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Bis[(diaminomethylidene)azanium] 5-(1-oxido-1*H*-1,2,3,4-tetrazol-5-yl)-1*H*-1,2,3,4-tetrazol-1-olate

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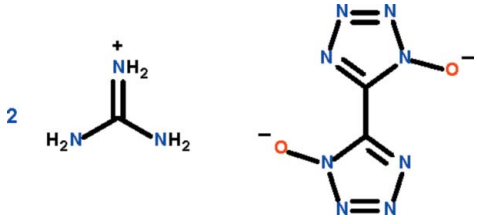
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 11.5.

The anion of the title salt, $2[C(NH_2)_3]^+\cdot C_2N_8O_2^{2-}$, lies on a center of inversion and its two five-membered rings are coplanar. The guanidinium cation forms N—H···O and N—H···N hydrogen bonds to the anion, generating an eight-membered ring. Other hydrogen bonds lead to the formation of a three-dimensional network.

Related literature

For the synthesis of 1,1′-dihydroxy-5,5′-bistetrazole, see: Tselinskii *et al.* (2001).



Experimental

Crystal data

$2CH_6N_3^+\cdot C_2N_8O_2^{2-}$
M_r = 288.28
Monoclinic, $P2_1/c$
a = 3.6477 (3) Å
b = 16.9661 (12) Å
c = 9.5465 (7) Å
β = 97.465 (1)°
V = 585.80 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.12 × 0.11 × 0.08 mm

Data collection

Bruker SMART APEX diffractometer
3402 measured reflections
1328 independent reflections
1229 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.02
1328 reflections
115 parameters
6 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H1···O1	0.89 (1)	1.94 (1)	2.821 (2)	173 (2)
N5—H2···N3^{i}	0.88 (1)	2.40 (1)	3.196 (2)	152 (2)
N6—H3···N3^{i}	0.88 (1)	2.34 (1)	3.126 (2)	149 (2)
N6—H4···N4^{ii}	0.88 (1)	2.12 (1)	2.975 (2)	164 (2)
N7—H5···O1^{iii}	0.87 (1)	1.97 (1)	2.754 (2)	150 (2)
N7—H6···N2	0.88 (1)	2.23 (1)	3.104 (2)	177 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5870).

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supplementary materials

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Comment

The depronated ion of 1,1′-dihydroxy-5,5′-bistetrazole is an example of an organic compound having no hydrogen atoms, and is an appropriate building block for the synthesis of coordination polymers that require metal–nitrogen linkages. To date, no crystal structure of the metal derivatives have been reported. The ion is obtained by the reaction of 1,1′-dihydroxy-5,5′-bistetrazole with guanidine. In the salt (Scheme I), the anion lies on a center of inversion; its two five-membered rings are necessarily coplanar. The guanidinium cation forms N–H···O and N–H···N hydrogen bonds to the anion to generate an eight-membered ring (Fig. 1). Other hydrogen bonds lead to the formation of a three-dimensional network (Table 1).

Experimental

Guanidine carbonate (180 mg, 1 mmol) was added to a methanol solution (10 ml) of 1,1′-dihydroxy-5,5′-bistetrazole (206 mg, 1 mmol). The mixture was stirred for 2 h. The white precipitate that formed was filtered and washed with methanol; yield 0.245 g (90%). CH&N Elemental analysis for $C_4H_8N_{14}O_2$: Calc. C 16.90, H 2.84, N 69.00%. Found C 16.74, H 2.87, N 68.23%. Diethyl ether was used to recrystallize the compound.

Refinement

The amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their displacement parameters were refined.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

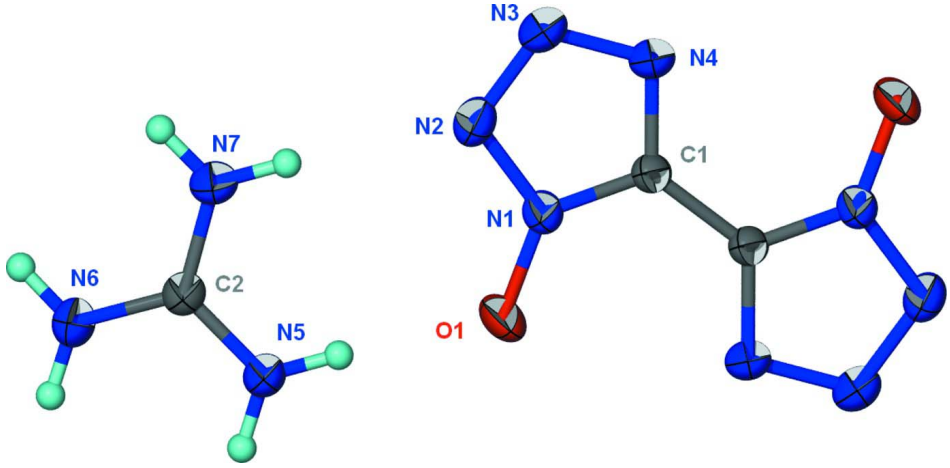

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $2[C(NH_2)_3]^+$ $(C_2N_8O_2)^{2-}$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The dianion lies on a center of inversion; symmetry-related atoms are not labeled.

Bis[(diaminomethylidene)azanium] 5-(1-oxido-1*H*-1,2,3,4-tetrazol-5-yl)- 1*H*-1,2,3,4-tetrazol-1-olate

Crystal data

$2CH_6N_3^+·C_2N_8O_2^{2-}$
M_r = 288.28
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 3.6477 (3) Å
b = 16.9661 (12) Å
c = 9.5465 (7) Å
β = 97.465 (1)°
V = 585.80 (8) Å^3
Z = 2
$F(000)$ = 300
D_x = 1.634 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2581 reflections
θ = 2.5–28.5°
μ = 0.13 mm^{-1}
T = 293 K
Prism, colorless
0.12 × 0.11 × 0.08 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
3402 measured reflections
1328 independent reflections
1229 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −4→4
k = −20→21
l = −7→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.02
1328 reflections
115 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.1424P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3597 (3)	0.63225 (6)	0.54252 (10)	0.0478 (3)
N1	0.1684 (3)	0.59470 (6)	0.43882 (10)	0.0326 (3)
N2	0.1200 (3)	0.62317 (6)	0.30734 (11)	0.0409 (3)
N3	−0.0687 (3)	0.56977 (7)	0.22907 (12)	0.0431 (3)
N4	−0.1457 (3)	0.50828 (6)	0.30661 (11)	0.0377 (3)
N5	0.5844 (4)	0.79085 (7)	0.52906 (12)	0.0447 (3)
N6	0.5689 (4)	0.90966 (7)	0.41611 (13)	0.0441 (3)
N7	0.2983 (4)	0.80194 (7)	0.30217 (12)	0.0427 (3)
C1	0.0040 (3)	0.52416 (6)	0.43820 (11)	0.0283 (3)
C2	0.4842 (3)	0.83418 (7)	0.41568 (12)	0.0324 (3)
H1	0.525 (5)	0.7400 (6)	0.528 (2)	0.056 (5)*
H2	0.703 (5)	0.8143 (11)	0.6033 (15)	0.064 (5)*
H3	0.699 (5)	0.9327 (11)	0.4888 (16)	0.065 (5)*
H4	0.485 (5)	0.9392 (9)	0.3427 (15)	0.061 (5)*
H5	0.242 (5)	0.8300 (10)	0.2262 (15)	0.061 (5)*
H6	0.248 (5)	0.7514 (6)	0.3000 (19)	0.057 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0689 (7)	0.0371 (5)	0.0365 (5)	−0.0201 (4)	0.0028 (4)	−0.0089 (4)
N1	0.0422 (5)	0.0260 (5)	0.0292 (5)	−0.0022 (4)	0.0034 (4)	−0.0017 (4)
N2	0.0550 (7)	0.0330 (5)	0.0343 (6)	−0.0011 (4)	0.0043 (5)	0.0064 (4)
N3	0.0527 (7)	0.0423 (6)	0.0321 (6)	−0.0019 (5)	−0.0028 (5)	0.0072 (4)
N4	0.0456 (6)	0.0365 (5)	0.0285 (5)	−0.0041 (4)	−0.0049 (4)	0.0021 (4)
N5	0.0657 (8)	0.0332 (6)	0.0318 (6)	−0.0096 (5)	−0.0073 (5)	0.0051 (4)
N6	0.0572 (7)	0.0303 (5)	0.0422 (6)	−0.0014 (5)	−0.0037 (5)	0.0048 (4)
N7	0.0584 (7)	0.0391 (6)	0.0287 (5)	−0.0054 (5)	−0.0021 (5)	0.0004 (4)
C1	0.0324 (5)	0.0244 (5)	0.0270 (5)	0.0011 (4)	−0.0004 (4)	−0.0007 (4)
C2	0.0360 (6)	0.0315 (5)	0.0294 (6)	−0.0006 (4)	0.0032 (4)	0.0016 (4)

Geometric parameters (Å, º)

O1—N1	1.3013 (13)	N5—H2	0.877 (9)
N1—N2	1.3350 (14)	N6—C2	1.3172 (16)
N1—C1	1.3383 (14)	N6—H3	0.879 (10)
N2—N3	1.3112 (16)	N6—H4	0.883 (9)
N3—N4	1.3301 (15)	N7—C2	1.3200 (16)
N4—C1	1.3305 (14)	N7—H5	0.869 (9)
N5—C2	1.3201 (16)	N7—H6	0.876 (9)
N5—H1	0.889 (9)	C1—C1^{i}	1.440 (2)
O1—N1—N2	122.03 (10)	H3—N6—H4	117.9 (18)
O1—N1—C1	129.56 (10)	C2—N7—H5	119.9 (13)
N2—N1—C1	108.36 (10)	C2—N7—H6	120.5 (12)
N3—N2—N1	106.34 (10)	H5—N7—H6	119.3 (18)
N2—N3—N4	110.97 (10)	N4—C1—N1	108.29 (10)

N3—N4—C1	106.04 (10)	N4—C1—C1^{i}	127.50 (13)
C2—N5—H1	119.3 (12)	N1—C1—C1^{i}	124.20 (13)
C2—N5—H2	117.7 (13)	N6—C2—N7	120.04 (11)
H1—N5—H2	123.0 (18)	N6—C2—N5	119.94 (11)
C2—N6—H3	122.5 (14)	N7—C2—N5	120.01 (11)
C2—N6—H4	119.5 (12)		
O1—N1—N2—N3	177.18 (11)	N3—N4—C1—C1^{i}	179.84 (15)
C1—N1—N2—N3	−0.51 (14)	O1—N1—C1—N4	−177.19 (12)
N1—N2—N3—N4	0.58 (15)	N2—N1—C1—N4	0.26 (14)
N2—N3—N4—C1	−0.42 (14)	O1—N1—C1—C1^{i}	3.0 (2)
N3—N4—C1—N1	0.09 (13)	N2—N1—C1—C1^{i}	−179.50 (13)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H1···O1	0.89 (1)	1.94 (1)	2.821 (2)	173 (2)
N5—H2···N3^{ii}	0.88 (1)	2.40 (1)	3.196 (2)	152 (2)
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N6—H4···N4^{iii}	0.88 (1)	2.12 (1)	2.975 (2)	164 (2)
N7—H5···O1^{iv}	0.87 (1)	1.97 (1)	2.754 (2)	150 (2)
N7—H6···N2	0.88 (1)	2.23 (1)	3.104 (2)	177 (2)

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