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(*S*)-*N*-(1-Phenylethylpiperidin-1-ium-3-ylmethyl)-*N*-phenylpropionamide trifluoroacetate

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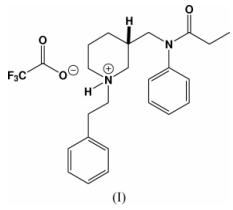
The synthesis and crystal structure of the title compound, $C_{25}H_{31}F_3N_2O_3$, are reported.



(I)

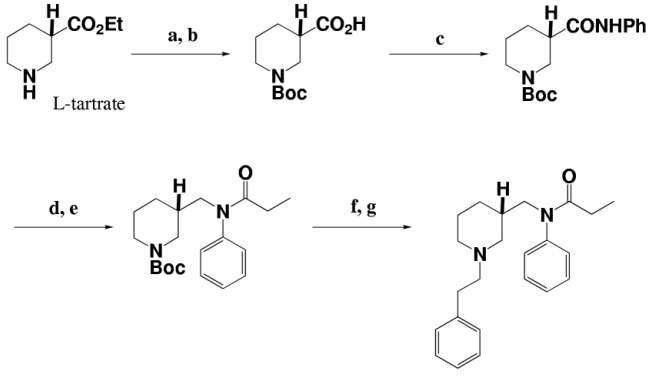
Key indicators

Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.047
wR factor = 0.125
Data-to-parameter ratio = 10.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Experimental

The title compound was synthesized from ethyl (*S*)-nipecotate L-tartrate (see reaction Scheme). Crystals were grown from an ethyl acetate solution.



(**a**) Boc_2O, NEt_3, Dioxan-H_2O, r.t., 4h. (**b**) LiOH, H_2O, 0 °C. (**c**) $PhNH_2$, DCC, CH_2Cl_2, r.t. (**d**) Borane, THF, reflux, 2h. (**e**) EtCOCl, $^iPr_2NEt$, CH_2Cl_2, r.t. (**f**) TFA, CH_2Cl_2, 0 °C. (**g**) $PhCH_2CH_2Br$, K_2CO_3, CH_3CN, 65 °C.
Boc: tert-BuOCO-; DCC; 1,3-Dicyclohexylcarbodiimide.

Crystal data

$C_{23}H_{31}N_2O^+\cdot C_2F_3O_2^-$
M_r = 464.52
Orthorhombic, $P2_12_12_1$
a = 10.1973 (7) Å
b = 10.8295 (7) Å
c = 21.9100 (10) Å
V = 2419.6 (3) Å^3
Z = 4
D_x = 1.275 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 648 reflections
θ = 3.0–28.7°
μ = 0.10 mm^{-1}
T = 213 (2) K
Plate, colorless
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
15 557 measured reflections
3332 independent reflections
3113 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 28.3°
h = −13 → 13
k = −14 → 9
l = −28 → 26

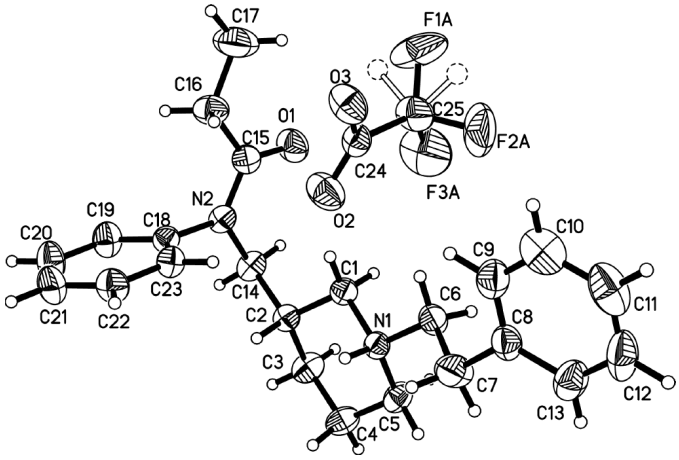


Figure 1
Displacement ellipsoid plot (50% probability level) of the title compound.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.125$
$S = 1.10$
3332 reflections
321 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 0.52P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.91 (3)	1.82 (3)	2.722 (2)	178 (2)

Symmetry code: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

Carbon-bound H atoms were placed in idealized positions and refined as riding, with C—H = 0.94 Å (0.98 Å for methyl H atoms). Nitrogen-bound H atoms were refined freely. Friedel pairs were merged for the final refinement since no atoms heavier than F are present and the absolute configuration was known.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL*97.

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