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(S)-1-Methoxycarbonyl-3-(4-nitrophenyl)propan-2-aminium bromide

Bo Wang

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: fudavid88@yahoo.com.cn

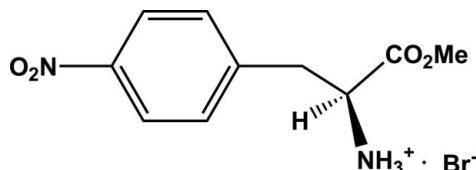
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.042; wR factor = 0.085; data-to-parameter ratio = 18.9.

In the crystal structure of the title compound, $\text{C}_{10}\text{H}_{13}\text{N}_2\text{O}_4^+\text{Br}^-$, intermolecular $\text{N}-\text{H}\cdots\text{Br}$ and $\text{N}-\text{H}\cdots(\text{O},\text{Br})$ hydrogen bonds link the cations and anions into a two-dimensional network parallel to the ab plane.

Related literature

For applications of metal-organic coordination compounds, see: Xiong *et al.* (1999); Fu, Zhang *et al.* (2008); Fu & Xiong (2008). For metal-organic frameworks with amino acid derivatives, see: Chen *et al.* (2000); Xie *et al.* (2002); Fu *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_{13}\text{N}_2\text{O}_4^+\text{Br}^-$
 $M_r = 305.13$

 Monoclinic, $P2_1$
 $a = 4.9323$ (10) Å

 $b = 8.6233$ (17) Å

 $c = 15.226$ (3) Å

 $\beta = 95.77$ (3)°

 $V = 644.3$ (2) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 3.20$ mm⁻¹
 $T = 298$ K

 $0.40 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 diffractometer

Absorption correction: multi-scan

 (*CrystalClear*; Rigaku, 2005)

 $T_{\min} = 0.90$, $T_{\max} = 1.00$

6658 measured reflections

2917 independent reflections

 2532 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.085$
 $S = 1.04$

2917 reflections

154 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.62$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Absolute structure: Flack (1983),

1202 Friedel pairs

Flack parameter: 0.008 (5)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{i}}$	0.87	2.61	3.031 (4)	111
$\text{N1}-\text{H1A}\cdots\text{Br1}^{\text{ii}}$	0.87	2.61	3.290 (3)	135
$\text{N1}-\text{H1B}\cdots\text{Br1}^{\text{iii}}$	0.93	2.51	3.303 (3)	143
$\text{N1}-\text{H1C}\cdots\text{Br1}^{\text{iv}}$	1.00	2.55	3.495 (3)	157

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

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/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC*.

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: CV2604).

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supplementary materials

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(S)-1-Methoxycarbonyl-3-(4-nitrophenyl)propan-2-aminium bromide

B. Wang

Comment

The construction of metal-organic coordination compounds has attracted much attention owing to potential functions, such as permittivity, fluorescence, magnetism and optical properties (Fu, Zhang *et al.*, 2008; Xiong *et al.*, 1999; Fu & Xiong, 2008). Amino acid derivatives constitute a class of excellent ligands for the construction of novel metal-organic frameworks (Fu *et al.*, 2007; Xie *et al.*, 2002; Chen *et al.*, 2000). We report here the crystal structure of the title compound.

The title compound is built up from a Br⁻ anion and a protonated amino group cation (Fig.1). The nitro group and the benzene ring are nearly coplanar being twisted to each other by 2.39 (6)°. The *S* absolute configuration at C8 is deduced from the synthetic pathway and confirmed by the X-ray analysis.

The crystal packing is stabilized by N—H···Br and N—H···O H-bonds (Table 1) building an infinite two-dimensional network parallel to *ab* plane (Fig.2).

Experimental

Under nitrogen protection, methyl 2-amino-3-(4-nitrophenyl)propanoate (30 mmol), nitric acid (50 mmol) and sulfuric acid (20 mmol) were added in a flask. The mixture was stirred at 110 °C for 3 hours. The resulting solution was poured into ice water (100mL), then filtered and washed with distilled water. The crude product was recrystallized with distilled water by adding 4ml HBr to yield colourless needle-like crystals, suitable for X-ray analysis.

Refinement

C-bound H atoms were positioned geometrically and treated as riding, with C-H = 0.93 Å (aromatic), C-H = 0.96 Å (methyl), C-H = 0.97 Å (methylene) and C-H = 0.98 Å (methine), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$. The H atoms of amine group were located in difference Fourier maps and at the last stage of refinement they were treated as riding, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$.

Figures

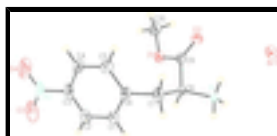


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

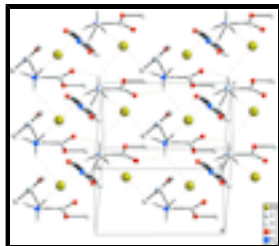


Fig. 2. A portion of the crystal packing, viewed along the *c* axis. Dashed lines denote N—H···Br and N—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity.

(S)-1-Methoxycarbonyl-3-(4-nitrophenyl)propan-2-aminium bromide

Crystal data

$C_{10}H_{13}N_2O_4^+ \cdot Br^-$	$F_{000} = 308$
$M_r = 305.13$	$D_x = 1.573 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2yb	Cell parameters from 2532 reflections
$a = 4.9323 (10) \text{ \AA}$	$\theta = 3.6\text{--}27.5^\circ$
$b = 8.6233 (17) \text{ \AA}$	$\mu = 3.20 \text{ mm}^{-1}$
$c = 15.226 (3) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 95.77 (3)^\circ$	Needle, colourless
$V = 644.3 (2) \text{ \AA}^3$	$0.40 \times 0.05 \times 0.05 \text{ mm}$
$Z = 2$	

Data collection

Rigaku Mercury2 diffractometer	2917 independent reflections
Radiation source: fine-focus sealed tube	2532 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.055$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 298 \text{ K}$	$\theta_{\text{min}} = 3.6^\circ$
CCD profile fitting scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$k = -11 \rightarrow 11$
$T_{\text{min}} = 0.90$, $T_{\text{max}} = 1.00$	$l = -19 \rightarrow 19$
6658 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2)]$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.62 \text{ e \AA}^{-3}$
2917 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
154 parameters	Extinction correction: none

1 restraint Absolute structure: Flack (1983), 1202 Friedel pairs
 Primary atom site location: structure-invariant direct methods Flack parameter: 0.008 (5)
 Secondary atom site location: difference Fourier map

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 (\AA^2)

	x	y	z	U_{iso}^*/U_{eq}
C6	0.4984 (8)	0.4001 (4)	0.7127 (2)	0.0390 (9)
C2	0.8039 (10)	0.2806 (6)	0.6203 (3)	0.0565 (12)
H2	0.9359	0.2063	0.6122	0.068*
C7	0.3794 (8)	0.4113 (5)	0.7995 (2)	0.0438 (10)
H7A	0.3349	0.3081	0.8188	0.053*
H7B	0.2118	0.4708	0.7916	0.053*
C3	0.7186 (9)	0.3798 (6)	0.5544 (3)	0.0527 (12)
C5	0.4128 (9)	0.5000 (5)	0.6421 (3)	0.0554 (11)
H5	0.2796	0.5742	0.6489	0.067*
C4	0.5236 (10)	0.4886 (6)	0.5644 (3)	0.0642 (15)
H4	0.4667	0.5550	0.5180	0.077*
C1	0.6940 (9)	0.2910 (5)	0.6986 (3)	0.0481 (11)
H1	0.7523	0.2226	0.7439	0.058*
O1	0.7970 (5)	0.6832 (4)	0.79876 (16)	0.0488 (7)
C9	0.6074 (6)	0.6629 (6)	0.85319 (19)	0.0360 (7)
O2	0.4710 (6)	0.7600 (3)	0.88109 (18)	0.0495 (7)
C8	0.5764 (8)	0.4886 (4)	0.8712 (2)	0.0325 (8)
H8	0.7551	0.4384	0.8731	0.039*
N2	0.8359 (11)	0.3691 (6)	0.4697 (3)	0.0759 (13)
O3	1.0072 (9)	0.2696 (7)	0.4603 (2)	0.1077 (17)
O4	0.7514 (11)	0.4561 (6)	0.4108 (3)	0.1200 (18)
C10	0.8330 (12)	0.8435 (6)	0.7701 (3)	0.0717 (15)
H10A	0.9736	0.8472	0.7308	0.107*
H10B	0.8837	0.9076	0.8206	0.107*
H10C	0.6653	0.8808	0.7399	0.107*
N1	0.4679 (6)	0.4728 (4)	0.95973 (18)	0.0391 (8)
H1A	0.6015	0.4736	1.0021	0.059*
H1B	0.3245	0.5428	0.9591	0.059*
H1C	0.3835	0.3672	0.9607	0.059*

supplementary materials

Br1 0.99538 (6) 0.66497 (4) 0.05395 (2) 0.04632 (13)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.045 (2)	0.043 (2)	0.029 (2)	-0.0124 (19)	0.0045 (17)	-0.0031 (18)
C2	0.065 (3)	0.061 (3)	0.045 (3)	0.003 (2)	0.009 (2)	-0.018 (2)
C7	0.042 (2)	0.050 (2)	0.041 (2)	-0.014 (2)	0.0121 (18)	-0.006 (2)
C3	0.064 (3)	0.064 (3)	0.032 (2)	-0.019 (2)	0.015 (2)	-0.014 (2)
C5	0.062 (3)	0.056 (3)	0.049 (3)	0.007 (2)	0.006 (2)	0.002 (2)
C4	0.096 (4)	0.064 (3)	0.033 (3)	-0.005 (3)	0.010 (3)	0.008 (2)
C1	0.063 (3)	0.041 (2)	0.040 (2)	0.003 (2)	0.005 (2)	-0.0061 (19)
O1	0.0659 (16)	0.0393 (17)	0.0455 (14)	-0.0094 (17)	0.0266 (12)	-0.0001 (15)
C9	0.0413 (16)	0.0386 (16)	0.0280 (16)	-0.001 (3)	0.0032 (13)	0.001 (2)
O2	0.0603 (18)	0.0379 (16)	0.0533 (18)	0.0130 (15)	0.0201 (14)	0.0078 (14)
C8	0.039 (2)	0.0319 (19)	0.0278 (19)	0.0033 (17)	0.0099 (15)	-0.0005 (16)
N2	0.098 (4)	0.094 (3)	0.039 (3)	-0.028 (3)	0.024 (3)	-0.021 (2)
O3	0.109 (4)	0.151 (5)	0.070 (3)	-0.003 (3)	0.044 (3)	-0.034 (3)
O4	0.177 (5)	0.142 (4)	0.049 (3)	-0.017 (4)	0.048 (3)	0.001 (3)
C10	0.103 (4)	0.058 (3)	0.059 (3)	-0.015 (3)	0.031 (3)	0.010 (2)
N1	0.054 (2)	0.0350 (17)	0.0299 (17)	0.0033 (15)	0.0137 (15)	0.0039 (14)
Br1	0.0471 (2)	0.0436 (2)	0.0490 (2)	0.0020 (3)	0.00881 (15)	-0.0088 (3)

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

C6—C1	1.380 (6)	O1—C9	1.322 (4)
C6—C5	1.408 (5)	O1—C10	1.466 (6)
C6—C7	1.503 (5)	C9—O2	1.180 (5)
C2—C3	1.353 (6)	C9—C8	1.538 (6)
C2—C1	1.363 (5)	C8—N1	1.506 (4)
C2—H2	0.9300	C8—H8	0.9800
C7—C8	1.538 (5)	N2—O4	1.210 (6)
C7—H7A	0.9700	N2—O3	1.223 (6)
C7—H7B	0.9700	C10—H10A	0.9600
C3—C4	1.363 (6)	C10—H10B	0.9600
C3—N2	1.468 (6)	C10—H10C	0.9600
C5—C4	1.356 (5)	N1—H1A	0.8742
C5—H5	0.9300	N1—H1B	0.9289
C4—H4	0.9300	N1—H1C	1.0023
C1—H1	0.9300		
C1—C6—C5	117.4 (4)	O2—C9—O1	126.6 (5)
C1—C6—C7	121.4 (4)	O2—C9—C8	124.0 (3)
C5—C6—C7	121.2 (4)	O1—C9—C8	109.3 (4)
C3—C2—C1	119.1 (4)	N1—C8—C9	107.4 (3)
C3—C2—H2	120.4	N1—C8—C7	109.9 (3)
C1—C2—H2	120.4	C9—C8—C7	111.4 (3)
C6—C7—C8	112.1 (3)	N1—C8—H8	109.4
C6—C7—H7A	109.2	C9—C8—H8	109.4

C8—C7—H7A	109.2	C7—C8—H8	109.4
C6—C7—H7B	109.2	O4—N2—O3	122.6 (5)
C8—C7—H7B	109.2	O4—N2—C3	118.3 (6)
H7A—C7—H7B	107.9	O3—N2—C3	119.0 (5)
C2—C3—C4	121.5 (4)	O1—C10—H10A	109.5
C2—C3—N2	119.4 (5)	O1—C10—H10B	109.5
C4—C3—N2	119.1 (5)	H10A—C10—H10B	109.5
C4—C5—C6	120.3 (4)	O1—C10—H10C	109.5
C4—C5—H5	119.8	H10A—C10—H10C	109.5
C6—C5—H5	119.8	H10B—C10—H10C	109.5
C5—C4—C3	119.9 (4)	C8—N1—H1A	110.5
C5—C4—H4	120.1	C8—N1—H1B	105.7
C3—C4—H4	120.1	H1A—N1—H1B	121.3
C2—C1—C6	121.8 (4)	C8—N1—H1C	106.3
C2—C1—H1	119.1	H1A—N1—H1C	106.2
C6—C1—H1	119.1	H1B—N1—H1C	105.9
C9—O1—C10	115.2 (4)		
C1—C6—C7—C8	75.9 (5)	C10—O1—C9—O2	-1.2 (5)
C5—C6—C7—C8	-104.6 (4)	C10—O1—C9—C8	175.3 (3)
C1—C2—C3—C4	-0.6 (7)	O2—C9—C8—N1	-29.0 (4)
C1—C2—C3—N2	-179.9 (4)	O1—C9—C8—N1	154.3 (3)
C1—C6—C5—C4	-0.8 (6)	O2—C9—C8—C7	91.3 (4)
C7—C6—C5—C4	179.7 (4)	O1—C9—C8—C7	-85.4 (3)
C6—C5—C4—C3	0.1 (7)	C6—C7—C8—N1	-170.5 (3)
C2—C3—C4—C5	0.6 (7)	C6—C7—C8—C9	70.7 (4)
N2—C3—C4—C5	179.9 (4)	C2—C3—N2—O4	178.0 (5)
C3—C2—C1—C6	-0.2 (7)	C4—C3—N2—O4	-1.3 (7)
C5—C6—C1—C2	0.9 (6)	C2—C3—N2—O3	0.6 (7)
C7—C6—C1—C2	-179.7 (4)	C4—C3—N2—O3	-178.7 (5)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O2 ⁱ	0.87	2.61	3.031 (4)	111
N1—H1A \cdots Br1 ⁱⁱ	0.87	2.61	3.290 (3)	135
N1—H1B \cdots Br1 ⁱⁱⁱ	0.93	2.51	3.303 (3)	143
N1—H1C \cdots Br1 ^{iv}	1.00	2.55	3.495 (3)	157

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

Fig. 1

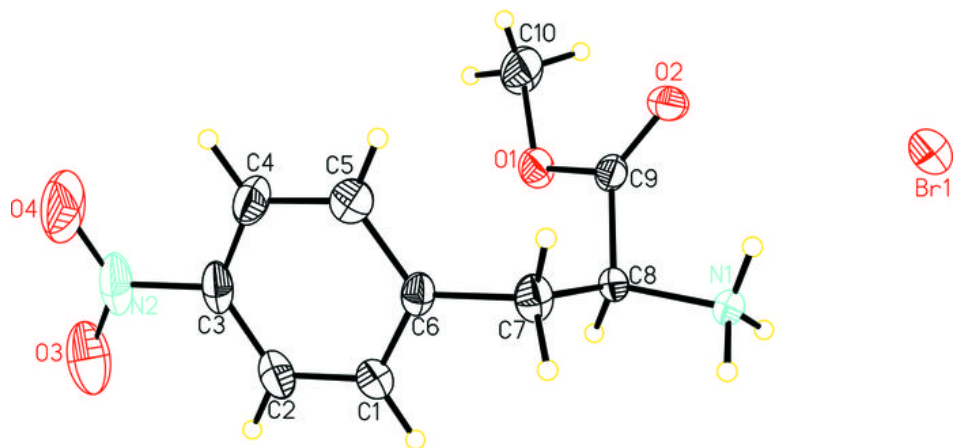


Fig. 2

