Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.
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Electronic Supporting Information

Taming of Tetranitroethane: A Promising Precursor to High Performance Energetic Ingredients

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Table 1 Crystal data and structure refinement for 10.

Formula	$C_2H_{12}Ag_2N_8O_8$
CCDC #	1847183
$D_{calc.}$/ g cm^{-3}	2.705
μ/mm^{-1}	3.301
Formula Weight	491.94
Color	yellow
Shape	needle
Size/mm^3	0.28×0.19×0.09
T/K	173(2)
Crystal System	tetragonal
Space Group	I4/m
a/Å	11.6936(9)
b/Å	11.6936(9)
c/Å	8.8330(8)
α/°	90
β/°	90
γ/°	90
V/Å^3	1207.8(2)
Z	4
Z'	0.25
Wavelength/Å	0.710730
Radiation type	MoK_α
Θ_{min}/°	2.463
Θ_{max}/°	25.180
Measured Refl.	9946
Independent Refl.	586
Reflections Used	584
R_{int}	0.0249
Parameters	71
Restraints	0
Largest Peak	1.422
Deepest Hole	-2.225
GooF	1.196
wR_2 (all data)	0.2378
wR_2	0.2378
R_1 (all data)	0.0663
R_1	0.0663

Table 2. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 10. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
Ag1	5000	5000	6896.7(18)	21.3(7)
Ag2	5000(2)	3578(2)	10000	20.8(8)
N3	6271(17)	3723(17)	6840(20)	21(2)
N4	6617(15)	3380(16)	9100(20)	21(2)
O1	6546(12)	556(14)	6498(18)	21(3)
O2	5920(14)	2235(12)	5856(19)	26(4)
O3	3451(12)	555(14)	3507(18)	22(3)
O4	4079(14)	2233(12)	4143(19)	26(4)
N1	5827(15)	1193(17)	5840(20)	21(2)
N2	4173(15)	1191(17)	4170(20)	21(2)
C1	4998(12)	602(14)	5000	15(3)

Table 3. Anisotropic Displacement Parameters (×10⁴) 10. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ag1	23.1(8)	23.1(8)	17.6(11)	0	0	0
Ag2	19.4(13)	23.1(14)	19.7(14)	0	0	0.0(8)
N3	21(5)	26(5)	16(4)	-2(4)	2(4)	-1(4)
N4	21(5)	26(5)	16(4)	-2(4)	2(4)	-1(4)
O1	13(7)	32(9)	18(7)	4(7)	-9(6)	8(6)
O2	38(9)	13(7)	28(9)	-7(7)	12(8)	-6(6)
O3	13(7)	34(9)	18(7)	-3(7)	-9(6)	-7(6)
O4	38(9)	12(7)	28(9)	4(7)	14(8)	7(6)
N1	21(5)	26(5)	16(4)	-2(4)	2(4)	-1(4)
N2	21(5)	26(5)	16(4)	-2(4)	2(4)	-1(4)
C1	10(6)	28(8)	7(6)	0	0	-1(6)

Table 4. Bond Lengths in Å for 10.

Atom	Atom	Length/Å
Ag1	Ag1[1]	3.351(3)
Ag1	Ag2	3.2063(19)
Ag1	Ag2[2]	3.2064(19)
Ag1	Ag2[3]	3.2064(19)
Ag1	Ag2[4]	3.2063(19)
Ag1	N3[2]	2.11(2)
Ag1	N3[5]	2.11(2)
Ag1	N3	2.11(2)
Ag1	N3[6]	2.11(2)
Ag2	Ag1[3]	3.2064(19)
Ag2	Ag2[4]	2.352(3)
Ag2	Ag2[2]	2.352(3)
Ag2	Ag2[3]	3.327(5)
Ag2	N4[4]	2.068(18)
Ag2	N4	2.065(18)
Ag2	N4[6]	2.068(18)
Ag2	N4[7]	2.065(18)
N4	Ag2[2]	2.068(18)
N4	N4[7]	1.60(4)
O1	O3[8]	1.30(2)
O1	N1	1.27(2)
O2	O2[9]	1.51(3)
O2	N1	1.22(3)
O3	O1[8]	1.30(2)
O3	N2	1.27(2)
O4	O4[9]	1.51(3)
O4	N2	1.22(2)
N1	N1[9]	1.48(4)
N1	C1	1.40(2)
N2	N2[9]	1.47(4)
N2	C1	1.40(2)
C1	N1[9]	1.40(2)
C1	N2[9]	1.40(2)
C1	C1[8]	1.41(3)

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[1]1-X,1-Y,1-Z; [2]1-Y,+X,+Z; [3]1-X,1-Y,2-Z; [4]+Y,1-X,2-Z; [5]1-X,1-Y,+Z; [6]+Y,1-X,+Z; [7]+X,+Y,2-Z; [8]1-X,-Y,1-Z; [9]+X,+Y,1-Z

Table 5. Bond Angles in ° for 10.

Atom	Atom	Atom	Angle/°
Ag2[1]	Ag1	Ag1[2]	148.75(4)
Ag2[3]	Ag1	Ag1[2]	148.75(4)
Ag2	Ag1	Ag1[2]	148.75(4)
Ag2[4]	Ag1	Ag1[2]	148.75(4)
Ag2[1]	Ag1	Ag2[3]	62.50(8)
Ag2	Ag1	Ag2[3]	43.04(5)
Ag2[4]	Ag1	Ag2[3]	43.04(5)
Ag2	Ag1	Ag2[1]	43.04(5)
Ag2	Ag1	Ag2[4]	62.50(8)
Ag2[1]	Ag1	Ag2[4]	43.04(5)
N3[5]	Ag1	Ag1[2]	88.7(5)
N3[6]	Ag1	Ag1[2]	88.7(5)
N3[1]	Ag1	Ag1[2]	88.7(5)
N3	Ag1	Ag1[2]	88.7(5)
N3[1]	Ag1	Ag2[3]	112.8(5)
N3[6]	Ag1	Ag2[4]	69.7(5)
N3[5]	Ag1	Ag2[3]	69.7(5)
N3[5]	Ag1	Ag2	69.8(5)
N3	Ag1	Ag2[4]	112.8(5)
N3[6]	Ag1	Ag2[3]	69.8(5)
N3	Ag1	Ag2[1]	69.8(5)
N3[6]	Ag1	Ag2	112.8(5)
N3[5]	Ag1	Ag2[4]	112.7(5)
N3[6]	Ag1	Ag2[1]	112.7(5)
N3[1]	Ag1	Ag2	112.7(5)
N3[5]	Ag1	Ag2[1]	112.8(5)
N3	Ag1	Ag2	69.7(5)
N3	Ag1	Ag2[3]	112.7(5)
N3[1]	Ag1	Ag2[1]	69.7(5)
N3[1]	Ag1	Ag2[4]	69.8(5)
N3[6]	Ag1	N3	177.3(10)
N3[5]	Ag1	N3	89.97(3)
N3[1]	Ag1	N3[6]	89.97(3)
N3[1]	Ag1	N3	89.97(3)
N3[5]	Ag1	N3[6]	89.97(3)
N3[5]	Ag1	N3[1]	177.3(10)
Ag1	Ag2	Ag1[4]	117.50(8)
Ag1[4]	Ag2	Ag2[4]	58.75(4)
Ag1	Ag2	Ag2[4]	58.75(4)
Ag2[3]	Ag2	Ag1[4]	68.48(3)
Ag2[1]	Ag2	Ag1	68.48(3)
Ag2[1]	Ag2	Ag1[4]	68.48(3)
Ag2[3]	Ag2	Ag1	68.48(3)
Ag2[1]	Ag2	Ag2[4]	45.0
Ag2[3]	Ag2	Ag2[4]	45.0
Ag2[1]	Ag2	Ag2[3]	90.0
N4	Ag2	Ag1[4]	112.9(5)
N4	Ag2	Ag1	74.2(5)
N4[5]	Ag2	Ag1[4]	112.8(5)
N4[3]	Ag2	Ag1	112.8(5)
N4[5]	Ag2	Ag1	74.1(5)
N4[3]	Ag2	Ag1[4]	74.1(5)
N4[7]	Ag2	Ag1[4]	74.2(5)
N4[7]	Ag2	Ag1	112.9(5)

Atom	Atom	Atom	Angle/°
N4	Ag2	Ag2[1]	55.4(5)
N4	Ag2	Ag2[3]	136.6(5)
N4[3]	Ag2	Ag2[1]	136.5(5)
N4[7]	Ag2	Ag2[4]	96.4(5)
N4[3]	Ag2	Ag2[3]	55.3(5)
N4[5]	Ag2	Ag2[3]	55.3(5)
N4[5]	Ag2	Ag2[4]	96.3(5)
N4[7]	Ag2	Ag2[1]	55.4(5)
N4[7]	Ag2	Ag2[3]	136.6(5)
N4[3]	Ag2	Ag2[4]	96.3(5)
N4	Ag2	Ag2[4]	96.4(5)
N4[5]	Ag2	Ag2[1]	136.5(5)
N4[7]	Ag2	N4[3]	132.6(9)
N4	Ag2	N4[7]	45.5(10)
N4	Ag2	N4[3]	167.3(6)
N4[5]	Ag2	N4[3]	45.4(10)
N4	Ag2	N4[5]	132.6(9)
N4[7]	Ag2	N4[5]	167.3(6)
Ag2	N4	Ag2[1]	69.4(6)
N4[7]	N4	Ag2[1]	67.3(5)
N4[7]	N4	Ag2	67.3(5)
N1	O1	O3[8]	126.0(16)
N1	O2	O2[9]	89.3(12)
N2	O3	O1[8]	125.9(16)
N2	O4	O4[9]	89.1(12)
O1	N1	N1[9]	117.4(11)
O1	N1	C1	114.4(18)
O2	N1	O1	121.4(19)
O2	N1	N1[9]	90.7(12)
O2	N1	C1	124.0(18)
C1	N1	N1[9]	58.1(8)
O3	N2	N2[9]	117.4(11)
O3	N2	C1	114.5(18)
O4	N2	O3	121.1(19)
O4	N2	N2[9]	90.9(12)
O4	N2	C1	124.2(18)
C1	N2	N2[9]	58.1(8)
N1[9]	C1	N1	63.9(16)
N1[9]	C1	C1[8]	119.4(15)
N1	C1	C1[8]	119.4(15)
N2	C1	N1[9]	87.4(12)
N2	C1	N1	120.9(16)
N2[9]	C1	N1[9]	120.9(16)
N2[9]	C1	N1	87.4(12)
N2	C1	N2[9]	63.7(16)
N2[9]	C1	C1[8]	119.7(15)
N2	C1	C1[8]	119.7(15)

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[1]1-Y,+X,+Z; [2]1-X,1-Y,1-Z; [3]+Y,1-X,2-Z; [4]1-X,1-Y,2-Z; [5]+Y,1-X,+Z; [6]1-X,1-Y,+Z; [7]+X,+Y,2-Z; [8]1-X,-Y,1-Z; [9]+X,+Y,1-Z

Table 6. Torsion Angles in ° for 10.

Atom	Atom	Atom	Atom	Angle/°
O1[1]	O3	N2	O4	-177.7(18)
O1[1]	O3	N2	N2[2]	-68(2)
O1[1]	O3	N2	C1	-3(3)
O1	N1	C1	N1[2]	-108.2(15)
O1	N1	C1	N2[2]	125.3(17)
O1	N1	C1	N2	-177.6(15)
O1	N1	C1	C1[1]	2(2)
O2[2]	O2	N1	O1	123.1(18)
O2[2]	O2	N1	N1[2]	-0.001(3)
O2[2]	O2	N1	C1	-51.0(16)
O2	N1	C1	N1[2]	66(2)
O2	N1	C1	N2[2]	-60(2)
O2	N1	C1	N2	-3(2)
O2	N1	C1	C1[1]	176.8(16)
O3[1]	O1	N1	O2	-177.0(18)
O3[1]	O1	N1	N1[2]	-68(2)
O3[1]	O1	N1	C1	-2(3)
O3	N2	C1	N1[2]	125.3(17)
O3	N2	C1	N1	-177.5(15)
O3	N2	C1	N2[2]	-108.2(14)
O3	N2	C1	C1[1]	3(2)
O4[2]	O4	N2	O3	123.2(17)
O4[2]	O4	N2	N2[2]	-0.001(4)
O4[2]	O4	N2	C1	-51.1(16)
O4	N2	C1	N1[2]	-60(2)
O4	N2	C1	N1	-3(2)
O4	N2	C1	N2[2]	66(2)
O4	N2	C1	C1[1]	177.2(16)
N1[2]	N1	C1	N2[2]	-126.5(12)
N1[2]	N1	C1	N2	-69.4(12)
N1[2]	N1	C1	C1[1]	110.5(14)
N2[2]	N2	C1	N1	-69.3(12)
N2[2]	N2	C1	N1[2]	-126.6(12)
N2[2]	N2	C1	C1[1]	110.8(14)

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[1]1-X,-Y,1-Z; [2]+X,+Y,1-Z

Table 7. Hydrogen Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 10. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H3A	6415	3525	5863	25
H3B	6025	3099	7361	25
H3C	6923	3995	7273	25
H4A	7133	3315	9862	25
H4B	6794	3998	8516	25
H4C	6636	2738	8516	25

Table 8. Hydrogen Bond information for 10.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N3	H3A	O2[1]	0.91	2.22	2.98(3)	140.9
N3	H3A	O4[2]	0.91	2.29	2.98(3)	133.3
N3	H3B	O3[3]	0.91	2.15	3.05(2)	175.3
N3	H3C	O1[4]	0.91	2.16	3.06(2)	172.3

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[1]+X,+Y,1-Z; [2]1-Y,+X,+Z; [3]1/2+Y,1/2-X,1/2+Z; [4]3/2-X,1/2-Y,3/2-Z

Table 9. Atomic Occupancies for all atoms that are not fully occupied in 10.

Atom	Occupancy
Ag2	0.5
N3	0.5
H3A	0.5
H3B	0.5
H3C	0.5
N4	0.5
H4A	0.5
H4B	0.5
H4C	0.5
O1	0.5
O2	0.5
O3	0.5
O4	0.5
N1	0.5
N2	0.5

Table 10. Crystal data and structure refinement for 11.

Identification code	**11**
CCDC #	1847186
Empirical formula	$C_2H_8N_6O_8$
Formula weight	244.14
Temperature/K	130
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	9.2546(11)
b/Å	7.8000(9)
c/Å	13.1353(14)
α/°	90
β/°	107.923(3)
γ/°	90
Volume/Å^3	902.17(18)
Z	4
ρ_{calc}g/cm^3	1.797
μ/mm^{-1}	0.180
F(000)	504.0
Crystal size/mm^3	0.15 × 0.15 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.158 to 61.08
Index ranges	-13 ≤ h ≤ 13, -11 ≤ k ≤ 11, -18 ≤ l ≤ 18
Reflections collected	20104
Independent reflections	2763 [R_{int} = 0.0391, R_{sigma} = 0.0282]
Data/restraints/parameters	2763/0/177
Goodness-of-fit on F^2	1.067
Final R indexes [I>=2σ (I)]	R_1 = 0.0392, wR_2 = 0.0826
Final R indexes [all data]	R_1 = 0.0589, wR_2 = 0.0897
Largest diff. peak/hole / e Å^{-3}	0.37/-0.30

Table 11. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 11. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O1	6978.2(10)	220.4(11)	3517.6(7)	16.5(2)
O2	4713.7(10)	1113.8(13)	3396.1(7)	20.0(2)
O3	4461.9(9)	2264.8(12)	5183.5(6)	16.6(2)
O4	6552.1(10)	3188.6(11)	6342.9(6)	13.94(18)
O5	8403.6(10)	151.6(11)	6586.7(7)	17.7(2)
O6	10473.5(10)	1583.6(12)	6770.9(7)	18.1(2)
O7	10621.1(9)	2782.4(13)	4953.2(7)	19.2(2)
O8	8441.1(10)	3493.7(11)	3814.2(6)	14.44(19)
N1	6084.8(11)	1031.9(13)	3869.2(8)	11.9(2)
N2	5880.8(10)	2469.5(12)	5442.3(7)	10.17(19)
N3	9150.1(11)	1213.2(13)	6254.2(8)	11.4(2)
N4	9196.1(10)	2803.7(13)	4691.3(7)	10.6(2)
C1	6758.3(12)	1914.4(15)	4856.3(9)	10.2(2)
C2	8387.2(12)	2037.3(15)	5276.3(9)	10.7(2)
N6	6753.6(12)	6571.9(15)	3136.7(9)	13.4(2)
N5	2134.2(13)	3473.3(14)	3373.3(9)	13.5(2)

Table 12. Anisotropic Displacement Parameters ($Å^2×10^3$) for 11. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	20.5(4)	14.6(4)	18.5(4)	-5.4(3)	11.8(4)	-1.0(3)
O2	13.0(4)	30.2(5)	14.6(4)	-6.6(4)	0.8(3)	-2.4(4)
O3	9.2(4)	27.6(5)	14.1(4)	-0.8(4)	5.1(3)	0.9(3)
O4	19.9(4)	13.7(4)	8.3(4)	-3.4(3)	4.6(3)	-2.3(3)
O5	20.8(4)	15.4(4)	17.8(4)	6.3(3)	7.3(4)	-3.1(3)
O6	10.8(4)	27.2(5)	14.3(4)	5.5(4)	1.1(3)	-0.2(3)
O7	9.7(4)	32.5(5)	16.7(4)	5.3(4)	5.8(3)	-1.1(4)
O8	18.8(4)	14.5(4)	9.0(4)	3.4(3)	2.9(3)	0.1(3)
N1	14.2(5)	11.6(5)	11.1(5)	-1.4(4)	5.7(4)	-2.7(4)
N2	11.5(4)	10.2(4)	9.0(4)	0.7(4)	3.6(3)	-0.4(3)
N3	11.9(4)	11.8(5)	11.4(5)	1.4(4)	4.9(4)	1.9(4)
N4	11.7(4)	11.0(4)	9.4(4)	0.3(4)	3.7(4)	-0.3(4)
C1	10.5(5)	11.3(5)	9.1(5)	-0.9(4)	3.5(4)	-1.0(4)
C2	10.3(5)	12.4(5)	9.8(5)	1.8(4)	3.7(4)	-0.4(4)
N6	14.6(5)	13.7(5)	13.4(5)	-0.5(4)	6.4(4)	1.1(4)
N5	15.5(5)	13.6(5)	11.7(5)	0.2(4)	4.8(4)	-2.2(4)

Table 13. Bond Lengths for 11.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	N1	1.2380(12)	O8	N4	1.2687(12)
O2	N1	1.2309(13)	N1	C1	1.4304(14)
O3	N2	1.2612(12)	N2	C1	1.3506(14)
O4	N2	1.2837(12)	N3	C2	1.4153(14)
O5	N3	1.2407(13)	N4	C2	1.3646(14)
O6	N3	1.2380(12)	C1	C2	1.4403(16)
O7	N4	1.2565(12)			

Table 14. Bond Angles for 11.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	N1	C1	115.43(9)	O7	N4	O8	119.51(9)
O2	N1	O1	122.77(10)	O7	N4	C2	123.47(9)
O2	N1	C1	121.78(10)	O8	N4	C2	116.93(9)
O3	N2	O4	118.17(9)	N1	C1	C2	119.47(10)
O3	N2	C1	124.37(10)	N2	C1	N1	120.01(10)
O4	N2	C1	117.39(9)	N2	C1	C2	120.16(10)
O5	N3	C2	116.51(10)	N3	C2	C1	119.23(10)
O6	N3	O5	121.79(10)	N4	C2	N3	120.21(10)
O6	N3	C2	121.64(10)	N4	C2	C1	120.29(10)

Table 15. Hydrogen Bonds for 11.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N6	H6A	O3[1]	0.897(18)	2.035(19)	2.9129(14)	165.8(15)
N6	H6B	O8[2]	0.881(18)	2.060(18)	2.9261(14)	167.4(15)
N6	H6C	O6[3]	0.90(2)	2.270(19)	2.9118(14)	127.6(15)
N6	H6C	O7[3]	0.90(2)	2.166(19)	2.9496(14)	144.6(16)
N6	H6D	O6[4]	0.853(19)	2.254(19)	3.0613(15)	157.8(16)
N5	H5A	O4[1]	0.903(18)	1.961(19)	2.8491(14)	167.3(15)
N5	H5B	O7[5]	0.915(17)	2.016(18)	2.8890(14)	159.0(15)
N5	H5C	O4[4]	0.871(17)	2.022(17)	2.8634(14)	161.9(15)
N5	H5D	O2	0.873(19)	2.254(19)	3.0072(15)	144.4(15)
N5	H5D	O3	0.873(19)	2.091(18)	2.8337(14)	142.5(15)

[1]1-X,1-Y,1-Z; [2]3/2-X,1/2+Y,1/2-Z; [3]2-X,1-Y,1-Z; [4]-1/2+X,1/2-Y,-1/2+Z; [5]-1+X,+Y,+Z

Table 16. Torsion Angles for 11.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	N1	C1	N2	-163.40(10)	O6	N3	C2	N4	22.66(16)
O1	N1	C1	C2	9.74(15)	O6	N3	C2	C1	-163.32(10)
O2	N1	C1	N2	18.15(16)	O7	N4	C2	N3	2.35(17)

O2 N1 C1 C2 -168.70(10)	O7 N4 C2 C1 -171.61(10)
O3 N2 C1 N1 0.13(17)	O8 N4 C2 N3 178.85(10)
O3 N2 C1 C2 -172.97(10)	O8 N4 C2 C1 4.90(16)
O4 N2 C1 N1 177.10(9)	N1 C1 C2 N3 -114.93(12)
O4 N2 C1 C2 4.00(15)	N1 C1 C2 N4 59.08(15)
O5 N3 C2 N4 -160.14(10)	N2 C1 C2 N3 58.21(15)
O5 N3 C2 C1 13.88(15)	N2 C1 C2 N4 -127.78(12)

Table 17. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 11.

Atom	*x*	*y*	*z*	U(eq)
H6A	6220(20)	6880(20)	3576(14)	33(5)
H6B	6617(18)	7270(20)	2587(13)	27(4)
H6C	7750(20)	6620(20)	3509(15)	37(5)
H6D	6547(19)	5550(30)	2913(14)	32(5)
H5A	2413(19)	4590(20)	3398(13)	29(4)
H5B	1443(19)	3340(20)	3737(13)	28(4)
H5C	1778(18)	3110(20)	2716(14)	23(4)
H5D	2950(20)	2880(20)	3684(14)	34(5)

Table 18. Crystal data and structure refinement for 12.

CCDC #	1847185
Formula	$C_2H_9N_5O_7$
$D_{calc.}$/ g cm^{-3}	1.815
μ/mm^{-1}	0.180
Formula Weight	215.14
Color	yellow
Shape	needle
Size/mm^3	0.40×0.13×0.08
T/K	173(2)
Crystal System	triclinic
Space Group	P-1
a/Å	3.7131(15)
b/Å	9.097(4)
c/Å	12.626(5)
α/°	108.745(7)
β/°	96.500(5)
γ/°	98.409(5)
V/Å^3	393.6(3)
Z	2
Z'	1
Wavelength/Å	0.710730
Radiation type	MoK□
Θ_{min}/°	1.729
Θ_{max}/°	25.327
Measured Refl.	6578
Independent Refl.	1451
Reflections Used	1169
R_{int}	0.0287
Parameters	163
Restraints	0
Largest Peak	0.301
Deepest Hole	-0.233
GooF	1.118
wR_2 (all data)	0.1123
wR_2	0.1024
R_1 (all data)	0.0477
R_1	0.0383

Table 19. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 12. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
O1	7822(4)	3247.0(17)	-851.9(11)	25.9(4)
O2	9204(4)	5483.6(15)	2488.6(11)	28.3(4)
O3	6778(4)	4638.9(15)	3720(1)	27.5(4)
O4	2051(4)	2049.1(16)	2928.8(11)	29.5(4)
O5	2132(5)	582.7(17)	1212.2(12)	43.3(5)
N1	7923(4)	3821.9(18)	331.3(12)	21.8(4)
N2	7146(4)	4435.8(17)	2712.0(12)	20.3(4)
N3	3158(4)	1860.9(18)	2008.7(13)	23.0(4)
C1	5985(5)	2815(2)	651.9(16)	20.8(4)
C2	5495(5)	3079(2)	1813.3(15)	19.3(4)
O6	1264(4)	8517.9(15)	5001.1(11)	26.6(4)
O7	4352(4)	10843.5(16)	6641.9(12)	30.8(4)
N4	3525(5)	7371.5(19)	4827.6(15)	22.9(4)
N5	1838(5)	11233(2)	7384.2(16)	26.2(4)

Table 20. Anisotropic Displacement Parameters (×10^4) 12. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	32.7(8)	23.6(7)	15.6(7)	2.9(6)	5.6(6)	-4.6(6)
O2	36.5(8)	21.6(7)	22.2(7)	6.0(6)	5.1(6)	-4.8(6)
O3	39.1(8)	24.9(7)	15.4(7)	4.0(5)	6.7(6)	2.3(6)
O4	33.9(8)	32.0(8)	24.4(8)	13.0(6)	8.7(6)	1.0(6)
O5	63.5(11)	25.8(8)	26.9(8)	0.9(6)	9.5(7)	-16.7(7)
N1	24.0(8)	23.3(8)	15.5(8)	3.8(6)	3.8(6)	3.0(6)
N2	22.5(8)	19.5(8)	18.8(8)	6.4(6)	2.8(6)	4.5(6)
N3	24.1(8)	22.9(8)	22.3(9)	9.6(7)	2.6(7)	2.8(6)
C1	21.4(9)	18.2(9)	20.8(10)	5.0(8)	1.7(8)	2.6(7)
C2	20.6(9)	18.3(9)	19.1(9)	7.5(7)	2.2(7)	2.6(7)
O6	23.1(7)	24.9(7)	27.9(8)	3.6(6)	3.1(6)	6.0(6)
O7	23.3(7)	34.9(8)	24.6(8)	-1.5(6)	2.4(6)	3.5(6)
N4	25.2(9)	21.2(8)	21.4(9)	7.7(7)	1.8(7)	2.7(7)
N5	23.2(9)	22.6(9)	30.7(10)	7.3(8)	4.8(8)	2.6(7)

Table 21. Bond Lengths in Å for 12.

Atom	Atom	Length/Å
O1	N1	1.409(2)
O2	N2	1.2571(19)
O3	N2	1.252(2)
O4	N3	1.244(2)
O5	N3	1.241(2)
N1	C1	1.277(2)
N2	C2	1.381(2)
N3	C2	1.409(2)
C1	C2	1.445(3)
O6	N4	1.411(2)
O7	N5	1.399(2)

Table 22. Bond Angles in ° for 12.

Atom	Atom	Atom	Angle/°
C1	N1	O1	111.18(15)
O2	N2	C2	116.95(15)
O3	N2	O2	119.55(14)
O3	N2	C2	123.46(15)
O4	N3	C2	122.20(15)
O5	N3	O4	120.27(15)
O5	N3	C2	117.50(15)
N1	C1	C2	124.06(17)
N2	C2	N3	119.67(15)
N2	C2	C1	123.88(16)
N3	C2	C1	116.45(15)

Table 23. Torsion Angles in ° for 12.

Atom	Atom	Atom	Atom	Angle/°
O1	N1	C1	C2	-178.76(15)
O2	N2	C2	N3	180.00(14)
O2	N2	C2	C1	-0.5(2)
O3	N2	C2	N3	2.3(3)
O3	N2	C2	C1	-178.21(16)
O4	N3	C2	N2	11.4(2)
O4	N3	C2	C1	-168.17(16)
O5	N3	C2	N2	-170.51(17)
O5	N3	C2	C1	9.9(2)
N1	C1	C2	N2	-1.7(3)
N1	C1	C2	N3	177.86(16)

Table 24. Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 12. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1	9060(70)	3950(30)	-950(20)	39(7)
H1A	4740(60)	1800(30)	116(18)	28(5)
H4A	5950(80)	7760(30)	4750(20)	46(7)
H4B	2500(80)	6520(40)	4160(30)	68(9)
H4C	3650(70)	6990(30)	5390(20)	44(7)
H5A	3020(80)	12160(30)	8000(20)	53(7)
H5B	-160(90)	11380(30)	7050(20)	63(9)
H5C	1120(70)	10500(30)	7610(20)	45(8)
H6	2770(90)	9650(40)	5830(30)	85(10)

Table 25. Hydrogen Bond information for **12**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O1	H1	N1[1]	0.78(3)	2.02(3)	2.728(2)	149(2)
N4	H4A	O6[2]	0.95(3)	1.95(3)	2.868(2)	164(2)
N5	H5A	O1[3]	0.95(3)	2.06(3)	2.884(2)	145(2)
N5	H5B	O7[4]	0.86(3)	1.99(3)	2.764(3)	149(3)
O6	H6	O7	1.22(3)	1.24(3)	2.4531(19)	177(3)

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[1]2-X,1-Y,-Z; [2]1+X,+Y,+Z; [3]+X,1+Y,1+Z; [4]-1+X,+Y,+Z

Table 26. Crystal data and structure refinement for 14.

Formula	$C_4H_{15}N_9O_{12}$
CCDC #	1847184
$D_{calc.}$/ g cm^{-3}	1.784
□/mm^{-1}	0.175
Formula Weight	381.25
Color	yellow
Shape	chunk
Size/mm^3	0.30×0.25×0.19
T/K	173(2)
Crystal System	triclinic
Space Group	*P*-1
a/Å	8.1131(10)
b/Å	8.6292(10)
c/Å	11.1334(13)
α/°	106.9700(10)
β/°	93.5300(10)
γ/°	105.5540(10)
V/Å^3	709.89(15)
Z	2
Z'	1
Wavelength/Å	0.710730
Radiation type	MoK□
Θ_{min}/°	1.935
Θ_{max}/°	25.388
Measured Refl.	5829
Independent Refl.	2596
Reflections Used	2144
R_{int}	0.0201
Parameters	286
Restraints	0
Largest Peak	0.414
Deepest Hole	-0.292
GooF	1.078
wR_2 (all data)	0.1369
wR_2	0.1253
R_1 (all data)	0.0532
R_1	0.0453

Table 27. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
O1	8675.5(19)	6915.3(18)	6423.5(14)	24.4(4)
O2	7859.5(18)	10652.2(18)	5181.9(13)	25.2(4)
O3	9261.3(19)	13326.4(17)	5963.6(14)	26.8(4)
O4	11483.5(18)	14225.3(17)	7930.2(13)	24.0(4)
O5	12082(2)	12245.3(19)	8516.0(15)	34.3(4)
N1	8561(2)	8361(2)	6101.8(16)	20.6(4)
N2	9012(2)	11835(2)	5976.2(15)	19.7(4)
N3	11228(2)	12688(2)	7788.0(16)	20.6(4)
C1	9680(3)	9687(3)	6859.5(19)	19.7(4)
C2	9968(2)	11407(2)	6840.5(18)	18.8(4)
O6	4668.4(19)	6331.1(19)	9321.6(14)	25.4(4)
O7	4849(2)	9351.3(19)	7134.9(14)	31.5(4)
O8	6486.5(18)	11930.8(17)	7605.6(13)	24.7(4)
O9	8845.2(18)	12982.8(18)	9535.3(14)	25.5(4)
O10	8694(2)	11428(2)	10743.9(16)	44.9(5)
N4	4837(2)	7545(2)	8674.3(16)	21.8(4)
N5	6016(2)	10575(2)	7858.6(16)	20.0(4)
N6	8147(2)	11646(2)	9754.7(16)	22.9(4)
C3	6140(3)	8831(3)	9270.0(19)	21.9(5)
C4	6758(2)	10351(2)	8931.8(18)	18.5(4)
O11	5292(2)	6058.4(19)	3354.6(13)	27.4(4)
N7	5285(2)	7466(2)	4404.3(16)	20.7(4)
O12	3200.1(17)	5549.8(16)	5571.6(12)	21.0(3)
N8	2184(2)	6340(2)	6386.1(17)	20.5(4)
N9	7885(3)	5515(2)	8641.2(19)	25.7(4)

Table 28. Anisotropic Displacement Parameters (×10^4) 14. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	30.4(8)	15.8(7)	25.5(8)	7.7(6)	-3.3(6)	5.0(6)
O2	25.3(8)	21.2(8)	23.3(8)	6.1(6)	-7.5(6)	1.3(6)
O3	29.5(8)	18.3(8)	31.9(9)	12.2(6)	-5.1(6)	3.2(6)
O4	27.6(8)	15.6(7)	24.6(8)	6.3(6)	-1.1(6)	1.0(6)
O5	37.9(9)	23.3(8)	35.3(9)	10.7(7)	-18.1(7)	2.6(7)
N1	23.3(9)	18.8(9)	21.3(9)	8.5(7)	1.6(7)	7.2(7)
N2	18.7(8)	19.9(9)	19.8(9)	7.4(7)	0.3(7)	4.1(7)
N3	21.1(9)	20.2(9)	20.0(9)	7.9(7)	0.0(7)	4.3(7)
C1	19.9(10)	21.3(11)	18.9(10)	7.0(8)	-0.4(8)	7.9(8)
C2	19.1(10)	18.6(10)	18.6(10)	6.5(8)	0.6(8)	5.5(8)
O6	25.7(8)	20.8(8)	28.5(8)	13.5(7)	-3.0(7)	0.2(6)
O7	33.6(9)	23.8(8)	25.8(8)	8.7(7)	-12.5(7)	-6.6(7)
O8	29.9(8)	19.0(8)	25.6(8)	11.7(6)	-1.1(6)	4.2(6)
O9	25.3(8)	20.1(8)	27.8(8)	11.2(6)	-1.5(6)	-1.3(6)
O10	47.7(11)	37.6(10)	37.6(10)	23.0(8)	-23.8(8)	-11.8(8)
N4	24.1(9)	17.9(9)	25.1(9)	10.1(7)	2.3(7)	5.6(7)
N5	20.7(9)	19.8(9)	18.8(9)	6.1(7)	1.4(7)	5.3(7)
N6	22.8(9)	22.9(9)	21.9(9)	8.8(7)	-1.5(7)	4.1(7)
C3	23.7(11)	23.2(11)	20.0(11)	7.3(9)	-0.1(8)	8.9(9)
C4	20.4(10)	18.2(10)	14.5(10)	4.4(8)	-1.3(8)	3.6(8)
O11	36.1(9)	28.9(8)	18.9(8)	4.5(6)	-0.6(6)	17.1(7)
N7	21.6(9)	18.8(9)	19.4(9)	4.4(7)	-1.1(7)	5.2(7)
O12	23.1(7)	18.6(7)	21.4(8)	5.2(6)	3.1(6)	7.8(6)
N8	19.1(9)	20.0(9)	23.0(10)	8.3(8)	1.8(8)	5.6(7)
N9	27.2(10)	19.7(10)	27.0(10)	6.6(8)	-3.6(8)	4.4(8)

Table 29. Bond Lengths in Å for 14.

Atom	Atom	Length/Å
O1	N1	1.418(2)
O2	N2	1.248(2)
O3	N2	1.252(2)
O4	N3	1.247(2)
O5	N3	1.240(2)
N1	C1	1.277(3)
N2	C2	1.391(3)
N3	C2	1.400(3)

Atom	Atom	Length/Å
C1	C2	1.446(3)
O6	N4	1.418(2)
O7	N5	1.238(2)
O8	N5	1.249(2)
O9	N6	1.242(2)
O10	N6	1.245(2)
N4	C3	1.282(3)
N5	C4	1.391(3)
N6	C4	1.399(3)
C3	C4	1.439(3)
O11	N7	1.420(2)
O12	N8	1.414(2)

Table 30. Bond Angles in ° for 14.

Atom	Atom	Atom	Angle/°
C1	N1	O1	109.44(16)
O2	N2	O3	119.69(16)
O2	N2	C2	117.10(16)
O3	N2	C2	123.22(17)
O4	N3	C2	122.49(16)
O5	N3	O4	120.03(16)
O5	N3	C2	117.47(16)
N1	C1	C2	126.19(19)
N2	C2	N3	119.66(17)
N2	C2	C1	123.33(18)
N3	C2	C1	116.94(17)
C3	N4	O6	108.05(16)
O7	N5	O8	120.02(16)
O7	N5	C4	117.12(16)
O8	N5	C4	122.86(16)
O9	N6	O10	119.38(17)
O9	N6	C4	122.80(16)
O10	N6	C4	117.83(17)
N4	C3	C4	126.07(18)
N5	C4	N6	120.25(17)
N5	C4	C3	122.93(17)
N6	C4	C3	116.81(17)

Table 31. Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1	8020(30)	6050(30)	5770(30)	42(7)
H1A	10370(30)	9620(30)	7470(20)	27(6)
H6	3740(30)	5470(30)	8850(20)	32(7)
H3	6830(30)	8840(30)	10000(20)	27(6)
H11	5790(40)	5510(30)	3780(20)	41(7)
H7A	6450(40)	8080(30)	4900(20)	43(7)
H7B	4830(30)	8130(30)	3980(20)	32(6)
H7C	4500(30)	7000(30)	4940(20)	40(7)
H8A	1560(30)	6820(30)	5940(20)	27(6)
H8B	1350(30)	5510(30)	6600(20)	35(7)
H8C	2940(30)	7100(30)	7080(20)	30(6)
H9A	7800(40)	4340(40)	8450(30)	55(9)
H9B	7030(50)	5740(40)	9020(30)	84(12)
H9C	8930(50)	6140(50)	9200(30)	79(11)
H9D	7910(40)	5860(40)	7900(30)	60(9)

Table 32. Hydrogen Bond information for 14.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O1	H1	O12[1]	0.89(3)	1.73(3)	2.620(2)	176(3)
O6	H6	O4[2]	0.91(3)	1.90(3)	2.779(2)	162(2)
O11	H11	O12[1]	0.90(3)	1.66(3)	2.5595(19)	175(3)
N7	H7A	O2	0.99(3)	2.12(3)	2.825(2)	127(2)
N7	H7B	O8[3]	0.97(3)	1.98(3)	2.834(2)	145(2)
N7	H7C	O12	0.99(3)	1.75(3)	2.703(2)	161(2)
N8	H8B	O4[2]	0.94(3)	2.11(3)	2.829(2)	132(2)
N8	H8C	O7	0.91(3)	2.12(2)	2.761(2)	126.5(19)
N9	H9A	O8[4]	0.96(3)	1.97(3)	2.838(2)	149(3)

[1]1-x,1-y,1-z; [2]-1+x,-1+y,+z; [3]1-x,2-y,1-z; [4]+x,-1+y,+z