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2-Anilino-4,6-dimethylpyrimidinium chloroacetate

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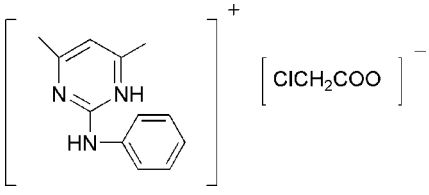
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.141; data-to-parameter ratio = 13.5.

In the crystal structure of the title compound, $C_{12}H_{14}N_3^+\cdot C_2H_2ClO_2^-$, the chloroacetate anion is linked to the *N*-(4,6-dimethylpyrimidin-2-yl)aniline cation by N—H···O hydrogen bonding. Within the cation, the pyrimidine ring is twisted with respect to the phenyl ring by a dihedral angle of 7.59 (4)°.

Related literature

For general background, see: Xue *et al.* (2000); Li *et al.* (1996); Stock *et al.* (1997).



Experimental

Crystal data

$C_{12}H_{14}N_3^+\cdot C_2H_2ClO_2^-$
M_r = 293.75
Tetragonal, $P4_2/n$
a = 19.604 (4) Å
c = 7.542 (3) Å
V = 2898.6 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 293 (2) K
0.68 × 0.35 × 0.33 mm

Data collection

Bruker APEX area-dectector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.839, T_{max} = 0.917
9030 measured reflections
2541 independent reflections
1991 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.141
S = 1.03
2541 reflections
188 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	1.98	2.833 (3)	173
N2—H2A···O2^{i}	0.98 (4)	1.61 (4)	2.572 (2)	166 (4)

Symmetry code: (i) $x, y, z+1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2368).

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  doi:10.1107/S1600536807064422

supplementary materials

Acta Cryst. (2008). E**64**, o318 [**doi:10.1107/S1600536807064422**]

2-Anilino-4,6-dimethylpyrimidinium chloroacetate

J.-C. Li, X.-Q. Qiu, Y.-H. Feng and Q. Lin

Comment

The 2-anilino-4,6-dimethylpyrimidine has a good and wide fungicidal activity (Xue *et al.*, 2000; Li *et al.*, 1996). The pyriminethanil could be combined with certain acids to form pyrimethanil salts that have a reduced vapor pressure that increased the persistence of the compounds on the crop to be protected from fungal attack, and increased activity (Stock *et al.*, 1997).

The crystal of the title compound consists of 2-phenylamino-4,6-dimethylpyrimidinium cations and chloroacetate anions (Fig. 1). All bond lengths and angles are normal. The atoms of the pyrimidine ring are coplanar, the largest deviation from the mean plane being 0.005 (2)Å (N3). The dihedral angle between the pyrimidine and phenyl rings is 7.59 (4)°. The cation links with the anion *via* N—H···O hydrogen bonding (Table 1, Fig. 2).

Experimental

The title compound was prepared by the reaction of *N*-(4,6-dimethylpyrimidin-2-yl)aniline (0.01 mol) and chloroacetic acid (0.01 mol) in anhydrous alcohol at room temperature for 1 h. Single crystals of suitable for X-ray measurements were obtained by by slow evaporation of anhydrous alcohol at room temperature.

Refinement

The H atoms attached to N2 was located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, N—H = 0.86 Å, and included in final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C) for methyl H atoms.

Figures

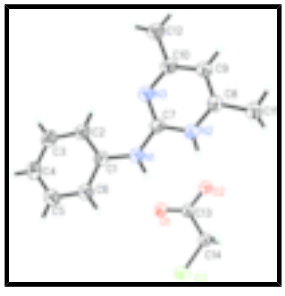

Fig. 1. The molecular structure of the title compound with 35% probability ellipsoid.

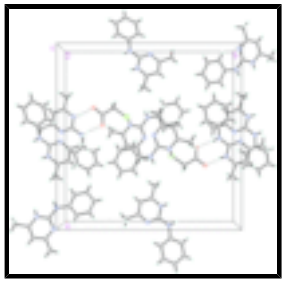

Fig. 2. The molecular packing of the title compound viewed along the *c* axis with 35% probability ellipsoid. Hydrogen bonds are shown as dashed lines.

2-Anilino-4,6-dimethylpyrimidinium chloroacetate

Crystal data

$C_{12}H_{14}N_3^+ \cdot C_2H_2ClO_2^-$	$Z = 8$
$M_r = 293.75$	$F_{000} = 1232$
Tetragonal, $P4_2/n$	$D_x = 1.346$ Mg m^{-3}
Hall symbol: -P 4bc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 19.604$ (4) Å	Cell parameters from 2794 reflections
$b = 19.604$ (4) Å	$\theta = 2.6$–24.3º
$c = 7.542$ (3) Å	$\mu = 0.27$ mm^{-1}
$\alpha = 90$º	$T = 293$ (2) K
$\beta = 90$º	Block, colorless
$\gamma = 90$º	0.68 × 0.35 × 0.33 mm
$V = 2898.6$ (13) Å^3	

Data collection

Bruker APEX area-dectector diffractometer	2541 independent reflections
Radiation source: fine-focus sealed tube	1991 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 293$(2) K	$\theta_{max} = 25.0$º
φ and ω–scan	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$h = -23 \rightarrow 7$
$T_{min} = 0.839$, $T_{max} = 0.917$	$k = -21 \rightarrow 21$
9030 measured reflections	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 1.1159P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$(\Delta/\sigma)_{max} = 0.003$
$wR(F^2) = 0.141$	$\Delta\rho_{max} = 0.25$ e Å^{-3}

S = 1.03	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
2541 reflections	Extinction correction: SHELXL
188 parameters	Extinction coefficient: 0.025 (2)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.87158 (3)	0.61011 (4)	0.05379 (12)	0.0780 (3)
N2	0.56643 (10)	0.61105 (9)	0.9030 (2)	0.0464 (5)
N3	0.49318 (9)	0.52683 (9)	0.7827 (2)	0.0478 (5)
O1	0.73448 (9)	0.56305 (9)	−0.0534 (3)	0.0768 (6)
C7	0.55484 (10)	0.54846 (11)	0.8321 (3)	0.0426 (5)
C10	0.44090 (11)	0.57048 (12)	0.8040 (3)	0.0483 (6)
O2	0.68383 (9)	0.66274 (9)	−0.0258 (3)	0.0831 (7)
C1	0.61714 (11)	0.44082 (11)	0.7549 (3)	0.0442 (5)
N1	0.61051 (9)	0.50841 (9)	0.8161 (3)	0.0490 (5)
H1A	0.6481	0.5272	0.8486	0.059*
C13	0.73486 (12)	0.62373 (12)	−0.0210 (3)	0.0541 (6)
C8	0.51423 (12)	0.65435 (11)	0.9262 (3)	0.0484 (6)
C6	0.68113 (12)	0.41108 (12)	0.7776 (3)	0.0543 (6)
H6	0.7162	0.4360	0.8296	0.065*
C2	0.56552 (12)	0.40296 (12)	0.6751 (3)	0.0528 (6)
H2	0.5227	0.4222	0.6575	0.063*
C3	0.57816 (14)	0.33658 (12)	0.6220 (3)	0.0607 (7)
H3	0.5434	0.3113	0.5698	0.073*
C9	0.44996 (11)	0.63484 (12)	0.8768 (3)	0.0525 (6)
H9	0.4131	0.6641	0.8918	0.063*
C14	0.79893 (13)	0.66082 (13)	0.0334 (5)	0.0778 (9)
H14A	0.7906	0.6831	0.1462	0.093*
H14B	0.8081	0.6962	−0.0532	0.093*
C5	0.69275 (14)	0.34523 (13)	0.7238 (4)	0.0681 (8)
H5	0.7356	0.3258	0.7401	0.082*
C4	0.64120 (14)	0.30741 (13)	0.6452 (4)	0.0680 (8)
H4	0.6492	0.2628	0.6087	0.082*
C12	0.37230 (12)	0.54599 (14)	0.7465 (4)	0.0608 (7)
H12A	0.3705	0.5444	0.6194	0.091*

H12B	0.3379	0.5766	0.7896	0.091*
H12C	0.3643	0.5012	0.7936	0.091*
C11	0.53038 (14)	0.72231 (12)	1.0070 (4)	0.0633 (7)
H11A	0.5505	0.7157	1.1217	0.095*
H11B	0.4892	0.7484	1.0190	0.095*
H11C	0.5618	0.7464	0.9321	0.095*
H2A	0.614 (2)	0.6267 (18)	0.915 (5)	0.118 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0474 (4)	0.0703 (5)	0.1163 (7)	0.0027 (3)	−0.0106 (4)	0.0018 (4)
N2	0.0411 (10)	0.0406 (10)	0.0575 (12)	−0.0030 (8)	−0.0024 (8)	−0.0031 (8)
N3	0.0413 (10)	0.0492 (11)	0.0528 (11)	−0.0028 (8)	−0.0031 (8)	−0.0022 (8)
O1	0.0539 (11)	0.0468 (10)	0.1298 (18)	−0.0024 (8)	−0.0187 (10)	−0.0190 (10)
C7	0.0395 (11)	0.0417 (12)	0.0467 (12)	−0.0033 (9)	−0.0014 (9)	0.0022 (9)
C10	0.0415 (12)	0.0543 (14)	0.0490 (13)	−0.0009 (10)	−0.0019 (10)	0.0032 (10)
O2	0.0454 (10)	0.0489 (10)	0.155 (2)	0.0019 (8)	−0.0176 (11)	−0.0166 (11)
C1	0.0457 (12)	0.0406 (12)	0.0463 (12)	−0.0048 (9)	0.0017 (9)	0.0004 (9)
N1	0.0375 (10)	0.0430 (10)	0.0665 (13)	−0.0031 (8)	−0.0052 (8)	−0.0055 (9)
C13	0.0460 (13)	0.0427 (14)	0.0734 (16)	−0.0030 (10)	−0.0047 (11)	−0.0029 (11)
C8	0.0491 (13)	0.0442 (12)	0.0520 (14)	−0.0003 (10)	0.0002 (10)	0.0010 (10)
C6	0.0452 (13)	0.0476 (13)	0.0701 (16)	−0.0016 (10)	−0.0045 (11)	−0.0058 (11)
C2	0.0447 (13)	0.0535 (14)	0.0603 (15)	−0.0049 (10)	−0.0022 (11)	−0.0064 (11)
C3	0.0593 (15)	0.0519 (14)	0.0707 (17)	−0.0116 (12)	−0.0018 (13)	−0.0128 (12)
C9	0.0427 (13)	0.0512 (13)	0.0635 (15)	0.0040 (10)	−0.0017 (11)	−0.0016 (11)
C14	0.0483 (15)	0.0464 (14)	0.139 (3)	−0.0015 (11)	−0.0165 (16)	−0.0052 (16)
C5	0.0552 (15)	0.0543 (15)	0.095 (2)	0.0090 (12)	−0.0048 (14)	−0.0090 (14)
C4	0.0685 (17)	0.0450 (14)	0.091 (2)	−0.0015 (12)	0.0033 (15)	−0.0144 (13)
C12	0.0430 (13)	0.0668 (16)	0.0727 (17)	−0.0024 (11)	−0.0064 (12)	−0.0068 (13)
C11	0.0610 (15)	0.0468 (14)	0.0821 (18)	0.0018 (11)	−0.0048 (14)	−0.0108 (12)

Geometric parameters (Å, °)

Cl1—C14	1.744 (3)	C6—C5	1.372 (3)
N2—C8	1.341 (3)	C6—H6	0.9300
N2—C7	1.358 (3)	C2—C3	1.384 (3)
N2—H2A	0.98 (4)	C2—H2	0.9300
N3—C7	1.334 (3)	C3—C4	1.373 (4)
N3—C10	1.345 (3)	C3—H3	0.9300
O1—C13	1.214 (3)	C9—H9	0.9300
C7—N1	1.350 (3)	C14—H14A	0.9700
C10—C9	1.387 (3)	C14—H14B	0.9700
C10—C12	1.492 (3)	C5—C4	1.386 (4)
O2—C13	1.260 (3)	C5—H5	0.9300
C1—C2	1.392 (3)	C4—H4	0.9300
C1—C6	1.394 (3)	C12—H12A	0.9600
C1—N1	1.409 (3)	C12—H12B	0.9600
N1—H1A	0.8600	C12—H12C	0.9600

C13—C14	1.508 (3)	C11—H11A	0.9600
C8—C9	1.368 (3)	C11—H11B	0.9600
C8—C11	1.499 (3)	C11—H11C	0.9600
C8—N2—C7	119.73 (19)	C4—C3—H3	119.5
C8—N2—H2A	121 (2)	C2—C3—H3	119.5
C7—N2—H2A	118 (2)	C8—C9—C10	118.7 (2)
C7—N3—C10	117.06 (19)	C8—C9—H9	120.7
N3—C7—N1	121.52 (19)	C10—C9—H9	120.7
N3—C7—N2	123.28 (19)	C13—C14—Cl1	115.43 (18)
N1—C7—N2	115.19 (18)	C13—C14—H14A	108.4
N3—C10—C9	121.9 (2)	Cl1—C14—H14A	108.4
N3—C10—C12	116.6 (2)	C13—C14—H14B	108.4
C9—C10—C12	121.5 (2)	Cl1—C14—H14B	108.4
C2—C1—C6	119.0 (2)	H14A—C14—H14B	107.5
C2—C1—N1	125.2 (2)	C6—C5—C4	120.6 (2)
C6—C1—N1	115.86 (19)	C6—C5—H5	119.7
C7—N1—C1	130.60 (18)	C4—C5—H5	119.7
C7—N1—H1A	114.7	C3—C4—C5	119.2 (2)
C1—N1—H1A	114.7	C3—C4—H4	120.4
O1—C13—O2	125.7 (2)	C5—C4—H4	120.4
O1—C13—C14	122.1 (2)	C10—C12—H12A	109.5
O2—C13—C14	112.1 (2)	C10—C12—H12B	109.5
N2—C8—C9	119.4 (2)	H12A—C12—H12B	109.5
N2—C8—C11	117.0 (2)	C10—C12—H12C	109.5
C9—C8—C11	123.6 (2)	H12A—C12—H12C	109.5
C5—C6—C1	120.4 (2)	H12B—C12—H12C	109.5
C5—C6—H6	119.8	C8—C11—H11A	109.5
C1—C6—H6	119.8	C8—C11—H11B	109.5
C3—C2—C1	119.8 (2)	H11A—C11—H11B	109.5
C3—C2—H2	120.1	C8—C11—H11C	109.5
C1—C2—H2	120.1	H11A—C11—H11C	109.5
C4—C3—C2	121.1 (2)	H11B—C11—H11C	109.5
C10—N3—C7—N1	179.9 (2)	N1—C1—C6—C5	179.5 (2)
C10—N3—C7—N2	0.7 (3)	C6—C1—C2—C3	0.8 (4)
C8—N2—C7—N3	0.1 (3)	N1—C1—C2—C3	−179.4 (2)
C8—N2—C7—N1	−179.2 (2)	C1—C2—C3—C4	−0.6 (4)
C7—N3—C10—C9	−1.1 (3)	N2—C8—C9—C10	0.1 (3)
C7—N3—C10—C12	179.4 (2)	C11—C8—C9—C10	−179.7 (2)
N3—C7—N1—C1	−1.7 (4)	N3—C10—C9—C8	0.7 (4)
N2—C7—N1—C1	177.6 (2)	C12—C10—C9—C8	−179.7 (2)
C2—C1—N1—C7	8.5 (4)	O1—C13—C14—Cl1	−2.1 (4)
C6—C1—N1—C7	−171.7 (2)	O2—C13—C14—Cl1	177.1 (2)
C7—N2—C8—C9	−0.4 (3)	C1—C6—C5—C4	0.3 (4)
C7—N2—C8—C11	179.3 (2)	C2—C3—C4—C5	0.2 (4)
C2—C1—C6—C5	−0.7 (4)	C6—C5—C4—C3	−0.1 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1A···O1^{i}	0.86	1.98	2.833 (3)	173
N2—H2A···O2^{i}	0.98 (4)	1.61 (4)	2.572 (2)	166 (4)

Symmetry codes: (i) *x*, *y*, *z*+1.

Fig. 1

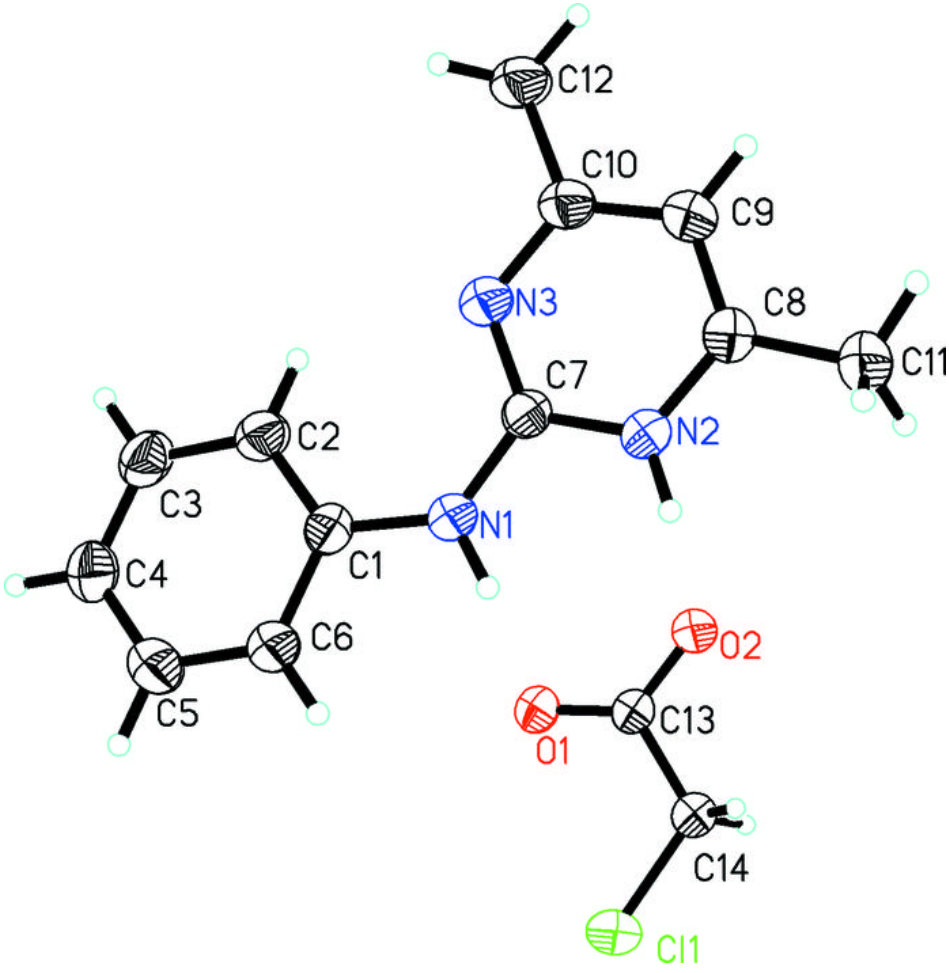

Fig. 2