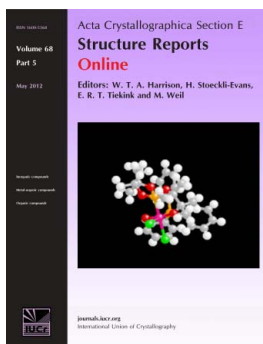


# Ammonium 4-methoxybenzenesulfonate

Sebastián Suarez, Fabio Doctorovich and Ricardo Baggio

*Acta Cryst.* (2012). **E68**, o2228–o2229

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## Ammonium 4-methoxybenzenesulfonate

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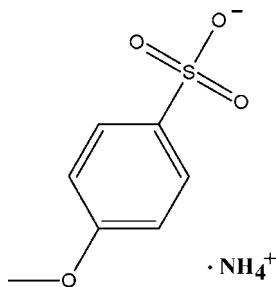
Received 14 June 2012; accepted 20 June 2012

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.119; data-to-parameter ratio = 12.8.

The molecular structure of the title compound,  $\text{NH}_4^+ \cdot \text{C}_7\text{H}_7\text{O}_4\text{S}^-$ , is featureless [the methoxy C atom deviating 0.173 (6) Å from the phenyl mean plane] with interatomic distances and angles in the expected ranges. The main feature of interest is the packing mode. Hydrophilic ( $\text{SO}_3$  and  $\text{NH}_4$ ) and hydrophobic ( $\text{PhOCH}_3$ ) parts in the structure segregate, the former interacting through a dense hydrogen-bonding scheme, leading to a well connected two-dimensional structure parallel to (100) and the latter hydrophobic groups acting as spacers for an interplanar separation of  $c/2 = 10.205$  (2) Å. In spite of being aligned along [110], the benzene rings stack in a far from parallel fashion [*viz.* consecutive ring centers determine a broken line with a 164.72 (12)° zigzag angle], thus preventing any possible  $\pi$ - $\pi$  interaction.

### Related literature

For literature on the role of weak interactions in supra-molecular structures, see: Desiraju (2007). For related structures, see: Fewings *et al.* (2001); Wang *et al.* (2007). For the Cambridge Structural Database, see: Allen (2002). For the synthesis, see: Porcheddu *et al.* (2009).



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### Experimental

#### Crystal data

$\text{NH}_4^+ \cdot \text{C}_7\text{H}_7\text{O}_4\text{S}^-$   
 $M_r = 205.23$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 6.2664$  (12) Å  
 $b = 7.1342$  (12) Å  
 $c = 20.410$  (2) Å  
 $V = 912.4$  (2) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.34$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.20 \times 0.10 \times 0.10$  mm

#### Data collection

Oxford Diffraction Gemini CCD S Ultra diffractometer  
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.958$ ,  $T_{\max} = 0.965$   
 4265 measured reflections  
 1732 independent reflections  
 1548 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.119$   
 $S = 1.04$   
 1732 reflections  
 135 parameters  
 21 restraints  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.47$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.36$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983), 637 Friedel pairs  
 Flack parameter:  $-0.11$  (14)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1N} \cdots \text{O1}^{\text{i}}$	0.88 (2)	1.99 (2)	2.851 (3)	170 (3)
$\text{N1}-\text{H4N} \cdots \text{O2}^{\text{ii}}$	0.86 (2)	1.98 (2)	2.797 (3)	160 (3)
$\text{N1}-\text{H2N} \cdots \text{O3}^{\text{iii}}$	0.88 (2)	1.98 (2)	2.824 (3)	162 (3)
$\text{N1}-\text{H3N} \cdots \text{O3}$	0.87 (2)	2.04 (2)	2.890 (3)	164 (3)

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ ; (iii)  $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ .

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *SHELXL97* and *PLATON* (Spek, 2009).

The authors acknowledge ANPCyT (project No. PME 2006-01113) for the purchase of the Oxford Gemini CCD diffractometer and the Spanish Research Council (CSIC) for the provision of a free-of-charge licence to the Cambridge Structural Database (Allen, 2002).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QM2074).

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**E63**, o3481.

## supplementary materials

*Acta Cryst.* (2012). E68, o2228–o2229 [doi:10.1107/S1600536812028103]

**Ammonium 4-methoxybenzenesulfonate****Sebastián Suarez, Fabio Doctorovich and Ricardo Baggio****Comment**

The study of supramolecular systems determined by weak interactions such as hydrogen bonding,  $\pi$ - $\pi$  stacking or dipole-dipole interactions have been, and currently are, active fields of structural research due to their implications in crystal engineering, self-assembly and, above all, biological systems (Desiraju, 2007). Derivatives of the benzenesulfonate anion are extremely suited to this end due to the possibility of  $\pi$ -interactions between arene rings, as well as hydrogen bonding between the sulphonate groups and any H donor eventually available (Water, ammonium, *etc.*). With this latter  $\text{NH}_4$  partner a number of structures of the sort have been published (among many others, ammonium *p*-toluenesulfonate, Fewings *et al.*, 2001, (II); ammonium 4-hydroxybenzenesulfonate, Wang *et al.*, 2007, (III), *etc.*), the vast majority displaying, as expected, an extremely complex non-bonding interactions scheme. We present herein one further member in this family, ammonium 4-methoxybenzenesulfonate,  $\text{C}_7\text{H}_7\text{O}_4\text{S}\cdot\text{H}_4\text{N}$  (I), which ended up being isostructural to (II) but different from (III), in spite of the very similar formulations.

The molecular structure in (I) (Fig 1) is featureless, with interatomic bond and angles in the expected ranges, and its main interest resides in the packing mode. Hydrophilic ( $\text{SO}_3$ ,  $\text{NH}_4$ ) and hydrophobic ( $\text{PhOCH}_3$ ) parts in the structure segregate, the former one interacting through a dense H-bonding scheme (Table 1) leading to a well connected two-dimensional structure, parallel to (100) (Fig 2a) and the latter hydrophobic groups acting as spacers (Figs 2 b, 2c), for an interplanar separation of  $C/2 = 10.205$  (2) Å. In spite of the deceiving views in Figs 2 b/2c, Ph groups stack in a far from parallel fashion, defining dihedral angles of  $37^\circ$  and thus preventing any possible  $\pi$ - $\pi$  interaction.

**Experimental**

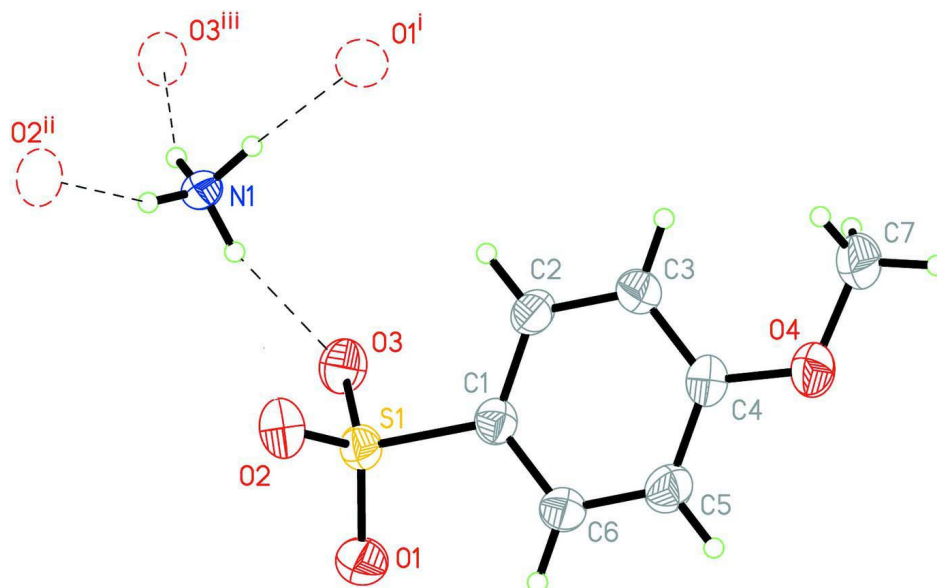
The title compound was obtained as a byproduct in the synthesis of *N*-hydroxy-4-methoxybenzenesulfonamide, following the procedure described in Porcheddu *et al.*, 2009. A few light yellow crystals were obtained after evaporating an acetonitrile solution.

**Refinement**

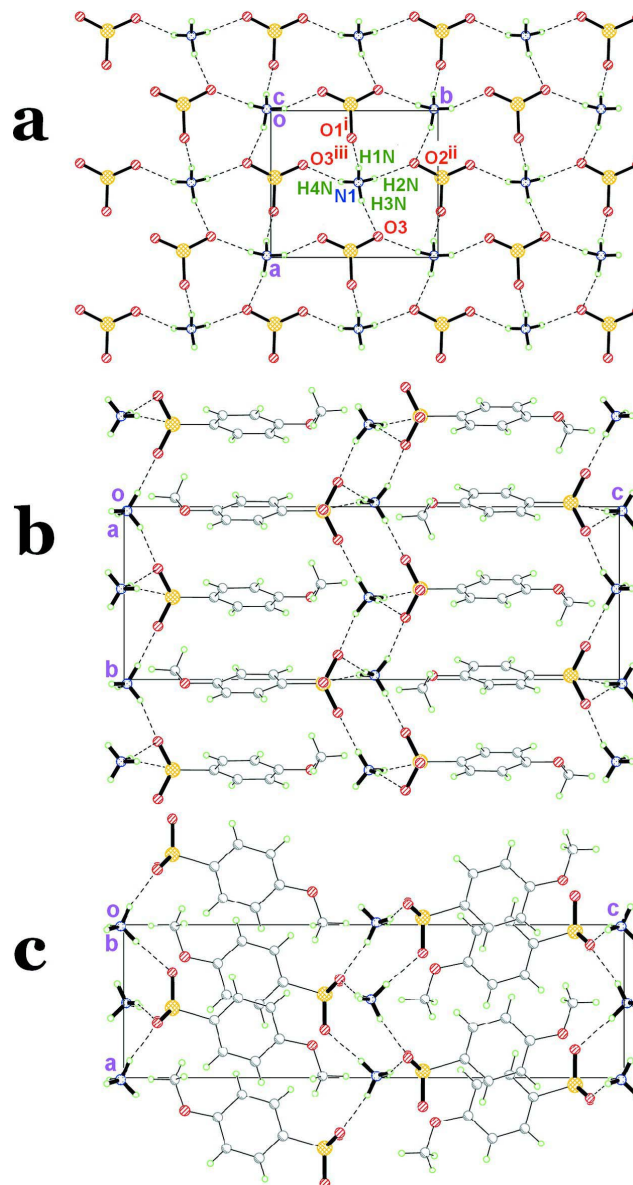
All H atoms were found in a difference map, though treated differently in refinement: C—H atoms were idealized and allowed to ride, with displacement parameters taken as  $U_{\text{iso}}(\text{H}) = X \times U_{\text{eq}}(\text{C})$  [ $(\text{C—H})_{\text{methyl}} = 0.96 \text{ \AA}^\circ$ ,  $X = 1.5$ ;  $(\text{C—H})_{\text{arom}} = 0.93 \text{ \AA}^\circ$ ,  $X = 1.2$ ] ( $\text{CH}_3$  groups were also free to rotate as well). Ammonium H's were refined with restrained N—H = 0.85 (1) Å, H $\cdots$ H = 1.35 (2) Å distances and free isotropic displacement factors.

**Computing details**

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); 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: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

**Figure 1**

Ellipsoid plot of (I), drawn with displacement factors at a 50% probability level. Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $x - 1/2, -y + 1/2, -z + 1$ ; (iii)  $x - 1/2, -y + 3/2, -z + 1$ .


**Figure 2**

Packing views of (I). a) Projection parallel to (001) showing the hydrophilic part only and the H-bonding interactions taking place therein. Symmetry codes: as in Fig 1. b) A packing view with the whole structure, projected down [100]. Hydrophilic/hydrophobic parts (seen in projection) drawn in heavy/weak lining, respectively. c) Same as b) viewed along [010].

### Ammonium 4-methoxybenzenesulfonate

#### Crystal data

 $\text{NH}_4^+ \cdot \text{C}_7\text{H}_7\text{O}_4\text{S}^-$ 
 $M_r = 205.23$ 

 Orthorhombic,  $P2_12_12_1$ 

Hall symbol: P 2ac 2ab

 $a = 6.2664 (12) \text{ \AA}$ 
 $b = 7.1342 (12) \text{ \AA}$ 
 $c = 20.410 (2) \text{ \AA}$ 
 $V = 912.4 (2) \text{ \AA}^3$ 
 $Z = 4$ 
 $F(000) = 432$ 
 $D_x = 1.494 \text{ Mg m}^{-3}$ 

 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2823 reflections  
 $\theta = 2.1\text{--}25.9^\circ$   
 $\mu = 0.34\text{ mm}^{-1}$

$T = 298\text{ K}$   
 Blocks, yellow  
 $0.20 \times 0.10 \times 0.10\text{ mm}$

*Data collection*

Oxford Diffraction Gemini CCD S Ultra diffractometer  
 Graphite monochromator  
 $\omega$  scans, thick slices  
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.958$ ,  $T_{\max} = 0.965$   
 4265 measured reflections

1732 independent reflections  
 1548 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$   
 $\theta_{\max} = 26.2^\circ$ ,  $\theta_{\min} = 2.0^\circ$   
 $h = -7 \rightarrow 6$   
 $k = -8 \rightarrow 8$   
 $l = -20 \rightarrow 25$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.119$   
 $S = 1.04$   
 1732 reflections  
 135 parameters  
 21 restraints  
 Primary atom site location: structure-invariant direct methods  
 Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites  
 H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0842P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.47\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.36\text{ e \AA}^{-3}$   
 Absolute structure: Flack (1983), 637 Friedel pairs  
 Flack parameter:  $-0.11(14)$

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.95549 (11)	0.47643 (10)	0.59811 (3)	0.0305 (2)
O1	1.1864 (3)	0.4832 (4)	0.59781 (11)	0.0483 (6)
O2	0.8694 (4)	0.3085 (3)	0.56840 (11)	0.0422 (6)
O3	0.8578 (4)	0.6413 (3)	0.56824 (11)	0.0379 (6)
O4	0.7122 (4)	0.4778 (4)	0.87726 (9)	0.0426 (6)
C1	0.8765 (4)	0.4753 (4)	0.68155 (13)	0.0295 (6)
C2	0.6702 (4)	0.5282 (5)	0.69849 (13)	0.0324 (6)
H2	0.5734	0.5616	0.6660	0.039*
C3	0.6087 (4)	0.5311 (5)	0.76355 (13)	0.0338 (6)
H3	0.4713	0.5677	0.7751	0.041*
C4	0.7550 (5)	0.4783 (4)	0.81175 (13)	0.0324 (6)
C5	0.9583 (6)	0.4232 (4)	0.79423 (15)	0.0375 (7)
H5	1.0544	0.3863	0.8265	0.045*
C6	1.0207 (5)	0.4223 (4)	0.72898 (14)	0.0335 (6)
H6	1.1583	0.3864	0.7174	0.040*
C7	0.5125 (6)	0.5513 (6)	0.89746 (15)	0.0498 (8)

H7A	0.5091	0.5598	0.9444	0.075*
H7B	0.4001	0.4700	0.8828	0.075*
H7C	0.4931	0.6737	0.8789	0.075*
N1	0.4879 (3)	0.5244 (3)	0.49420 (10)	0.0264 (5)
H1N	0.391 (3)	0.526 (4)	0.5251 (9)	0.034 (8)*
H2N	0.463 (5)	0.619 (3)	0.4678 (12)	0.068 (13)*
H3N	0.612 (3)	0.542 (5)	0.5125 (10)	0.039 (9)*
H4N	0.484 (5)	0.421 (3)	0.4729 (13)	0.069 (14)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0297 (3)	0.0358 (3)	0.0261 (3)	-0.0015 (3)	0.0015 (3)	-0.0013 (3)
O1	0.0310 (11)	0.0745 (17)	0.0394 (12)	0.0018 (12)	0.0032 (9)	-0.0021 (15)
O2	0.0533 (16)	0.0399 (12)	0.0336 (12)	-0.0030 (10)	0.0042 (12)	-0.0044 (10)
O3	0.0454 (13)	0.0370 (11)	0.0313 (12)	-0.0024 (10)	-0.0004 (11)	0.0042 (10)
O4	0.0491 (12)	0.0521 (13)	0.0265 (10)	0.0068 (12)	-0.0010 (9)	0.0003 (11)
C1	0.0305 (12)	0.0307 (13)	0.0273 (13)	-0.0035 (12)	0.0009 (11)	-0.0001 (12)
C2	0.0300 (13)	0.0393 (14)	0.0279 (13)	-0.0026 (13)	-0.0045 (11)	0.0008 (14)
C3	0.0286 (13)	0.0402 (15)	0.0327 (14)	0.0001 (12)	0.0023 (11)	-0.0037 (14)
C4	0.0390 (14)	0.0313 (13)	0.0268 (13)	-0.0036 (13)	-0.0006 (11)	-0.0015 (13)
C5	0.0416 (16)	0.0377 (15)	0.0331 (15)	0.0081 (14)	-0.0069 (14)	0.0024 (12)
C6	0.0331 (15)	0.0333 (13)	0.0342 (14)	0.0060 (12)	-0.0021 (12)	-0.0019 (11)
C7	0.0438 (17)	0.076 (2)	0.0292 (15)	-0.0005 (18)	0.0048 (14)	-0.0050 (17)
N1	0.0247 (10)	0.0303 (10)	0.0241 (10)	0.0038 (9)	-0.0026 (9)	0.0019 (10)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S1—O2	1.447 (2)	C4—C5	1.380 (4)
S1—O1	1.448 (2)	C5—C6	1.388 (4)
S1—O3	1.459 (2)	C5—H5	0.9300
S1—C1	1.773 (3)	C6—H6	0.9300
O4—C4	1.364 (3)	C7—H7A	0.9600
O4—C7	1.418 (4)	C7—H7B	0.9600
C1—C6	1.377 (4)	C7—H7C	0.9600
C1—C2	1.390 (4)	N1—H1N	0.876 (15)
C2—C3	1.383 (4)	N1—H2N	0.877 (16)
C2—H2	0.9300	N1—H3N	0.873 (16)
C3—C4	1.396 (4)	N1—H4N	0.858 (16)
C3—H3	0.9300		
O2—S1—O1	113.48 (17)	C4—C5—C6	120.7 (3)
O2—S1—O3	109.62 (13)	C4—C5—H5	119.7
O1—S1—O3	112.99 (16)	C6—C5—H5	119.7
O2—S1—C1	107.15 (14)	C1—C6—C5	119.2 (3)
O1—S1—C1	106.45 (13)	C1—C6—H6	120.4
O3—S1—C1	106.71 (14)	C5—C6—H6	120.4
C4—O4—C7	117.2 (2)	O4—C7—H7A	109.5
C6—C1—C2	120.6 (3)	O4—C7—H7B	109.5
C6—C1—S1	119.6 (2)	H7A—C7—H7B	109.5



C2—C1—S1	119.8 (2)	O4—C7—H7C	109.5
C3—C2—C1	120.1 (3)	H7A—C7—H7C	109.5
C3—C2—H2	119.9	H7B—C7—H7C	109.5
C1—C2—H2	119.9	H1N—N1—H2N	108 (3)
C2—C3—C4	119.3 (3)	H1N—N1—H3N	108 (3)
C2—C3—H3	120.4	H2N—N1—H3N	108 (3)
C4—C3—H3	120.4	H1N—N1—H4N	111 (3)
O4—C4—C5	115.8 (3)	H2N—N1—H4N	110 (3)
O4—C4—C3	124.2 (3)	H3N—N1—H4N	111 (3)
C5—C4—C3	120.0 (3)		

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1N...O1 <sup>i</sup>	0.88 (2)	1.99 (2)	2.851 (3)	170 (3)
N1—H4N...O2 <sup>ii</sup>	0.86 (2)	1.98 (2)	2.797 (3)	160 (3)
N1—H2N...O3 <sup>iii</sup>	0.88 (2)	1.98 (2)	2.824 (3)	162 (3)
N1—H3N...O3	0.87 (2)	2.04 (2)	2.890 (3)	164 (3)

Symmetry codes: (i)  $x-1, y, z$ ; (ii)  $x-1/2, -y+1/2, -z+1$ ; (iii)  $x-1/2, -y+3/2, -z+1$ .