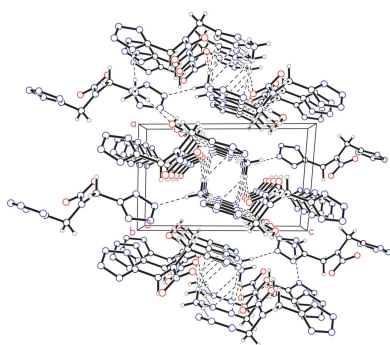# Crystal structures of the three closely related compounds: bis[(1*H*-tetrazol-5-yl)methyl]-nitramide, triaminoguanidinium 5-({[(1*H*-tetrazol-5-yl)methyl](nitro)amino}methyl)tetrazol-1-ide, and diammonium bis[(tetrazol-1-id-5-yl)methyl]-nitramide monohydrate

Lauren A. Mitchell,[a] Gregory H. Imler,[a] Damon A. Parrish,[a]* Jeffrey R. Deschamps,[a] Philip W. Leonard[b] and David E. Chavez[b]

[a]Center for Biomolecular Science and Engineering, Naval Research Laboratory, Washington, DC 20375, USA, and [b]Los Alamos National Laboratory, Los Alamos, NM 87545, USA. *Correspondence e-mail: damon.parrish@nrl.navy.mil
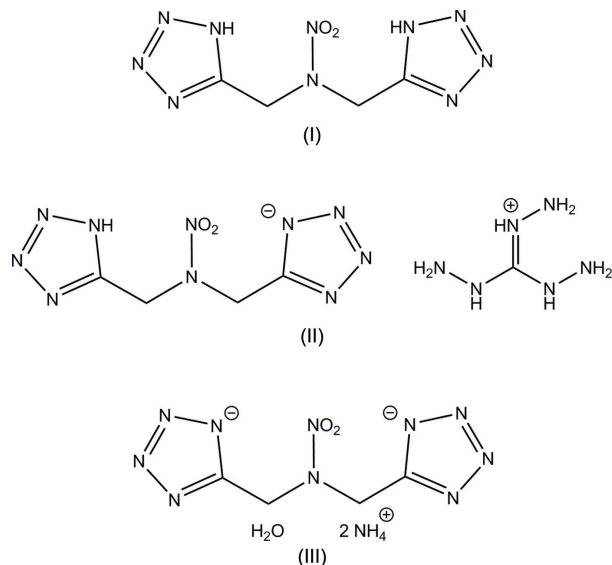
In the molecule of neutral bis[(1*H*-tetrazol-5-yl)methyl]nitramide, (I), $C_4H_6N_{10}O_2$, there are two intramolecular N—H···O hydrogen bonds. In the crystal, N—H···N hydrogen bonds link molecules, forming a two-dimensional network parallel to ($\bar{2}$01) and weak C—H···O, C—H···N hydrogen bonds, and intermolecular π–π stacking completes the three-dimensional network. The anion in the molecular salt, triaminoguanidinium 5-({[(1*H*-tetrazol-5-yl)meth-yl](nitro)amino}methyl)tetrazol-1-ide, (II), $CH_9N_6^+\cdot C_4H_5N_{10}O_2^-$, displays intra-molecular π–π stacking and in the crystal, N—H···N and N—H···O hydrogen bonds link the components of the structure, forming a three-dimensional network. In the crystal of diammonium bis[(tetrazol-1-id-5-yl)methyl]nitramide monohydrate, (III), $2NH_4^+\cdot C_4H_4N_{10}O_2^{2-}\cdot H_2O$, O—H···N, N—H···N, and N—H···O hydrogen bonds link the components of the structure into a three-dimensional network. In addition, there is intermolecular π–π stacking. In all three structures, the central N atom of the nitramide is mainly sp^2-hybridized. Bond lengths indicate delocalization of charges on the tetrazole rings for all three compounds. Compound (II) was found to be a non-merohedral twin and was solved and refined in the major component.

1. Chemical context

Materials which release large amount of energy during chemical transformations are characterized as energetic materials. Interest is high in improving energetics to reduce environmental impact and to improve safety and performance (Talawar *et al.*, 2009). These materials can pose a hazard if they have high sensitivities to friction, heat, electrostatic discharge or impact. Compounds containing both tetrazole and nitro groups have frequently been used in the development of energetic materials (Klapötke *et al.*, 2009; Wei *et al.*, 2015). Tetrazoles have been of special interest because of their high nitrogen content, which lead to high heats of formation and to more environmentally benign decomposition products like N_2 (Jaidann *et al.*, 2010). Nitro groups have been commonly utilized to achieve an optimum oxygen balance (Wu *et al.*, 2014). Herein is a discussion of the X-ray crystal structures of three nitro-containing tetrazole complexes. Structure (I), bis[(1*H*-tetrazol-5-yl)methyl]nitramide, is the neutral form,

structure (II), triaminoguanidinium 5-({[(1*H*-tetrazol-5-yl)methyl](nitro)amino}methyl)tetrazol-1-ide, has one deprotonated tetrazole ring with a triaminoguanididinium counterion, and structure (III), diammonium bis[(tetrazol-1-id-5-yl)methyl]nitramide monohydrate, has both tetrazole rings deprotonated with ammonium counter-ions.

(I)

(II)

(III)

2. Structural commentary

In the molecule of complex (I), two intramolecular hydrogen bonds, N4—H4···O15 and N10—H10···O16, both between tetrazole donors and nitro acceptors are present (Fig. 1). This
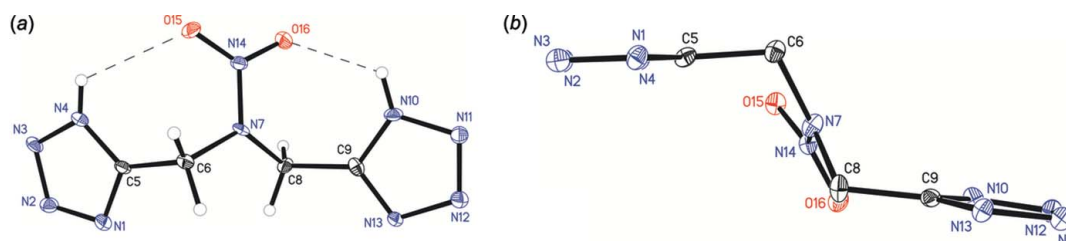

Figure 3
The molecular structure of structure (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. (*a*) Front view. (*b*) Side view, H atoms and cation omitted for clarity.

molecule adopts a chair-like conformation where the tetrazole rings are *trans* to one another. Molecule (III) adopts a similar conformation, despite not having any similar intramolecular hydrogen bonds (Fig. 2). Surprisingly, while structures (I) and (III) are both in a chair conformation, with respect to the tetrazole rings, structure (II) is bent into a boat where the tetrazole rings are *cis* to one another (Fig. 3).

This unusual conformation is likely due to the intramolecular π–π stacking interactions observed between the tetrazole rings [centroid–centroid distance = 3.4978 (10) Å]. Both tetrazole rings are nearly planar with an r.m.s. deviation of 0.0007 for the protonated ring and 0.00004 Å for the deprotonated ring.

Figure 1
The molecular structure of structure (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. (*a*) Front view, dashed lines indicate intramolecular hydrogen bonds. (*b*) Side view, H atoms omitted for clarity.
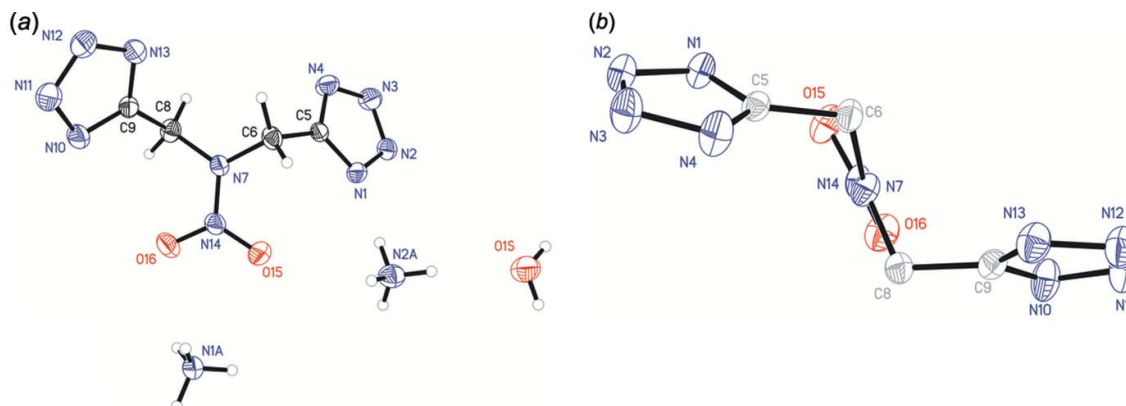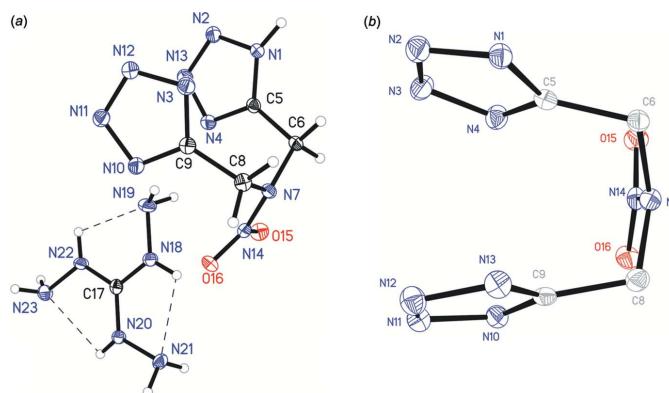

Figure 2
The molecular structure of structure (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. (*a*) Front view. (*b*) Side view, H atoms, cations, and solvent omitted for clarity.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4···N2^i	0.80 (6)	2.19 (6)	2.957 (5)	160 (4)
N4—H4···O15	0.80 (6)	2.45 (5)	2.924 (5)	119 (4)
C6—H6B···O15^{ii}	0.99	2.37	3.264 (5)	150
C8—H8B···N11^{iii}	0.99	2.44	3.316 (5)	147
N10—H10···N13^{iv}	0.87 (1)	1.99 (3)	2.770 (5)	149 (4)
N10—H10···O16	0.87 (1)	2.28 (4)	2.796 (4)	118 (4)

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1···N10^i	0.929 (19)	1.804 (19)	2.713 (2)	165.6 (17)
N1—H1···N11^i	0.929 (19)	2.673 (19)	3.422 (2)	138.2 (14)
N1—H1···O16^i	0.929 (19)	2.596 (18)	2.9952 (18)	106.5 (13)
N18—H18···O15	0.84 (2)	2.569 (19)	3.1451 (18)	126.4 (16)
N18—H18···N21	0.84 (2)	2.292 (19)	2.650 (2)	105.9 (15)
N19—H19A···N4	0.92 (2)	2.29 (2)	3.026 (2)	137.3 (17)
N19—H19B···N13^{ii}	0.91 (2)	2.54 (2)	3.275 (2)	138.5 (16)
N20—H20···N13^{iii}	0.86 (2)	2.09 (2)	2.867 (2)	149.0 (17)
N20—H20···N23	0.86 (2)	2.358 (18)	2.660 (2)	100.9 (14)
N21—H21A···N11^{ii}	0.89 (2)	2.46 (2)	3.143 (2)	134.4 (16)
N21—H21B···O15^{iv}	0.89 (2)	2.31 (2)	3.090 (2)	146.3 (18)
N22—H22···N2^v	0.86 (2)	2.40 (2)	3.118 (2)	142.3 (17)
N22—H22···N19	0.86 (2)	2.325 (19)	2.650 (2)	102.9 (15)
N23—H23A···N11^{vi}	0.89 (2)	2.22 (2)	3.087 (2)	166.5 (18)
N23—H23B···N3^{vi}	0.92 (2)	2.38 (2)	3.091 (2)	133.9 (17)

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

For all three compounds, the C—N (ranging from 1.321 to 1.338 Å) and N—N (ranging from 1.301 to 1.362 Å) bond lengths for the tetrazole rings were found to match more closely with bonds of multiple character than of discrete single and double bonds, signifying a delocalized aromatic system (Allen *et al.*, 1987).

In structure (II), the N18—C17, N20—C17, and N22—C17 bond lengths for the triaminoguandidinium cation were all found to be relatively equal (maximum difference 0.006 Å), indicating delocalization of the charge over all three branches.

The pyramidality of the amine functionality for the central tertiary amine was examined for all three structures by examining χ_n, the angle between the $N_{amine}-N_{nitro}$ vector and the $C_{methylene1}/N_{amine}/C_{methylene2}$ plane, described by Allen *et al.* (1995). Structure (I) has a χ_n of 13.1 (5)° for vector N2–N1 and plane C11/C5/N1, structure (II) has a χ_n of 26.11 (18)° for vector N14–N7 and plane C6/N7/C8, and structure (III) has a χ_n of 6.21 (11)° for vector N7A–N7 and plane C6/N7/C8. This indicated the hybridization of the central nitrogen atom is mainly sp^2 hybridized for all three structures (sp^2 $\chi_n \simeq 0°$, sp^3 $\chi_n \simeq 60°$).

3. Supramolecular features

The packing and intermolecular hydrogen bonding vary greatly between the three structures. Structure (I) has four intermolecular hydrogen bonds (Table 1). The tetrazole rings of adjacent molecules are linked *via* N—H···N bonds, forming a two-dimensional network parallel to ($\overline{2}$01). These interactions cause the tetrazole rings to lie in the same plane, resulting in the alignment of the tetrazole rings seen when viewed along the *b* axis (Fig. 4). Additionally, there is one weak C—H···N and one weak C—H···O hydrogen bond linking the molecules into a three-dimensional network.

Structure (II) does not have any non-classical intermolecular hydrogen bonds (Table 2). There are twelve N—H···N bonds and three N—H···O bonds, with the majority of

Figure 4
Packing diagram for structure (I) viewed along the *b* axis. Dashed lines indicate intermolecular hydrogen bonds.
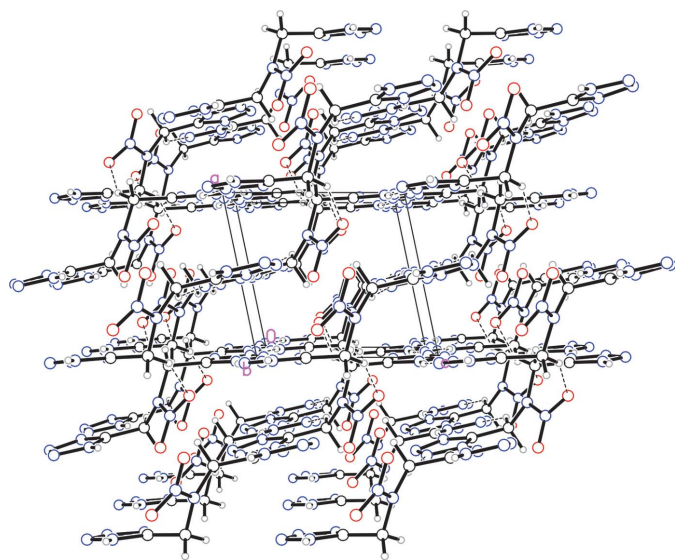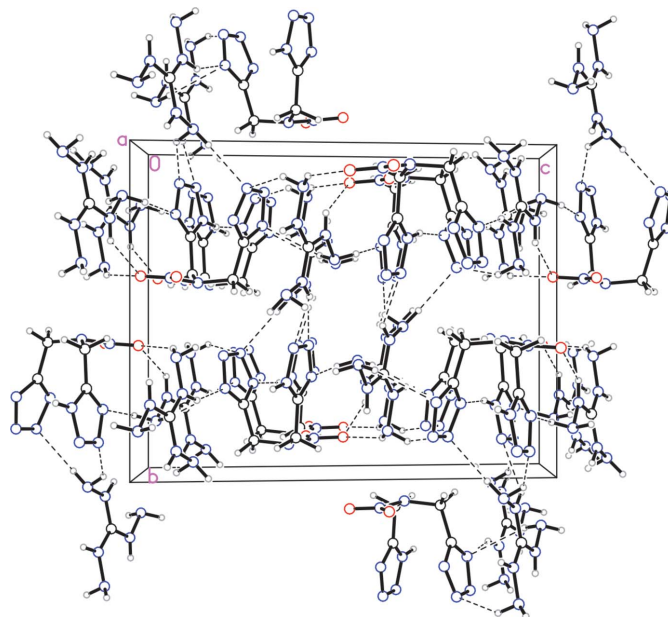

Figure 5
Packing diagram for structure (II) viewed along the *a* axis. Dashed lined indicate intermolecular hydrogen bonds.

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1S—H1SA$\cdots$N13[i]	0.88 (2)	2.06 (2)	2.9253 (12)	168.0 (18)
O1S—H1SB$\cdots$N3[ii]	0.83 (2)	2.31 (2)	2.9498 (13)	134.8 (17)
N1A—H1A$\cdots$N12[iii]	0.859 (16)	2.211 (16)	3.0533 (13)	166.7 (14)
N1A—H1B$\cdots$O16	0.847 (16)	2.388 (16)	3.0079 (13)	130.5 (13)
N1A—H1B$\cdots$N13[iv]	0.847 (16)	2.540 (15)	3.2862 (14)	147.6 (13)
N1A—H1B$\cdots$N12[iv]	0.847 (16)	2.585 (15)	3.2472 (14)	136.0 (13)
N2A—H2A$\cdots$O1S	0.880 (16)	2.030 (16)	2.9062 (14)	173.2 (14)
N2A—H2B$\cdots$N1[v]	0.854 (16)	2.179 (16)	3.0243 (13)	170.3 (14)
N1A—H1C$\cdots$N2[v]	0.882 (16)	2.107 (16)	2.9654 (12)	164.2 (14)
N2A—H2C$\cdots$O1S[vi]	0.849 (17)	2.147 (17)	2.9766 (13)	165.2 (14)
N2A—H2D$\cdots$N1	0.896 (16)	2.117 (16)	3.0096 (13)	174.0 (13)
N1A—H1D$\cdots$N10[vii]	0.906 (16)	2.045 (16)	2.9273 (13)	164.2 (13)

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $x-1, y+1, z$; (iii) $-x+1, -y, -z+1$; (iv) $x, y+1, z$; (v) $-x+1, -y+1, -z$; (vi) $-x, -y+1, -z$; (vii) $-x+1, -y+1, -z+1$.

the interactions between the main residue and the triamino-guandidinium counter-ion. The additional hydrogen bonds link the molecules into a three-dimensional network. The compound packs into columns of alternating anions and cations along the c axis (Fig. 5).

Structure (III) contains several intermolecular hydrogen bonds, which also form a three-dimensional network (Table 3). There are seven N—H$\cdots$N bonds between ammonium donors and tetrazole acceptors, two O—H$\cdots$N bonds between water donors and tetrazole acceptors, two N—H$\cdots$O bonds between ammonium donors and water acceptors, and one N—H$\cdots$O bond between an ammonium donor and a nitro acceptor. The ions and molecules pack into columns along the b axis (Fig. 6).

Although compounds (I) and (III) do not exhibit any intramolecular π–π stacking, intermolecular π–π stacking is present between tetrazole rings of adjacent molecules. Compound (I) displays head-to-tail stacking interactions with
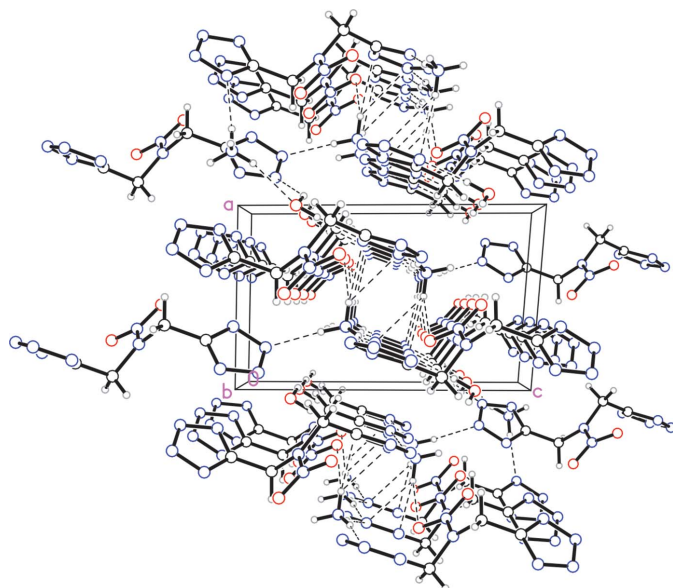
a centroid–centroid distance of 3.627 (2) Å. Compound (II) displays head-to-head and tail-to-tail stacking with a centroid–centroid distance of 3.8472 (10) Å for plane N1/N2/N3/N4/C5 to N1/N2/N3/N4/C5 and 4.0025 (8) Å for plane C9/N10/N11/N12/N13 to C9/N10/N11/N12/N13. There is no intermolecular π–π stacking for compound (II), which contains the larger counter-ion, triaminoguandidinium.

The neutral complex, compound (I), has a density of 1.825 g cm^{-3} (173 K). This is similar to the density, determined by X-ray crystallography, of the well known energetics RDX (α-hexahydro-1,3,5-trinitro-1,3,5-triazine) and HMX (1,3,5,7-tetra-nitro-1,3,5,7-tetraazacyclooctane) at 1.794 g cm^{-3} (298 K) and 1.948 g cm^{-3} (120 K) respectively (Zhurov *et al.*, 2011). The ionic compounds have much lower densities. The density of compound (II) is 1.611 g cm^{-3} (293 K), and the density of compound (III) is 1.579 g cm^{-3} (296 K).

4. Database survey

A search of the Cambridge Structural Database (version 5.36, last updated May 2015; Groom *et al.*, 2016) found 392 complexes that contained both tetrazole and nitro groups. The most similar compounds were 5-nitro-2H-tetrazole (Klapötke *et al.*, 2009), ammonium 5-nitrotetrazolate (Klapötke *et al.*, 2008), and triaminoguanidinium 5-nitrotetrazolate (Klapötke *et al.*, 2008). A search for triaminoguandidinium containing compounds found 47 hits. The compounds from the CSD had similar bond lengths and angles to the triaminoguandidinium cation in complex (II). The average difference in C—N bond lengths for the triaminoguandidinium complexes in the CSD was found to be 0.015 Å, indicating a high level of charge delocalization, similar to that seen in complex (II).

5. Synthesis and crystallization

Compound (I):

A 100 ml round-bottom flask was charged with *N,N*-bis(cyanomethyl)nitramide (2.5 g, 18 mmol), zinc bromide (3.9 g, 17 mmol), 30 ml water, and a magnetic stirbar. The reaction was heated to 323 K with stirring. Sodium azide (2.5 g, 38 mmol) was dissolved in 30 ml water and added to the heated reaction. A reflux condenser was fitted to the flask and the reaction was heated to 363 K for 1 h causing a gradual color change to light brown and the formation of a precipitate. The reaction was allowed to cool to room temperature, then 37% HCl (5 ml) was added and the mixture was allowed to stir for 30 min. The product was collected by vacuum filtration using a Buchner funnel and recrystallized from hot water. Yield 95%, 4 g. Melting point 475–477 K (dec.). CHN: Expected: C, 21.24; H, 2.67; N, 61.93. Found: C, 21.82(0.08); H, 2.96(0.08); N, 62.20(0.30). ^{1}H NMR (DMSO-d_6): 4.15 (2, *s*), 5.49 (4, *s*) ppm. ^{13}C NMR (DMSO-d_6): 40.33, 152.74 ppm. IR: 637, 685, 765, 875, 933, 1042, 1088, 1111, 1246, 1284, 1408, 1481, 1524, 1557, 2864, 3022 cm^{-1}.

Compound (II):

A 50 ml round-bottom flask was charged with a stir bar, barium hydroxide octahydrate (3.2 g, 10 mmol) and 20 mmol

Figure 6
Packing diagram for structure (III) viewed along the b axis. Dashed lined indicate intermolecular hydrogen bonds.

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Mitchell *et al.* $\cdot$ C$_4$H$_6$N$_{10}$O$_2$, CH$_9$N$_6^+ \cdot$C$_4$H$_5$N$_{10}$O$_2^-$ and 2NH$_4^+ \cdot$C$_4$H$_4$N$_{10}$O$_2^{2-} \cdot$H$_2$O   **1059**

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_4H_6N_{10}O_2$	$CH_9N_6^+ \cdot C_4H_5N_{10}O_2^-$	$2NH_4^+ \cdot C_4H_4N_{10}O_2^{2-} \cdot H_2O$
M_r	226.19	330.32	278.27
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1/c$	Triclinic, $P\bar{1}$
Temperature (K)	173	100	296
a, b, c (Å)	6.3640 (17), 9.627 (3), 6.8627 (18)	6.5312 (11), 12.682 (2), 16.183 (3)	7.5893 (11), 7.6077 (11), 11.2319 (15)
α, β, γ (°)	90, 101.805 (4), 90	90, 97.118 (3), 90	85.564 (4), 85.555 (4), 65.007 (4)
V (Å^3)	411.57 (19)	1330.0 (4)	585.29 (14)
Z	2	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.15	0.13	0.13
Crystal size (mm)	$0.36 \times 0.32 \times 0.01$	$0.52 \times 0.06 \times 0.02$	$0.75 \times 0.63 \times 0.24$
Data collection			
Diffractometer	Bruker SMART APEXII CCD	Bruker SMART APEXII CCD	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (TWINABS; Bruker, 2008)	Multi-scan (SADABS; Bruker, 2008)	Multi-scan (SADABS; Bruker, 2008)
T_{min}, T_{max}	0.615, 0.745	0.674, 0.745	0.687, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	889, 889, 835	11821, 2733, 2141	38379, 3178, 3000
R_{int}	0.038	0.037	0.057
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.625	0.628	0.688
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.102, 1.14	0.037, 0.093, 1.00	0.036, 0.106, 1.12
No. of reflections	889	2733	3178
No. of parameters	151	238	202
No. of restraints	2	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.25, −0.32	0.23, −0.25	0.29, −0.27

Computer programs: APEX2, SAINT and XPREP (Bruker, 2008), SHELXTL (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015) within WinGX (Farrugia, 2012) and publCIF (Westrip, 2010).

water. The base was stirred until fully dissolved. Compound (I) (4.5 g, 20 mmol) was added to the basic solution, dissolved, and the mixture was stirred 30 min as the color darkened to brown. The brown mixture was filtered to remove insoluble material, the filtrate was returned to the 50 ml round-bottom flask and stirred. Triaminoguanidinium sulfate (3.06 g, 10 mmol) was added to the stirring solution, causing immediate precipitation of barium sulfate. The mixture was stirred for 30 min and then allowed to stand for 10 min. Barium sulfate was removed by Buchner filtration and the filtrate was rotovapped until a precipitate formed. After isolating the product by filtration, it was recrystallized from water/ethanol solution. Yield 34%, 1.35 g. Melting point 428–430 K (dec.). ^{1}H NMR (DMSO-d_6): 4.65 (8, s), 5.20 (4, s), 8.6 (1, s) ppm. ^{13}C NMR (DMSO-d_6): 46.95, 157.60, 159.64 ppm. IR: 637, 685, 765, 875, 933, 1042, 1088, 1111, 1246, 1284, 1408, 1481, 1524, 1557, 2864, 3022 cm^{-1}.

Compound (III):

A 50 ml round-bottom flask was charged with (I) (2.5 g, 11 mmol), 10 ml water, and a magnetic stir bar and then stirred. An ammonium hydroxide solution (30%, 3 ml) was added to the reaction. After stirring for 1 h at 298 K, 10 ml ethanol was added and the resulting precipitate was collected by Buchner filtration. The product was recrystallized from water/methanol solution. Yield 80%, 2.3 g. Melting point 389–393 K (dec.). ^{1}H NMR (DMSO-d_6): 5.13 (4, s), 3.70 (broad) ppm. ^{13}C NMR (DMSO-d_6): 40.05; 155.80 ppm. IR: 2908; 2149; 1869; 1844; 1717; 1700; 1684; 1676; 1653; 1636; 1617; 1540; 1521; 1456; 1419; 1364; 1270; 1209; 1159; 1140; 1113; 1076; 920; 877; 809; 706; 612 cm^{-1}.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The methylene H atoms were positioned geometrically and refined using a riding model, with C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C). All other H atoms were located in a difference Fourier map using. Compound (II) was found to be a non-merohedral twin and was solved and refined in the major component. The N10—H10 bond length in structure (I) was restrained.

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supporting information

Crystal structures of the three closely related compounds: bis[(1*H*-tetrazol-5-yl)methyl]nitramide, triaminoguanidinium 5-({[(1*H*-tetrazol-5-yl)methyl](nitro)-amino}methyl)tetrazol-1-ide, and diammonium bis[(tetrazol-1-id-5-yl)methyl]-nitramide monohydrate

Lauren A. Mitchell, Gregory H. Imler, Damon A. Parrish, Jeffrey R. Deschamps, Philip W. Leonard and David E. Chavez

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2008); cell refinement: *APEX2* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008) and *XPREP* (Bruker, 2008); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) within *WinGX* (Farrugia, 2012); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I) Bis[(1*H*-tetrazol-5-yl)methyl]nitramide

Crystal data

$C_4H_6N_{10}O_2$	$F(000) = 232$
$M_r = 226.19$	$D_x = 1.825$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.3640$ (17) Å	Cell parameters from 2717 reflections
$b = 9.627$ (3) Å	$\theta = 3.0–26.2°$
$c = 6.8627$ (18) Å	$\mu = 0.15$ mm^{-1}
$\beta = 101.805$ (4)°	$T = 173$ K
$V = 411.57$ (19) Å^3	Thin plate, colorless
$Z = 2$	$0.36 \times 0.32 \times 0.01$ mm

Data collection

Bruker SMART APEXII CCD diffractometer	889 measured reflections
	889 independent reflections
Radiation source: fine focus sealed tube	835 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.038$
ω scans	$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
Absorption correction: multi-scan (TWINABS; Bruker, 2008)	$h = -7 \rightarrow 7$
	$k = 0 \rightarrow 12$
$T_{min} = 0.615$, $T_{max} = 0.745$	$l = 0 \rightarrow 8$

Refinement

Refinement on F^2	$S = 1.14$
Least-squares matrix: full	889 reflections
$R[F^2 > 2\sigma(F^2)] = 0.036$	151 parameters
$wR(F^2) = 0.102$	2 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent
  and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.4169P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.0174 (5)	0.3550 (3)	0.2473 (5)	0.0132 (7)
N2	−0.0013 (5)	0.3439 (4)	0.0467 (5)	0.0133 (7)
N3	0.0000 (5)	0.4659 (4)	−0.0356 (5)	0.0124 (7)
N4	0.0208 (5)	0.5581 (4)	0.1139 (5)	0.0117 (7)
H4	0.028 (7)	0.640 (7)	0.099 (6)	0.014*
C5	0.0317 (6)	0.4901 (4)	0.2851 (5)	0.0108 (7)
C6	0.0466 (6)	0.5533 (4)	0.4880 (5)	0.0125 (7)
H6A	−0.0542	0.6326	0.4769	0.015*
H6B	0.0009	0.4834	0.5768	0.015*
N7	0.2620 (5)	0.6017 (3)	0.5783 (5)	0.0122 (7)
C8	0.4251 (6)	0.5139 (4)	0.6986 (5)	0.0129 (8)
H8A	0.5573	0.5178	0.6438	0.015*
H8B	0.3738	0.4165	0.6889	0.015*
C9	0.4788 (5)	0.5559 (5)	0.9148 (5)	0.0113 (7)
N10	0.5184 (5)	0.6848 (3)	0.9857 (5)	0.0118 (7)
H10	0.510 (7)	0.764 (3)	0.924 (6)	0.014*
N11	0.5665 (5)	0.6792 (3)	1.1860 (5)	0.0128 (7)
N12	0.5574 (5)	0.5489 (4)	1.2341 (4)	0.0127 (7)
N13	0.5023 (5)	0.4701 (4)	1.0666 (4)	0.0110 (7)
N14	0.3273 (5)	0.7226 (3)	0.5122 (4)	0.0112 (7)
O15	0.2034 (4)	0.7844 (3)	0.3794 (4)	0.0142 (6)
O16	0.5086 (4)	0.7636 (3)	0.5919 (4)	0.0154 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0148 (14)	0.0102 (17)	0.0139 (15)	−0.0015 (13)	0.0011 (12)	−0.0015 (13)
N2	0.0141 (15)	0.0093 (18)	0.0164 (17)	0.0004 (13)	0.0029 (12)	0.0010 (13)
N3	0.0141 (14)	0.0082 (17)	0.0144 (16)	−0.0017 (13)	0.0017 (12)	−0.0040 (13)
N4	0.0139 (15)	0.0074 (16)	0.0135 (15)	−0.0021 (13)	0.0021 (11)	−0.0019 (14)
C5	0.0104 (16)	0.0071 (18)	0.0139 (17)	−0.0023 (14)	0.0003 (13)	0.0004 (15)
C6	0.0139 (18)	0.0113 (18)	0.0122 (16)	−0.0044 (16)	0.0020 (13)	0.0004 (16)
N7	0.0152 (16)	0.0061 (15)	0.0142 (15)	−0.0045 (13)	0.0003 (12)	−0.0008 (13)
C8	0.022 (2)	0.0062 (18)	0.0093 (16)	0.0041 (14)	0.0013 (14)	0.0004 (13)

C9	0.0091 (16)	0.0115 (18)	0.0130 (17)	0.0008 (15)	0.0015 (13)	−0.0036 (16)
N10	0.0171 (16)	0.0060 (17)	0.0131 (15)	0.0011 (13)	0.0049 (12)	0.0014 (13)
N11	0.0155 (16)	0.0101 (17)	0.0122 (14)	0.0020 (13)	0.0013 (11)	0.0001 (13)
N12	0.0136 (15)	0.0112 (15)	0.0131 (15)	−0.0003 (14)	0.0019 (11)	0.0003 (15)
N13	0.0131 (15)	0.0079 (18)	0.0112 (15)	−0.0010 (13)	0.0007 (11)	−0.0005 (13)
N14	0.0147 (15)	0.0078 (15)	0.0115 (14)	−0.0016 (12)	0.0037 (12)	−0.0003 (12)
O15	0.0182 (13)	0.0099 (13)	0.0136 (12)	0.0031 (12)	0.0012 (10)	0.0029 (11)
O16	0.0165 (13)	0.0148 (14)	0.0144 (12)	−0.0052 (10)	0.0021 (10)	−0.0007 (11)

Geometric parameters (Å, º)

N1—C5	1.326 (5)	C8—C9	1.508 (5)
N1—N2	1.361 (4)	C8—H8A	0.9900
N2—N3	1.304 (5)	C8—H8B	0.9900
N3—N4	1.343 (5)	C9—N13	1.314 (5)
N4—C5	1.334 (5)	C9—N10	1.338 (5)
N4—H4	0.80 (6)	N10—N11	1.347 (4)
C5—C6	1.505 (5)	N10—H10	0.867 (11)
C6—N7	1.460 (5)	N11—N12	1.301 (5)
C6—H6A	0.9900	N12—N13	1.362 (5)
C6—H6B	0.9900	N14—O15	1.230 (4)
N7—N14	1.346 (4)	N14—O16	1.236 (4)
N7—C8	1.457 (5)		
C5—N1—N2	105.2 (3)	N7—C8—C9	113.2 (3)
N3—N2—N1	111.1 (3)	N7—C8—H8A	108.9
N2—N3—N4	105.8 (3)	C9—C8—H8A	108.9
C5—N4—N3	109.1 (4)	N7—C8—H8B	108.9
C5—N4—H4	127 (3)	C9—C8—H8B	108.9
N3—N4—H4	124 (3)	H8A—C8—H8B	107.8
N1—C5—N4	108.7 (4)	N13—C9—N10	108.2 (3)
N1—C5—C6	124.5 (3)	N13—C9—C8	125.3 (4)
N4—C5—C6	126.7 (3)	N10—C9—C8	126.5 (4)
N7—C6—C5	113.4 (3)	C9—N10—N11	108.7 (3)
N7—C6—H6A	108.9	C9—N10—H10	130 (3)
C5—C6—H6A	108.9	N11—N10—H10	121 (3)
N7—C6—H6B	108.9	N12—N11—N10	106.6 (3)
C5—C6—H6B	108.9	N11—N12—N13	109.9 (3)
H6A—C6—H6B	107.7	C9—N13—N12	106.7 (3)
N14—N7—C8	117.4 (3)	O15—N14—O16	124.9 (3)
N14—N7—C6	117.4 (3)	O15—N14—N7	118.2 (3)
C8—N7—C6	123.6 (3)	O16—N14—N7	116.9 (3)
C5—N1—N2—N3	−0.4 (4)	N7—C8—C9—N13	135.9 (4)
N1—N2—N3—N4	0.3 (4)	N7—C8—C9—N10	−46.8 (5)
N2—N3—N4—C5	0.0 (4)	N13—C9—N10—N11	−0.4 (4)
N2—N1—C5—N4	0.3 (4)	C8—C9—N10—N11	−178.1 (3)
N2—N1—C5—C6	177.6 (3)	C9—N10—N11—N12	0.5 (4)

N3—N4—C5—N1	−0.2 (4)	N10—N11—N12—N13	−0.4 (4)
N3—N4—C5—C6	−177.4 (3)	N10—C9—N13—N12	0.1 (4)
N1—C5—C6—N7	104.7 (4)	C8—C9—N13—N12	177.8 (3)
N4—C5—C6—N7	−78.6 (5)	N11—N12—N13—C9	0.2 (4)
C5—C6—N7—N14	77.3 (4)	C8—N7—N14—O15	165.5 (3)
C5—C6—N7—C8	−87.9 (4)	C6—N7—N14—O15	−0.6 (5)
N14—N7—C8—C9	82.6 (4)	C8—N7—N14—O16	−15.0 (4)
C6—N7—C8—C9	−112.2 (4)	C6—N7—N14—O16	178.9 (3)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N4—H4⋯N2[i]	0.80 (6)	2.19 (6)	2.957 (5)	160 (4)
N4—H4⋯O15	0.80 (6)	2.45 (5)	2.924 (5)	119 (4)
C6—H6B⋯O15[ii]	0.99	2.37	3.264 (5)	150
C8—H8B⋯N11[iii]	0.99	2.44	3.316 (5)	147
N10—H10⋯N13[iv]	0.87 (1)	1.99 (3)	2.770 (5)	149 (4)
N10—H10⋯O16	0.87 (1)	2.28 (4)	2.796 (4)	118 (4)

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

(II) Triaminoguanidinium 5-({[(1*H*-tetrazol-5-yl)methyl](nitro)amino}methyl)tetrazol-1-ide

Crystal data

$CH_9N_6{}^+ \cdot C_4H_5N_{10}O_2{}^-$	$F(000) = 688$
$M_r = 330.32$	$D_x = 1.650$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.5312$ (11) Å	Cell parameters from 2664 reflections
$b = 12.682$ (2) Å	$\theta = 3.0–25.8°$
$c = 16.183$ (3) Å	$\mu = 0.13$ mm^{-1}
$\beta = 97.118$ (3)°	$T = 100$ K
$V = 1330.0$ (4) Å^3	Thin plate, colorless
$Z = 4$	$0.52 \times 0.06 \times 0.02$ mm

Data collection

Bruker SMART APEXII CCD diffractometer	11821 measured reflections
Radiation source: fine focus sealed tube	2733 independent reflections
Graphite monochromator	2141 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (SADABS; Bruker, 2008)	$\theta_{max} = 26.5°$, $\theta_{min} = 2.1°$
$T_{min} = 0.674$, $T_{max} = 0.745$	$h = -8 \rightarrow 8$
	$k = -15 \rightarrow 15$
	$l = -20 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.037$	
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.6423P]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
2733 reflections	$(\Delta/\sigma)_{max} < 0.001$
238 parameters	$\Delta\rho_{max} = 0.23$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.25$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.8295 (2)	0.71404 (10)	0.35827 (9)	0.0141 (3)
H1	0.937 (3)	0.7311 (14)	0.3283 (12)	0.017*
N2	0.8025 (2)	0.61115 (11)	0.37468 (9)	0.0181 (3)
N3	0.6329 (2)	0.60429 (11)	0.40878 (9)	0.0178 (3)
N4	0.5480 (2)	0.70155 (10)	0.41515 (9)	0.0162 (3)
C5	0.6732 (2)	0.76797 (12)	0.38346 (10)	0.0128 (3)
C6	0.6426 (2)	0.88578 (12)	0.37832 (11)	0.0142 (3)
H6A	0.697088	0.918049	0.432299	0.017*
H6B	0.721587	0.914793	0.335095	0.017*
N7	0.42480 (19)	0.91361 (10)	0.35817 (9)	0.0132 (3)
C8	0.3282 (2)	0.90418 (12)	0.27155 (10)	0.0151 (3)
H8A	0.416201	0.939648	0.234379	0.018*
H8B	0.192815	0.940317	0.265472	0.018*
C9	0.2981 (2)	0.79121 (12)	0.24561 (10)	0.0128 (3)
N10	0.1481 (2)	0.72878 (10)	0.26607 (9)	0.0152 (3)
N11	0.1891 (2)	0.63274 (11)	0.23517 (9)	0.0164 (3)
N12	0.3573 (2)	0.63861 (11)	0.19785 (9)	0.0176 (3)
N13	0.4297 (2)	0.73864 (11)	0.20361 (9)	0.0165 (3)
N14	0.3000 (2)	0.89963 (10)	0.41915 (9)	0.0140 (3)
O15	0.38074 (18)	0.89379 (9)	0.49195 (7)	0.0185 (3)
O16	0.11190 (17)	0.89812 (9)	0.39797 (8)	0.0189 (3)
C17	−0.0806 (2)	0.68873 (12)	0.58804 (10)	0.0133 (3)
N18	0.0711 (2)	0.72677 (11)	0.54903 (9)	0.0170 (3)
H18	0.086 (3)	0.7929 (16)	0.5481 (12)	0.020*
N19	0.2228 (2)	0.65585 (12)	0.52819 (11)	0.0209 (3)
H19A	0.255 (3)	0.6740 (16)	0.4767 (14)	0.027*
H19B	0.337 (3)	0.6698 (16)	0.5647 (14)	0.027*
N20	−0.2213 (2)	0.75296 (10)	0.61240 (9)	0.0145 (3)
H20	−0.311 (3)	0.7310 (14)	0.6435 (12)	0.017*
N21	−0.1977 (2)	0.86203 (11)	0.59874 (10)	0.0181 (3)
H21A	−0.139 (3)	0.8920 (16)	0.6454 (14)	0.024*
H21B	−0.324 (3)	0.8875 (15)	0.5871 (13)	0.024*
N22	−0.0899 (2)	0.58573 (11)	0.60267 (9)	0.0160 (3)
H22	−0.002 (3)	0.5432 (15)	0.5856 (12)	0.019*
N23	−0.2648 (2)	0.54700 (12)	0.63607 (11)	0.0212 (3)
H23A	−0.221 (3)	0.4982 (17)	0.6733 (14)	0.028*
H23B	−0.346 (3)	0.5116 (16)	0.5941 (14)	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0119 (6)	0.0132 (7)	0.0177 (8)	−0.0008 (5)	0.0036 (6)	−0.0010 (6)
N2	0.0145 (7)	0.0147 (7)	0.0253 (9)	−0.0003 (5)	0.0024 (6)	−0.0011 (6)
N3	0.0144 (7)	0.0140 (7)	0.0249 (8)	0.0012 (5)	0.0026 (6)	0.0029 (6)
N4	0.0144 (7)	0.0144 (7)	0.0202 (8)	0.0007 (5)	0.0036 (6)	−0.0001 (6)
C5	0.0110 (7)	0.0148 (8)	0.0122 (8)	−0.0010 (6)	0.0002 (6)	−0.0010 (6)
C6	0.0097 (7)	0.0134 (8)	0.0195 (9)	−0.0002 (6)	0.0017 (6)	−0.0005 (7)
N7	0.0112 (6)	0.0136 (7)	0.0151 (7)	0.0000 (5)	0.0031 (5)	0.0009 (5)
C8	0.0158 (8)	0.0152 (8)	0.0140 (9)	−0.0002 (6)	0.0012 (6)	0.0019 (6)
C9	0.0130 (7)	0.0153 (8)	0.0095 (8)	0.0007 (6)	−0.0015 (6)	0.0007 (6)
N10	0.0145 (7)	0.0145 (7)	0.0163 (8)	−0.0004 (5)	0.0010 (6)	−0.0007 (6)
N11	0.0171 (7)	0.0158 (7)	0.0161 (8)	−0.0004 (5)	0.0015 (6)	−0.0013 (6)
N12	0.0167 (7)	0.0168 (7)	0.0196 (8)	−0.0012 (5)	0.0038 (6)	−0.0020 (6)
N13	0.0168 (7)	0.0169 (7)	0.0159 (8)	−0.0007 (6)	0.0029 (6)	−0.0002 (6)
N14	0.0144 (7)	0.0103 (6)	0.0180 (8)	−0.0004 (5)	0.0042 (6)	−0.0007 (5)
O15	0.0210 (6)	0.0207 (6)	0.0135 (6)	0.0011 (5)	0.0013 (5)	−0.0004 (5)
O16	0.0098 (6)	0.0205 (6)	0.0267 (7)	−0.0007 (4)	0.0032 (5)	−0.0027 (5)
C17	0.0137 (8)	0.0148 (8)	0.0104 (8)	−0.0011 (6)	−0.0021 (6)	0.0000 (6)
N18	0.0167 (7)	0.0125 (7)	0.0233 (8)	0.0000 (5)	0.0082 (6)	0.0006 (6)
N19	0.0184 (8)	0.0223 (8)	0.0240 (9)	0.0020 (6)	0.0104 (7)	−0.0017 (7)
N20	0.0141 (7)	0.0115 (7)	0.0186 (8)	0.0001 (5)	0.0049 (6)	0.0020 (6)
N21	0.0183 (7)	0.0113 (7)	0.0237 (9)	0.0015 (6)	−0.0016 (6)	0.0005 (6)
N22	0.0142 (7)	0.0127 (7)	0.0226 (8)	0.0013 (5)	0.0081 (6)	0.0014 (6)
N23	0.0197 (8)	0.0147 (7)	0.0312 (10)	−0.0033 (6)	0.0112 (7)	0.0024 (7)

Geometric parameters (Å, º)

N1—C5	1.334 (2)	N12—N13	1.3530 (19)
N1—N2	1.3475 (19)	N14—O15	1.2319 (18)
N1—H1	0.929 (19)	N14—O16	1.2337 (17)
N2—N3	1.300 (2)	C17—N20	1.324 (2)
N3—N4	1.3614 (19)	C17—N18	1.330 (2)
N4—C5	1.321 (2)	C17—N22	1.330 (2)
C5—C6	1.508 (2)	N18—N19	1.410 (2)
C6—N7	1.463 (2)	N18—H18	0.84 (2)
C6—H6A	0.9900	N19—H19A	0.92 (2)
C6—H6B	0.9900	N19—H19B	0.91 (2)
N7—N14	1.3673 (19)	N20—N21	1.4123 (19)
N7—C8	1.469 (2)	N20—H20	0.86 (2)
C8—C9	1.499 (2)	N21—H21A	0.89 (2)
C8—H8A	0.9900	N21—H21B	0.89 (2)
C8—H8B	0.9900	N22—N23	1.4112 (19)
C9—N10	1.333 (2)	N22—H22	0.86 (2)
C9—N13	1.338 (2)	N23—H23A	0.89 (2)
N10—N11	1.3558 (19)	N23—H23B	0.92 (2)
N11—N12	1.3196 (19)		

C5—N1—N2	108.22 (13)	N12—N11—N10	109.37 (13)
C5—N1—H1	134.4 (11)	N11—N12—N13	108.97 (13)
N2—N1—H1	117.0 (11)	C9—N13—N12	105.14 (13)
N3—N2—N1	106.71 (13)	O15—N14—O16	123.94 (14)
N2—N3—N4	110.36 (13)	O15—N14—N7	118.36 (13)
C5—N4—N3	105.71 (13)	O16—N14—N7	117.62 (14)
N4—C5—N1	109.01 (14)	N20—C17—N18	120.25 (15)
N4—C5—C6	124.68 (14)	N20—C17—N22	120.16 (15)
N1—C5—C6	126.31 (14)	N18—C17—N22	119.59 (15)
N7—C6—C5	111.70 (12)	C17—N18—N19	117.99 (14)
N7—C6—H6A	109.3	C17—N18—H18	117.6 (13)
C5—C6—H6A	109.3	N19—N18—H18	122.9 (13)
N7—C6—H6B	109.3	N18—N19—H19A	107.8 (13)
C5—C6—H6B	109.3	N18—N19—H19B	105.4 (13)
H6A—C6—H6B	107.9	H19A—N19—H19B	106.1 (18)
N14—N7—C6	117.27 (13)	C17—N20—N21	117.55 (14)
N14—N7—C8	117.01 (13)	C17—N20—H20	121.3 (12)
C6—N7—C8	118.94 (13)	N21—N20—H20	120.1 (12)
N7—C8—C9	111.78 (13)	N20—N21—H21A	109.3 (13)
N7—C8—H8A	109.3	N20—N21—H21B	105.9 (13)
C9—C8—H8A	109.3	H21A—N21—H21B	108.4 (19)
N7—C8—H8B	109.3	C17—N22—N23	117.82 (14)
C9—C8—H8B	109.3	C17—N22—H22	120.9 (13)
H8A—C8—H8B	107.9	N23—N22—H22	120.6 (13)
N10—C9—N13	111.61 (14)	N22—N23—H23A	107.1 (13)
N10—C9—C8	124.97 (14)	N22—N23—H23B	107.8 (13)
N13—C9—C8	123.25 (14)	H23A—N23—H23B	105.5 (19)
C9—N10—N11	104.91 (13)		
C5—N1—N2—N3	0.17 (18)	C8—C9—N10—N11	−175.43 (14)
N1—N2—N3—N4	−0.10 (18)	C9—N10—N11—N12	−0.01 (17)
N2—N3—N4—C5	−0.01 (18)	N10—N11—N12—N13	0.01 (18)
N3—N4—C5—N1	0.11 (18)	N10—C9—N13—N12	0.00 (18)
N3—N4—C5—C6	−179.14 (15)	C8—C9—N13—N12	175.53 (14)
N2—N1—C5—N4	−0.18 (19)	N11—N12—N13—C9	0.00 (17)
N2—N1—C5—C6	179.06 (15)	C6—N7—N14—O15	20.04 (19)
N4—C5—C6—N7	−38.1 (2)	C8—N7—N14—O15	170.96 (13)
N1—C5—C6—N7	142.82 (16)	C6—N7—N14—O16	−162.96 (13)
C5—C6—N7—N14	72.31 (18)	C8—N7—N14—O16	−12.05 (19)
C5—C6—N7—C8	−78.03 (17)	N20—C17—N18—N19	176.83 (15)
N14—N7—C8—C9	−78.88 (16)	N22—C17—N18—N19	−3.0 (2)
C6—N7—C8—C9	71.53 (17)	N18—C17—N20—N21	−2.8 (2)
N7—C8—C9—N10	77.91 (19)	N22—C17—N20—N21	177.00 (15)
N7—C8—C9—N13	−97.02 (18)	N20—C17—N22—N23	6.8 (2)
N13—C9—N10—N11	0.01 (18)	N18—C17—N22—N23	−173.34 (15)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N10[i]	0.929 (19)	1.804 (19)	2.713 (2)	165.6 (17)
N1—H1···N11[i]	0.929 (19)	2.673 (19)	3.422 (2)	138.2 (14)
N1—H1···O16[i]	0.929 (19)	2.596 (18)	2.9952 (18)	106.5 (13)
N18—H18···O15	0.84 (2)	2.569 (19)	3.1451 (18)	126.4 (16)
N18—H18···N21	0.84 (2)	2.292 (19)	2.650 (2)	105.9 (15)
N19—H19*A*···N4	0.92 (2)	2.29 (2)	3.026 (2)	137.3 (17)
N19—H19*B*···N13[ii]	0.91 (2)	2.54 (2)	3.275 (2)	138.5 (16)
N20—H20···N13[iii]	0.86 (2)	2.09 (2)	2.867 (2)	149.0 (17)
N20—H20···N23	0.86 (2)	2.358 (18)	2.660 (2)	100.9 (14)
N21—H21*A*···N11[ii]	0.89 (2)	2.46 (2)	3.143 (2)	134.4 (16)
N21—H21*B*···O15[iv]	0.89 (2)	2.31 (2)	3.090 (2)	146.3 (18)
N22—H22···N2[v]	0.86 (2)	2.40 (2)	3.118 (2)	142.3 (17)
N22—H22···N19	0.86 (2)	2.325 (19)	2.650 (2)	102.9 (15)
N23—H23*A*···N11[vi]	0.89 (2)	2.22 (2)	3.087 (2)	166.5 (18)
N23—H23*B*···N3[vi]	0.92 (2)	2.38 (2)	3.091 (2)	133.9 (17)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) *x*−1, −*y*+3/2, *z*+1/2; (iv) *x*−1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1; (vi) −*x*, −*y*+1, −*z*+1.

(III) Diammonium bis[(tetrazol-1-id-5-yl)methyl]nitramide monohydrate

Crystal data

2NH$_4$$^+$·C$_4H_4N_{10}O_2$$^{2-}$·H$_2$O	$Z = 2$
$M_r = 278.27$	$F(000) = 292$
Triclinic, $P\overline{1}$	$D_x = 1.579$ Mg m^{-3}
$a = 7.5893$ (11) Å	Mo *Kα* radiation, $\lambda = 0.71073$ Å
$b = 7.6077$ (11) Å	Cell parameters from 9690 reflections
$c = 11.2319$ (15) Å	$\theta = 3.0–29.3°$
$\alpha = 85.564$ (4)°	$\mu = 0.13$ mm^{-1}
$\beta = 85.555$ (4)°	$T = 296$ K
$\gamma = 65.007$ (4)°	Irregular, colorless
$V = 585.29$ (14) Å^3	0.75 × 0.63 × 0.24 mm

Data collection

Bruker SMART APEXII CCD diffractometer	38379 measured reflections
Radiation source: fine-focus sealed tube	3178 independent reflections
Graphite monochromator	3000 reflections with $I > 2\sigma(I)$
ω and *φ* scans	$R_{int} = 0.057$
Absorption correction: multi-scan (SADABS; Bruker, 2008)	$\theta_{max} = 29.3°$, $\theta_{min} = 1.8°$
$T_{min} = 0.687$, $T_{max} = 0.746$	$h = -10 \rightarrow 10$
	$k = -10 \rightarrow 10$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	202 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: mixed
$wR(F^2) = 0.106$	H atoms treated by a mixture of independent
$S = 1.12$	and constrained refinement
3178 reflections	

$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.0988P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. Output from intergration and final cell refinement: A B C Alpha Beta Gamma Vol 7.59208 7.60543 11.22509 85.5941 85.5165 64.9686 584.79 0.00008 0.00008 0.00012 0.0004 0.0004 0.0004 0.01 Corrected for goodness of fit: 0.00040 0.00041 0.00058 0.0020 0.0022 0.0019 0.07

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
N1	0.63637 (12)	0.17356 (11)	−0.03243 (7)	0.02642 (17)
N2	0.74077 (12)	0.11482 (12)	−0.13592 (7)	0.02770 (18)
N3	0.86114 (14)	−0.06786 (13)	−0.12320 (8)	0.0330 (2)
N4	0.84018 (14)	−0.13474 (12)	−0.01067 (8)	0.03243 (19)
C5	0.70207 (12)	0.01714 (12)	0.04216 (7)	0.02088 (17)
C6	0.62734 (14)	0.01115 (14)	0.16882 (8)	0.02520 (18)
H6A	0.4887	0.0929	0.1739	0.030*
H6B	0.6459	−0.1207	0.1923	0.030*
N7	0.72326 (12)	0.07574 (11)	0.25251 (7)	0.02516 (17)
C8	0.88186 (13)	−0.06259 (14)	0.32289 (8)	0.02571 (18)
H8A	0.9528	0.0041	0.3523	0.031*
H8B	0.9712	−0.1626	0.2717	0.031*
C9	0.81243 (12)	−0.15606 (13)	0.42648 (8)	0.02285 (17)
N10	0.76291 (15)	−0.08633 (13)	0.53514 (7)	0.0334 (2)
N11	0.70900 (16)	−0.21454 (14)	0.59893 (8)	0.0382 (2)
N12	0.72763 (14)	−0.35383 (13)	0.53058 (8)	0.0343 (2)
N13	0.79270 (14)	−0.32036 (13)	0.41982 (8)	0.03096 (19)
N14	0.65021 (12)	0.26410 (12)	0.27363 (7)	0.02663 (17)
O15	0.52737 (13)	0.37956 (11)	0.20606 (7)	0.03769 (19)
O16	0.71072 (13)	0.31354 (12)	0.35853 (7)	0.03772 (19)
O1S	0.02418 (14)	0.53216 (13)	−0.20467 (7)	0.0398 (2)
H1SA	0.086 (3)	0.482 (3)	−0.2719 (18)	0.060*
H1SB	−0.028 (3)	0.650 (3)	−0.2242 (18)	0.060*
N1A	0.36192 (14)	0.69706 (13)	0.37735 (8)	0.02941 (18)
H1A	0.317 (2)	0.615 (2)	0.4054 (13)	0.035*
H1B	0.484 (2)	0.643 (2)	0.3830 (13)	0.035*
H1C	0.336 (2)	0.730 (2)	0.3016 (14)	0.035*
H1D	0.318 (2)	0.807 (2)	0.4177 (13)	0.035*
N2A	0.24096 (13)	0.50259 (13)	0.00386 (9)	0.03122 (19)
H2A	0.184 (2)	0.504 (2)	−0.0618 (14)	0.037*
H2B	0.267 (2)	0.601 (2)	0.0049 (14)	0.037*
H2C	0.181 (2)	0.493 (2)	0.0693 (15)	0.037*
H2D	0.356 (2)	0.399 (2)	−0.0029 (13)	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0331 (4)	0.0229 (4)	0.0194 (3)	−0.0083 (3)	−0.0005 (3)	0.0001 (3)
N2	0.0362 (4)	0.0283 (4)	0.0188 (3)	−0.0141 (3)	0.0003 (3)	−0.0004 (3)
N3	0.0396 (4)	0.0297 (4)	0.0238 (4)	−0.0096 (3)	0.0059 (3)	−0.0034 (3)
N4	0.0401 (4)	0.0235 (4)	0.0250 (4)	−0.0057 (3)	0.0027 (3)	−0.0005 (3)
C5	0.0251 (4)	0.0211 (4)	0.0183 (4)	−0.0112 (3)	−0.0024 (3)	−0.0011 (3)
C6	0.0331 (4)	0.0290 (4)	0.0186 (4)	−0.0181 (4)	0.0001 (3)	−0.0009 (3)
N7	0.0336 (4)	0.0236 (4)	0.0188 (3)	−0.0122 (3)	−0.0026 (3)	−0.0015 (3)
C8	0.0245 (4)	0.0294 (4)	0.0217 (4)	−0.0103 (3)	0.0015 (3)	−0.0005 (3)
C9	0.0242 (4)	0.0232 (4)	0.0196 (4)	−0.0082 (3)	−0.0023 (3)	−0.0009 (3)
N10	0.0513 (5)	0.0298 (4)	0.0210 (4)	−0.0194 (4)	0.0034 (3)	−0.0031 (3)
N11	0.0540 (6)	0.0349 (5)	0.0256 (4)	−0.0203 (4)	0.0061 (4)	0.0008 (3)
N12	0.0430 (5)	0.0329 (4)	0.0302 (4)	−0.0197 (4)	−0.0012 (3)	0.0038 (3)
N13	0.0420 (5)	0.0293 (4)	0.0251 (4)	−0.0181 (3)	−0.0024 (3)	−0.0016 (3)
N14	0.0365 (4)	0.0257 (4)	0.0188 (3)	−0.0147 (3)	0.0027 (3)	−0.0019 (3)
O15	0.0502 (5)	0.0281 (4)	0.0272 (4)	−0.0092 (3)	−0.0055 (3)	0.0028 (3)
O16	0.0533 (5)	0.0357 (4)	0.0295 (4)	−0.0222 (4)	−0.0043 (3)	−0.0086 (3)
O1S	0.0512 (5)	0.0321 (4)	0.0275 (4)	−0.0099 (3)	0.0021 (3)	0.0005 (3)
N1A	0.0372 (4)	0.0266 (4)	0.0222 (4)	−0.0108 (3)	−0.0040 (3)	−0.0008 (3)
N2A	0.0307 (4)	0.0263 (4)	0.0381 (5)	−0.0132 (3)	−0.0010 (4)	−0.0022 (3)

Geometric parameters (Å, º)

N1—C5	1.3325 (11)	N10—N11	1.3475 (13)
N1—N2	1.3480 (11)	N11—N12	1.3095 (14)
N2—N3	1.3051 (12)	N12—N13	1.3485 (12)
N3—N4	1.3488 (12)	N14—O16	1.2373 (11)
N4—C5	1.3312 (12)	N14—O15	1.2375 (11)
C5—C6	1.4948 (12)	O1S—H1SA	0.88 (2)
C6—N7	1.4611 (12)	O1S—H1SB	0.83 (2)
C6—H6A	0.9700	N1A—H1A	0.859 (16)
C6—H6B	0.9700	N1A—H1B	0.847 (16)
N7—N14	1.3334 (11)	N1A—H1C	0.882 (16)
N7—C8	1.4593 (12)	N1A—H1D	0.906 (16)
C8—C9	1.4935 (12)	N2A—H2A	0.880 (16)
C8—H8A	0.9700	N2A—H2B	0.854 (16)
C8—H8B	0.9700	N2A—H2C	0.849 (17)
C9—N13	1.3284 (12)	N2A—H2D	0.896 (16)
C9—N10	1.3315 (12)		
C5—N1—N2	104.67 (7)	N13—C9—C8	123.16 (8)
N3—N2—N1	109.51 (7)	N10—C9—C8	124.72 (8)
N2—N3—N4	109.43 (8)	C9—N10—N11	104.58 (8)
C5—N4—N3	104.71 (8)	N12—N11—N10	109.26 (8)
N4—C5—N1	111.67 (8)	N11—N12—N13	109.62 (8)
N4—C5—C6	124.00 (8)	C9—N13—N12	104.41 (8)

N1—C5—C6	124.32 (8)	O16—N14—O15	123.83 (8)
N7—C6—C5	113.19 (7)	O16—N14—N7	118.24 (8)
N7—C6—H6A	108.9	O15—N14—N7	117.93 (8)
C5—C6—H6A	108.9	H1SA—O1S—H1SB	102.1 (18)
N7—C6—H6B	108.9	H1A—N1A—H1B	107.3 (14)
C5—C6—H6B	108.9	H1A—N1A—H1C	111.5 (14)
H6A—C6—H6B	107.8	H1B—N1A—H1C	108.9 (14)
N14—N7—C8	119.43 (8)	H1A—N1A—H1D	114.4 (14)
N14—N7—C6	118.69 (8)	H1B—N1A—H1D	106.7 (14)
C8—N7—C6	121.50 (8)	H1C—N1A—H1D	107.9 (13)
N7—C8—C9	112.80 (7)	H2A—N2A—H2B	111.5 (14)
N7—C8—H8A	109.0	H2A—N2A—H2C	116.2 (14)
C9—C8—H8A	109.0	H2B—N2A—H2C	108.9 (15)
N7—C8—H8B	109.0	H2A—N2A—H2D	103.5 (13)
C9—C8—H8B	109.0	H2B—N2A—H2D	106.1 (14)
H8A—C8—H8B	107.8	H2C—N2A—H2D	110.1 (14)
N13—C9—N10	112.13 (8)		
C5—N1—N2—N3	0.47 (10)	N7—C8—C9—N13	90.37 (11)
N1—N2—N3—N4	−0.27 (12)	N7—C8—C9—N10	−89.46 (11)
N2—N3—N4—C5	−0.06 (12)	N13—C9—N10—N11	−0.21 (12)
N3—N4—C5—N1	0.37 (11)	C8—C9—N10—N11	179.64 (9)
N3—N4—C5—C6	179.15 (8)	C9—N10—N11—N12	0.43 (12)
N2—N1—C5—N4	−0.52 (11)	N10—N11—N12—N13	−0.50 (13)
N2—N1—C5—C6	−179.30 (8)	N10—C9—N13—N12	−0.08 (11)
N4—C5—C6—N7	95.25 (11)	C8—C9—N13—N12	−179.93 (8)
N1—C5—C6—N7	−86.12 (11)	N11—N12—N13—C9	0.35 (11)
C5—C6—N7—N14	88.66 (10)	C8—N7—N14—O16	−5.08 (12)
C5—C6—N7—C8	−98.39 (9)	C6—N7—N14—O16	168.02 (8)
N14—N7—C8—C9	96.03 (10)	C8—N7—N14—O15	174.04 (8)
C6—N7—C8—C9	−76.87 (10)	C6—N7—N14—O15	−12.86 (12)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1S—H1SA···N13[i]	0.88 (2)	2.06 (2)	2.9253 (12)	168.0 (18)
O1S—H1SB···N3[ii]	0.83 (2)	2.31 (2)	2.9498 (13)	134.8 (17)
N1A—H1A···N12[iii]	0.859 (16)	2.211 (16)	3.0533 (13)	166.7 (14)
N1A—H1B···O16	0.847 (16)	2.388 (16)	3.0079 (13)	130.5 (13)
N1A—H1B···N13[iv]	0.847 (16)	2.540 (15)	3.2862 (14)	147.6 (13)
N1A—H1B···N12[iv]	0.847 (16)	2.585 (15)	3.2472 (14)	136.0 (13)
N2A—H2A···O1S	0.880 (16)	2.030 (16)	2.9062 (14)	173.2 (14)
N2A—H2B···N1[v]	0.854 (16)	2.179 (16)	3.0243 (13)	170.3 (14)
N1A—H1C···N2[v]	0.882 (16)	2.107 (16)	2.9654 (12)	164.2 (14)
N2A—H2C···O1S[vi]	0.849 (17)	2.147 (17)	2.9766 (13)	165.2 (14)

N2*A*—H2*D*···N1	0.896 (16)	2.117 (16)	3.0096 (13)	174.0 (13)
N1*A*—H1*D*···N10[vii]	0.906 (16)	2.045 (16)	2.9273 (13)	164.2 (13)

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) *x*−1, *y*+1, *z*; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*, *y*+1, *z*; (v) −*x*+1, −*y*+1, −*z*; (vi) −*x*, −*y*+1, −*z*; (vii) −*x*+1, −*y*+1, −*z*+1.