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1-Methoxycarbonyl-2-(4-nitrophenyl)-ethanaminium nitrate

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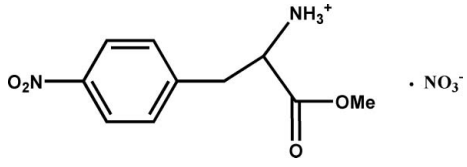
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.056; wR factor = 0.161; data-to-parameter ratio = 9.3.

In the title compound, $C_{10}H_{13}O_4N_2^+\cdot NO_3^-$, the nitro group and the benzene ring are essentially coplanar. The dihedral angle between the benzene ring and the methylcarboxylate plane is 49.6 (3)°. The crystal structure is stabilized by cation–anion N—H···O and N—H···N hydrogen bonds, building sheets parallel to (001).

Related literature

For details of α-amino acid derivatives, see: Lucchese *et al.* (2007); Arki *et al.* (2004); Hauck *et al.* (2006); Dai & Fu (2008); Wen (2008); Azim *et al.* (2006).



Experimental

Crystal data

$C_{10}H_{13}N_2O_4^+\cdot NO_3^-$
M_r = 287.23
Monoclinic, $P2_1$
a = 5.3722 (11) Å
b = 8.4244 (17) Å
c = 15.380 (3) Å
β = 93.67 (3)°
V = 694.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.94, T_{max} = 0.96
7232 measured reflections
1682 independent reflections
1164 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.160
S = 1.04
1682 reflections
181 parameters
7 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O5^{i}	0.89	1.90	2.771 (4)	166
N2—H2B···N3^{i}	0.89	2.60	3.402 (4)	151
N2—H2B···O6^{i}	0.89	2.61	3.218 (5)	126
N2—H2C···O6^{ii}	0.89	2.35	2.950 (5)	124
N2—H2C···O3^{iii}	0.89	2.41	2.929 (5)	117
N2—H2C···O7^{iii}	0.89	2.47	3.176 (5)	137
N2—H2A···O7	0.89	2.12	2.993 (5)	166
N2—H2A···O5	0.89	2.26	2.917 (4)	130
N2—H2A···N3	0.89	2.56	3.402 (4)	158

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by a start-up grant from Southeast University to Professor Ren-Gen Xiong.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2667).

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supporting information

Acta Cryst. (2008). E**64**, o1905 [doi:10.1107/S1600536808028390]

1-Methoxycarbonyl-2-(4-nitrophenyl)ethanaminium nitrate

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S1. Comment

Amino acid derivatives are important compounds due to their biological activities, and there has been an increased interest in the enantiomeric preparation of α-amino acid derivatives as precursors for the synthesis of novel biologically active molecules (Lucchese *et al.*, 2007; Arki *et al.*, 2004; Hauck *et al.*, 2006; Azim *et al.*, 2006; Dai *et al.*, 2008; Wen, 2008). Here we report the crystal structure of the title compound.

The asymmetric unit of the title compound contains a organic cation and a NO_3^- anion (Fig. 1). The nitro group and the benzene ring are essentially coplanar, and the methyl 2-aminopropanoate substituent group is in an extended conformation. The dihedral angle between the C1–C6 and C8–C10/O3/O4 planes is 49.6 (3)°. The *S* absolute configuration at C8 was deduced from the synthetic pathway.

The crystal packing is stabilized by cation–anion N—H···O and N—H···N hydrogen bonds (Table 1) building sheets parallel to the (001) (Fig. 2).

S2. Experimental

Under nitrogen protection, 2-amino-3-phenylpropanoic acid (30 mmol), nitric acid (50 mmol) and sulfuric acid (20 mmol) were added in a flask. The mixture was stirred at 383 K for 3 h. The resulting solution was poured into ice water (100 ml), then filtered and washed with distilled water. The nitration amino acid was esterified with H_2SO_4 and CH_3OH at 383 K for 12 h. The crude product obtained by evaporation of the solution was recrystallized with distilled water (15 ml)-HNO_3 (1 ml) to yield colourless block-like crystals, suitable for X-ray analysis.

S3. Refinement

All H atoms attached to C atoms and N atom were placed geometrically and treated as riding with C—H = 0.96 Å (methyl), 0.97 Å (methylene), 0.98 Å (methine), 0.93 Å (aromatic) and N—H = 0.89 Å with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}$(N and methyl C). In the absence of significant anomalous scattering, Friedel pairs were merged.

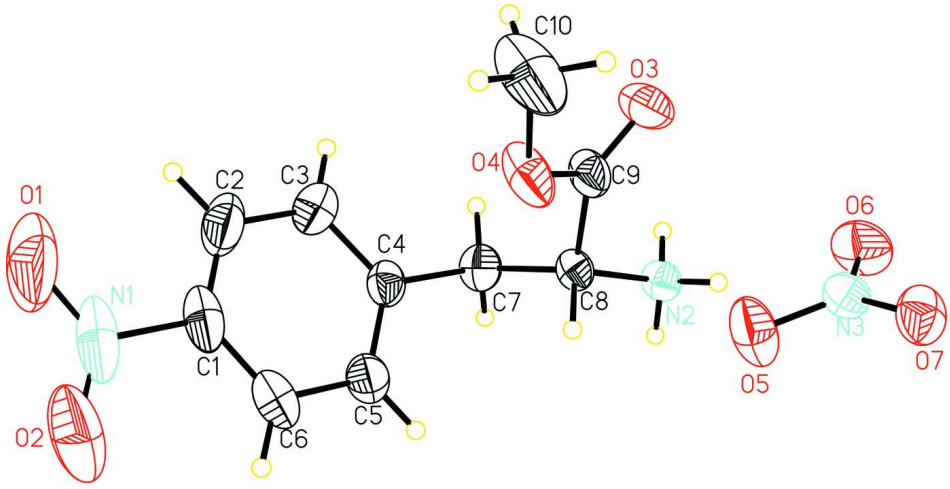

Figure 1

The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

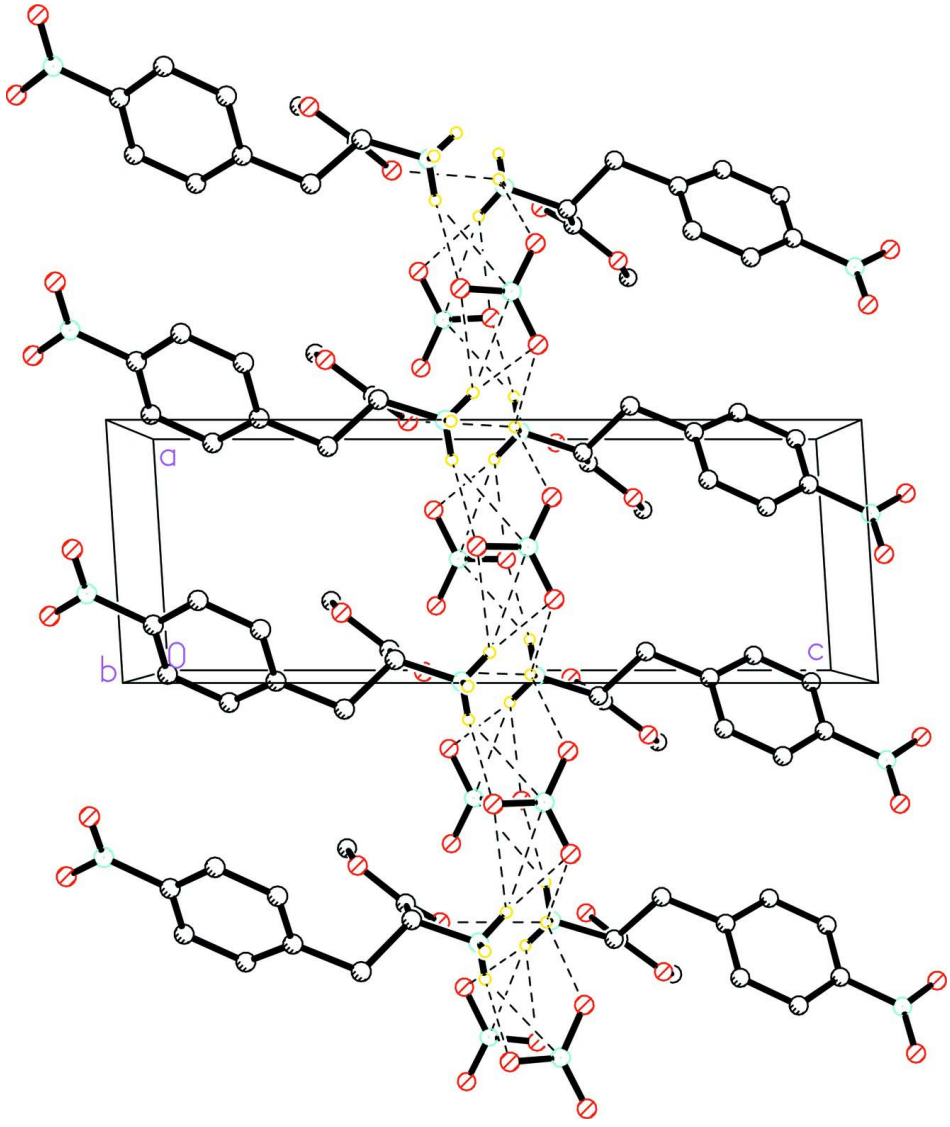

Figure 2

The crystal packing of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

1-Methoxycarbonyl-2-(4-nitrophenyl)ethanaminium nitrate

Crystal data

$C_{10}H_{13}N_2O_4^+\cdot NO_3^-$
$M_r = 287.23$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 5.3722\ (11)$ Å
$b = 8.4244\ (17)$ Å
$c = 15.380\ (3)$ Å
$\beta = 93.67\ (3)°$
$V = 694.6\ (2)$ Å^3
$Z = 2$

$F(000) = 300$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1657 reflections
$\theta = 3.6–27.5°$
$\mu = 0.12$ mm^{-1}
$T = 298$ K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.94, T_{max} = 0.96
7232 measured reflections
1682 independent reflections
1164 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.5°, θ_{min} = 3.6°
h = −6→6
k = −10→10
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.160
S = 1.04
1682 reflections
181 parameters
7 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
$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2 + 0.1127P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
O6	0.2827 (6)	0.2359 (4)	0.41323 (19)	0.0739 (9)
N2	0.9977 (5)	0.4443 (4)	0.55140 (18)	0.0502 (8)
H2A	0.8876	0.4035	0.5118	0.075*
H2B	1.1460	0.3995	0.5461	0.075*
H2C	1.0094	0.5485	0.5431	0.075*
O4	0.7381 (10)	0.2064 (5)	0.7129 (2)	0.1100 (13)
N3	0.4793 (6)	0.2783 (4)	0.4504 (2)	0.0551 (8)
C8	0.9152 (6)	0.4134 (5)	0.6393 (2)	0.0450 (8)
H8	0.7528	0.4645	0.6444	0.054*
O7	0.6843 (6)	0.2597 (5)	0.4190 (2)	0.0895 (11)
O3	0.9747 (8)	0.1395 (4)	0.6053 (2)	0.0883 (12)
C4	0.9935 (7)	0.4905 (5)	0.7975 (2)	0.0549 (10)
C7	1.1003 (7)	0.4825 (6)	0.7094 (2)	0.0600 (10)
H7A	1.2495	0.4175	0.7135	0.072*
H7B	1.1478	0.5885	0.6921	0.072*
O5	0.4776 (5)	0.3493 (7)	0.5199 (2)	0.0973 (15)

C9	0.8833 (9)	0.2365 (6)	0.6494 (2)	0.0620 (11)
C2	0.9717 (13)	0.4069 (9)	0.9457 (3)	0.0971 (19)
H2	1.0311	0.3444	0.9924	0.117*
C5	0.7944 (10)	0.5923 (6)	0.8092 (3)	0.0721 (13)
H5	0.7337	0.6558	0.7631	0.086*
C3	1.0808 (10)	0.3984 (8)	0.8659 (3)	0.0818 (14)
H3	1.2137	0.3297	0.8593	0.098*
C1	0.7795 (11)	0.5067 (8)	0.9543 (3)	0.0828 (16)
N1	0.6608 (16)	0.5128 (10)	1.0390 (3)	0.120 (2)
C6	0.6851 (10)	0.6006 (7)	0.8881 (3)	0.0839 (15)
H6	0.5514	0.6682	0.8957	0.101*
O2	0.4950 (14)	0.6095 (13)	1.0471 (4)	0.172 (3)
C10	0.6934 (16)	0.0396 (7)	0.7309 (4)	0.1130 (13)
H10A	0.5863	0.0308	0.7784	0.170*
H10B	0.8492	−0.0121	0.7463	0.170*
H10C	0.6150	−0.0099	0.6801	0.170*
O1	0.7474 (15)	0.4254 (10)	1.0972 (3)	0.179 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O6	0.081 (2)	0.067 (2)	0.0724 (19)	−0.0210 (17)	−0.0044 (16)	−0.0037 (17)
N2	0.0498 (16)	0.0528 (19)	0.0501 (16)	−0.0008 (14)	0.0186 (13)	0.0031 (14)
O4	0.196 (4)	0.0643 (19)	0.0772 (17)	−0.047 (2)	0.069 (2)	−0.0111 (15)
N3	0.0503 (18)	0.057 (2)	0.059 (2)	0.0003 (15)	0.0103 (15)	−0.0039 (17)
C8	0.0437 (17)	0.051 (2)	0.0413 (17)	−0.0039 (16)	0.0150 (14)	−0.0033 (16)
O7	0.074 (2)	0.102 (3)	0.095 (2)	0.019 (2)	0.0339 (18)	−0.010 (2)
O3	0.143 (3)	0.0526 (18)	0.075 (2)	0.0141 (19)	0.044 (2)	0.0034 (17)
C4	0.064 (2)	0.054 (2)	0.0467 (19)	−0.0102 (19)	0.0012 (16)	−0.0040 (18)
C7	0.055 (2)	0.069 (3)	0.056 (2)	−0.006 (2)	0.0083 (17)	0.005 (2)
O5	0.0501 (16)	0.153 (4)	0.089 (2)	0.005 (2)	0.0032 (16)	−0.057 (3)
C9	0.092 (3)	0.053 (2)	0.0432 (18)	−0.004 (2)	0.0229 (19)	−0.0008 (19)
C2	0.138 (5)	0.105 (5)	0.047 (2)	−0.012 (5)	−0.002 (3)	0.009 (3)
C5	0.095 (3)	0.066 (3)	0.054 (2)	0.003 (3)	0.006 (2)	−0.003 (2)
C3	0.095 (3)	0.090 (4)	0.060 (3)	0.007 (3)	−0.001 (2)	0.012 (3)
C1	0.103 (4)	0.094 (4)	0.053 (3)	−0.030 (3)	0.022 (3)	−0.020 (3)
N1	0.166 (6)	0.144 (6)	0.052 (3)	−0.059 (5)	0.028 (3)	−0.027 (4)
C6	0.099 (4)	0.089 (4)	0.066 (3)	0.001 (3)	0.018 (3)	−0.027 (3)
O2	0.183 (6)	0.240 (9)	0.102 (4)	−0.022 (6)	0.070 (4)	−0.058 (5)
C10	0.199 (4)	0.067 (2)	0.0810 (18)	−0.046 (2)	0.067 (2)	−0.0103 (17)
O1	0.265 (8)	0.211 (8)	0.065 (3)	−0.043 (6)	0.046 (4)	0.005 (4)

Geometric parameters (Å, º)

O6—N3	1.222 (4)	C7—H7A	0.97
N2—C8	1.473 (4)	C7—H7B	0.97
N2—H2A	0.89	C2—C1	1.344 (8)
N2—H2B	0.89	C2—C3	1.396 (7)

N2—H2C	0.89	C2—H2	0.93
O4—C9	1.312 (5)	C5—C6	1.384 (6)
O4—C10	1.455 (7)	C5—H5	0.93
N3—O5	1.225 (5)	C3—H3	0.93
N3—O7	1.241 (4)	C1—C6	1.361 (8)
C8—C9	1.509 (6)	C1—N1	1.488 (7)
C8—C7	1.534 (6)	N1—O2	1.219 (11)
C8—H8	0.98	N1—O1	1.227 (10)
O3—C9	1.189 (5)	C6—H6	0.93
C4—C3	1.366 (6)	C10—H10A	0.96
C4—C5	1.392 (7)	C10—H10B	0.96
C4—C7	1.506 (5)	C10—H10C	0.96

C8—N2—H2A	109.5	O3—C9—C8	124.5 (4)
C8—N2—H2B	109.5	O4—C9—C8	110.1 (4)
H2A—N2—H2B	109.5	C1—C2—C3	119.1 (5)
C8—N2—H2C	109.5	C1—C2—H2	120.4
H2A—N2—H2C	109.5	C3—C2—H2	120.4
H2B—N2—H2C	109.5	C6—C5—C4	121.2 (5)
C9—O4—C10	116.2 (4)	C6—C5—H5	119.4
O6—N3—O5	119.8 (3)	C4—C5—H5	119.4
O6—N3—O7	122.9 (4)	C4—C3—C2	120.4 (5)
O5—N3—O7	117.2 (4)	C4—C3—H3	119.8
N2—C8—C9	108.2 (3)	C2—C3—H3	119.8
N2—C8—C7	111.0 (3)	C2—C1—C6	122.8 (5)
C9—C8—C7	112.0 (4)	C2—C1—N1	118.8 (7)
N2—C8—H8	108.5	C6—C1—N1	118.4 (7)
C9—C8—H8	108.5	O2—N1—O1	124.9 (7)
C7—C8—H8	108.5	O2—N1—C1	118.0 (8)
C3—C4—C5	118.6 (4)	O1—N1—C1	117.0 (9)
C3—C4—C7	122.4 (4)	C1—C6—C5	117.9 (5)
C5—C4—C7	118.9 (4)	C1—C6—H6	121.0
C4—C7—C8	112.4 (3)	C5—C6—H6	121.0
C4—C7—H7A	109.1	O4—C10—H10A	109.5
C8—C7—H7A	109.1	O4—C10—H10B	109.5
C4—C7—H7B	109.1	H10A—C10—H10B	109.5
C8—C7—H7B	109.1	O4—C10—H10C	109.5
H7A—C7—H7B	107.8	H10A—C10—H10C	109.5
O3—C9—O4	125.4 (4)	H10B—C10—H10C	109.5

C3—C4—C7—C8	112.3 (5)	C5—C4—C3—C2	0.0 (8)
C5—C4—C7—C8	−66.0 (5)	C7—C4—C3—C2	−178.3 (5)
N2—C8—C7—C4	165.5 (3)	C1—C2—C3—C4	0.1 (9)
C9—C8—C7—C4	−73.4 (5)	C3—C2—C1—C6	0.1 (9)
C10—O4—C9—O3	1.7 (9)	C3—C2—C1—N1	178.8 (5)
C10—O4—C9—C8	−179.2 (6)	C2—C1—N1—O2	176.4 (7)
N2—C8—C9—O3	19.3 (6)	C6—C1—N1—O2	−4.9 (9)
C7—C8—C9—O3	−103.4 (5)	C2—C1—N1—O1	0.3 (8)

N2—C8—C9—O4	−159.9 (4)	C6—C1—N1—O1	179.1 (6)
C7—C8—C9—O4	77.5 (5)	C2—C1—C6—C5	−0.3 (8)
C3—C4—C5—C6	−0.2 (7)	N1—C1—C6—C5	−179.0 (5)
C7—C4—C5—C6	178.2 (4)	C4—C5—C6—C1	0.4 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O5^{i}	0.89	1.90	2.771 (4)	166
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Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) −*x*+2, *y*+1/2, −*z*+1.