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Supporting information for article:

Energetic propane-1,3-diaminium and butane-1,4-diaminium salts of *N*,*N*′-dinitroethylenediazanide: syntheses, crystal structures and thermal properties

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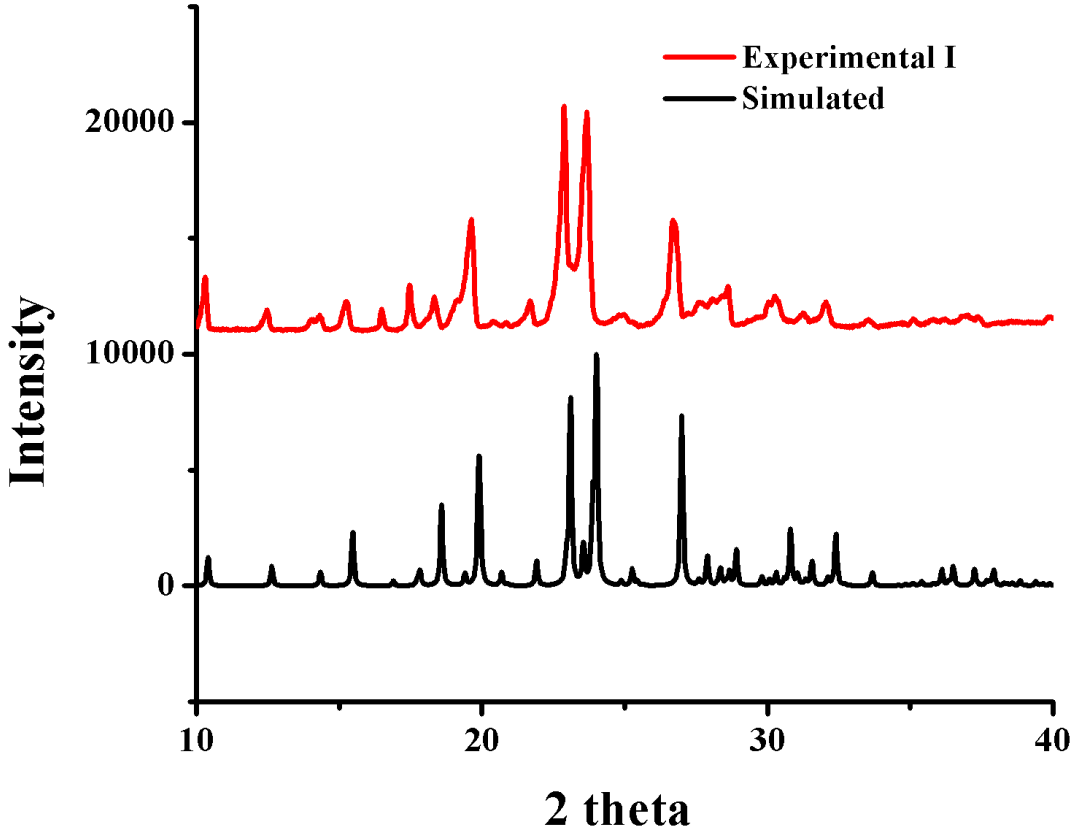

Figure S1 Powder XRD pattern for 1,3-propanediaminium ethane-bis-nitraminate (**I**)

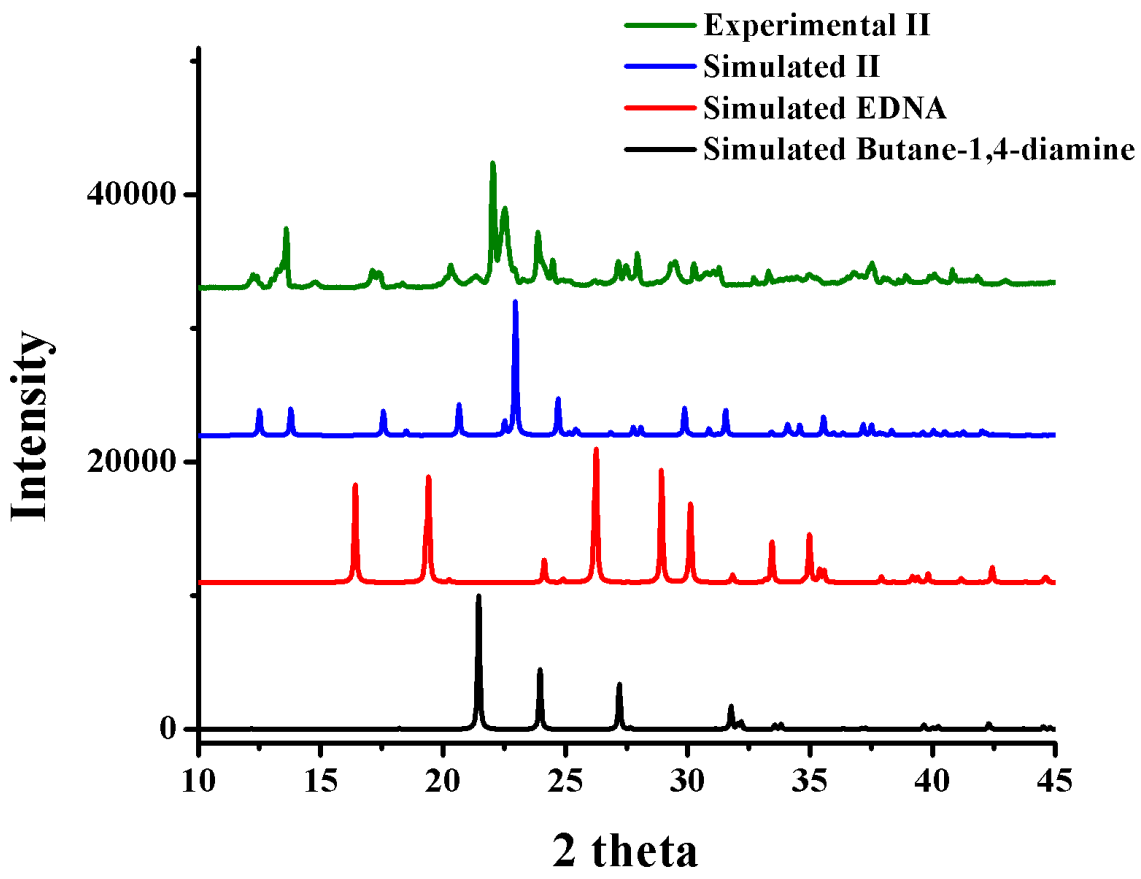

Figure S2 Powder XRD pattern for 1,4-butanediaminium ethane-bis-nitraminate (**II**)

Data collection: *APEX2* V2014-11 (Bruker, 2016) for Compound I; *APEX2* V2014-11 (Bruker, 2014) for Compound II. Cell refinement: *SAINT* V8.38A (Bruker, 2016) for Compound I; *SAINT* v8.37A (Bruker, 2015) for Compound II. Data reduction: *SAINT* V8.38A (Bruker, 2016) for Compound I; *SAINT* v8.37A (Bruker, 2015) for Compound II. For both compounds, program(s) used to solve structure: SHELXT 2014/5 (Sheldrick, 2014). Program(s) used to refine structure: *SHELXL2017*/1 (Sheldrick, 2017) for Compound I; *SHELXL* (Sheldrick, 2015) for Compound II. For both compounds, molecular graphics: Olex2 (Dolomanov *et al.*, 2009). Software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009); *PLATON* (Spek, 2009) for Compound I; Olex2 (Dolomanov *et al.*, 2009) for Compound II.

Data for 1,3-propanediaminium ethane-bis-nitraminate (I)

Table S1 Crystal Data

$C_2H_4N_4O_4 \cdot C_3H_{12}N_2$	D_x = 1.428 Mg m^{-3}

M_r = 224.24	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, *Pbca*	Cell parameters from 2241 reflections
a = 12.3448 (14) Å	θ = 2.4–24.2°
b = 9.9435 (13) Å	μ = 0.12 mm^{-1}
c = 16.9904 (19) Å	*T* = 100 K
V = 2085.6 (4) Å^3	Plate, colourless
Z = 8	0.26 × 0.17 × 0.08 mm
F(000) = 960	

Table S2 Data collection

Bruker APEX-II CCD diffractometer	1828 reflections with $I > 2\sigma(I)$
ϕ and ω scans	R_{int} = 0.077
Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0728 before and 0.0668 after correction. The Ratio of minimum to maximum transmission is 0.8792. The λ/2 correction factor is 0.00150.	θ_{max} = 28.3°, θ_{min} = 2.4°
T_{min} = 0.656, T_{max} = 0.746	*h* = -16→16
18809 measured reflections	*k* = -9→13
2591 independent reflections	*l* = -22→22

Table S3 Refinement

Refinement on F^2	Primary atom site location: dual
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.6306P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
2591 reflections	$\Delta\rangle_{max}$ = 0.24 e Å^{-3}
160 parameters	$\Delta\rangle_{min}$ = -0.25 e Å^{-3}
6 restraints	

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.

Table S4 Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.52445 (10)	0.84025 (13)	0.72260 (7)	0.0297 (3)
O2	0.62676 (11)	0.85073 (13)	0.61563 (7)	0.0370 (4)
O3	0.56105 (9)	0.18981 (13)	0.55971 (6)	0.0261 (3)
O4	0.67822 (10)	0.19857 (13)	0.46112 (7)	0.0297 (3)
N1	0.56838 (11)	0.78109 (16)	0.66152 (8)	0.0256 (3)
N2	0.55013 (11)	0.65589 (15)	0.65373 (8)	0.0239 (3)
N3	0.64024 (11)	0.38529 (15)	0.52363 (7)	0.0230 (3)
N4	0.62552 (11)	0.25971 (15)	0.51576 (7)	0.0232 (3)
N5	0.64848 (11)	-0.07170 (16)	0.44556 (8)	0.0218 (3)
H5A	0.7032 (13)	-0.111 (2)	0.4714 (10)	0.035 (5)*
H5B	0.5844 (12)	-0.1040 (19)	0.4607 (10)	0.031 (5)*
H5C	0.6523 (16)	0.0172 (14)	0.4549 (11)	0.035 (6)*
N6	0.89704 (11)	-0.10009 (17)	0.21727 (8)	0.0224 (3)
H6A	0.9411 (15)	-0.149 (2)	0.2470 (11)	0.041 (6)*
H6B	0.9196 (16)	-0.0147 (14)	0.2187 (11)	0.035 (6)*
H6C	0.9024 (16)	-0.129 (2)	0.1678 (8)	0.035 (5)*
C1	0.60335 (14)	0.59631 (18)	0.58573 (9)	0.0232 (4)
H1A	0.682372	0.612859	0.587960	0.028*
H1B	0.574704	0.636047	0.536522	0.028*
C2	0.58044 (13)	0.44706 (18)	0.58800 (9)	0.0242 (4)
H2A	0.604022	0.408707	0.638970	0.029*
H2B	0.501852	0.430337	0.581751	0.029*
C3	0.65865 (12)	-0.09557 (18)	0.35959 (9)	0.0223 (4)
H3A	0.607638	-0.036108	0.331128	0.027*
H3B	0.638830	-0.189875	0.347697	0.027*
C4	0.77355 (12)	-0.06866 (18)	0.33093 (9)	0.0215 (4)
H4A	0.790641	0.028115	0.336666	0.026*
H4B	0.825733	-0.120459	0.363156	0.026*
C5	0.78367 (13)	-0.10960 (19)	0.24521 (9)	0.0246 (4)
H5D	0.757743	-0.203187	0.238836	0.030*
H5E	0.737193	-0.050622	0.212599	0.030*

Table S5 Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

O1	0.0322 (7)	0.0338 (8)	0.0232 (6)	0.0002 (6)	0.0068 (5)	0.0004 (5)
O2	0.0510 (8)	0.0317 (8)	0.0282 (7)	-0.0132 (6)	0.0154 (6)	0.0016 (5)
O3	0.0217 (6)	0.0323 (8)	0.0243 (6)	-0.0079 (5)	0.0001 (5)	0.0081 (5)
O4	0.0286 (7)	0.0291 (7)	0.0315 (6)	0.0004 (5)	0.0067 (5)	0.0016 (5)
N1	0.0232 (7)	0.0334 (9)	0.0202 (7)	-0.0021 (6)	0.0000 (5)	0.0041 (6)
N2	0.0204 (7)	0.0291 (9)	0.0223 (7)	-0.0039 (6)	-0.0004 (5)	0.0028 (6)
N3	0.0209 (7)	0.0270 (8)	0.0211 (7)	-0.0052 (6)	-0.0002 (5)	0.0013 (6)
N4	0.0182 (7)	0.0298 (9)	0.0217 (7)	-0.0040 (6)	-0.0017 (5)	0.0050 (6)
N5	0.0163 (7)	0.0264 (9)	0.0225 (7)	0.0010 (6)	0.0019 (5)	0.0036 (6)
N6	0.0192 (7)	0.0305 (9)	0.0175 (6)	0.0014 (6)	0.0006 (6)	0.0028 (6)
C1	0.0209 (8)	0.0309 (10)	0.0179 (7)	-0.0047 (7)	-0.0015 (6)	0.0028 (7)
C2	0.0215 (8)	0.0322 (10)	0.0189 (7)	-0.0074 (7)	-0.0007 (6)	0.0010 (7)
C3	0.0172 (8)	0.0285 (10)	0.0213 (7)	-0.0013 (7)	0.0004 (6)	-0.0027 (7)
C4	0.0170 (8)	0.0293 (10)	0.0183 (7)	-0.0019 (7)	-0.0006 (6)	0.0023 (6)
C5	0.0186 (8)	0.0317 (10)	0.0236 (8)	-0.0022 (7)	0.0001 (6)	-0.0030 (7)

Table S6 Geometric parameters (Å, º)

O1—N1	1.3103 (18)	N6—H6C	0.889 (12)
O2—N1	1.2676 (18)	C1—C2	1.511 (2)
O3—N4	1.2939 (17)	C1—H1A	0.9900
O4—N4	1.2863 (18)	C1—H1B	0.9900
N1—N2	1.272 (2)	C2—H2A	0.9900
N2—C1	1.455 (2)	C2—H2B	0.9900
N3—N4	1.269 (2)	C3—C4	1.523 (2)
N3—C2	1.455 (2)	C3—H3A	0.9900
N5—C3	1.485 (2)	C3—H3B	0.9900
N5—H5A	0.894 (13)	C4—C5	1.517 (2)
N5—H5B	0.891 (12)	C4—H4A	0.9900
N5—H5C	0.899 (13)	C4—H4B	0.9900
N6—C5	1.481 (2)	C5—H5D	0.9900
N6—H6A	0.887 (13)	C5—H5E	0.9900
N6—H6B	0.894 (13)		
O2—N1—N2	124.85 (14)	H1A—C1—H1B	108.5
O2—N1—O1	118.50 (15)	N3—C2—C1	107.47 (13)
N2—N1—O1	116.64 (13)	N3—C2—H2A	110.2
N1—N2—C1	113.65 (13)	C1—C2—H2A	110.2
N4—N3—C2	114.95 (13)	N3—C2—H2B	110.2
N3—N4—O4	117.95 (13)	C1—C2—H2B	110.2
N3—N4—O3	123.77 (14)	H2A—C2—H2B	108.5

O4—N4—O3	118.28 (14)	N5—C3—C4	111.42 (13)
C3—N5—H5A	110.4 (12)	N5—C3—H3A	109.3
C3—N5—H5B	107.5 (12)	C4—C3—H3A	109.3
H5A—N5—H5B	111.8 (18)	N5—C3—H3B	109.3
C3—N5—H5C	109.1 (12)	C4—C3—H3B	109.3
H5A—N5—H5C	107.6 (18)	H3A—C3—H3B	108.0
H5B—N5—H5C	110.4 (18)	C5—C4—C3	109.66 (13)
C5—N6—H6A	111.2 (14)	C5—C4—H4A	109.7
C5—N6—H6B	110.3 (13)	C3—C4—H4A	109.7
H6A—N6—H6B	108.2 (19)	C5—C4—H4B	109.7
C5—N6—H6C	110.7 (13)	C3—C4—H4B	109.7
H6A—N6—H6C	108.6 (19)	H4A—C4—H4B	108.2
H6B—N6—H6C	107.8 (18)	N6—C5—C4	111.62 (13)
N2—C1—C2	107.16 (13)	N6—C5—H5D	109.3
N2—C1—H1A	110.3	C4—C5—H5D	109.3
C2—C1—H1A	110.3	N6—C5—H5E	109.3
N2—C1—H1B	110.3	C4—C5—H5E	109.3
C2—C1—H1B	110.3	H5D—C5—H5E	108.0
O2—N1—N2—C1	0.1 (2)	N4—N3—C2—C1	-177.19 (13)
O1—N1—N2—C1	-178.69 (13)	N2—C1—C2—N3	-175.71 (12)
C2—N3—N4—O4	-178.29 (13)	N5—C3—C4—C5	173.46 (14)
C2—N3—N4—O3	1.4 (2)	C3—C4—C5—N6	-173.42 (15)
N1—N2—C1—C2	176.08 (13)		

Data for 1,4-butanediaminium ethane-bis-nitraminate (II)

Table S7 Crystal data

$C_2H_4N_4O_4 \cdot C_4H_{14}N_2$	$F(000) = 256$
$M_r = 238.26$	$D_x = 1.436$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 5.408$ (3) Å	Cell parameters from 6899 reflections
$b = 7.203$ (4) Å	$\theta = 2.9–28.6°$
$c = 14.288$ (8) Å	$\mu = 0.12$ mm^{-1}
$\beta = 98.03$ (3)°	$T = 100$ K
$V = 551.0$ (5) Å^3	Block, colourless
$Z = 2$	0.44 × 0.41 × 0.26 mm

Table S8 Data collection

Bruker APEX-II CCD diffractometer	1258 reflections with $I > 2\sigma(I)$
ϕ and ω scans	$R_{int} = 0.044$
Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0625 before and 0.0557 after correction. The Ratio of minimum to maximum transmission is 0.9347. The $\lambda/2$ correction factor is 0.00150.	$\theta_{max} = 28.3°$, $\theta_{min} = 2.9°$
$T_{min} = 0.697$, $T_{max} = 0.746$	$h = -7 \rightarrow 7$
11975 measured reflections	$k = -9 \rightarrow 9$
1371 independent reflections	$l = -18 \rightarrow 19$

Table S9 Refinement

Refinement on F^2	3 restraints
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.1936P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1371 reflections	$\Delta\rangle_{max} = 0.42$ e Å^{-3}
85 parameters	$\Delta\rangle_{min} = -0.22$ 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.

Table S10 Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.57312 (12)	0.50962 (10)	0.36574 (5)	0.01429 (17)
O2	0.69613 (12)	0.32306 (10)	0.25666 (5)	0.01567 (18)
N2	0.98232 (14)	0.42748 (11)	0.37090 (5)	0.01234 (19)
N3	0.75026 (14)	0.42316 (10)	0.33261 (5)	0.01133 (18)
C3	1.03227 (16)	0.54830 (13)	0.45476 (6)	0.0120 (2)
H3A	0.932326	0.663346	0.443575	0.014*

H3B	1.210936	0.583654	0.464370	0.014*
N1	0.26171 (14)	0.13317 (11)	0.29615 (5)	0.01154 (18)
H1A	0.406 (2)	0.1838 (18)	0.2778 (10)	0.023 (3)*
H1B	0.174 (2)	0.2286 (17)	0.3206 (9)	0.019 (3)*
H1C	0.164 (2)	0.0814 (19)	0.2444 (9)	0.026 (4)*
C1	0.32151 (18)	-0.01024 (13)	0.37207 (6)	0.0145 (2)
H1D	0.411206	-0.114987	0.347103	0.017*
H1E	0.164198	-0.058994	0.390608	0.017*
C2	0.48284 (17)	0.07085 (12)	0.45915 (6)	0.0130 (2)
H2A	0.402579	0.184429	0.479772	0.016*
H2B	0.648299	0.105826	0.442502	0.016*

Table S11 Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0104 (3)	0.0184 (4)	0.0143 (3)	0.0033 (2)	0.0024 (2)	0.0007 (2)
O2	0.0150 (3)	0.0199 (4)	0.0115 (3)	-0.0031 (3)	-0.0001 (2)	-0.0043 (3)
N2	0.0094 (3)	0.0152 (4)	0.0119 (4)	-0.0003 (3)	-0.0003 (3)	-0.0013 (3)
N3	0.0108 (3)	0.0123 (4)	0.0109 (3)	-0.0008 (3)	0.0015 (3)	0.0012 (3)
C3	0.0113 (4)	0.0132 (4)	0.0114 (4)	-0.0021 (3)	0.0011 (3)	-0.0015 (3)
N1	0.0111 (3)	0.0123 (4)	0.0107 (4)	0.0000 (3)	-0.0004 (3)	0.0006 (3)
C1	0.0182 (4)	0.0121 (4)	0.0117 (4)	-0.0011 (3)	-0.0032 (3)	0.0017 (3)
C2	0.0148 (4)	0.0121 (4)	0.0111 (4)	-0.0009 (3)	-0.0015 (3)	0.0005 (3)

Table S12 Geometric parameters (Å, º)

O1—N3	1.2869 (11)	N1—H1C	0.924 (11)
O2—N3	1.3017 (11)	N1—C1	1.5000 (12)
N2—N3	1.2974 (12)	C1—H1D	0.9900
N2—C3	1.4749 (12)	C1—H1E	0.9900
C3—C3^{i}	1.5503 (18)	C1—C2	1.5320 (13)
C3—H3A	0.9900	C2—C2^{ii}	1.5420 (18)
C3—H3B	0.9900	C2—H2A	0.9900
N1—H1A	0.931 (11)	C2—H2B	0.9900
N1—H1B	0.931 (10)		
N3—N2—C3	114.41 (8)	C1—N1—H1B	107.9 (8)
O1—N3—O2	118.84 (8)	C1—N1—H1C	110.0 (9)
O1—N3—N2	124.00 (8)	N1—C1—H1D	109.3
N2—N3—O2	117.16 (8)	N1—C1—H1E	109.3
N2—C3—C3^{i}	112.06 (10)	N1—C1—C2	111.44 (8)

N2—C3—H3A	109.2	H1D—C1—H1E	108.0
N2—C3—H3B	109.2	C2—C1—H1D	109.3
C3^{i}—C3—H3A	109.2	C2—C1—H1E	109.3
C3^{i}—C3—H3B	109.2	C1—C2—C2^{ii}	110.92 (10)
H3A—C3—H3B	107.9	C1—C2—H2A	109.5
H1A—N1—H1B	108.0 (12)	C1—C2—H2B	109.5
H1A—N1—H1C	109.8 (13)	C2^{ii}—C2—H2A	109.5
H1B—N1—H1C	109.5 (12)	C2^{ii}—C2—H2B	109.5
C1—N1—H1A	111.6 (8)	H2A—C2—H2B	108.0
N3—N2—C3—C3^{i}	81.04 (11)	C3—N2—N3—O2	178.03 (7)
C3—N2—N3—O1	-2.72 (12)	N1—C1—C2—C2^{ii}	173.34 (9)

Symmetry codes: (i) -*x*+2, -*y*+1, -*z*+1; (ii) -*x*+1, -*y*, -*z*+1.