Supporting Information:

Formamidinium Nitroformate: An Insensitive RDX Alternative

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Experimental Details

Caution! The compounds described herein are energetic materials that can explode under certain conditions (e.g., elevated temperatures, impact, friction, or electric discharge). Furthermore, the combinations of these compounds with organic solvents can be potentially hazardous. Although the authors did not experience any explosions during this work, nitroform and its metal salts are known explosives. Therefore, appropriate safety precautions such as the use of blast shields in a well-ventilated fume hood and personal protective equipment (safety glasses, face shields, ear plugs, as well as gloves and suits made from leather and/or Kevlar), should be taken at all times when handling these materials.

Materials and apparatus: All chemicals and solvents were obtained from commercial sources and were used as received. NMR spectra were recorded at 298 K on a Bruker AMX500 or a Varian NMRS-500 spectrometer. Chemical shifts are given relative to neat tetramethylsilane (^{1}H, ^{13}C) or neat CH_3NO_2 (^{14}N). Raman spectra were recorded at ambient temperature in Pyrex glass tubes in the range 4000−80 cm^{-1} on a Bruker Optics Vertex 70/RAM II FT-RA spectrometer using a Nd:YAG laser at 1064 nm. Infrared spectra were recorded in the range 4000−400 cm^{-1} on a Bruker Optics Alpha FT-IR spectrometer. Differential thermal analysis (DTA) curves were recorded with a purge of dry nitrogen gas on an OZM Research DTA552-Ex instrument with a heating rate of 5°C/min with Meavy 2.2.0 software. The impact and friction sensitivity data were determined on an OZM Research BAM fall hammer BFH-10 and an OZM Research BAM friction apparatus FSKM-10, respectively, through five individual measurements that were averaged. Both instruments were calibrated with RDX.

Synthesis of nitroform $HC(NO_2)_3$:[1-2] 4.010 g (35.8 mmol) 4,6-dihydroxypyrimidine was added to concentrated sulfuric acid (20 mL) in portions with stirring. When the solid dissolved, the solution was cooled with an ice bath and concentrated nitric acid (6.0 mL) was added very slowly over the course of 15 minutes with stirring. When the addition was complete, the reaction mixture was allowed to slowly come to room temperature as the ice bath melted and was stirred for 20 h. After this time, the reaction mixture became pale yellow in color and was poured onto 150 mL of ice water. Nitroform was extracted with dichloromethane (3 x 25mL) and dried with $MgSO_4$. At this point, the crude nitroform solution was stored at 0°C and its isolation performed in smaller batches so that smaller amounts of solid potassium nitroformate (KNF) could be handled more safely. 25 mL of the crude nitroform/dichloromethane solution was cooled in an ice bath and stirred while a saturated solution of KOH in ethanol was added dropwise. A yellow precipitate immediately formed. The addition was continued in this way until no more precipitate was formed. The yellow solid was carefully filtered off and washed with ether. Mass of KNF: 2.050 g (10.8 mmol). KNF was suspended in hexane (50 mL) and cooled in an ice bath. Concentrated H_2SO_4 (10 mL) was added dropwise with stirring. When the addition was complete, the solution was stirred further for 1 h. at 0°C. The hexane layer was separated, and nitroform extracted with hexane (3 x 15 mL). The combined hexane layers were cooled to -78°C, and a colorless precipitate formed. The hexane was carefully decanted off, and the residual hexane removed under dynamic vacuum at -78°C overnight leaving behind colorless nitroform crystals. Mass of the colorless

crystalline solid: 0.938 g (6.21 mmol). NMR: ^{14}N ($CDCl_3$) δ = -28.2 (s, $\Delta\upsilon_{1/2}$ = 11 Hz). IR (ATR) 3356 (s, br), 1594 (m), 1308 (w), 1165 (m), 1042 (m), 877 (w), 574 (w).

Synthesis of methylhydrazinium nitroformate $[CH_3NH_2NH_2][C(NO_2)_3]$ (1):[3] A solution of methylhydrazine (140 µL, 2.66 mmol) in 2 mL of dichloromethane was added dropwise to a solution of nitroform (2.62 mmol) in dichloromethane (5 mL) cooled with an ice bath under N_2 flow. When the addition was complete, the yellow solution was stirred at 0°C for 1.5 h. and then at ambient temperature for 1 h. The volatiles were then removed under dynamic vacuum at -20°C. Mass of the yellow solid: 507 mg; mass expected for 2.62 mmol $[CH_3NH_2NH_2][C(NO_2)_3]$ 524 mg. The yellow solid is deliquescent; however, the water can subsequently be removed under vacuum, recovering the yellow solid. This was also evident by the unchanging IR spectrum in air except for the increasing intensity of the water bands over time. **1** was dissolved in methanol which was removed *in vacuo* at -20°C affording yellow crystals suitable for X-ray diffraction. NMR: (CD_3NO_2) ^1H: δ = 3.14 (s, $[C\mathbf{H_3}NH_2NH_2]^+$); ^{13}C: δ = 149.5 (s, $[\mathbf{C}(NO_2)_3]^-$), 38.6 (s, $[\mathbf{C}H_3NH_2NH_2]^+$); (MeOH-d4) ^{14}N: δ = -30.6 (s, $\Delta\upsilon_{1/2}$ = 11 Hz, $[C(\mathbf{N}O_2)_3]$). Raman (25 mW) = 3040 (0.6), 2972 (1.5), 1621 (1.1), 1613 (1.1), 1555 (0.8), 1548 (1.0), 1469 (2.0), 1407 (3.9), 1353 (2.8), 1338 (3.1), 1273 (3.2), 1163 (2.3), 1152 (5.1), 998 (0.6), 941 (1.2), 892 (1.2), 870 (10.0), 839 (1.2), 790 (2.2), 471 (2.4), 430 (2.1), 420 (2.0), 386 (1.8), 374 (1.6), 290 (1.6), 249 (2.1), 236 (2.3). IR (ATR) $\bar{V}$ = 3335 (m), 3287 (w), 3118 (s), 2892 (w), 2785 (vw), 2522 (vw), 1679 (vw), 1617 (vw), 1594 (m), 1535 (s), 1475 (s), 1455 (vw), 1373 (s), 1246 (vs), 1134 (vs), 1084 (m), 1010 (m), 864 (s), 784 (vs), 729 (vs), 531 (w), 440 (w), 417 (m).

Synthesis of formamidinium nitroformate $[NH_2CHNH_2][C(NO_2)_3]$ (2): Formamidine acetate (55.0 mg, 0.528 mmol) was dissolved in methanol (2 mL). A solution of nitroform (93.0 mg, 0.616 mmol) in methanol (1 mL) was added dropwise at 0°C. The solution was warmed to ambient temperature and stirred for 30 minutes. The volatile compounds were removed *in vacuo* first at -20°C and then at ambient temperature affording yellow crystals suitable for X-ray diffraction. Mass of the yellow crystalline solid: 96 mg; mass expected for 0.528 mmol $[NH_2CHNH_2][C(NO_2)_3]$: 103 mg. NMR: (CD_3OD) ^1H: δ = 7.81 (s, $[NH_2C\mathbf{H}NH_2]^+$); ^{13}C: δ = 180.9 (s, $[NH_2\mathbf{C}HNH_2]^+$); ^{14}N: δ = -32.6 (s, $\Delta\upsilon_{1/2}$ = 12 Hz, $[C(\mathbf{N}O_2)_3]$). Raman (25 mW) = 3012 (0.3), 2936 (1.3), 1650 (0.3), 1548 (0.8), 1466 (0.4), 1394 (0.3), 1382 (5.0), 1353 (2.6), 1334 (0.5), 1280 (1.3), 1160 (1.1), 1150 (7.4), 1123 (1.2), 1047 (0.5), 917 (3.3), 871 (10.0), 787 (1.2), 715 (0.2), 653 (1.2), 612 (0.3), 571 (1.5), 546 (1.2), 477 (2.6), 431 (1.4), 420 (0.7). IR (ATR) = 3359 (vw), 3189 (vs), 3019 (vw), 2927 (vw), 2899 (vw), 2837 (vw), 2763 (w), 1716 (s), 1686 (w), 1540 (s), 1480 (m), 1385 (s), 1344 (s), 1258 (vs), 1141 (vs), 1068 (m), 1044 (w), 1011 (m), 918 (m), 824 (m), 783 (s), 725 (w), 649 (vs), 569 (s), 543 (m), 417 (w).

Synthesis of ethylenediammonium dinitroformate $[NH_3CH_2CH_2NH_3][C(NO_2)_3]_2$ (3): A solution of ethylenediamine (16.0 mg, 0.266 mmol) in methanol (1 mL) was added dropwise to a solution of nitroform (91.5 mg, 0.606 mmol) in methanol (5 mL) with stirring at 0°C. After the addition, the solution was stirred for an additional 30 minutes, and then at room temperature for 1 hour. The volatile compounds were removed under dynamic vacuum at -45°C until a constant mass was achieved affording crystals suitable for X-ray diffraction. When the volatile materials were removed at warmer temperatures, only the monocation was formed rather than the dication

even when a large excess of nitroform was used. NMR (CD_3OD) ^1H: δ = 2.68 (s, $[NH_3(C\mathbf{H_2})_2NH_3]^{2+}$), ^{13}C: δ = 36.5 (s, $[NH_3(\mathbf{C}H_2)_2NH_3]^{2+}$), ^{14}N: δ = -32.9 (s, $\Delta\upsilon_{1/2}$ = 15 Hz, $[C(\mathbf{N}O_2)_3]_2^-$), -355.8 (s, $\Delta\upsilon_{1/2}$ = 156 Hz, $[\mathbf{N}H_3(CH_2)_2\mathbf{N}H_3]^{2+}$). Raman (25 mW) = 3025 (0.1), 2995 (0.6), 2420 (0.3), 2319 (0.2), 2138 (0.1), 2083 (0.9), 1557 (0.7), 1488 (0.3), 1462 (0.7), 1388 (5.5), 1357 (1.5), 1340 (0.4), 1262 (1.5), 1157 (0.8), 1146 (3.3), 1132 (1.5), 1030 (0.1), 1009 (0.2), 971 (0.2), 870 (10.0), 789 (1.5), 750 (0.5), 735 (0.4), 677 (0.1), 482 (2.4), 465 (0.1), 428 (1.6), 421 (0.2). IR (ATR) = 3450 (w), 3249 (s), 3203 (w), 3068 (vs), 3033 (vw), 2942 (w), 2817 (w), 2661 (w), 2631 (vw), 2508 (m), 2411 (w), 1950 (m), 1719 (w), 1608 (s), 1542 (s), 1478 (m), 1452 (s), 1376 (s), 1271 (vs), 1145 (vs), 1059 (s), 1024 (m), 1008 (s), 941 (vw), 868 (s), 837 (m), 778 (vs), 732 (s), 672 (m), 625 (m), 565 (w), 539 (m), 482 (m), 416 (s).

Synthesis of methylammonium nitroformate $[CH_3NH_3][C(NO_2)_3]$ (4):[4] A solution of methylamine (0.50 mmol) in methanol (2 mL) was added to a solution of nitroform (0.20 mmol) in dichloromethane (1 mL) at 0°C. The solution was warmed to ambient temperature and stirred overnight. The volatile compounds were removed *in vacuo* from -40°C to ambient temperature affording yellow crystals suitable for X-ray diffraction. Mass of the yellow solid: 32 mg; mass expected for 0.20 mmol $[CH_3NH_3][C(NO_2)_3]$: 36 mg. NMR: (CD_3OD), ^1H: δ = 2.33 (s, $[C\mathbf{H_3}NH_3]^+$), ^{13}C: δ = 24.7 (s, $[\mathbf{C}H_3NH_3]^+$), ^{14}N: δ = -30.7 (s, $\Delta\upsilon_{1/2}$ = 11 Hz, $[C(\mathbf{N}O_2)_3]^-$); -364.6 (s, $\Delta\upsilon_{1/2}$ = 38 Hz, $[CH_3\mathbf{N}H_3]^+$). Raman (25 mW) = 3154 (0.1), 3053 (0.3), 2986 (1.1), 2912 (0.1), 2839 (0.2), 2538 (0.1), 1613 (0.2), 1529 (0.3), 1497 (0.1), 1468 (1.2), 1391 (6.2), 1323 (0.4), 1292 (1.7), 1260 (0.6), 1166 (0.9), 1146 (4.1), 990 (2.3), 941 (1.1), 873 (10.0), 789 (1.8), 734 (1.6), 469 (2.3), 431 (0.8), 416 (0.4). IR (ATR) = 3223 (w), 3191 (vs), 2860 (w), 2756 (vw), 2756 (vw), 2544 (m), 2435 (w), 1612 (m), 1531 (m), 1448 (s), 1424 (m), 1390 (s), 1238 (vs), 1139 (vs), 989 (s), 938 (s), 871 (s), 785 (vs), 731 (vs), 513 (m), 430 (w), 416 (vw).

Synthesis of dimethylammonium nitroformate $[(CH_3)_2NH_2][C(NO_2)_3]$ (5): Dimethylamine (0.305 mmol) was condensed onto a solution of nitroform (0.420 mmol) in dichloromethane (2 mL) at -196°C. The solution was allowed to warm from -40°C to -20°C for 1 h. A yellow precipitate formed, and the volatile materials were removed *in vacuo* from -20°C to 0°C. A yellow solid remained (55 mg, mass expected for 0.305 mmol $[(CH_3)_2NH_2][C(NO_2)_3]$ 60. mg). A small amount of the solid was again dissolved in methanol, cooled to -35°C and placed under vacuum affording crystals suitable for X-ray diffraction. NMR: (CD_3CN) ^1H: δ = 8.14 (br, $[(CH_3)_2N\mathbf{H_2}]^+$), 2.53 (t, $^3J_{HH}$ = 5.6 Hz, $[(C\mathbf{H_3})_2NH_2]^+$); (DMSO-d6) ^{13}C: δ = 35.3 (s, $[(\mathbf{C}H_3)_2NH_2]^+$); ^{14}N: δ = -29.6 (s, $\Delta\upsilon_{1/2}$ = 9 Hz, $[C(\mathbf{N}O_2)_3]^-$), -355.5 (t, $^1J_{NH}$ = 55.6 Hz, $\Delta\upsilon_{1/2}$ = 134 Hz, $[(CH_3)_2\mathbf{N}H_2]^+$). Raman (25 mW) = 3046 (1.0), 2977 (2.5), 2921 (0.2), 2858 (0.3), 2821 (0.4), 1605 (0.2), 1546 (0.9), 1463 (1.3), 1410 (0.9), 1390 (6.0), 1336 (1.5), 1271 (1.4), 1157 (3.9), 1143 (3.2), 1083 (0.3), 1045 (0.4), 1017 (0.3), 942 (0.3), 886 (3.4), 870 (10.0), 850 (1.1), 790 (1.1), 784 (0.6), 731 (0.2), 677 (0.2), 473 (2.4), 427 (1.4), 417 (1.0). IR (ATR) = 3469 (vw), 3170 (s), 2974 (w), 2811 (w), 2521 (vw), 2428 (m), 1730 (vw), 1590 (s), 1536 (s), 1487 (m), 1467 (s), 1438 (m), 1416 (m), 1387 (w), 1363 (m), 1334 (vw), 1267 (vs), 1247 (m), 1157 (s), 1143 (vs), 1080 (m), 1016 (s), 984 (w), 939 (m), 883 (m), 868 (s), 836 (s), 782 (vs), 727 (s), 625 (m), 570 (m), 524 (m), 471 (m), 417 (s).

X-ray Crystal Structure Determination: The single-crystal X-ray diffraction data were collected on a Bruker SMART APEX DUO 3-circle platform diffractometer, equipped with an APEX II CCD,

using Mo Kα radiation (TRIUMPH curved-crystal monochromator) from a fine-focus tube. The diffractometer was equipped with an Oxford Cryosystems Cryostream 700 apparatus for low-temperature data collection. The frames were integrated using the SAINT algorithm to give the hkl files corrected for Lp/decay.[5] The absorption correction was performed using the SADABS program.[6] Using Olex2[7] the structure was solved with the ShelXT[8] structure solution program using Intrinsic Phasing and refined with the ShelXL[9] refinement package using Least Squares minimization. All non-hydrogen atoms were refined anisotropically. ORTEP drawings were prepared using the CSD Mercury software.[10] Further crystallographic details for compounds 1-5 can be obtained from the Cambridge Crystallographic Data Centre [CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K.; fax (+44) 1223-336-033; e-mail deposit@ccdc.cam.ac.uk] on quoting the deposition numbers 1847171 - 1847175.

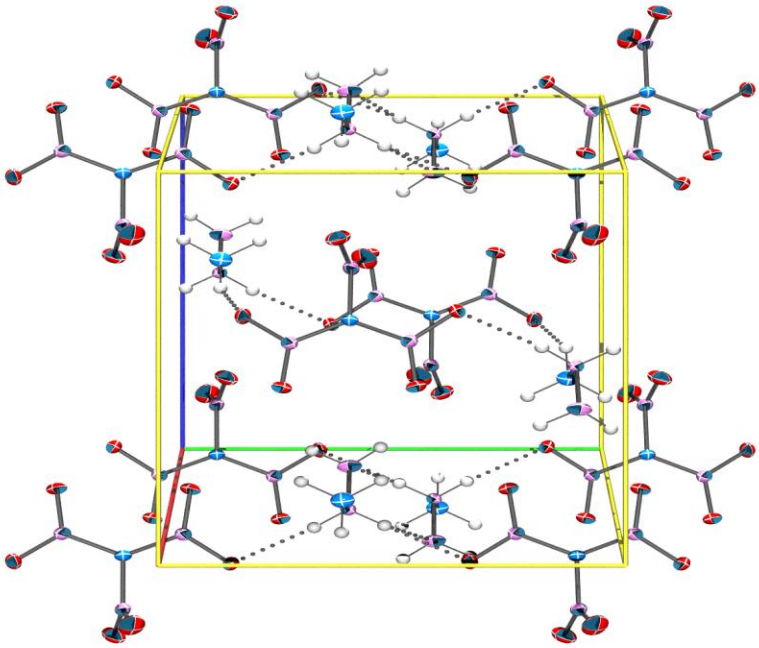

Figure S1. Unit cell of **1** normal to 100.

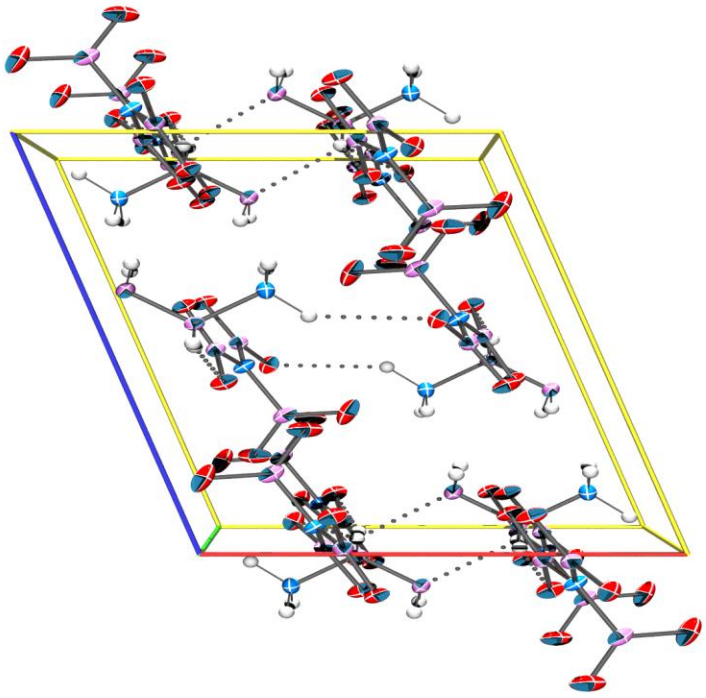

Figure S2. Unit cell of **1** normal to 010.

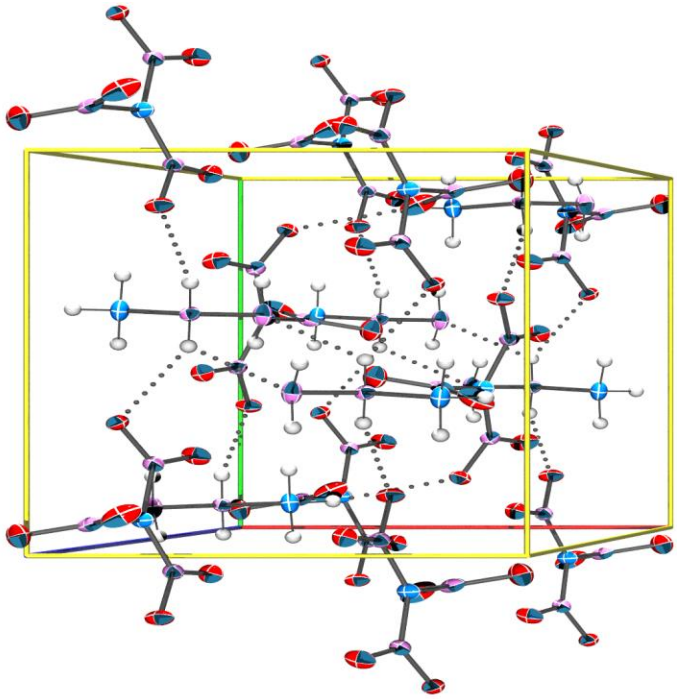

Figure S3. Unit cell of **1** normal to 001.

Table S1. Crystal data and structure refinement for 1.

Identification code	first_a
Empirical formula	$C_2H_7N_5O_6$
Formula weight	197.13
Temperature/K	100.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	8.5003(11)
b/Å	9.2107(12)
c/Å	10.3713(14)
α/°	90
β/°	108.348(2)
γ/°	90
Volume/Å^3	770.73(18)
Z	4
ρ_{calc}g/cm^3	1.699
μ/mm^{-1}	0.166
F(000)	408.0
Crystal size/mm^3	0.179 × 0.146 × 0.09
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.428 to 61.18
Index ranges	$-12 \leq h \leq 12$, $-13 \leq k \leq 13$, $-14 \leq l \leq 14$
Reflections collected	18851
Independent reflections	2358 [R_{int} = 0.0498, R_{sigma} = 0.0292]
Data/restraints/parameters	2358/0/131
Goodness-of-fit on F^2	1.037
Final R indexes [I>=2σ (I)]	R_1 = 0.0338, wR_2 = 0.0769
Final R indexes [all data]	R_1 = 0.0525, wR_2 = 0.0855
Largest diff. peak/hole / e Å^{-3}	0.38/-0.33

Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 1. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C2	7591.0(14)	4072.2(12)	5602.2(11)	14.8(2)
C1	6274.6(14)	8927.8(14)	3735.5(12)	18.3(2)
N1	8057.4(11)	9029.7(10)	4533.2(9)	12.23(18)
N2	9184.9(12)	9045.8(13)	3740.6(10)	18.4(2)
N4	7161.1(13)	4094.2(10)	6859.5(10)	17.2(2)
N3	8007.4(11)	5377.6(10)	5172.0(9)	13.45(19)
N5	7532.8(12)	2739(1)	4996.8(9)	13.60(19)
O5	7089(1)	1701.9(9)	5584.2(8)	17.02(17)
O6	7925.3(11)	2579.6(9)	3946.6(8)	20.68(19)
O1	8270.4(10)	5523.9(9)	4065.5(8)	17.72(18)
O2	8096.6(11)	6426.1(9)	5974.7(8)	18.60(18)
O4	5720.5(13)	4379.3(11)	6769.0(11)	31.6(2)
O3	8254.0(13)	3821.9(11)	7913.5(9)	28.5(2)

Table S3. Anisotropic Displacement Parameters ($Å^2×10^3$) for 1. 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}
C2	21.2(5)	13.3(5)	13.4(5)	-0.6(4)	10.4(4)	-0.3(4)
C1	12.2(5)	24.9(6)	18.3(5)	-1.3(4)	5.3(4)	-1.7(4)
N1	14.5(4)	11.4(4)	11.3(4)	0.2(3)	4.8(3)	-1.0(3)
N2	15.1(4)	27.4(6)	14.7(5)	-1.9(4)	7.3(4)	-1.5(4)
N4	26.1(5)	11.9(4)	17.8(5)	-3.5(4)	12.8(4)	-6.4(4)
N3	15.1(4)	12.6(4)	13.4(4)	1.1(3)	5.4(3)	1.0(3)
N5	15.0(4)	12.9(4)	13.3(4)	0.0(3)	5.0(3)	1.4(3)
O5	19.5(4)	12.0(4)	22.5(4)	0.4(3)	10.8(3)	-1.9(3)
O6	32.8(5)	18.4(4)	14.6(4)	-0.9(3)	12.9(4)	3.5(3)
O1	23.7(4)	18.7(4)	13.5(4)	3.0(3)	9.8(3)	0.7(3)
O2	29.0(4)	12.3(4)	16.8(4)	-2.6(3)	10.5(3)	-2.2(3)
O4	31.7(5)	30.0(5)	44.6(6)	-6.2(4)	28.4(5)	-0.9(4)
O3	41.0(6)	26.8(5)	14.5(4)	1.6(4)	4.3(4)	-13.1(4)

Table S4. Bond Lengths for 1.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C2	N4	1.4609(14)	N4	O3	1.2172(14)
C2	N3	1.3673(14)	N3	O1	1.2436(12)
C2	N5	1.3729(14)	N3	O2	1.2616(12)
C1	N1	1.4834(15)	N5	O5	1.2533(12)
N1	N2	1.4459(13)	N5	O6	1.2442(12)
N4	O4	1.2268(14)			

Table S5. Bond Angles for 1.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N3	C2	N4	116.25(9)	O1	N3	C2	122.45(9)
N3	C2	N5	127.81(9)	O1	N3	O2	122.35(9)
N5	C2	N4	115.94(9)	O2	N3	C2	115.19(9)
N2	N1	C1	115.29(9)	O5	N5	C2	115.63(9)
O4	N4	C2	117.31(10)	O6	N5	C2	121.66(9)
O3	N4	C2	117.65(10)	O6	N5	O5	122.70(9)
O3	N4	O4	125.03(11)				

Table S6. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 1.

Atom	*x*	*y*	*z*	U(eq)
H1A	5958.44	9768.7	3129.87	28
H1B	5610.55	8912.65	4355.3	28
H1C	6076.98	8034.76	3194.45	28
H1D	8240(17)	9834(16)	5014(14)	15
H1E	8308(17)	8271(16)	5090(14)	15
H2A	8776(19)	9745(18)	3116(15)	22
H2B	8970(19)	8211(18)	3262(16)	22

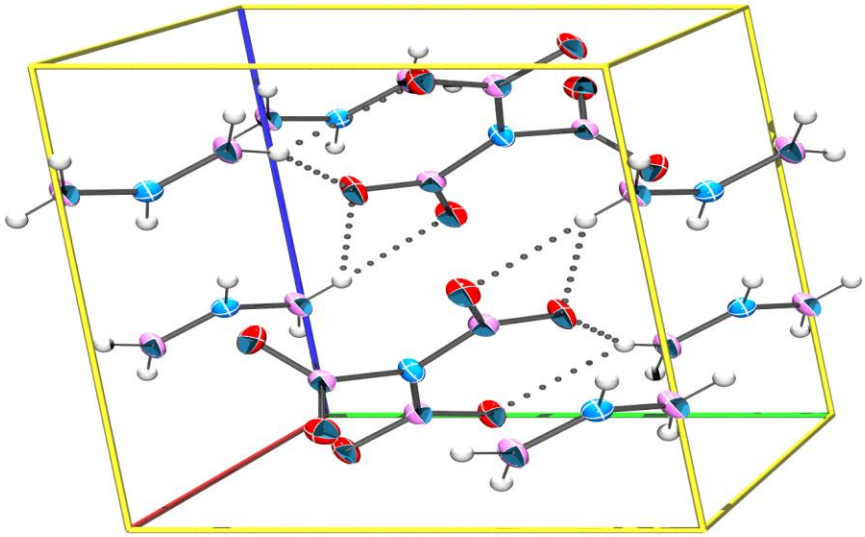

Figure S4. Unit cell of **2** normal to 100.

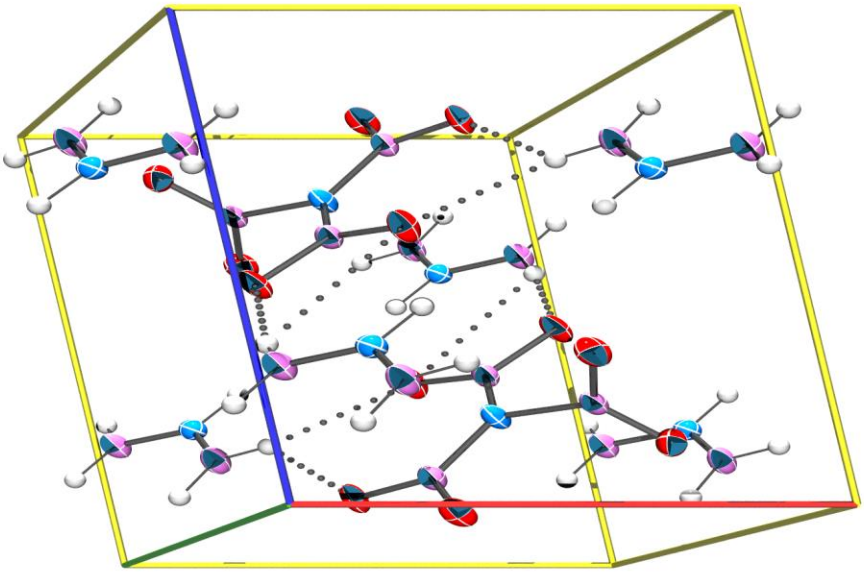

Figure S5. Unit cell of **2** normal to 010.

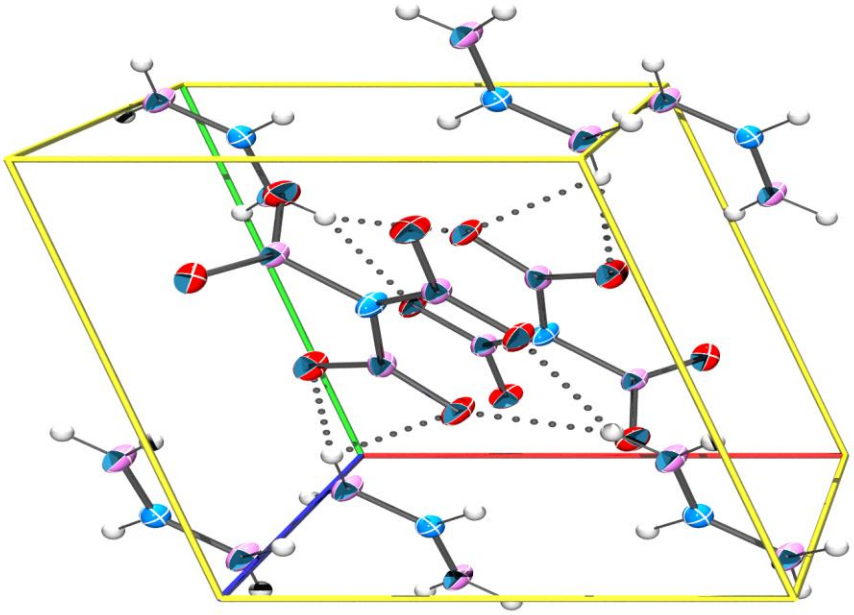

Figure S6. Unit cell of **2** normal to 001.

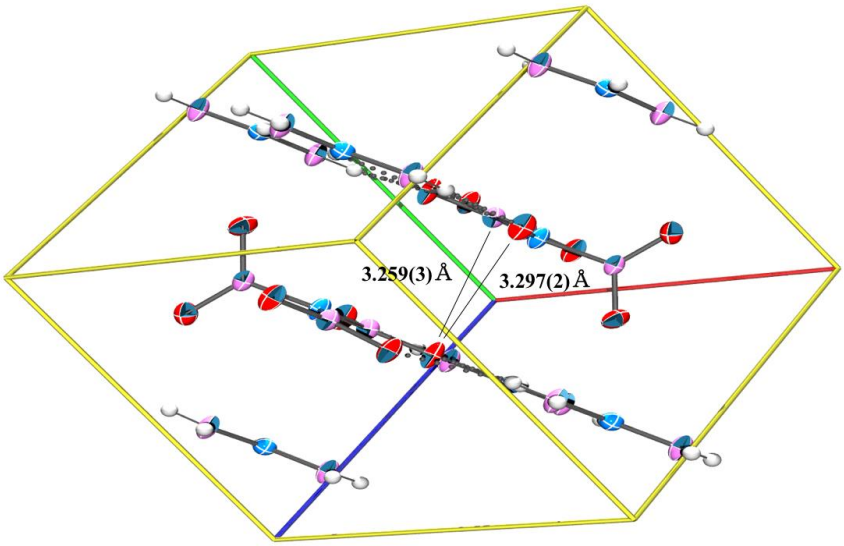

Figure S7. Unit cell of **2** showing the nearly planar layers.

Table S7. Crystal data and structure refinement for 2.

Identification code	IM04
Empirical formula	$C_2H_5N_5O_6$
Formula weight	195.11
Temperature/K	99.99
Crystal system	triclinic
Space group	P-1
a/Å	6.764(4)
b/Å	7.374(5)
c/Å	8.058(5)
α/°	98.976(11)
β/°	100.324(11)
γ/°	109.945(10)
Volume/Å^3	361.3(4)
Z	2
ρ_{calc}g/cm^3	1.794
μ/mm^{-1}	0.177
F(000)	200.0
Crystal size/mm^3	0.214 × 0.16 × 0.149
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.286 to 61.502
Index ranges	$-9 \leq h \leq 9$, $-10 \leq k \leq 10$, $-11 \leq l \leq 11$
Reflections collected	9189
Independent reflections	2204 [R_{int} = 0.0861, R_{sigma} = 0.0946]
Data/restraints/parameters	2204/0/138
Goodness-of-fit on F^2	0.967
Final R indexes [I>=2σ (I)]	R_1 = 0.0525, wR_2 = 0.0989
Final R indexes [all data]	R_1 = 0.1238, wR_2 = 0.1255
Largest diff. peak/hole / e Å^{-3}	0.30/-0.36

Table S8. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 2. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	4294(3)	6054(3)	7565(3)	17.6(5)
C2	1778(4)	9169(3)	3020(3)	16.4(5)
N1	6042(3)	6736(3)	8976(2)	17.0(4)
N2	2924(3)	7218(3)	7480(2)	15.8(4)
N3	3537(3)	4291(3)	6361(2)	17.0(4)
N4	433(3)	10077(3)	2804(3)	20.7(4)
N5	1496(3)	7484(3)	2050(3)	20.5(5)
O1	7191(2)	5750(2)	9272(2)	21.0(4)
O2	6406(3)	8383(2)	9937(2)	22.1(4)
O3	1494(2)	6851(2)	8245(2)	22.4(4)
O4	3305(3)	8487(2)	6639(2)	23.9(4)
O5	1792(2)	3930(2)	5261(2)	22.8(4)
O6	4517(2)	3141(2)	6329(2)	23.4(4)

Table S9. Anisotropic Displacement Parameters ($Å^2×10^3$) for 2. 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}
C1	17.8(10)	17.1(11)	16.4(11)	-0.8(9)	-2.8(9)	10.2(9)
C2	15.8(10)	18.0(12)	14.7(11)	4.3(9)	2.7(9)	6.0(9)
N1	14.8(9)	17.3(10)	17.2(10)	1.2(8)	-0.2(7)	7.3(8)
N2	15.4(9)	16.4(10)	14.0(9)	-0.5(8)	-1.4(7)	8.2(8)
N3	13.6(9)	18.6(10)	16(1)	0.7(8)	-0.1(7)	6.4(8)
N4	20.6(10)	18.9(11)	19.8(11)	-3.4(9)	-2.5(8)	11.4(9)
N5	19.9(10)	18.9(11)	21.7(11)	-1.4(9)	-0.9(8)	11.8(9)
O1	17.5(8)	20.6(9)	24.2(9)	1.0(7)	-2.9(6)	12.1(7)
O2	23.3(8)	16.6(9)	19.9(9)	-5.8(7)	-4.9(7)	9.2(7)
O3	20.1(8)	26.5(9)	23.0(9)	4.1(7)	5.5(7)	12.3(7)
O4	25.5(9)	22.8(9)	27.3(10)	10.8(8)	4.1(7)	13.2(7)
O5	18.8(8)	22.6(9)	20.2(9)	-3.7(7)	-7.5(7)	9.7(7)
O6	21.9(8)	18.8(9)	26.7(9)	-4.5(7)	-2.8(7)	13.1(7)

Table S10. Bond Lengths for 2.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	N1	1.370(3)	N1	O2	1.253(2)
C1	N2	1.462(3)	N2	O3	1.215(2)
C1	N3	1.367(3)	N2	O4	1.224(2)
C2	N4	1.304(3)	N3	O5	1.260(2)
C2	N5	1.294(3)	N3	O6	1.241(2)
N1	O1	1.250(2)			

Table S11. Bond Angles for 2.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N1	C1	N2	115.84(18)	O3	N2	C1	117.66(19)
N3	C1	N1	127.23(18)	O3	N2	O4	124.96(18)
N3	C1	N2	116.35(18)	O4	N2	C1	117.37(18)
N5	C2	N4	124.3(2)	O5	N3	C1	115.22(17)
O1	N1	C1	121.15(18)	O6	N3	C1	122.55(18)
O1	N1	O2	122.58(18)	O6	N3	O5	122.22(18)
O2	N1	C1	116.27(17)				

Table S12. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 2.

Atom	*x*	*y*	*z*	U(eq)
H4A	680(50)	11120(50)	3440(40)	44(9)
H4B	-740(50)	9530(40)	1920(40)	42(9)
H2	3110(40)	9860(30)	3950(30)	21(6)
H5A	2410(50)	6940(40)	2280(40)	38(8)
H5B	370(50)	6790(40)	1120(40)	45(9)

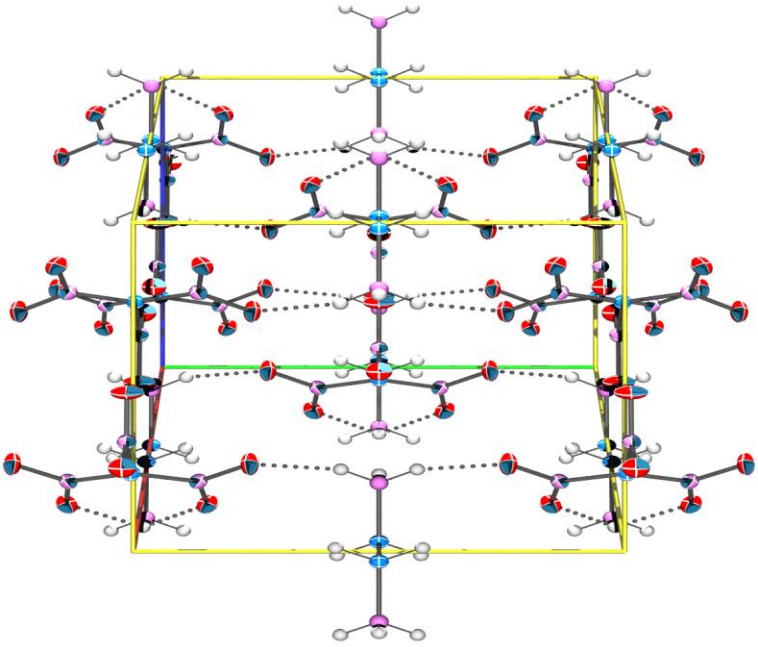

Figure S8. Unit cell of **3** normal to 100.

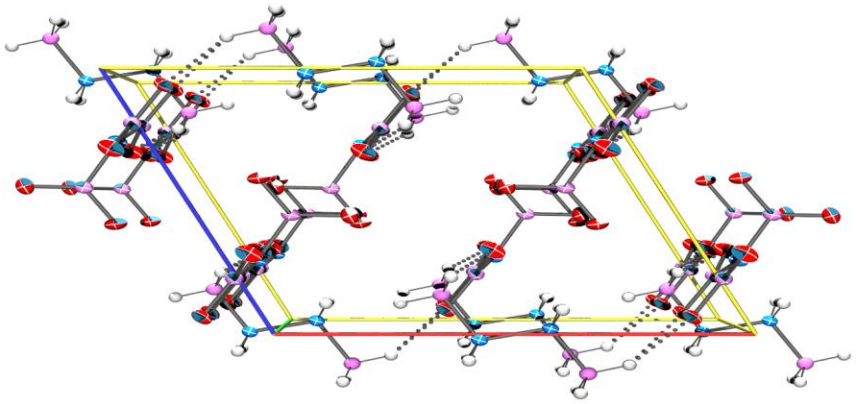

Figure S9. Unit cell of **3** normal to 010.

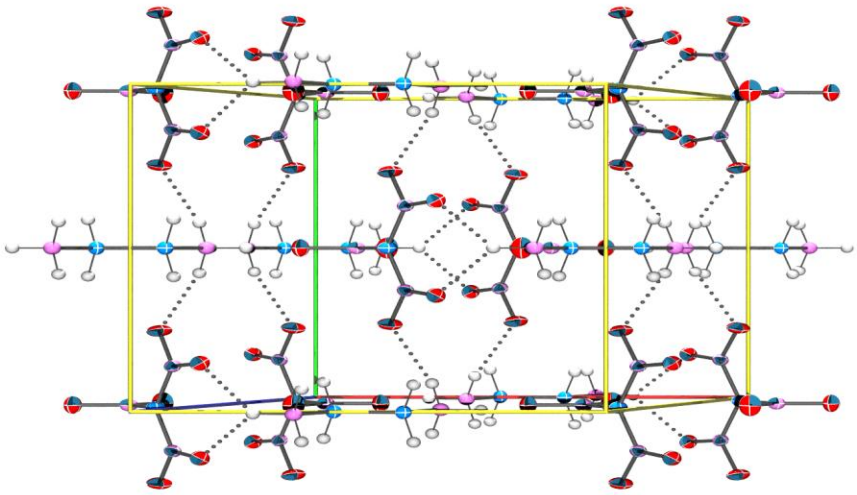

Figure S10. Unit cell of **3** normal to 001.

Table S13. Crystal data and structure refinement for 3.

Identification code	AB3_248_1
Empirical formula	$C_4H_{10}N_8O_{12}$
Formula weight	362.20
Temperature/K	100.0
Crystal system	monoclinic
Space group	C2/m
a/Å	10.023(4)
b/Å	9.240(3)
c/Å	8.250(3)
α/°	90
β/°	116.017(5)
γ/°	90
Volume/Å^3	686.6(4)
Z	2
ρ_{calc}g/cm^3	1.752
μ/mm^{-1}	0.174
F(000)	372.0
Crystal size/mm^3	0.2 × 0.15 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.494 to 61.076
Index ranges	$-14 \leq h \leq 13$, $-13 \leq k \leq 13$, $-11 \leq l \leq 11$
Reflections collected	8579
Independent reflections	1116 [R_{int} = 0.0533, R_{sigma} = 0.0312]
Data/restraints/parameters	1116/0/71
Goodness-of-fit on F^2	1.052
Final R indexes [I>=2σ (I)]	R_1 = 0.0327, wR_2 = 0.0843
Final R indexes [all data]	R_1 = 0.0386, wR_2 = 0.0880
Largest diff. peak/hole / e Å^{-3}	0.38/-0.33

Table S14. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 3. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	6161.1(8)	3542.8(8)	9280.7(10)	17.65(18)
O2	4308.1(9)	2605.4(8)	6926.4(11)	21.9(2)
O3	3203.5(13)	5000	4135.8(14)	25.3(3)
O4	1826.4(11)	5000	5552.1(15)	21.3(2)
N1	5985.2(14)	0	8514.8(16)	14.3(2)
N2	4992.7(9)	3676.9(9)	7862.8(11)	14.04(19)
N3	3034.1(13)	5000	5509.0(16)	14.1(2)
C1	5817.8(14)	0	10220.2(17)	12.9(3)
C2	4359.8(15)	5000	7240.8(18)	13.6(3)

Table S15. Anisotropic Displacement Parameters ($Å^2×10^3$) for 3. 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	12.8(3)	18.1(4)	16.4(4)	4.3(3)	1.3(3)	1.0(3)
O2	23.0(4)	11.6(4)	22.8(4)	-1.7(3)	2.2(3)	-2.7(3)
O3	23.7(6)	40.4(7)	11.1(5)	0	6.9(4)	0
O4	14.6(5)	26.7(6)	21.4(5)	0	6.7(4)	0
N1	15.1(5)	13.9(6)	13.9(5)	0	6.4(4)	0
N2	14.0(4)	13.0(4)	14.0(4)	1.3(3)	5.2(3)	-0.4(3)
N3	14.9(5)	12.9(5)	11.9(5)	0	3.5(4)	0
C1	11.9(6)	14.9(6)	10.6(6)	0	3.6(5)	0
C2	14.0(6)	12.5(6)	10.4(6)	0	1.8(5)	0

Table S16. Bond Lengths for 3.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	N2	1.2464(11)	N1	C1	1.4883(18)
O2	N2	1.2581(11)	N2	C2	1.3691(11)
O3	N3	1.2170(16)	N3	C2	1.4633(17)
O4	N3	1.2265(16)	C1	C1[1]	1.516(3)

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

Table S17. Bond Angles for 3.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	N2	O2	122.20(8)	O4	N3	C2	117.17(12)
O1	N2	C2	122.05(9)	N1	C1	C1[1]	109.39(13)
O2	N2	C2	115.74(9)	N2[2]	C2	N2	126.47(12)
O3	N3	O4	124.71(12)	N2	C2	N3	116.03(6)
O3	N3	C2	118.12(12)	N2[2]	C2	N3	116.03(6)

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

Table S18. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3.

Atom	*x*	*y*	*z*	U(eq)
H1C	6303.09	868.2	10941.29	16
H1D	6303.09	-868.2	10941.28	16
H1A	5553(16)	-781(16)	7839(19)	25(4)
H1B	6960(30)	0	8840(30)	44(7)

Table S19. Atomic Occupancy for 3.

Atom	*Occupancy*	Atom	*Occupancy*
H1C	0.5	H1D	0.5

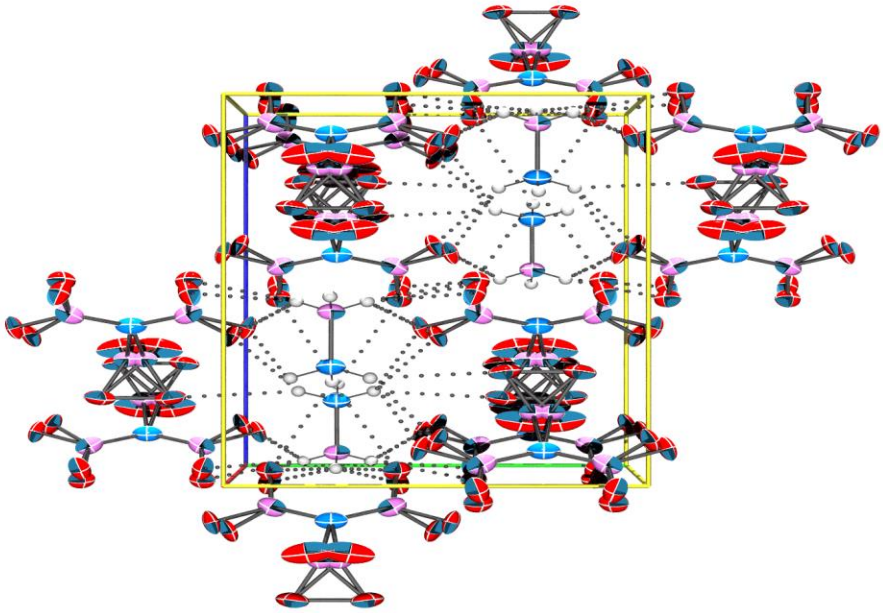

Figure S11. Unit cell of **4** normal to 100.

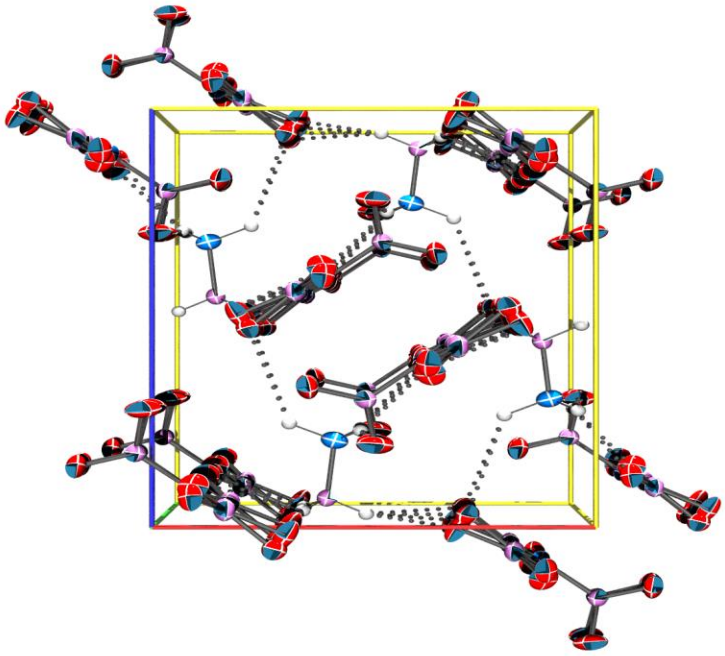

Figure S12. Unit cell of **4** normal to 010.

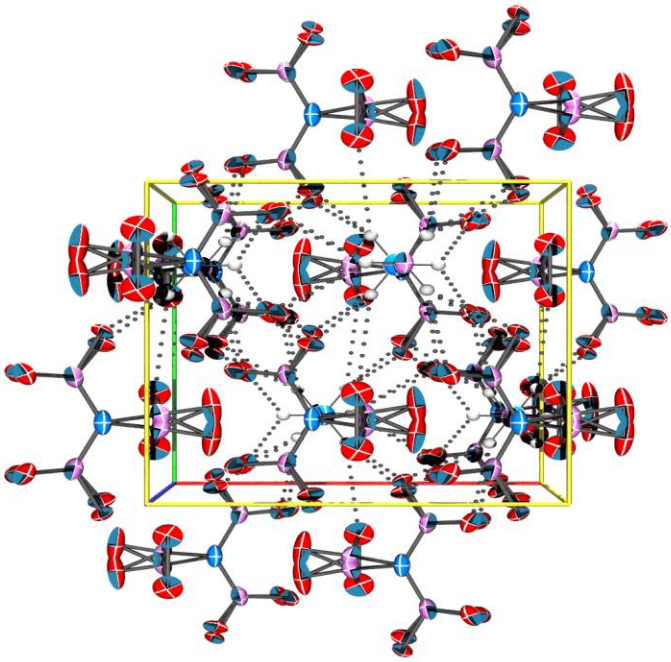

Figure S13. Unit cell of **4** normal to 001.

Table S20. Crystal data and structure refinement for 4.

Identification code	mo_IM26_0m
Empirical formula	$C_2H_6N_4O_6$
Formula weight	182.11
Temperature/K	99.99
Crystal system	orthorhombic
Space group	Pnma
a/Å	8.226(2)
b/Å	8.403(2)
c/Å	10.367(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	716.6(3)
Z	4
ρ_{calc}g/cm^3	1.688
μ/mm^{-1}	0.168
F(000)	376.0
Crystal size/mm^3	0.418 × 0.286 × 0.194
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.242 to 61
Index ranges	$-11 \leq h \leq 11$, $-11 \leq k \leq 11$, $-14 \leq l \leq 14$
Reflections collected	16233
Independent reflections	1158 [R_{int} = 0.0403, R_{sigma} = 0.0146]
Data/restraints/parameters	1158/0/106
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0350, wR_2 = 0.0906
Final R indexes [all data]	R_1 = 0.0428, wR_2 = 0.0967
Largest diff. peak/hole / e Å^{-3}	0.28/-0.27

Table S21. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 4. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O(1)	7870(9)	5958(8)	5245(8)	27.4(7)
O(2)	6183(8)	5042(16)	3776(9)	28.9(10)
N(1)	11091.3(15)	7500	5563.6(11)	22.9(3)
N(2)	6796.4(10)	6053.3(12)	4349.3(9)	26.7(2)
C(2)	6137.2(16)	7500	4041.1(14)	27.1(3)
C(1)	10918(2)	7500	6989.1(14)	28.4(3)
N(3)	4809.2(17)	7274(6)	3114.8(14)	22.4(10)
O(4)	3546.0(18)	7976(3)	3414.5(16)	53.6(9)
O(3)	5022(2)	8381(2)	2088.2(14)	36.6(4)
O(2A)	6147(6)	4635(14)	3992(9)	25.1(11)
O(1A)	8101(13)	5936(8)	4920(17)	32.5(17)

Table S22. Anisotropic Displacement Parameters ($Å^2×10^3$) for 4. 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_{12}	U_{13}	U_{23}
O(1)	22.4(13)	34.4(14)	25.4(17)	-7.8(10)	-2.0(12)	-1.9(13)
O(2)	32.6(12)	17(2)	36.7(19)	-5.0(14)	-4.2(11)	-10.7(16)
N(1)	16.3(5)	32.9(6)	19.5(5)	-0	-1.0(4)	0
N(2)	15.6(4)	36.1(5)	28.5(4)	-4.3(3)	0.1(3)	-7.4(4)
C(2)	14.0(6)	45.5(9)	21.7(6)	-0	-2.1(5)	0
C(1)	28.5(7)	36.9(8)	19.8(6)	-0	3.1(5)	0
N(3)	17.0(5)	32(3)	18.5(6)	-4.9(8)	-0.4(4)	-0.3(7)
O(4)	16.4(6)	111(3)	33.5(7)	12.6(9)	-3.8(6)	-15.1(10)
O(3)	46.4(10)	46.6(10)	16.7(6)	0.9(8)	-2.7(6)	5.0(6)
O(2A)	20.4(10)	15(2)	40.1(19)	-5.4(12)	-0.7(10)	-9.5(16)
O(1A)	25(2)	23.3(11)	49(4)	-5.8(12)	-18(2)	8.4(18)

Table S23. Bond Lengths for 4.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O(1)	N(2)	1.285(6)	N(2)	O(1A)	1.229(5)
O(2)	N(2)	1.153(8)	C(2)	N(3)[1]	1.467(2)
N(1)	C(1)	1.4847(19)	C(2)	N(3)	1.467(2)
N(2)	C(2)	1.3689(12)	N(3)	O(4)	1.235(3)
N(2)	O(2A)	1.357(9)	N(3)	O(3)	1.424(4)

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

⊤ Table S24. Bond Angles for 4.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O(2)	N(2)	O(1)	128.9(7)	N(3)	C(2)	N(2)[1]	124.3(2)
O(1A)	N(2)	O(2A)	113.9(6)	N(3)[1]	C(2)	N(3)	14.9(4)
N(2)[1]	C(2)	N(2)	125.25(12)	O(4)	N(3)	C(2)[1]	113.6(2)
N(3)	C(2)	N(2)	109.44(19)	O(3)	N(3)	C(2)[1]	108.2(2)
N(3)[1]	C(2)	N(2)	124.3(2)	O(3)	N(3)	O(4)	88.8(3)
N(3)[1]	C(2)	N(2)[1]	109.44(19)				

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

Table S25. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 4.

Atom	*x*	*y*	*z*	U(eq)
H1A	10180(30)	7500	5170(20)	35(5)
H1B	11640(20)	8350(20)	5300(16)	50(5)
H1C	10310(20)	8443(19)	7256(17)	56(5)
H1D	11970(30)	7500	7400(30)	51(6)

Table S26. Atomic Occupancy for 4.

Atom	***Occupancy***	**Atom**	***Occupancy***	**Atom**	***Occupancy***
O(1)	0.50(4)	O(2)	0.50(4)	N(3)	0.500000
O(4)	0.500000	O(3)	0.500000	O(2A)	0.50(4)
O(1A)	0.50(4)				

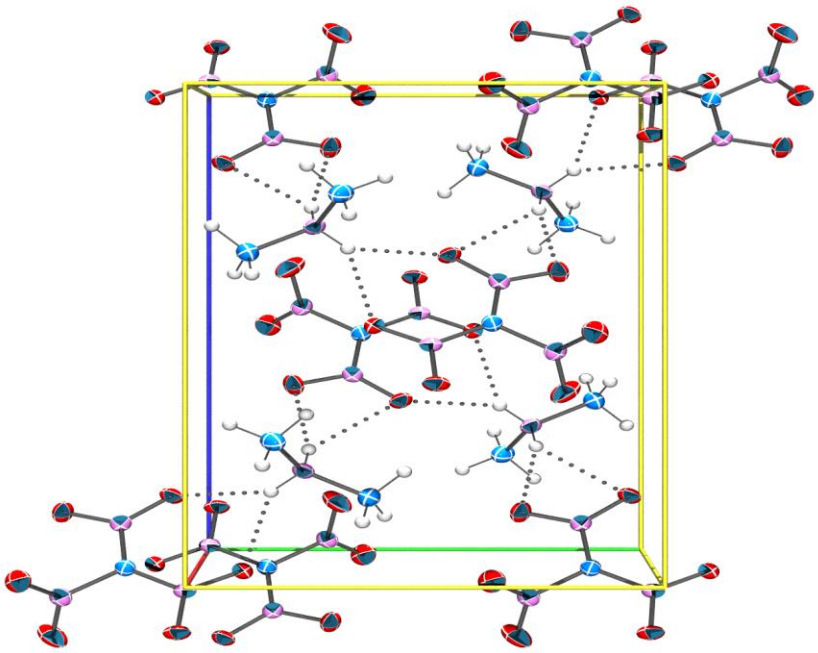

Figure S14. Unit cell of **5** normal to 100.

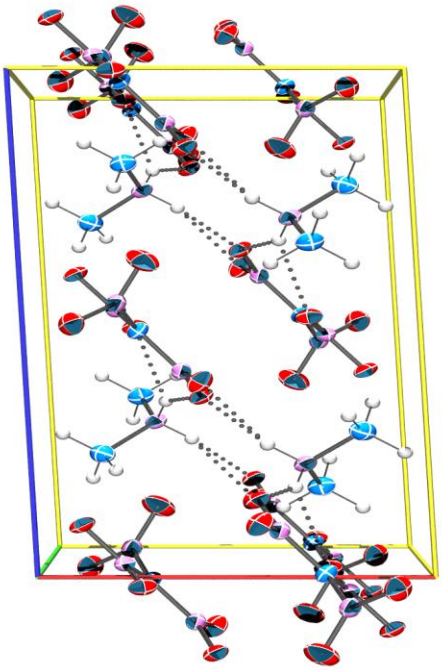

Figure S15. Unit cell of **5** normal to 010.

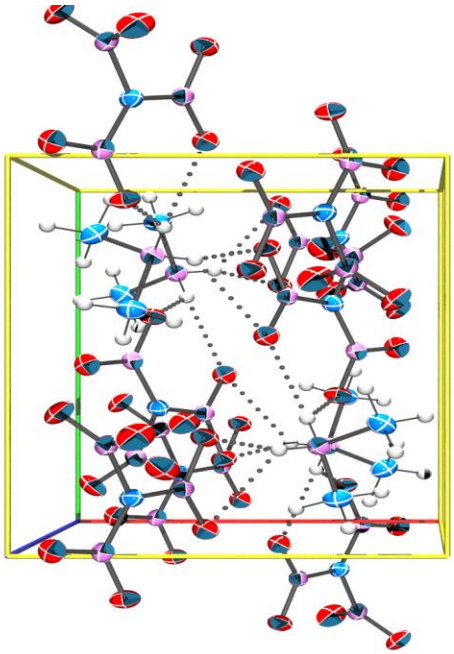

Figure S16. Unit cell of **5** normal to 001.

Table S27. Crystal data and structure refinement for 5.

Identification code	mo_AB3_244_0m
Empirical formula	$C_3H_8N_4O_6$
Formula weight	196.13
Temperature/K	100.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	7.409(3)
b/Å	9.031(4)
c/Å	12.671(5)
α/°	90
β/°	92.666(7)
γ/°	90
Volume/Å^3	846.8(6)
Z	4
ρ_{calc}g/cm^3	1.538
μ/mm^{-1}	0.148
F(000)	408.0
Crystal size/mm^3	0.172 × 0.166 × 0.151
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.504 to 61.13
Index ranges	$-10 \leq h \leq 10$, $-12 \leq k \leq 12$, $-18 \leq l \leq 17$
Reflections collected	19784
Independent reflections	2578 [R_{int} = 0.0450, R_{sigma} = 0.0304]
Data/restraints/parameters	2578/0/128
Goodness-of-fit on F^2	0.920
Final R indexes [I>=2σ (I)]	R_1 = 0.0385, wR_2 = 0.1120
Final R indexes [all data]	R_1 = 0.0672, wR_2 = 0.1330
Largest diff. peak/hole / e Å^{-3}	0.22/-0.25

Table S28. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 5. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O2	5649.2(13)	5535.6(10)	6539.3(7)	22.2(2)
O3	7345.1(13)	3913.8(10)	5115.0(7)	21.9(2)
O4	8815.1(13)	5236.8(11)	3992.0(7)	23.7(2)
O1	5639.8(14)	7910.5(10)	6201.1(8)	26.0(2)
O6	8837.0(14)	8588.6(12)	5029.6(9)	30.6(3)
N3	7799.9(14)	5127.7(12)	4747.4(8)	17.4(2)
O5	6929.8(17)	8027.3(13)	3750.8(9)	38.4(3)
N2	6125.6(14)	6612.5(11)	6002.5(8)	18.1(2)
N1	6966.6(16)	2620.9(12)	7136.2(9)	19.7(2)
N4	7694.1(15)	7786.9(12)	4610.2(9)	20.7(2)
C3	7208.9(18)	6441.2(14)	5166.8(10)	18.9(3)
C2	7394(2)	1194.2(15)	6617.2(11)	25.8(3)
C1	8477(2)	3224.1(16)	7817.1(11)	26.6(3)

Table S29. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 5. 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}
O2	24.7(5)	23.2(5)	19.2(4)	4.4(3)	7.4(4)	0.6(4)
O3	29.6(5)	17.6(4)	18.6(4)	1.3(3)	3.0(4)	-2.4(4)
O4	25.2(5)	30.4(5)	16.3(4)	-1.0(4)	8.6(4)	-0.4(4)
O1	27.0(5)	19.1(5)	32.6(5)	-4.0(4)	10.3(4)	2.0(4)
O6	27.4(6)	29.5(6)	35.2(6)	-3.1(4)	3.5(4)	-13.2(4)
N3	16.8(5)	21.1(5)	14.3(5)	0.1(4)	1.3(4)	-1.1(4)
O5	42.6(7)	37.9(6)	33.4(6)	16.1(5)	-12.2(5)	-8.2(5)
N2	16.0(5)	19.1(5)	19.3(5)	-1.5(4)	1.7(4)	-0.3(4)
N1	21.2(6)	20.4(5)	17.9(5)	3.2(4)	4.9(4)	0.1(4)
N4	17.5(5)	21.5(5)	23.3(5)	2.1(4)	3.2(4)	-1.9(4)
C3	18.8(6)	18.1(6)	20.4(6)	-0.1(4)	5.4(5)	-1.6(4)
C2	31.8(7)	21.2(7)	24.8(6)	-1.5(5)	3.4(6)	1.9(5)
C1	29.5(7)	25.8(7)	24.3(6)	2.2(5)	-0.2(6)	-5.7(6)

Table S30. Bond Lengths for 5.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O2	N2	1.2471(14)	O5	N4	1.2232(16)
O3	N3	1.2437(14)	N2	C3	1.3669(17)
O4	N3	1.2484(14)	N1	C2	1.4874(18)
O1	N2	1.2552(14)	N1	C1	1.4842(19)
O6	N4	1.2172(15)	N4	C3	1.4585(17)
N3	C3	1.3794(17)			

Table S31. Bond Angles for 5.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O3	N3	O4	122.69(11)	O6	N4	O5	124.54(12)
O3	N3	C3	121.16(11)	O6	N4	C3	117.81(11)
O4	N3	C3	116.16(11)	O5	N4	C3	117.65(11)
O2	N2	O1	121.86(11)	N3	C3	N4	116.13(11)
O2	N2	C3	121.71(11)	N2	C3	N3	127.16(11)
O1	N2	C3	116.43(11)	N2	C3	N4	116.56(11)
C1	N1	C2	113.87(11)				

Table S32. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 5.

Atom	*x*	*y*	*z*	U(eq)
H2A	8440.95	1327.22	6181.54	39
H2B	6352.09	873.5	6170.03	39
H2C	7671.45	441.91	7158.13	39
H1C	8808.44	2507.89	8373.96	40
H1D	8097.73	4153.88	8138.64	40
H1E	9520.74	3408.79	7388.44	40
H1A	6650(20)	3330(18)	6687(14)	25(4)
H1B	6010(30)	2530(20)	7519(14)	35(5)

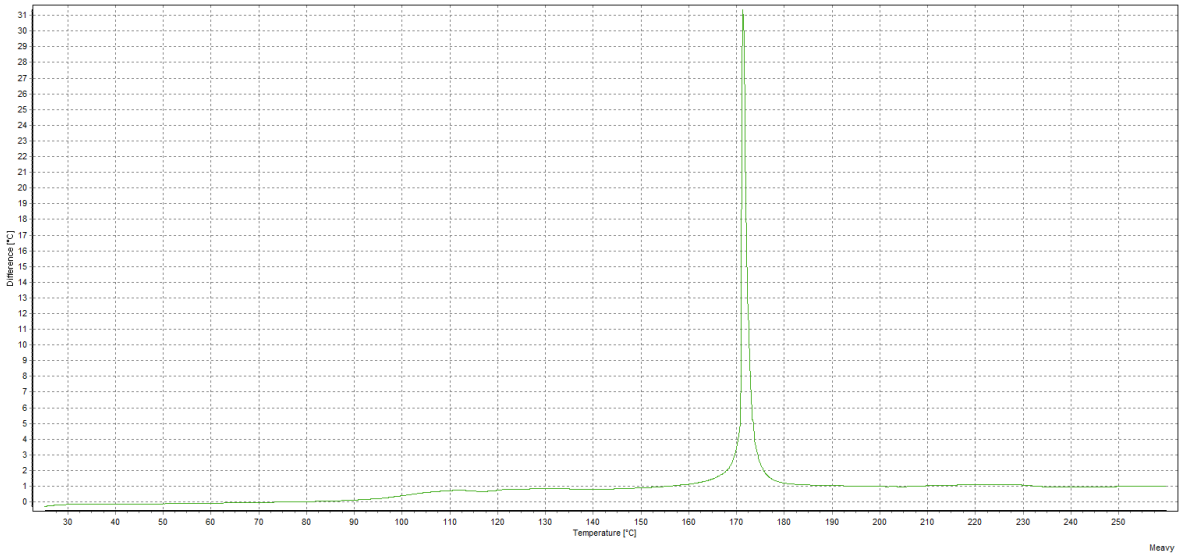

Figure S17. DTA trace of $[(NH_2)_2CH][nf]$, **2**.

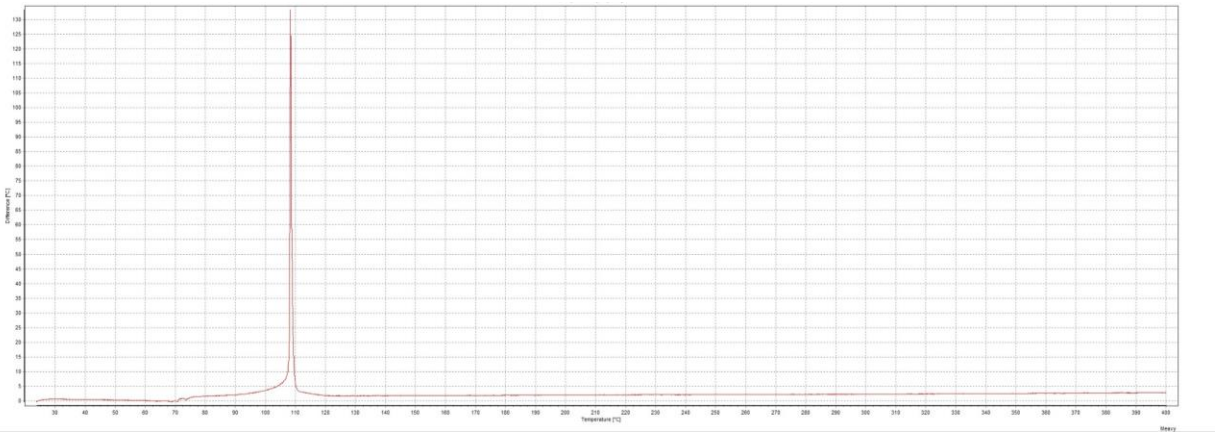

Figure S18. DTA trace of $[(NH_3CH_2)_2][nf]_2$, **3**.

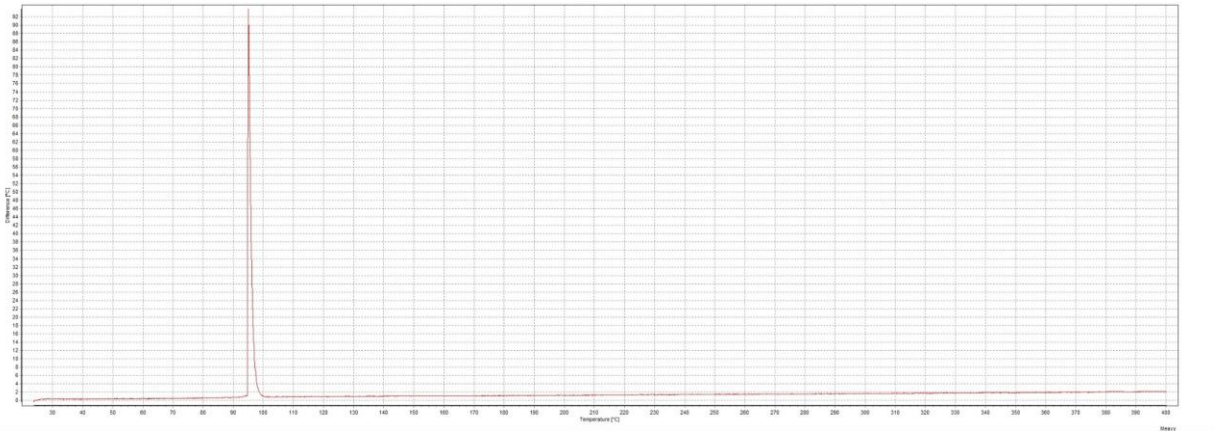

Figure S19. DTA trace of $[MeCH_3][nf]$, **4**.

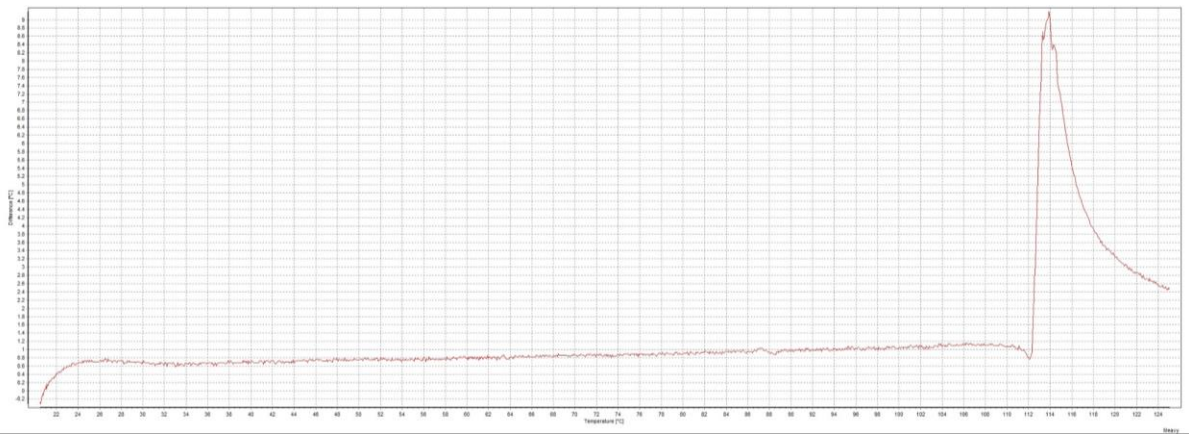

Figure S20. DTA trace of $[Me_2NH_2][nf]$, **5**.

References

1. Langlet, A.; Latypov, N. V.; Wellmar, U.; Goede, P.; Bergman, J., Formation of Nitroform in the Nitration of Gem-Dinitro Compounds. *Propellants Explos. Pyrotech.* **2004,** *29* (6), 344-348.
2. Huang, Y.; Gao, H.; Twamley, B.; Shreeve, J. M., Synthesis and Characterization of New Energetic Nitroformate Salts. *Eur. J. Inorg. Chem.* **2007,** *2007* (14), 2025-2030.
3. Jadhav, H. S. T., M. B.; Dhavale, D. D.; Asthana, S. N.; Krishnamurthy, V. N., Synthesis, characterization and thermal behavior of hydrazinium nitroformate (HNF) and its new N-alkyl substituted derivatives. *Indian J. Chem. Technol.* **2005,** *12* (2), 187-192.
4. Matuszko, A. J.; Chang, M. S. Methylamine Nitroform Oxidizer in Nitrocellulose, Aluminum and Metriol Trinitrate Propellants. US3222233, 1965.
5. *SAINT+ Vol. 8.27B*, Bruker AXS: Madison, WI, 2011.
6. *SADABS Vol. 2012-1*, Bruker, AXS: Madison, WI, 2012.
7. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H., OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Cryst.* **2009,** *42*, 339-341.
8. Sheldrick, G. M., SHELXT – Integrated space-group and crystal-structure determination. *Acta Cryst. A* **2015,** *A71*, 3-8.
9. Sheldrick, G. M., Crystal structure refinement with SHELXL. *Acta Cryst. C* **2015,** *C71*, 3-8.
10. *Mercury CSD 3.10*, 2017.