

Phenylmethanaminium chloroacetate

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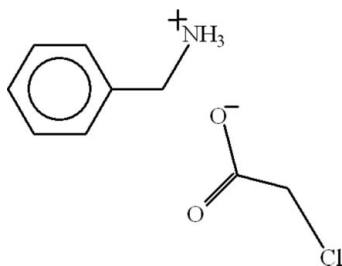
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.121; data-to-parameter ratio = 19.4.

In the title compound, $\text{C}_7\text{H}_{10}\text{N}^+\cdot\text{C}_2\text{H}_2\text{ClO}_2^-$, the planar chloroacetate ion [with a maximum deviation of 0.025 (3) Å] is oriented at a dihedral angle of 31.07 (4)° with respect to the planar [maximum deviation of 0.022 (3) Å] phenylmethanaminium cation. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a network. A weak $\text{C}-\text{H}\cdots\pi$ interaction is also present.

Related literature

For related structures, see: Amini *et al.* (2007); Houlemare-Druot & Coquerel (1998); Rademeyer (2003). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_7\text{H}_{10}\text{N}^+\cdot\text{C}_2\text{H}_2\text{ClO}_2^-$

$M_r = 201.65$

Orthorhombic, *Pbca*

$a = 11.1653$ (9) Å

$b = 8.0295$ (5) Å

$c = 22.3714$ (18) Å

$V = 2005.6$ (3) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\mu = 0.35$ mm⁻¹

$T = 296$ K

0.28 × 0.14 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.941$, $T_{\max} = 0.958$

11668 measured reflections

2485 independent reflections

1592 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.121$

$S = 1.03$

2485 reflections

128 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.21$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.894 (16)	1.919 (17)	2.779 (2)	160.9 (16)
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{ii}}$	0.869 (19)	2.026 (19)	2.798 (2)	147.5 (17)
$\text{N1}-\text{H1C}\cdots\text{O2}^{\text{iii}}$	0.94 (2)	1.80 (2)	2.740 (2)	175.5 (16)
$\text{C5}-\text{H5}\cdots\text{Cg1}^{\text{iv}}$	0.93	2.93	3.777	152

Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (iii) $x + 1, y, z$; (iv) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$. Cg1 is the centroid of the benzene ring.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2686).

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supplementary materials

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Phenylmethanaminium chloroacetate

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Comment

Organic ammonium salts have many applications such as phase transfer catalysis, photo base-generators *etc.* We have prepared a scheme for synthesizing various ammonium salts, which will differ due to the moiety attached to NH₃ group and due to the anion. We reported herein the crystal structure of the title compound, (I), in this regard. The crystal structures of benzylammonium nitrate, (II) (Rademeyer, 2003), bis(benzylammonium) sulfate, (III) (Amini *et al.*, 2007) and (±)- α -methylbenzylammonium chloroacetate, (IV) (Houllemare-Druot & Coquerel, 1998) have been reported.

The asymmetric unit of the title compound contains one cation and one anion (Fig. 1), in which the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The planar chloroacetate ion [with a maximum deviation of 0.025 (3) Å for atom C8] is oriented with respect to the planar phenylmethane moiety [with a maximum deviation of -0.022 (3) Å for atom C7] at a dihedral angle of 31.07 (4)°, and atom N1 is 1.3132 (24) Å away from the plane of the phenylmethane moiety.

In the crystal structure, strong intermolecular N-H...O hydrogen bonds (Table 1) link the molecules into a network (Fig. 2), in which they may be effective in the stabilization of the structure. There also exists a weak C—H... π interaction (Table 1).

Experimental

For the preparation of the title compound, benzylamine (1.09 ml, 0.01 mol) was added dropwise to a solution of chloroacetic acid (0.945 g, 0.01 mol) in dichloromethane (20 ml), and stirred for 30 min. The product precipitated, filtered out and washed with n-hexane. Crystals suitable for X-ray analysis were obtained from a mixture of n-hexane/chloroform (1:1).

Refinement

H atoms (for NH₃) were located in difference Fourier synthesis and refined isotropically. The remaining H atoms were positioned geometrically with C-H = 0.93 and 0.97 Å, for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

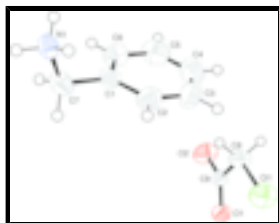


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

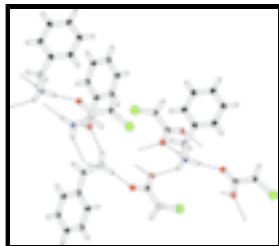
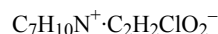


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

Phenylmethanaminium chloroacetate

Crystal data



$M_r = 201.65$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 11.1653$ (9) Å

$b = 8.0295$ (5) Å

$c = 22.3714$ (18) Å

$V = 2005.6$ (3) Å³

$Z = 8$

$F_{000} = 848$

$D_x = 1.336$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2485 reflections

$\theta = 2.6\text{--}28.3^\circ$

$\mu = 0.35$ mm⁻¹

$T = 296$ K

Needle, colorless

$0.28 \times 0.14 \times 0.12$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 7.40 pixels mm⁻¹

$T = 296$ K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.941$, $T_{\max} = 0.958$

11668 measured reflections

2485 independent reflections

1592 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 28.3^\circ$

$\theta_{\min} = 2.6^\circ$

$h = -12 \rightarrow 14$

$k = -7 \rightarrow 10$

$l = -29 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.121$

$S = 1.03$

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2834P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.21$ e Å⁻³

2485 reflections $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$
 128 parameters Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.0052 (11)
 Secondary atom site location: difference Fourier map

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.22460 (4)	0.33171 (7)	0.07550 (3)	0.0665 (2)
O1	-0.01235 (10)	0.22583 (15)	0.03185 (6)	0.0435 (4)
O2	-0.11363 (11)	0.44211 (18)	0.06754 (7)	0.0586 (5)
N1	0.64467 (14)	0.4356 (2)	0.04653 (7)	0.0368 (5)
C1	0.59953 (15)	0.4607 (2)	0.15510 (8)	0.0376 (5)
C2	0.69962 (18)	0.4421 (3)	0.19030 (10)	0.0519 (7)
C3	0.7096 (2)	0.5291 (3)	0.24323 (11)	0.0684 (9)
C4	0.6222 (3)	0.6365 (3)	0.26080 (10)	0.0693 (9)
C5	0.5239 (2)	0.6594 (3)	0.22598 (11)	0.0634 (8)
C6	0.51145 (17)	0.5705 (2)	0.17352 (9)	0.0492 (6)
C7	0.58321 (18)	0.3615 (2)	0.09867 (9)	0.0495 (7)
C8	0.08928 (14)	0.4447 (2)	0.08390 (9)	0.0436 (6)
C9	-0.02110 (14)	0.3609 (2)	0.05846 (7)	0.0333 (5)
H1A	0.6185 (16)	0.540 (2)	0.0418 (8)	0.0442*
H1B	0.6233 (17)	0.376 (2)	0.0159 (9)	0.0442*
H1C	0.7280 (18)	0.436 (2)	0.0517 (8)	0.0442*
H2	0.76068	0.37060	0.17840	0.0623*
H3	0.77687	0.51420	0.26716	0.0821*
H4	0.62979	0.69415	0.29664	0.0832*
H5	0.46510	0.73487	0.23747	0.0761*
H6	0.44308	0.58455	0.15031	0.0590*
H7A	0.49832	0.35274	0.08999	0.0593*
H7B	0.61368	0.24974	0.10502	0.0593*
H8A	0.07616	0.46464	0.12616	0.0523*
H8B	0.09865	0.55221	0.06471	0.0523*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0303 (3)	0.0642 (4)	0.1049 (5)	0.0065 (2)	-0.0105 (3)	-0.0122 (3)
O1	0.0383 (7)	0.0417 (7)	0.0504 (7)	-0.0084 (5)	0.0054 (5)	-0.0141 (6)
O2	0.0301 (7)	0.0623 (10)	0.0834 (11)	0.0074 (6)	-0.0015 (6)	-0.0179 (8)
N1	0.0312 (7)	0.0368 (9)	0.0424 (9)	0.0039 (7)	-0.0020 (7)	-0.0106 (7)
C1	0.0378 (9)	0.0337 (9)	0.0412 (10)	-0.0065 (8)	0.0030 (8)	0.0045 (8)
C2	0.0450 (11)	0.0533 (13)	0.0573 (13)	0.0005 (9)	-0.0035 (9)	0.0111 (10)
C3	0.0693 (15)	0.0801 (17)	0.0558 (14)	-0.0243 (14)	-0.0217 (12)	0.0170 (13)
C4	0.105 (2)	0.0624 (16)	0.0404 (12)	-0.0273 (15)	0.0086 (13)	-0.0014 (10)
C5	0.0784 (16)	0.0544 (14)	0.0575 (13)	0.0027 (12)	0.0264 (13)	-0.0040 (11)
C6	0.0416 (10)	0.0540 (12)	0.0519 (11)	0.0062 (9)	0.0067 (9)	0.0063 (9)
C7	0.0511 (11)	0.0411 (11)	0.0562 (12)	-0.0094 (9)	0.0043 (10)	-0.0049 (9)
C8	0.0320 (8)	0.0410 (11)	0.0577 (11)	0.0024 (8)	-0.0047 (8)	-0.0138 (9)
C9	0.0293 (8)	0.0355 (10)	0.0350 (9)	-0.0021 (7)	0.0009 (7)	-0.0020 (8)

Geometric parameters (\AA , $^\circ$)

C11—C8	1.7723 (17)	C4—C5	1.358 (4)
O1—C9	1.241 (2)	C5—C6	1.381 (3)
O2—C9	1.239 (2)	C2—H2	0.9300
N1—C7	1.478 (3)	C3—H3	0.9300
N1—H1A	0.894 (16)	C4—H4	0.9300
N1—H1B	0.869 (19)	C5—H5	0.9300
N1—H1C	0.94 (2)	C6—H6	0.9300
C1—C6	1.384 (2)	C7—H7A	0.9700
C1—C2	1.375 (3)	C7—H7B	0.9700
C1—C7	1.504 (3)	C8—C9	1.515 (2)
C2—C3	1.379 (3)	C8—H8A	0.9700
C3—C4	1.360 (4)	C8—H8B	0.9700
C11...O1	2.9455 (13)	C5...H3 ^{xii}	3.0000
C11...H7A	3.0800	C6...H3 ^{xii}	2.9700
C11...H1B ⁱ	2.871 (19)	C9...H1C ⁱⁱⁱ	2.87 (2)
C11...H8B ⁱⁱ	3.0000	C9...H1B ⁱ	2.998 (18)
O1...C11	2.9455 (13)	C9...H1A ⁱⁱ	2.822 (16)
O1...N1 ⁱ	2.798 (2)	C9...H8B ^{ix}	2.9700
O1...C7 ⁱ	3.187 (2)	H1A...O1 ^{iv}	1.919 (17)
O1...C7 ⁱⁱ	3.379 (2)	H1A...C9 ^{iv}	2.822 (16)
O1...N1 ⁱⁱ	2.779 (2)	H1B...C11 ^{vi}	2.871 (19)
O1...C6 ⁱⁱ	3.406 (2)	H1B...O1 ^{vi}	2.026 (19)
O2...N1 ⁱⁱⁱ	2.740 (2)	H1B...C9 ^{vi}	2.998 (18)
O1...H7A ⁱ	2.8000	H1C...O2 ^v	1.80 (2)
O1...H1A ⁱⁱ	1.919 (17)	H1C...C9 ^v	2.87 (2)
O1...H1B ⁱ	2.026 (19)	H2...H7B	2.5200

O2...H1C ⁱⁱⁱ	1.80 (2)	H2...O2 ^v	2.9100
O2...H2 ⁱⁱⁱ	2.9100	H2...C4 ^{vii}	2.9400
O2...H7B ^{iv}	2.6100	H3...C6 ^{xi}	2.9700
N1...O1 ^{iv}	2.779 (2)	H3...C5 ^{xi}	3.0000
N1...O2 ^v	2.740 (2)	H4...H8A ^{xi}	2.6000
N1...O1 ^{vi}	2.798 (2)	H5...C2 ^{xiii}	2.9600
C2...C4 ^{vii}	3.531 (4)	H5...C3 ^{xiii}	3.0900
C4...C2 ^{viii}	3.531 (4)	H5...C1 ^{xiii}	3.1000
C6...O1 ^{iv}	3.406 (2)	H6...H7A	2.3800
C6...C9 ^{iv}	3.475 (2)	H7A...C11	3.0800
C7...O1 ^{iv}	3.379 (2)	H7A...H6	2.3800
C7...O1 ^{vi}	3.187 (2)	H7A...O1 ^{vi}	2.8000
C9...C9 ^{ix}	3.472 (2)	H7B...H2	2.5200
C9...C6 ⁱⁱ	3.475 (2)	H7B...O2 ⁱⁱ	2.6100
C1...H5 ^x	3.1000	H8A...C4 ^{xii}	2.9300
C2...H5 ^x	2.9600	H8A...H4 ^{xii}	2.6000
C3...H5 ^x	3.0900	H8B...C9 ^{ix}	2.9700
C4...H2 ^{viii}	2.9400	H8B...C11 ^{iv}	3.0000
C4...H8A ^{xi}	2.9300		
H1B—N1—H1C	111.8 (16)	C5—C4—H4	120.00
C7—N1—H1C	111.4 (11)	C4—C5—H5	120.00
C7—N1—H1A	108.6 (12)	C6—C5—H5	120.00
C7—N1—H1B	105.8 (12)	C1—C6—H6	120.00
H1A—N1—H1B	109.5 (16)	C5—C6—H6	120.00
H1A—N1—H1C	109.6 (15)	N1—C7—H7A	109.00
C2—C1—C7	121.44 (17)	N1—C7—H7B	109.00
C2—C1—C6	118.44 (18)	C1—C7—H7A	109.00
C6—C1—C7	120.10 (16)	C1—C7—H7B	109.00
C1—C2—C3	120.2 (2)	H7A—C7—H7B	108.00
C2—C3—C4	120.7 (2)	C11—C8—C9	115.23 (12)
C3—C4—C5	120.0 (2)	O1—C9—O2	127.19 (15)
C4—C5—C6	119.9 (2)	O1—C9—C8	120.28 (14)
C1—C6—C5	120.74 (18)	O2—C9—C8	112.53 (14)
N1—C7—C1	113.14 (14)	C11—C8—H8A	108.00
C1—C2—H2	120.00	C11—C8—H8B	108.00
C3—C2—H2	120.00	C9—C8—H8A	108.00
C2—C3—H3	120.00	C9—C8—H8B	108.00
C4—C3—H3	120.00	H8A—C8—H8B	108.00
C3—C4—H4	120.00		
C6—C1—C2—C3	1.3 (3)	C1—C2—C3—C4	-1.3 (4)
C7—C1—C2—C3	-177.00 (19)	C2—C3—C4—C5	-0.2 (4)
C2—C1—C6—C5	0.1 (3)	C3—C4—C5—C6	1.6 (4)
C7—C1—C6—C5	178.42 (18)	C4—C5—C6—C1	-1.6 (3)
C2—C1—C7—N1	-83.7 (2)	C11—C8—C9—O1	-2.8 (2)
C6—C1—C7—N1	98.1 (2)	C11—C8—C9—O2	177.43 (13)

supplementary materials

Symmetry codes: (i) $x-1/2, -y+1/2, -z$; (ii) $-x+1/2, y-1/2, z$; (iii) $x-1, y, z$; (iv) $-x+1/2, y+1/2, z$; (v) $x+1, y, z$; (vi) $x+1/2, -y+1/2, -z$; (vii) $-x+3/2, y-1/2, z$; (viii) $-x+3/2, y+1/2, z$; (ix) $-x, -y+1, -z$; (x) $-x+1, y-1/2, -z+1/2$; (xi) $x+1/2, y, -z+1/2$; (xii) $x-1/2, y, -z+1/2$; (xiii) $-x+1, y+1/2, -z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^{iv}$	0.894 (16)	1.919 (17)	2.779 (2)	160.9 (16)
$N1-H1B\cdots O1^{vi}$	0.869 (19)	2.026 (19)	2.798 (2)	147.5 (17)
$N1-H1C\cdots O2^v$	0.94 (2)	1.80 (2)	2.740 (2)	175.5 (16)
$C5-H5\cdots Cg1^{xiii}$	0.93	2.93	3.777	152.00

Symmetry codes: (iv) $-x+1/2, y+1/2, z$; (vi) $x+1/2, -y+1/2, -z$; (v) $x+1, y, z$; (xiii) $-x+1, y+1/2, -z+1/2$.

Fig. 1

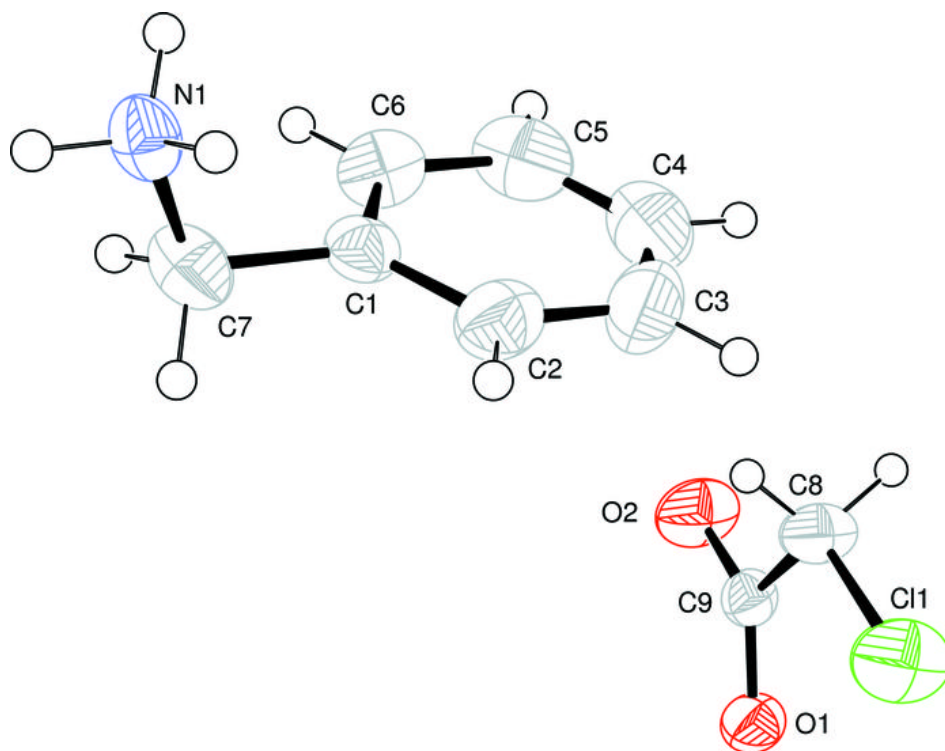


Fig. 2

