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Ammonium 2-(2,4-dichlorophenoxy)-acetate hemihydrate

Hui-Lian Liu,[a] Shu-Hua Guo,[a] Yun-Ying Li[b] and Fang-Fang Jian[c]*

[a]Microscale Science Institute, Biology Department, Weifang University, Weifang 261061, People's Republic of China, [b]The 7th Middle School, Weifang 261061, People's Republic of China, and [c]Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: ffjian2008@163.com

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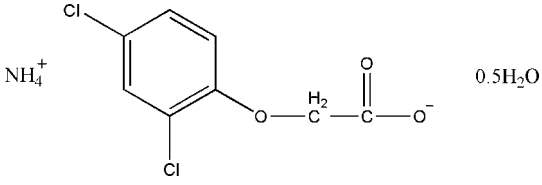
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.023; wR factor = 0.060; data-to-parameter ratio = 17.4.

The title compound, $NH_4^+\cdot C_8H_7Cl_2O_6^-\cdot 0.5H_2O$, was prepared by the reaction of 2-(2,4-dichlorophenoxy)acetic acid and ammonia in water at 367 K. The molecular structure and packing are stabilized by N—H···O and O—H···O intermolecular hydrogen-bond interactions.

Related literature

For the biological activity of 2-(2,4-dichlorophenoxy)acetic acid, see: Lv *et al.* (1998). Due to their versatile bonding modes with metal ions, they have also been used in the synthesis of mononuclear monomeric (Gao *et al.*, 2004*a*; Psomas *et al.*, 2000) and polymeric complexes (Liu *et al.*, 2004; Gao *et al.*, 2004*b*, 2005).



Experimental

Crystal data

$NH_4^+\cdot C_8H_5Cl_2O_3^-\cdot 0.5H_2O$
M_r = 247.07
Monoclinic, $C2/c$
a = 37.738 (8) Å
b = 4.3889 (9) Å
c = 12.900 (3) Å
β = 103.83 (3)°
V = 2074.7 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 293 K
0.15 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9447 measured reflections
2385 independent reflections
2216 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.060
S = 1.07
2385 reflections
137 parameters
90 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1···O2^{i}	0.85	1.97	3.2969 (15)	170
N1—H1A···O2^{i}	0.84	2.07	2.8908 (14)	168
N1—H1B···O3^{ii}	0.85	2.02	2.8578 (13)	168
N1—H1C···O3^{iii}	0.88	2.09	2.9310 (15)	161

Symmetry codes: (i) $x, -y, z+\frac{1}{2}$; (ii) $x, y+1, z$; (iii) $-x, -y, -z$.

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); 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: AT2826).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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Ammonium 2-(2,4-dichlorophenoxy)acetate hemihydrate

Hui-Lian Liu, Shu-Hua Guo, Yun-Ying Li and Fang-Fang Jian

S1. Comment

2-(2,4-Dichlorophenoxy)acetic acid is one of the important biologically active compounds that have been commonly used in herbicides and plant growth substances (Lv *et al.*, 1998). Due to their versatile bonding modes with metal ions, they have also been used in the synthesis of mononuclear monomeric (Gao *et al.*, 2004*a*; Psomas *et al.*, 2000) and polymeric complexes (Liu *et al.*, 2004; Gao *et al.*, 2004*b*, 2005). We synthesized the title compound, (I), and report here its crystal structure.

In the crystal structure of (I) (Fig. 1), all the non-H atoms of 2-(2,4-dichlorophenoxy)acetic acid are in the same plane, with the maximum deviation being 0.146Å for atom O3.

S2. Experimental

A mixture of 2-(2,4-dichlorophenoxy)acetic acid (4.42 g, 0.02 mol) and ammonia (1.0 ml, 0.02 mol) was stirred with water (50 ml) at 367 K for 2 h. Single crystals suitable for X-ray measurements were obtained by recrystallization from acetone and ethanol (1:1) at room temperature.

S3. Refinement

The H atoms of the water molecule were found from a difference Fourier map and refined freely. The remaining H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H and N—H distances of 0.93–0.96 and 0.86Å, respectively, and with U_{iso}(H)=1.2–1.5U_{eq}(C,N).

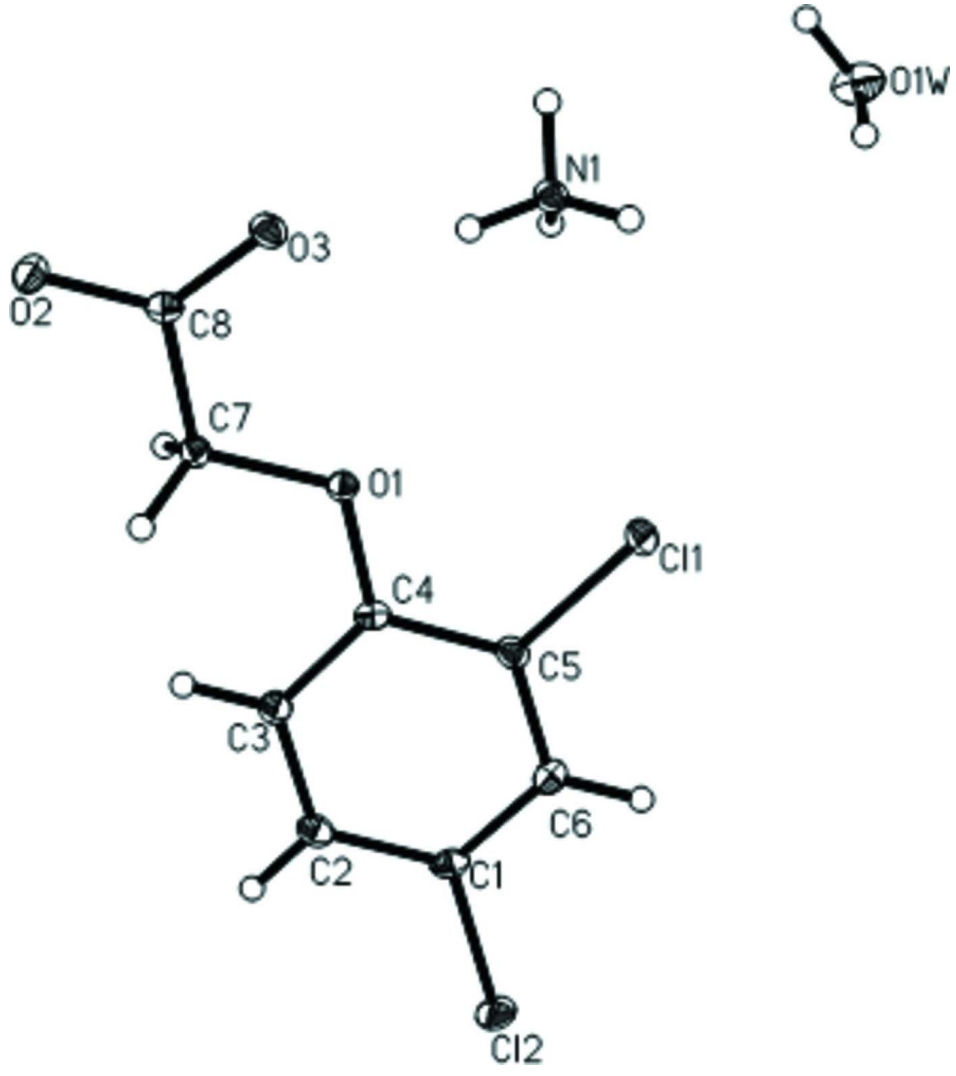

Figure 1

The molecular structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

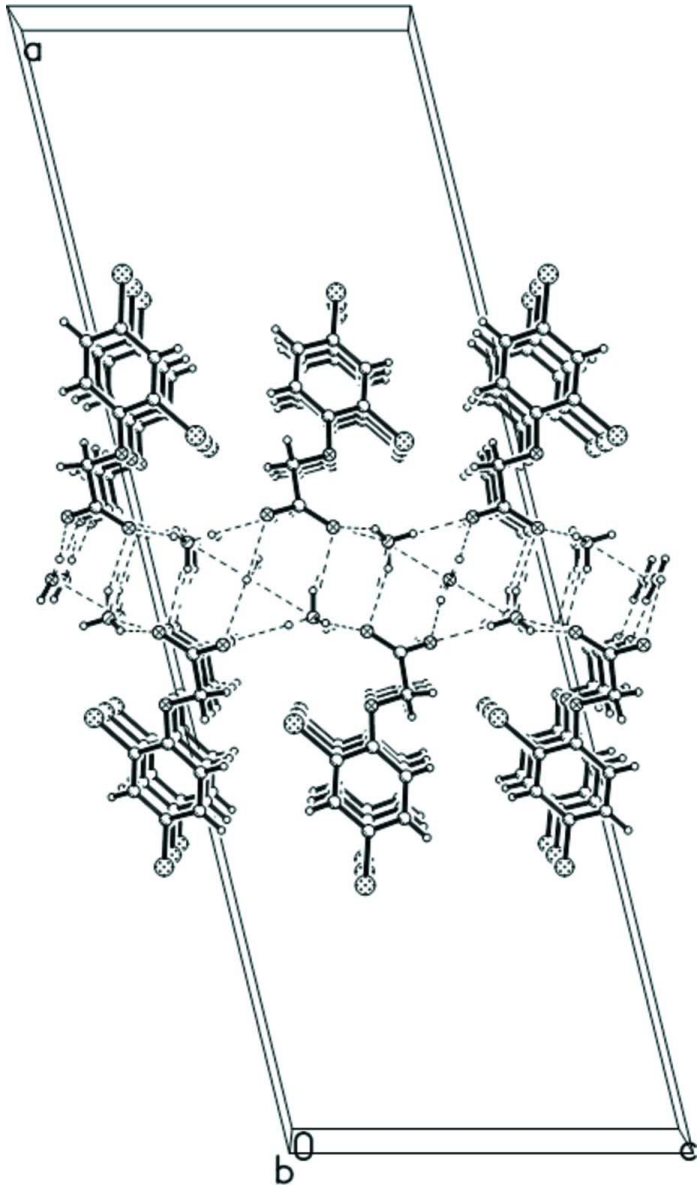

Figure 2

The crystal packing of (I), viewed along *b* axis. Hydrogen bonds are indicated by dashed lines.

Ammonium 2-(2,4-dichlorophenoxy)acetate hemihydrate

Crystal data

$NH_4^+ \cdot C_8H_5Cl_2O_3^- \cdot 0.5H_2O$
$M_r = 247.07$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 37.738\ (8)$ Å
$b = 4.3889\ (9)$ Å
$c = 12.900\ (3)$ Å
$\beta = 103.83\ (3)°$
$V = 2074.7\ (8)$ Å^3
$Z = 8$
$F(000) = 1016$
$D_x = 1.582$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2216 reflections
$\theta = 3.2–27.5°$
$\mu = 0.61$ mm^{-1}
$T = 293$ K
Bar, colourless
0.15 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
9447 measured reflections
2385 independent reflections
2216 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -48 \rightarrow 48$
$k = -5 \rightarrow 5$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.060$
$S = 1.07$
2385 reflections
137 parameters
90 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
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 1.6158P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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}
Cl2	0.252351 (7)	0.68291 (7)	0.14126 (2)	0.01935 (8)
Cl1	0.120306 (7)	0.46470 (7)	0.21755 (2)	0.01934 (8)
O3	0.04445 (2)	−0.21277 (17)	−0.01018 (6)	0.01408 (16)
O2	0.05453 (2)	−0.41964 (18)	−0.15912 (6)	0.01545 (16)
O1	0.10714 (2)	0.10909 (18)	0.02392 (6)	0.01371 (16)
C4	0.14091 (3)	0.2393 (2)	0.04524 (8)	0.0119 (2)
C6	0.18475 (3)	0.5588 (2)	0.16711 (8)	0.0146 (2)
H6A	0.1910	0.6774	0.2285	0.018*
C7	0.09822 (3)	−0.0753 (2)	−0.07012 (8)	0.0122 (2)
H7A	0.1178	−0.2195	−0.0687	0.015*
H7B	0.0961	0.0534	−0.1324	0.015*
C3	0.16595 (3)	0.2075 (2)	−0.01809 (9)	0.0141 (2)
H3A	0.1598	0.0924	−0.0803	0.017*
C8	0.06265 (3)	−0.2485 (2)	−0.07926 (8)	0.0113 (2)
C5	0.15090 (3)	0.4193 (2)	0.13763 (8)	0.0130 (2)
C2	0.20009 (3)	0.3460 (3)	0.01088 (9)	0.0152 (2)
H2A	0.2166	0.3227	−0.0316	0.018*

C1	0.20925 (3)	0.5183 (2)	0.10313 (9)	0.0142 (2)
N1	0.03308 (2)	0.2772 (2)	0.11575 (7)	0.01333 (18)
O1W	0.0000	−0.1574 (3)	0.2500	0.0237 (3)
H1A	0.0399	0.2902	0.1824	0.024 (4)*
H1B	0.0378	0.4414	0.0872	0.024 (4)*
H1WA	−0.0158	−0.2797	0.2154	0.044 (5)*
H1C	0.0093	0.2577	0.0997	0.029 (4)*
H1D	0.0423	0.1190	0.0853	0.029 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.01261 (13)	0.02457 (15)	0.02073 (14)	−0.00724 (10)	0.00367 (10)	−0.00251 (10)
Cl1	0.01548 (13)	0.02906 (16)	0.01553 (13)	−0.00433 (11)	0.00777 (10)	−0.00675 (10)
O3	0.0133 (3)	0.0140 (4)	0.0165 (4)	−0.0013 (3)	0.0066 (3)	−0.0002 (3)
O2	0.0164 (4)	0.0157 (4)	0.0137 (4)	−0.0026 (3)	0.0024 (3)	−0.0021 (3)
O1	0.0108 (3)	0.0175 (4)	0.0137 (4)	−0.0038 (3)	0.0047 (3)	−0.0044 (3)
C4	0.0099 (4)	0.0119 (5)	0.0134 (5)	−0.0002 (4)	0.0019 (4)	0.0022 (4)
C6	0.0150 (5)	0.0155 (5)	0.0125 (5)	−0.0014 (4)	0.0013 (4)	−0.0006 (4)
C7	0.0119 (5)	0.0132 (5)	0.0121 (5)	−0.0014 (4)	0.0042 (4)	−0.0020 (4)
C3	0.0137 (5)	0.0150 (5)	0.0140 (5)	−0.0012 (4)	0.0040 (4)	−0.0013 (4)
C8	0.0101 (4)	0.0101 (4)	0.0130 (5)	0.0014 (4)	0.0016 (4)	0.0033 (4)
C5	0.0125 (5)	0.0154 (5)	0.0121 (5)	0.0007 (4)	0.0048 (4)	0.0012 (4)
C2	0.0130 (5)	0.0171 (5)	0.0170 (5)	−0.0006 (4)	0.0064 (4)	0.0010 (4)
C1	0.0102 (5)	0.0151 (5)	0.0167 (5)	−0.0028 (4)	0.0019 (4)	0.0021 (4)
N1	0.0131 (4)	0.0134 (4)	0.0143 (4)	−0.0009 (3)	0.0050 (3)	0.0001 (3)
O1W	0.0199 (6)	0.0128 (5)	0.0358 (7)	0.000	0.0014 (5)	0.000

Geometric parameters (Å, º)

Cl2—C1	1.7400 (11)	C7—H7A	0.9700
Cl1—C5	1.7336 (12)	C7—H7B	0.9700
O3—C8	1.2584 (13)	C3—C2	1.3924 (15)
O2—C8	1.2523 (13)	C3—H3A	0.9300
O1—C4	1.3635 (13)	C2—C1	1.3824 (16)
O1—C7	1.4298 (12)	C2—H2A	0.9300
C4—C3	1.3962 (15)	N1—H1A	0.8385
C4—C5	1.4042 (15)	N1—H1B	0.8474
C6—C5	1.3851 (15)	N1—H1C	0.8757
C6—C1	1.3901 (16)	N1—H1D	0.9069
C6—H6A	0.9300	O1W—H1WA	0.8458
C7—C8	1.5225 (14)		
C4—O1—C7	115.30 (8)	O2—C8—C7	113.65 (9)
O1—C4—C3	124.81 (10)	O3—C8—C7	120.22 (9)
O1—C4—C5	117.10 (9)	C6—C5—C4	121.65 (10)
C3—C4—C5	118.09 (10)	C6—C5—Cl1	119.18 (8)
C5—C6—C1	118.74 (10)	C4—C5—Cl1	119.17 (8)

C5—C6—H6A	120.6	C1—C2—C3	119.65 (10)
C1—C6—H6A	120.6	C1—C2—H2A	120.2
O1—C7—C8	111.88 (9)	C3—C2—H2A	120.2
O1—C7—H7A	109.2	C2—C1—C6	121.08 (10)
C8—C7—H7A	109.2	C2—C1—Cl2	119.67 (9)
O1—C7—H7B	109.2	C6—C1—Cl2	119.24 (9)
C8—C7—H7B	109.2	H1A—N1—H1B	110.0
H7A—C7—H7B	107.9	H1A—N1—H1C	107.2
C2—C3—C4	120.77 (10)	H1B—N1—H1C	106.9
C2—C3—H3A	119.6	H1A—N1—H1D	116.2
C4—C3—H3A	119.6	H1B—N1—H1D	108.7
O2—C8—O3	126.13 (10)	H1C—N1—H1D	107.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1···O2^{i}	0.85	1.97	3.2969 (15)	170
N1—H1*A*···O2^{i}	0.84	2.07	2.8908 (14)	168
N1—H1*B*···O3^{ii}	0.85	2.02	2.8578 (13)	168
N1—H1*C*···O3^{iii}	0.88	2.09	2.9310 (15)	161

Symmetry codes: (i) $x, -y, z+1/2$; (ii) $x, y+1, z$; (iii) $-x, -y, -z$.