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Crystal structure of 2-(2-(2-amino-6-chloro-9H-purin-9-yl)ethyl)propane-1,3-diyl diacetate, $C_{14}H_{18}ClN_5O_4$

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	$0.20 \times 0.14 \times 0.14$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.27 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9428, 3019, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2613
$N(\text{param})_{\text{refined}}$:	219
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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Abstract

$C_{14}H_{18}ClN_5O_4$, triclinic, $P\bar{1}$, $a = 7.7966(3)$ Å, $b = 8.2167(4)$ Å, $c = 12.9574(7)$ Å, $\alpha = 82.444(4)^\circ$, $\beta = 81.604(4)^\circ$, $\gamma = 86.751(4)^\circ$, $V = 813.46(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0454$, $wR_{\text{ref}}(F^2) = 0.1131$, $T = 293(2)$ K.

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The 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 material

The title compound was synthesized with a total yield of 56% following the literature method with small modification [5]. Colorless crystals suitable for X-ray diffraction study were obtained from an acetone solution.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.09566(7)	0.54285(7)	0.28209(4)	0.03793(19)
O3	0.2554(4)	0.3960(3)	1.03291(17)	0.0931(9)
O4	0.7949(2)	1.0034(2)	0.76552(17)	0.0606(6)
N1	−0.0358(2)	0.7369(2)	0.48631(14)	0.0307(4)
N2	0.3871(2)	0.6096(2)	0.34179(14)	0.0310(4)
N3	0.4223(2)	0.7568(2)	0.48694(14)	0.0303(4)
N4	0.1444(2)	0.8398(2)	0.58018(14)	0.0285(4)
N5	0.6564(2)	0.6596(3)	0.37882(17)	0.0428(5)
H5A	0.7244	0.7002	0.4146	0.051*
H5B	0.6991	0.6085	0.3265	0.051*
O1	0.2491(2)	0.4748(2)	0.86300(13)	0.0420(4)
O2	0.52253(19)	0.93232(19)	0.82111(13)	0.0368(4)
C1	−0.0225(3)	0.8187(3)	0.56426(17)	0.0315(5)
H1	−0.1186	0.8596	0.6058	0.038*
C2	0.1362(3)	0.7004(3)	0.44724(16)	0.0266(5)
C3	0.2173(3)	0.6232(3)	0.36397(16)	0.0276(5)
C4	0.4833(3)	0.6760(3)	0.40464(18)	0.0303(5)
C5	0.2493(3)	0.7643(2)	0.50420(16)	0.0257(5)
C6	0.1950(3)	0.9318(3)	0.65951(17)	0.0315(5)
H6A	0.1091	1.0196	0.6723	0.038*
H6B	0.3051	0.9815	0.6325	0.038*
C7	0.2119(3)	0.8251(3)	0.76290(17)	0.0312(5)
H7A	0.1130	0.7553	0.7805	0.037*
H7B	0.2066	0.8960	0.8176	0.037*
C8	0.3775(3)	0.7163(3)	0.76392(17)	0.0311(5)
H8	0.3837	0.6498	0.7059	0.037*
C9	0.3736(3)	0.5991(3)	0.86436(19)	0.0387(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H9A	0.4878	0.5478	0.8693	0.046*
H9B	0.3400	0.6581	0.9247	0.046*
C10	0.5407(3)	0.8140(3)	0.74574(18)	0.0349(5)
H10A	0.6410	0.7410	0.7549	0.042*
H10B	0.5570	0.8707	0.6747	0.042*
C11	0.2050(3)	0.3780(3)	0.9532(2)	0.0446(6)
C12	0.0909(4)	0.2451(4)	0.9415(3)	0.0684(9)
H12A	0.0694	0.1747	1.0069	0.103*
H12B	0.1466	0.1822	0.8877	0.103*
H12C	−0.0171	0.2924	0.9222	0.103*
C13	0.6610(3)	1.0206(3)	0.82118(19)	0.0357(5)
C14	0.6269(4)	1.1438(3)	0.8975(2)	0.0515(7)
H14A	0.6762	1.1032	0.9601	0.077*
H14B	0.5040	1.1622	0.9150	0.077*
H14C	0.6787	1.2452	0.8664	0.077*

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C, aromatic ring, and methylene})$, $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{C, methyl})$, and $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{N, amino})$. The final refinement by using geometrical restraints, with C—H = 0.93 Å (aromatic ring), and C—H = 0.96 Å (methyl), C—H = 0.97 Å (methylene), N—H = 0.86 Å (amino).

Comment

Penciclovir and its oral form famciclovir are both potent and selective anti-herpesvirus agents [5]. The purity and the crystal type of the medicine is very important for pharmaceutical industrial synthesis. The title compound is a

key intermediate for the syntheses of penciclovir and famciclovir. Before the syntheses of related pharmaceuticals, the purification of the raw materials is to guarantee the high purity of the products. However, we did not find the crystal profile of the title compound in literatures up to now.

As shown in the figure the molecular structure of the title compound is composed of two acetoxy groups, isopentane, and the purine fragment. All the bond lengths are in the normal ranges [6]. There are intermolecular N—H...N hydrogen bonds, by which adjacent molecules are connected into one-dimensional chain along [100].

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