0.033*
H23C	0.2938	0.9733	0.3837	0.033*

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filtered, slow evaporation afforded colorless needle crystals of the title compound as an unexpected result.

Experimental details

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

Comment

Pharmaceutical cocrystal compounds [5] have been extensively studied in their structures and pharmacological activity. The stability of some drug molecules will be improved after forming cocrystals with other biocompatible molecules. That's very important for drug industry. 5-Fluorouracil (5-FU) and its derivatives as the first and the most important organic anticancer drugs have been widely investigated over sixty years [6]. Although our original intention is to combine some bioactive groups like 5-FU with coinage metal complexes [7–10], we fail to get the target silver complex. Only salt title compound has been obtained and is reported here. The dimethylammonium part of the title compound deduces from the decomposition of DMF [11].

The title compound has four anions and four cations in the unit cell. The bond lengths and angles are in the normal ranges [4]. Similar to bis(4-dimethylamino-pyridin-1-ium) tetrafluorosuccinate [12], the 5-fluorouracil-1-acetate and dimethylammonium form strong N–H···O hydrogen bonds. The adjacent 5-FU groups are forming 5-FU and 5-FU mismatch basepairs *via* N–H···O duplicate hydrogen bonds. This observation is rarely reported in the crystal structures of 5-FU derivatives.

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