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2-Amino-4,6-dimethoxypyrimidin-1-ium 2,2-dichloroacetate

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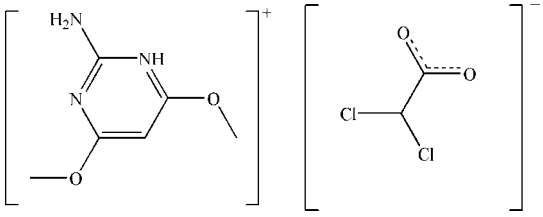
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.200; data-to-parameter ratio = 13.9.

In the title salt, $C_6H_{10}N_3O_2^+\cdot C_2HCl_2O_2^-$, two cations and two anions are linked by N—H···O hydrogen bonds, forming chains along the c axis.

Related literature

For the biological activity of heterocyclic compounds, see: Gilchrist (1998). For the bioactivity of pyrimidine derivatives, see: Xue *et al.* (1993). For a related structure, see: Hemamalini *et al.* (2005). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_6H_{10}N_3O_2^+\cdot C_2HCl_2O_2^-$
M_r = 284.10
Triclinic, $P\overline{1}$
a = 6.8502 (14) Å
b = 8.6667 (17) Å
c = 11.255 (2) Å
α = 67.480 (1)°
β = 87.320 (2)°
γ = 85.970 (2)°
V = 615.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.53 mm^{-1}
T = 293 K
0.45 × 0.43 × 0.35 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.795, T_{max} = 0.835
4710 measured reflections
2173 independent reflections
1806 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.200
S = 1.09
2173 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···O3^{i}	0.86	1.97	2.822 (3)	173
N3—H3A···O3^{ii}	0.86	2.07	2.848 (3)	149
N2—H2···O4^{i}	0.86	1.85	2.692 (3)	168

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, y+1, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5473).

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supporting information

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2-Amino-4,6-dimethoxypyrimidin-1-ium 2,2-dichloroacetate

Cui-Hua Lin and Nai-Sheng Liu

S1. Comment

Five and six-membered heterocyclic compounds are important constituents that often exist in biologically active natural products and synthetic compounds of medicinal interest (Gilchrist, 1998). As useful precursors to potentially bioactive pyrimidine derivatives, methylpyrimidine has attracted considerable attention for many years (Xue *et al.*, 1993). In recent years, new complexes of pyrimidine have been synthesized (Hemamalini *et al.*, 2005). Herein we report herein the crystal structure of the title compound (I).

The molecular structure of (I) is shown in Fig. 1. There is one cation and one anion in the asymmetric unit of (I). All bond lengths are within the normal ranges (Allen *et al.*, 1987). In the crystal, two cations and two anions are linked by intermolecular N—H···O hydrogen bonds to form centrosymmetric four component aggregates.

S2. Experimental

A mixture of 2-amino-4,6-dichloropyrimidine (0.1 mol) and sodium methoxide (0.1 mol) was stirred with methanol (30 ml) for 3 h to afford 2-Amino-4,6-dimethoxypyrimidine (yield 85%). The title compound was crystallized from an aqueous mixture containing 2-Amino-4,6-dimethoxypyrimidine and dichloroacetate in a 1:1 stoichiometric ratio at room temperature by the slow evaporation technique.

S3. Refinement

H atoms bonded to C atoms were fixed geometrically and and included in a riding-model approximation with C—H = 0.93–0.98 Å and N—H = 0.86Å with U_{iso}(H)=1.2–1.5U_{eq}(C).

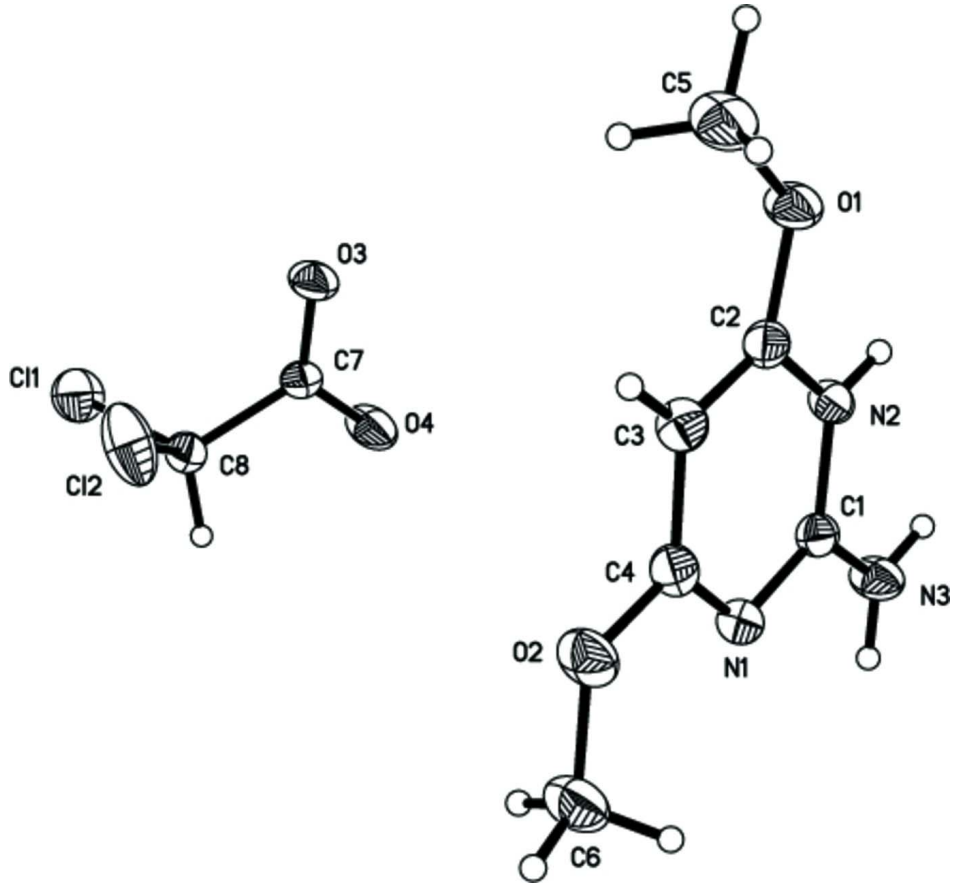

Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

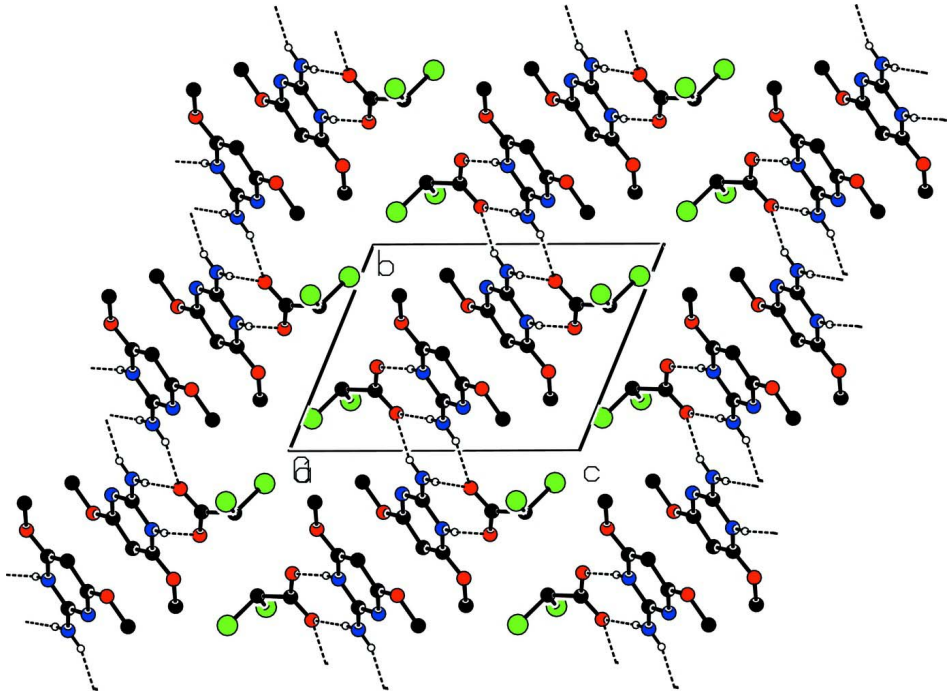

Figure 2

Part of the crystal structure with hydrogen bonds shown as dashed lines.

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Crystal data

$C_6H_{10}N_3O_2^+\cdot C_2HCl_2O_2^-$
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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.8502\ (14)$ Å
$b = 8.6667\ (17)$ Å
$c = 11.255\ (2)$ Å
$\alpha = 67.480\ (1)°$
$\beta = 87.320\ (2)°$
$\gamma = 85.970\ (2)°$
$V = 615.6\ (2)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.533$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3794 reflections
$\theta = 3.5–27.5°$
$\mu = 0.53$ mm^{-1}
$T = 293$ K
Block, colorless
0.45 × 0.43 × 0.35 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.795$, $T_{max} = 0.835$
4710 measured reflections
2173 independent reflections
1806 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.5°$
$h = -8\rightarrow7$
$k = -10\rightarrow10$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.200$
$S = 1.09$
2173 reflections
156 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1265P)^2 + 0.3628P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.11057 (16)	0.15674 (13)	0.03526 (9)	0.0641 (4)
Cl2	0.44050 (14)	0.24515 (19)	0.14101 (11)	0.0804 (5)
N1	0.5368 (4)	0.7901 (3)	0.4560 (2)	0.0365 (6)
N2	0.3706 (3)	0.6207 (3)	0.6442 (2)	0.0336 (6)
H2	0.2667	0.6043	0.6924	0.040*
N3	0.2252 (4)	0.8660 (3)	0.5053 (3)	0.0450 (7)
H3A	0.2264	0.9542	0.4356	0.054*
H3B	0.1235	0.8474	0.5555	0.054*
O1	0.4900 (3)	0.3802 (3)	0.7880 (2)	0.0487 (6)
O2	0.8446 (3)	0.7020 (3)	0.4156 (2)	0.0530 (7)
O3	0.0938 (4)	0.1810 (3)	0.3189 (2)	0.0511 (7)
O4	−0.0703 (4)	0.4064 (3)	0.1873 (2)	0.0653 (8)
C1	0.3790 (4)	0.7598 (3)	0.5350 (3)	0.0332 (6)
C2	0.5248 (4)	0.5073 (4)	0.6779 (3)	0.0367 (7)
C3	0.6887 (4)	0.5305 (4)	0.6026 (3)	0.0389 (7)
H3	0.7962	0.4540	0.6232	0.047*
C4	0.6855 (4)	0.6780 (4)	0.4910 (3)	0.0384 (7)
C5	0.6408 (6)	0.2493 (5)	0.8325 (4)	0.0651 (11)
H5A	0.6728	0.2056	0.7670	0.098*
H5B	0.5956	0.1614	0.9089	0.098*
H5C	0.7551	0.2931	0.8514	0.098*
C6	0.8456 (6)	0.8491 (5)	0.3002 (4)	0.0614 (10)
H6A	0.7688	0.8339	0.2367	0.092*
H6B	0.9777	0.8690	0.2682	0.092*
H6C	0.7910	0.9432	0.3181	0.092*

C7	0.0616 (4)	0.2935 (4)	0.2130 (3)	0.0378 (7)
C8	0.1932 (4)	0.2995 (4)	0.0968 (3)	0.0394 (7)
H8	0.1832	0.4127	0.0302	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0840 (8)	0.0668 (7)	0.0495 (6)	−0.0127 (5)	−0.0068 (5)	−0.0292 (5)
Cl2	0.0425 (6)	0.1419 (12)	0.0752 (7)	0.0083 (6)	−0.0034 (5)	−0.0639 (8)
N1	0.0382 (13)	0.0356 (13)	0.0345 (13)	−0.0043 (10)	0.0020 (10)	−0.0121 (10)
N2	0.0329 (12)	0.0350 (13)	0.0292 (12)	0.0017 (10)	0.0014 (9)	−0.0089 (10)
N3	0.0435 (14)	0.0386 (14)	0.0392 (14)	0.0085 (11)	0.0022 (11)	−0.0016 (11)
O1	0.0530 (13)	0.0411 (12)	0.0371 (12)	0.0138 (10)	0.0023 (10)	−0.0015 (9)
O2	0.0392 (12)	0.0607 (16)	0.0543 (14)	−0.0042 (11)	0.0145 (10)	−0.0181 (12)
O3	0.0588 (15)	0.0438 (13)	0.0337 (12)	0.0107 (10)	0.0032 (10)	0.0012 (10)
O4	0.0660 (16)	0.0588 (16)	0.0430 (13)	0.0296 (13)	0.0131 (12)	0.0046 (11)
C1	0.0372 (15)	0.0319 (14)	0.0311 (14)	−0.0031 (11)	−0.0025 (11)	−0.0124 (11)
C2	0.0424 (16)	0.0340 (15)	0.0334 (14)	0.0028 (12)	−0.0045 (12)	−0.0128 (12)
C3	0.0330 (15)	0.0410 (16)	0.0416 (16)	0.0042 (12)	−0.0039 (12)	−0.0152 (13)
C4	0.0348 (15)	0.0445 (17)	0.0396 (16)	−0.0071 (13)	0.0019 (12)	−0.0197 (13)
C5	0.070 (2)	0.059 (2)	0.0451 (19)	0.0295 (19)	−0.0043 (18)	−0.0025 (17)
C6	0.057 (2)	0.068 (3)	0.052 (2)	−0.0141 (18)	0.0182 (17)	−0.0157 (18)
C7	0.0431 (16)	0.0315 (15)	0.0340 (15)	0.0008 (12)	0.0002 (12)	−0.0076 (12)
C8	0.0445 (17)	0.0375 (16)	0.0339 (15)	0.0009 (12)	0.0003 (12)	−0.0116 (12)

Geometric parameters (Å, º)

Cl1—C8	1.767 (3)	O3—C7	1.234 (4)
Cl2—C8	1.768 (3)	O4—C7	1.241 (4)
N1—C4	1.320 (4)	C2—C3	1.353 (4)
N1—C1	1.342 (4)	C3—C4	1.408 (4)
N2—C2	1.354 (4)	C3—H3	0.9300
N2—C1	1.355 (4)	C5—H5A	0.9600
N2—H2	0.8600	C5—H5B	0.9600
N3—C1	1.315 (4)	C5—H5C	0.9600
N3—H3A	0.8600	C6—H6A	0.9600
N3—H3B	0.8600	C6—H6B	0.9600
O1—C2	1.330 (4)	C6—H6C	0.9600
O1—C5	1.433 (4)	C7—C8	1.539 (4)
O2—C4	1.327 (4)	C8—H8	0.9800
O2—C6	1.430 (5)		
C4—N1—C1	116.5 (2)	O1—C5—H5A	109.5
C2—N2—C1	120.4 (2)	O1—C5—H5B	109.5
C2—N2—H2	119.8	H5A—C5—H5B	109.5
C1—N2—H2	119.8	O1—C5—H5C	109.5
C1—N3—H3A	120.0	H5A—C5—H5C	109.5
C1—N3—H3B	120.0	H5B—C5—H5C	109.5

H3A—N3—H3B	120.0	O2—C6—H6A	109.5
C2—O1—C5	117.1 (3)	O2—C6—H6B	109.5
C4—O2—C6	117.9 (3)	H6A—C6—H6B	109.5
N3—C1—N1	119.5 (3)	O2—C6—H6C	109.5
N3—C1—N2	118.4 (3)	H6A—C6—H6C	109.5
N1—C1—N2	122.1 (3)	H6B—C6—H6C	109.5
O1—C2—C3	127.8 (3)	O3—C7—O4	126.9 (3)
O1—C2—N2	111.7 (3)	O3—C7—C8	119.0 (3)
C3—C2—N2	120.5 (3)	O4—C7—C8	114.1 (3)
C2—C3—C4	115.5 (3)	C7—C8—Cl1	108.5 (2)
C2—C3—H3	122.2	C7—C8—Cl2	111.4 (2)
C4—C3—H3	122.2	Cl1—C8—Cl2	109.21 (17)
N1—C4—O2	118.7 (3)	C7—C8—H8	109.3
N1—C4—C3	125.0 (3)	Cl1—C8—H8	109.3
O2—C4—C3	116.3 (3)	Cl2—C8—H8	109.3
C4—N1—C1—N3	−179.5 (3)	C1—N1—C4—O2	179.6 (3)
C4—N1—C1—N2	−0.8 (4)	C1—N1—C4—C3	0.9 (4)
C2—N2—C1—N3	179.4 (3)	C6—O2—C4—N1	0.1 (4)
C2—N2—C1—N1	0.7 (4)	C6—O2—C4—C3	179.0 (3)
C5—O1—C2—C3	−1.4 (5)	C2—C3—C4—N1	−0.7 (5)
C5—O1—C2—N2	178.5 (3)	C2—C3—C4—O2	−179.5 (3)
C1—N2—C2—O1	179.6 (3)	O3—C7—C8—Cl1	81.6 (3)
C1—N2—C2—C3	−0.5 (4)	O4—C7—C8—Cl1	−97.9 (3)
O1—C2—C3—C4	−179.6 (3)	O3—C7—C8—Cl2	−38.6 (4)
N2—C2—C3—C4	0.5 (4)	O4—C7—C8—Cl2	141.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*B*···O3^{i}	0.86	1.97	2.822 (3)	173
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