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Key indicators

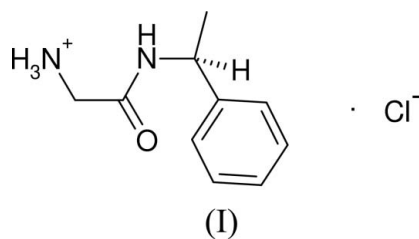
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.042
 wR factor = 0.127
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**{*N*-[(*S*)-1-Phenylethyl]carbamoyl}methylaminium
chloride**In the title compound, $\text{C}_{10}\text{H}_{15}\text{N}_2\text{O}^+\cdot\text{Cl}^-$, the crystal packing is
influenced by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds,
resulting in a layered structure.

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Comment

The known title compound, (I) (Fig. 1), was prepared as an
intermediate in the syntheses of new asymmetric catalysts,
following the literature procedure of Ho *et al.* (2001).

All the geometrical parameters for (I) lie within their expected ranges (Allen *et al.*, 1995). The absolute configuration of (I) is well defined and atom C7 has *S* configuration, as expected from the configuration of the equivalent C atom in the (*S*)-1-phenylethylamine starting material. The dihedral angle between the mean planes of the benzene ring (atoms C1–C6) and the C7/C9/C10/N1/O1 grouping is $66.14(13)^\circ$.

The crystal packing in (I) is influenced by hydrogen bonds (Table 1). An $\text{N}-\text{H}\cdots\text{O}$ bond arising from the N1 group links the cations into chains propagating in the *a* direction. The $-\text{NH}_3$ group participates in three $\text{N}-\text{H}\cdots\text{Cl}$ bonds [mean $\text{H}\cdots\text{Cl} = 2.32$ Å, mean $\text{N}\cdots\text{Cl} = 3.183(3)$ Å, mean $\text{N}-\text{H}\cdots\text{Cl} = 159^\circ$], which crosslink the [100] stacks in the *b* direction. The only intermolecular interactions in the *c*

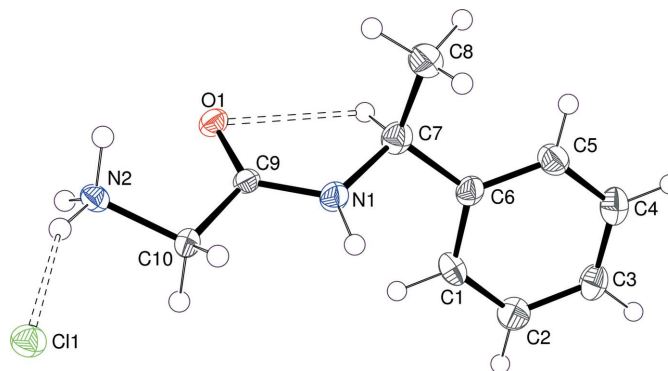


Figure 1

View of (I) (50% probability displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radii). The $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond and possible $\text{C}-\text{H}\cdots\text{O}$ interaction are indicated by dashed lines.

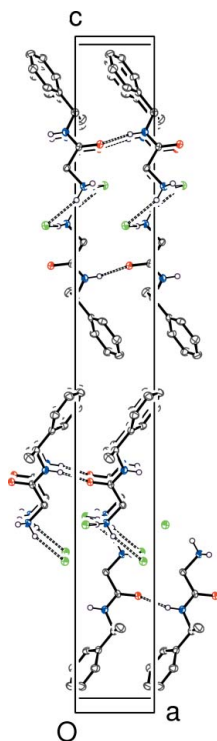


Figure 2
The packing of (I), viewed down [010], with all C-bound H atoms omitted for clarity and hydrogen bonds indicated by dashed lines.

direction are van der Waals forces (Fig. 2). A *PLATON* (Spek, 2003) analysis of (I) flagged a short intramolecular C—H...O distance (Fig. 1 and Table 1), although its structural significance – an attractive interaction or a repulsive steric contact – is not clear.

Experimental

N-Boc glycine (10 mmol, 1.75 g) was dissolved in dry THF (30 ml) in a dry flask under nitrogen. The solution was cooled to 195 K, and *N*-methyl morpholine (10 mmol, 1.01 g, 1.09 ml) was added with stirring. *t*Bu-chloroformate (10 mmol, 1.36 g, 1.30 ml) was added, and the solution stirred for 30 min. (*S*)-1-Phenylethylamine (10 mmol, 1.21 g, 1.29 ml) was added in one portion and the reaction mixture stirred at room temperature for 18 h. The solvent was removed *in vacuo*. The residue was taken up in EtOAc (30 ml), washed with 10% aqueous Na₂CO₃ (20 ml), 0.1 M aqueous HCl (20 ml) and saturated brine (20 ml), then dried over Na₂SO₄ and filtered, and the solvent was removed *in vacuo*. The resulting oil (1.37 g, 4.95 mmol) was dissolved in dry dichloromethane (DCM, 15 ml) and cooled to 273 K. Bubbling excess dry HCl through the reaction medium with stirring for 2 h allowed the collection of the desired product as a white precipitate, which was recrystallized from EtOH/Et₂O (1.09 g, 89%). Slow evaporation of a DCM solution of the purified material produced colourless needles of (I) suitable for diffraction; m.p. 446–449 K. [α]_D = –97.0°, *C* = 0.6 (MeOH); IR (KBr, cm^{–1}): ν_{\max} 3289 (C=O), 2967 (CH), 1660 (C=O), 1561 (C=O); ¹H NMR (250 MHz, CD₃OD): δ_{H} 9.2 (1H, *d*, *J* = 8.0 Hz, NH), 8.2 (3H, *s*, N⁺H₃), 7.3 (5H, *m*, Ph), 4.9 (1H, *q*, *J* = 7.0 Hz, CH), 3.6 (2H, *s*, CH₂), 1.3 (3H, *d*, *J* = 7.0 Hz, CH₃); ¹³C NMR (250 MHz, CD₃OD): δ_{C} 164.9 (C=O), 144.1, 128.3, 126.8, 126.1, 48.5 (CH), 40.1 (CH₂), 22.6 (CH₃); MS (ESI⁺):

calculated *m/z* 179.1179; found 179.1180 [M—Cl]⁺; (ESI[–]) 35.4 and 37.4 [Cl][–].

Crystal data

C₁₀H₁₅N₂O⁺·Cl[–]
M_r = 214.69
 Orthorhombic, *P*2₁2₁2₁
a = 4.6309 (3) Å
b = 5.8963 (4) Å
c = 39.939 (3) Å
V = 1090.54 (13) Å³
Z = 4
D_x = 1.308 Mg m^{–3}

Mo K α radiation
 Cell parameters from 856 reflections
 θ = 2.9–27.5°
 μ = 0.32 mm^{–1}
T = 120 (2) K
 Block cut from needle, colourless
 0.30 × 0.24 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2003)
*T*_{min} = 0.910, *T*_{max} = 0.951
 3682 measured reflections
 1768 independent reflections

1578 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.030
 θ_{\max} = 26.0°
h = –5 → 5
k = –6 → 7
l = –48 → 48

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.042
wR(*F*²) = 0.127
S = 1.14
 1768 reflections
 129 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 1.5926P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.41 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.39 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983),
 439 Friedel pairs
 Flack parameter: 0.08 (13)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...O1 ⁱ	0.88	2.01	2.839 (4)	156
N2—H2A...Cl1	0.91	2.32	3.181 (3)	157
N2—H2B...Cl1 ⁱⁱ	0.91	2.27	3.146 (3)	162
N2—H2C...Cl1 ⁱⁱⁱ	0.91	2.36	3.222 (3)	158
C7—H7...O1	1.00	2.45	2.809 (5)	101

Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) –*x*, *y* – ½, –*z* + ½; (iii) –*x*, *y* + ½, –*z* + ½.

All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å and N—H = 0.88–0.91 Å) and refined as riding on their carrier atoms, allowing for rotation of the rigid terminal –XH₃ groups. The constraint *U*_{iso}(H) = 1.2*U*_{eq}(carrier) or *U*_{iso}(H) = 1.5*U*_{eq}(methyl carrier) was applied as applicable.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997), *SCALEPACK* and *SORTAV* (Blessing 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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