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The crystal structure of 5-chloro-4,6-dimethoxypyrimidin-2-amine, $C_6H_8ClN_3O_2$

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Abstract

$C_6H_8ClN_3O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 5.066(3)$ Å, $b = 12.775(8)$ Å, $c = 12.587(8)$ Å, $\beta = 94.060(8)^\circ$, $V = 812.5(9)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0597$, $wR_{ref}(F^2) = 0.1990$, $T = 296(2)$ 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

Sodium dichloroisocyanurate (SDIC, 5.30 g) was added to deionized water (40.00 mL). The solution was stirred at 288 K

Table 1: Data collection and handling.

Crystal:	White rod
Size:	$0.12 \times 0.10 \times 0.08$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.43 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4854, 1823, 0.057
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 965
$N(\text{param})_{\text{refined}}$:	111
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.1897(2)	0.34398(8)	0.79001(9)	0.0537(4)
O1	0.2141(5)	0.2033(2)	0.6081(2)	0.0493(8)
O2	0.5749(5)	0.5083(2)	0.7582(2)	0.0458(7)
N2	0.5312(6)	0.2924(2)	0.5216(2)	0.0371(8)
N3	0.7199(5)	0.4496(2)	0.5985(2)	0.0356(7)
N6	0.8465(7)	0.3827(3)	0.4402(3)	0.0478(9)
H6A	0.835292	0.336295	0.390549	0.057*
H6B	0.955266	0.434073	0.436820	0.057*
C1	0.3819(7)	0.2849(3)	0.6027(3)	0.0376(9)
C2	0.3869(7)	0.3561(3)	0.6852(3)	0.0357(9)
C3	0.5640(7)	0.4385(3)	0.6785(3)	0.0367(9)
C4	0.7498(8)	0.5960(3)	0.7512(3)	0.0529(12)
H4A	0.928861	0.571440	0.750713	0.079*
H4B	0.733851	0.641145	0.811325	0.079*
H4C	0.703822	0.634026	0.686718	0.079*
C5	0.2104(10)	0.1284(4)	0.5248(4)	0.0702(15)
H5A	0.173189	0.162797	0.457624	0.105*
H5B	0.075883	0.077124	0.535007	0.105*
H5C	0.379641	0.094449	0.525401	0.105*
C6	0.6941(7)	0.3748(3)	0.5231(3)	0.0362(9)

to get a clear solution, after which 5.5 mL of glacial acetic acid and 2-amino-4,6-dimethoxypyrimidine aqueous solution (including 3.10 g of pyrimidine and 10 mL of deionized water) were sequentially added to the mixture. After reacting at room temperature for 4 h, the filtrate from filtering was put in the refrigerator overnight to be cooled. Colorless transparent crystals were obtained.

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Experimental details

H atoms were positioned geometrically (N–H = 0.86 Å, C–H = 0.96 Å) and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Comment

Aminopyrimidine derivatives have attracted extensive attention in molecular biology, as versatile intermediates in organic synthesis and because of their bactericidal action [4–13]. The crystal structures of some 2-amino-substituted pyrimidine compounds, such as 2-amino-4,6-dimethyl pyrimidiniumbromide [8] and 5-chloro-pyrimidin-2-amine [13] have previously been elucidated.

The title compound pyrimidine molecule (*cf.* the figure) has a symmetrical structure, which crystallises in space group $P2_1/n$. All the bond lengths and angles are within proper ranges and in great accord with that found for related structures [8, 14]. The two carbon atoms (C4, C5) of the methoxy substituent in the ring are 7.083 Å apart, while the chlorine atom (Cl1) and the nitrogen atom (N6) from amino are only 5.725 Å apart from each other. At the same time, all the atoms in the molecule (except the hydrogen atom) are basically in the same plane, where the dihedral angle between the planes defined by one methoxy group (C4, O2, C3, N3) and by the central ring is the largest, that is 2.6(5)°. Therefore, the title compound has an unexceptionable stacking property, which makes contributions to the packing scheme resulting in a high density and stability.

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