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Electronic Supplementary Information

pH-Controlled forms of 1-amino-1-hydrazino-2,2-dinitroethylene (HFOX): Selective reactivity of amine and hydrazinyl groups with aldehydes or ketones

Ajay Kumar Chinnam,[a] Richard J. Staples,[b] Jean'ne M. Shreeve*[a]

[a] Department of Chemistry, University of Idaho, Moscow, ID 83844-2343 USA, E-mail: jshreeve@uidaho.edu

[b] Department of Chemistry, Michigan State University, East Lansing, Michigan 48824, USA

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1. Experimental Section

Caution! 1-Amino-1-hydrazino-2,2-dinitroethylene is highly sensitive[1] and other new materials can potentially explode under certain conditions (e.g., impact, friction, or electric discharge). Although no explosions or detonations occurred during the preparation and manipulation of these materials, all reactions should be carried out on a small scale with appropriate safety precautions, such as the use of extra shields in a fume hood and personal protection equipment (safety glasses, face shields, ear plugs, as well as gloves) are strongly encouraged.

General Information

Reagents were purchased from Aldrich and Acros Organics and were used as received. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a 300 MHz (Bruker AVANCE 300) nuclear magnetic resonance spectrometer operating at 300.13, 75.48 and 282.40 MHz, respectively, by using DMSO-d_6 or acetone-d_6 or chloroform-d or acetonitrile-d_3 as the solvent and locking solvent. Tetramethyl silane, is used as a reference for ^{1}H, and ^{13}C, respectively. IR spectra were recorded using KBr pellets for solids on a Nicolet Thermo-model AVATAR 370-spectrometer. The melting and decomposition (onset) points were obtained on a differential scanning calorimeter (TA Instruments Co., model Q2000) at a scan rate of 5 °C min^{-1}. Density was measured at room temperature by employing a Micromeritics AccuPyc II 1340 gas pycnometer. Elemental analyses (C, H, N) were determined using a Vario Micro-cube Elementar Analyser. The sensitivities to impact (IS) and friction (FS) were determined according to BAM standards. Crystallographic data for the reported structures in this manuscript have been deposited with the Cambridge Crystallographic Data Centre. CCDC 1975378 (**11**), 1975379 (**9**), 2154055 (**5, form i**), 2154056 (**5, form ii**), 2154057 (**5c**), 2154058 (**5e**), and 2154059 (**12a**), contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

3-(2-(1-Amino-2,2-dinitrovinyl)hydrazono)butan-2-one (3): A mixture of HFOX (0.2 g, 1.23 mmol) and 2,3-butanedione (0.1g, 1.23 mmol) was stirred in distilled water (10.0 mL) and pH~2 adjusted using concentrated HCl. The resulting mixture was stirred at room temperature for 4 hours. A yellow precipitate formed and was collected by filtration. The solid was washed with excess water (15.0 mL) and dried at room temperature to give a yellow product **3** (0.26 g, 92%). T_d (onset): 165 °C; ^{1}H NMR (DMSO-d6, ppm): 12.42 (b, s, 1H, NH), 10.44 (s, 1H, NH), 9.86 (s, 1H, NH), 2.50 (s, 3H, CH_3), 2.04 (s, 3H, CH_3); ^{13}C NMR (DMSO-d6, ppm): 197.3, 157.2, 155.9, 125.7, 25.2, 11.6. IR (KBr pellet): υ 3421, 3310, 1696, 1618, 1558, 1492, 1426, 1360, 1303, 1216, 1139, 1076, 867, 800, 787, 749, 605, 541, 486, 486 cm^{-1}; Elemental analysis (%) calcd for $C_6H_9N_5O_5$ (231.06): C, 31.37; H, 3.92; N, 30.30; found: C, 31.43; H, 4.34; N, 30.30.

Potassium (5,6-dimethyl-1,2,4-triazin-3-yl)dinitromethanide (5): HFOX (0.30 g, 1.84 mmol) and 2,3-butanedione (0.16 g, 1.84 mmol) were mixed in acetonitrile (10.0 mL). To this, potassium hydroxide (0.10 g, 1.84 mmol) dissolved in the minimum amount of water was added, and the resulting mixture was stirred at room temperature for 6 hours. An orange precipitate formed and

was collected by filtration and dried at room temperature to give an orange solid **5** (0.39 g, 85%) T_d (onset): 236 °C; ^{1}H NMR (DMSO-d_6): 2.65 (s, 3H, CH_3), 2.53 (s, 3H, CH_3) ppm; ^{13}C NMR (DMSO-d_6): 159.8, 159.6, 155.9, 132.4, 21.4, 19.1 ppm. IR (KBr pellet): υ 3431, 1702, 1497, 1361, 1246, 1137, 984, 932, 820, 753, 653, 519 cm^{-1}; Elemental analysis (%) calcd for $C_6H_7N_5KO_{4.5}$ (251.01): C, 27.69; H, 2.71; N, 26.91; found: C, 27.14; H, 2.88; N, 27.07.

Sodium (5,6-dimethyl-1,2,4-triazin-3-yl)dinitromethanide (5a): HFOX (0.30 g, 1.84 mmol) and 2,3-butanedione (0.16 g, 1.84 mmol) were mixed in acetonitrile (10.0 mL). To this, sodium hydroxide (74 mg, 1.84 mmol) dissolved in the minimum amount of water was added, and the resulting mixture was stirred at room temperature for 6 hours. An orange precipitate formed and was collected by filtration and dried at room temperature to give an orange solid **5a** (0.38 g, 88%) T_d (onset): 260 °C; ^{1}H NMR (DMSO-d_6): 2.63 (s, 3H, CH_3), 2.51 (s, 3H, CH_3) ppm; ^{13}C NMR (DMSO-d_6): 159.9, 159.5, 155.9, 132.4, 21.4, 19.2 ppm. IR (KBr pellet): υ 3454, 2515, 1495, 1442, 1371, 1241, 1124, 1045, 983, 937, 830, 754, 652, 552 cm^{-1}; Elemental analysis (%) calcd for $C_6H_6N_5NaO_4$ (235.03): C, 30.52; H, 2.99; N, 29.66; found: C, 30.17; H, 3.12; N, 29.28.

Lithium (5,6-dimethyl-1,2,4-triazin-3-yl)dinitromethanide (5b): HFOX (0.2 g, 1.23 mmol) and 2,3-butanedione (0.1g, 1.23 mmol) were mixed in acetonitrile (10.0 mL). To this, lithium hydroxide (30 mg, 1.23 mmol) dissolved in the minimum amount of water was added, and the resulting mixture was stirred at room temperature for 6 hours. A yellow precipitate formed and was collected by filtration and dried at room temperature to give yellow solid **5b** (0.2 g, 76%). T_d (onset): 158 °C; ^{1}H NMR (DMSO-d_6): 2.63 (s, 3H, CH_3), 2.50 (s, 3H, CH_3) ppm; ^{13}C NMR (DMSO-d_6): 159.9, 156.8, 155.9, 132.3, 21.5, 19.2 ppm. IR (KBr pellet): υ 3463, 2339, 1636, 1508, 1392, 1343, 1256, 1135, 1034, 987, 930, 829, 755, 737, 589, 489 cm^{-1}; Elemental analysis (%) calcd for $C_6H_8N_5LiO_5$ (237.06): C, 30.27; H, 3.81; N, 29.41; found: C, 30.52; H, 4.01; N, 29.75.

Ammonium (5,6-dimethyl-1,2,4-triazin-3-yl)dinitromethanide (5c): HFOX (0.30 g, 1.84 mmol) and 2,3-butanedione (0.30 g, 1.84 mmol) were mixed in acetonitrile (10.0 mL). To this, aqueous ammonia (0.5 mL) was added dropwise, and the resulting mixture was stirred at room temperature for 6 hours. A yellow precipitate formed and was collected by filtration and dried at room temperature to give yellow solid **5c** (0.38 g, 90%). T_d (onset): 161 °C; ^{1}H NMR (DMSO-d_6): 7.11 (b, 4H, NH_4), 2.63 (s, 3H, CH_3), 2.50 (s, 3H, CH_3) ppm; ^{13}C NMR (DMSO-d_6): 159.9, 159.6, 155.9, 132.4, 21.4, 19.1 ppm. IR (KBr pellet): υ 3176, 1690, 1482, 1349, 1238, 1142, 1043, 1006, 984, 934, 822, 799, 752, 654, 613, 553 cm^{-1}; Elemental analysis (%) calcd for $C_6H_{10}N_6O_4$ (230.07): C, 31.31; H, 4.38; N, 36.51; found: C, 31.57; H, 4.59; N, 37.23.

Sodium dinitro(1,2,4-triazin-3-yl)methanide (5e): HFOX (0.28 g, 1.72 mmol) and 40% glyoxal (0.20 g, 1.72 mmol) were mixed in acetonitrile (10.0 mL). To this, sodium hydroxide (70 mg, 1.72 mmol) dissolved in water (1.0 mL) was added, and the resulting mixture was stirred at room temperature for 6 hours. An orange precipitate formed and was collected by filtration and dried at room temperature to give an orange solid **5e** (0.30 g, 84%). T_d (onset): 301 °C; ^{1}H NMR (DMSO-

d_6): 9.29 (s, 1H, CH), 8.92 (s, 1H, CH) ppm; ^{13}C NMR (DMSO-d_6): 162.0, 154.0, 148.0, 132.6 ppm. IR (KBr pellet): υ 3405, 3029, 1609, 1507, 1370, 1313, 1243, 1123, 1089, 1057, 887, 842, 751, 611, 461 cm^{-1}; Elemental analysis (%) calcd for $C_4H_2N_5NaO_4$ (207.00): C, 23.20; H, 0.97; N, 33.82; found: C, 22.61; H, 1.57; N, 34.18.

Lithium dinitro(1,2,4-triazin-3-yl)methanide (5f): HFOX (0.30 g, 1.84 mmol) and 40% glyoxal (0.25 g, 1.84 mmol) were mixed in acetonitrile (10.0 mL). To this, lithium hydroxide (44 mg, 1.84 mmol) dissolved in the minimum amount of water was added, and the resulting mixture was stirred at room temperature for 6 hours. An orange precipitate formed and was collected by filtration and dried at room temperature to give an orange solid **5f** (0.27 g, 77%). T_d (onset): 200 ℃; ^{1}H NMR (DMSO-d_6): 9.29 (s, 1H, CH), 8.91 (s, 1H, CH) ppm; ^{13}C NMR (DMSO-d_6): 162.0, 150.2, 147.8, 131.8 ppm. IR (KBr pellet): υ 3406, 3122, 1518, 1440, 1415, 1345, 1278, 1139, 1087, 1061, 987, 968, 885, 870, 844, 812, 748, 538, 481, 436 cm^{-1}; Elemental analysis (%) calcd for $C_4H_2N_5LiO_4$ (191.02): C, 25.15; H, 1.06; N, 36.66; found: C, 24.84; H, 1.73; N, 35.69.

Potassium-1-(1-amino-2,2-dinitrovinyl)-2-(3-oxobutan-2-ylidene)hydrazin-1-ide (6): To compound **3** (0.2 g, 0.93 mmol) in acetonitrile (10.0 mL) was added potassium hydride (52 mg, 0.93 mmol) in distilled water (1.0 mL). The resulting mixture was stirred at room temperature for 1 hour, and an orange precipitate formed and was collected by filtration and washed with acetonitrile (2.0 mL) and dried at room temperature to give an orange solid **6** (0.23 g, 98%). T_d (onset): 142 ℃; ^{1}H NMR (DMSO-d6, ppm): 8.06 (b, 2H, NH_2), 2.38 (s, 3H, CH_3), 1.89 (s, 3H, CH_3); ^{13}C NMR (DMSO-d6, ppm): 198.8, 155.8, 130.0, 24.9, 10.8. IR (KBr pellet): υ 3599, 3429, 3307, 3194, 1676, 1607, 1545, 1510, 1418, 1361, 1291, 1226, 1120, 1029, 946, 854, 793, 751, 709, 658, 611, 523 cm^{-1}; Elemental analysis (%) calcd for $C_6H_8N_5KO_5$ (269.01): C, 26.76; H, 2.99; N, 26.01; found: C, 25.94; H, 3.21; N, 26.09.

2,2-Dinitro-1-(2-((perfluorophenyl)methylene)hydrazineyl)ethen-1-amine (9): HFOX (0.2 g, 1.23 mmol) and pentafluorobenzaldehyde (0.24 g, 1.23 mmol) were mixed in distilled water (10 mL). A few drops of concentrated HCl were added to maintain a pH~2, and the resulting mixture was stirred at room temperature for 4 hours. A yellow precipitate was formed and collected by filtration and dried at room temperature to give a yellow solid **9** (0.40 g, 96%). T_d (onset): 160 ℃; ^{1}H NMR (DMSO-d_6): 13.36 (s, 1H, NH), 10.16 (s, 1H, NH_2), 9.43 (s, 1H, NH_2), 8.43 (s, 1H, CH) ppm; ^{13}C NMR (DMSO-d_6): 154.3, 146.9, 143.5, 142.4, 140.4, 139.2, 125.7, 108.4 ppm. ^{19}F NMR (DMSO-d_6): -139.69 (s, 1F), -141.95 (s, 1F), -149.99 (s, 1F), -162.19 to -162.29 (m, 2F) ppm; IR (KBr pellet): υ 3472, 1654, 1509, 1425, 1347, 1287, 1188, 1139, 1037, 987, 828, 828, 810, 712, 663, 595, 522, 453 cm^{-1}; Elemental analysis (%) calcd for $C_9H_4F_5N_5O_4$ (341.01): C, 31.69; H, 1.18; N, 20.53; found: C, 31.92; H, 1.34; N, 20.22.

1-(2-(1,3-Dichloropropan-2-ylidene)hydrazineyl)-2,2-dinitroethen-1-amine (10): HFOX (0.25 g, 1.53 mmol) and 1,3-dichloropropan-2-one (0.19 g, 1.53 mol) were mixed in distilled water (10 mL). To this, a few drops of concentrated HCl was added to maintain pH~2, and the resulting mixture was stirred at room temperature for 4 hours. A pale-yellow precipitate formed and was

collected by filtration and dried at room temperature to give compound **10** (0.41 g, 98%). T_m (onset): 117 °C; T_d (onset): 135 °C; ^{1}H NMR (DMSO-d_6): 12.55 (s, 1H, NH), 10.30 (s, 1H, NH_2), 9.85 (s, 12H, NH_2), 4.53 (s, 2H, CH_2), 4.51 (s, 2H, CH_2) ppm; ^{13}C NMR (DMSO-d_6): 155.6, 154.5, 125.3, 45.2, 34.9 ppm. IR (KBr pellet): υ 3418, 3308, 3017, 1540, 1360, 1225, 1141, 1062, 957, 906, 873, 851, 789, 755, 713, 699, 633 cm^{-1}; Elemental analysis (%) calcd for $C_5H_7Cl_2N_5O_4$ (270.98): C, 22.08; H, 2.59; N, 25.74; found: C, 22.19; H, 2.70; N, 26.38.

1-(2-(1,3-Diazidopropan-2-ylidene)hydrazineyl)-2,2-dinitroethen-1-amine (11): HFOX (0.15 g, 0.92 mmol) and diazo carbonyl compound (0.13 g, 0.92 mmol) were mixed in distilled water (10 mL). A few drops of concentrated HCl were added to this mixture to maintain pH~2, and the resulting mixture was stirred at room temperature for 6 hours. A yellow precipitate formed and was collected by filtration and dried at room temperature to give a yellow solid of **11** (0.27 g, 93%) T_d (onset): 160 °C; ^{1}H NMR (DMSO-d_6): 13.36 (s, 1H, NH), 10.16 (s, 1H, NH_2), 9.43 (s, 12H, NH_2), 8.43 (s, 1H, CH) ppm; ^{13}C NMR (DMSO-d_6): 154.3, 146.9, 143.5, 142.4, 140.4, 139.2, 125.7, 108.4 ppm. IR (KBr pellet): υ 3430, 3311, 3199, 2114, 1598, 1560, 1533, 1365, 1219, 1138, 1115, 1000, 907, 843, 787, 743, 683, 601, 457 cm^{-1}; Elemental analysis (%) calcd for $C_5H_7N_{11}O_4$ (285.06): C, 21.06; H, 2.47; N, 54.03; found: C, 21.18; H, 2.53; N, 54.06.

1-(1-Amino-2,2-dinitrovinyl)-3-methyl-5-(trifluoromethyl)-4,5-dihydro-1H-pyrazol-5-ol (12a): HFOX (0.24 g, 1.47 mmol) and 1,1,1-trifluoropentane-2,4-dione (0.23 g, 1.47 mmol) were mixed in distilled water (10 mL).. To this, a few drops of concentrated HCl was added to maintain a pH~2, and the resulting mixture was stirred at room temperature for 4 hours. A colorless precipitate formed and was collected by filtration and washed with an excess of water (5.0 mL) and dried at room temperature to give a colorless solid **12a** (0.33 g, 76%). T_d (onset): 184 °C; ^{1}H NMR (DMSO-d_6, ppm): 10.35 (s, 1H), 9.61 (s, 1H), 9.31 (s, 1H), 3.70-3.43 (q, 2H), 2.09 (s, 3H); ^{13}C NMR (DMSO-d6, ppm): 163.7, 156.8, 154.8, 124.6, 120.8, 93.4, 92.9, 92.5, 46.2, 15.8. ^{19}F NMR (DMSO-d_6, ppm): -81.2. IR (KBr pellet): υ 3426, 2706, 1638, 1532, 1489, 1433, 1382, 1300, 1195, 1140, 1065, 950, 477 cm^{-1}; $C_7H_8F_3N_5O_5$(299.03): C, 28.10; H, 2.70; N, 23.41; found: C, 27.34; H, 3.01; N, 23.93.

1-(1-Amino-2,2-dinitrovinyl)-3,5-bis(trifluoromethyl)-4,5-dihydro-1H-pyrazol-5-ol (12b): HFOX (0.30 g, 1.84 mmol) and hexafluoropentane-2,4-dione (0.38 g, 1.84 mmol) were mixed in distilled water (10 mL). To this, a few drops of concentrated HCl was added to maintain a pH~2, and the resulting mixture was stirred at ice-bath temperature for 30 minutes and then at room temperature for 6 hours. The solvent was removed using an air blower, which resulted in a white solid. The solid was washed with cold water (5.0 mL) and collected by filtration and dried at room temperature to give a colorless solid **12b** (0.41 g, 64%). T_d (onset): 171 °C; ^{1}H NMR (DMSO-d_6, ppm): 11.81 (s, 1H, OH), 10.03 (s, 2H, NH_2), 4.13-3.10 (m, 2H, CH_2); ^{13}C NMR (DMSO-d_6, ppm): 156.8, 151.8-149.6 (q), 126.6, 126.6-116.8 (q), 95.2-93.0 (q), 81.1, 41.10. ^{19}F NMR (DMSO-d_6, ppm): -66.87, -80.98. IR (KBr pellet): υ 3429, 1574, 1535, 1461, 1431, 1327, 1271, 1191, 1104, 1019, 879, 850, 677 cm^{-1}; Elemental analysis (%) calcd for $C_7H_6F_6N_5O_{5.5}$(362.02): C, 23.22; H, 1.67; N, 19.34; found: C, 23.15; H, 2.15; N, 18.78.

2. Theoretical Calculations

The heats of formation for anions of **5-5f** and neutral compounds **9-11**, and **12-12b** were calculated based on isodesmic reactions (Scheme 1). The calculations were carried out using Gaussian 03 (Revision D.01) suite of programs.[2] All the structures were optimized, and frequency analyses were calculated at the B3LYP/6-31+G** level and single energy points were calculated at the MP2/6- 311++G** level. The solid-state heats of formation for neutral compounds (**9**, **10**, **12a**, and **12b**) were calculated based on Trouton's rule according to equation 1 (*T* represents melting temperature).[3]

$$\Delta H_{sub} = 188/Jmol^{-1}K^{-1} \times T \qquad (1)$$

For salts, the solid-state enthalpy of formation is obtained using a Born−Haber energy cycle.[4]

Scheme 1

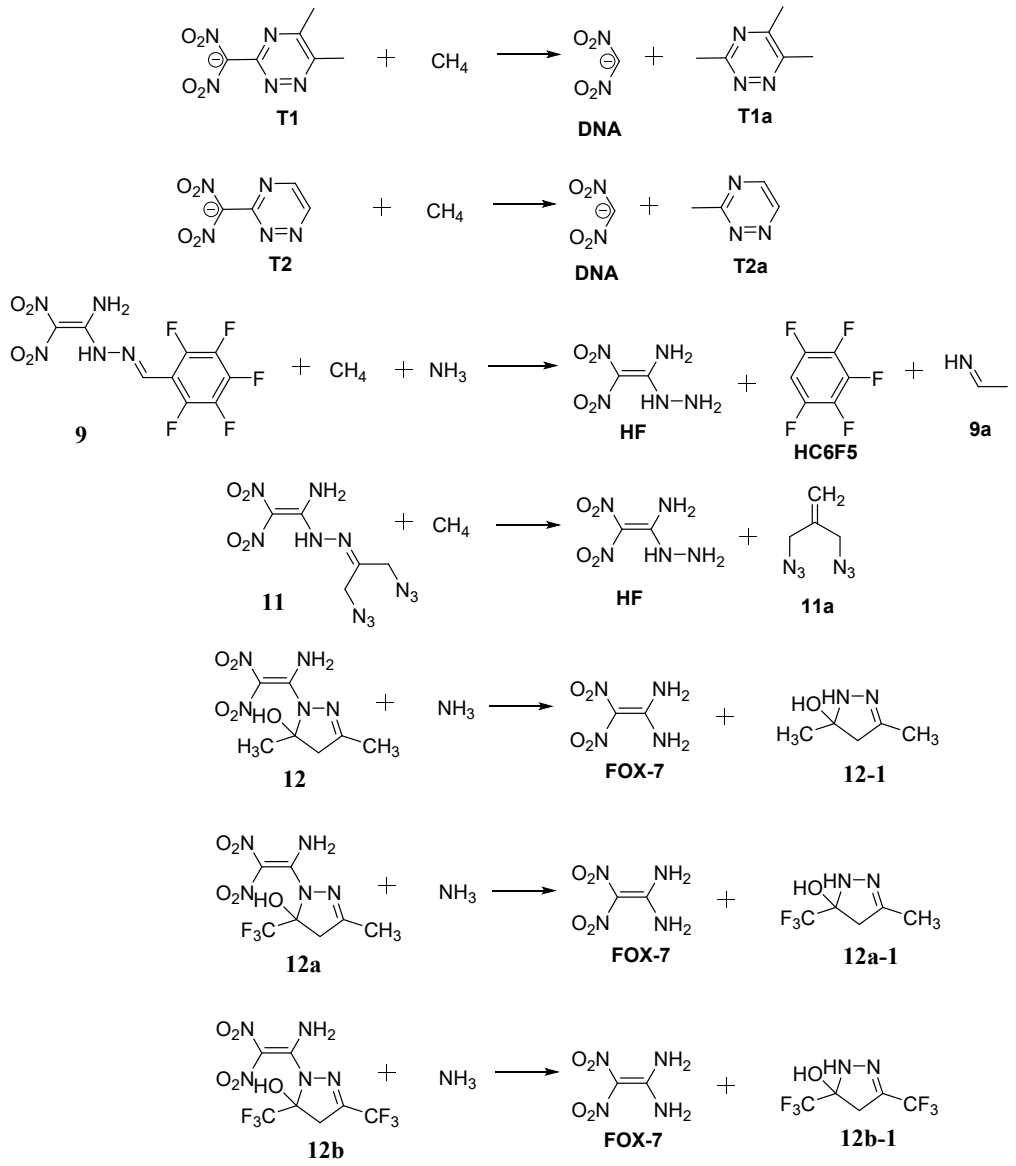


Table S1 Calculated zero point energy (ZPE), values of the correction (Hr), total energy (E0) and gas-state heats of formation (HOF).

Compound	ZPE [Hartree /Particle]	HT [Hartree /Particle]	E0 [kJ mol^{-1}]	HOF (gas) [kJ mol^{-1}]
T1	0.138464	0.153194	-804.8998915	-8.47
T1a	0.146460	0.156596	-397.2408086	173.25
T2	0.083633	0.094951	-726.4910676	86.27
T2a	0.091461	0.098313	-318.8300122	273.10
DNA	0.036739	0.042196	-447.9434569	-14.14
9	0.153515	0.174552	-1415.970394	-692.58
HF	0.109516	0.120809	-652.2173912	66.15
HC_6F_5	0.059621	0.069237	-726.9385768	-850.17

9a	0.068467	0.073344	-133.5862623	36.79
11	0.176603	0.196718	-1095.0752785	745.51
11a	0.104667	0.115516	-499.2526806	691.08
12	0.208621	0.225844	-920.8980753	-226.78
12a	0.184064	0.203903	-1218.1428151	-869.78
12b	0.160902	0.182745	-1515.3873553	-1469.47
12-1	0.152908	0.16227	-380.2818367	-122.71
12a-1	0.12944	0.140983	-677.5378422	-793.68
12b-1	0.106555	0.119911	-974.7875549	1406.75
FOX-7	0.09195	0.102165	-597.0259689	-134.1
NH_3	0.034384	0.038203	-56.4154647	-45.90
CH_4	0.044793	0.048605	-40.3796224	-74.60

[a] The enthalpy of sublimation was calculated by using Trouton's rule. Solid-state heats of formation of the resulting compounds were calculated with Equation (1) in which T_m is the melting temperature.

$$\Delta Hf = \Delta Hf(g) - \Delta Hsub = \Delta Hf(g) - 188[J\ mol^{-1}\ K^{-1}] \times T_m \quad (1)$$

3. X-ray Crystallographic data[5-11]

Table S2. Crystallographic data and structure refinement details for **5** (**Forms i and ii**), **5c** and **5e**

Compound	**5 (Form i)**	**5 (Form ii)**	**5c**	**5e**
Formula	$C_6H_6KN_5O_4$	$C_6H_6KN_5O_4$	$C_6H_{10}N_6O_4$	$C_4H_2N_5NaO_4$
$D_{calc.}$/ g cm^{-3}	1.743	1.728	1.540	1.931
μ/mm^{-1}	5.024	4.981	1.128	2.011
Formula Weight	251.26	251.26	230.20	207.10
Color	yellow	yellow	yellow	yellow
Shape	block-shaped	needle-shaped	block-shaped	needle-shaped
Size/mm^3	0.21×0.13×0.04	0.23×0.04×0.02	0.18×0.12×0.08	0.19×0.10×0.03
T/K	99.98(15)	99.98(16)	100.00(10)	100.00(10)
Crystal System	monoclinic	orthorhombic	triclinic	orthorhombic
Space Group	*C*2/*c*	$P2_12_12_1$	*P*-1	*Pnma*
a/Å	18.0180(2)	7.95992(19)	5.96567(19)	12.4451(2)
b/Å	12.9201(2)	12.8763(3)	7.3226(2)	7.51185(15)
c/Å	8.26330(11)	18.8416(5)	11.8671(4)	15.2435(2)
α/°	90	90	77.154(3)	90
β/°	95.5690(12)	90	81.278(3)	90
γ/°	90	90	81.757(3)	90
V/Å^3	1914.57(5)	1931.16(8)	496.34(3)	1425.06(4)
Z	8	8	2	8
Z'	1	2	1	1
Wavelength/Å	1.54184	1.54184	1.54184	1.54184
Radiation type	Cu K$_a$	Cu K$_a$	Cu K$_a$	Cu K$_a$
θ_{min}/°	4.217	4.159	3.850	4.587
θ_{max}/°	79.625	80.359	79.781	79.507
Measured Refl's.	13322	9448	5927	6395
Ind't Refl's	2054	3872	2094	1639
Refl's with I > 2(I)	1967	3633	1903	1471
R_{int}	0.0494	0.0465	0.0271	0.0247
Parameters	148	294	163	163
Restraints	0	0	0	0
Largest Peak	0.338	0.366	0.311	0.287
Deepest Hole	-0.321	-0.409	-0.289	-0.282
GooF	1.068	1.134	1.080	1.063
wR_2 (all data)	0.0808	0.1160	0.0989	0.0850
wR_2	0.0800	0.1146	0.0964	0.0829
R_1 (all data)	0.0313	0.0436	0.0382	0.0331
R_1	0.0304	0.0412	0.0358	0.0295
CCDC	**2154055**	**2154056**	**2154057**	**2154058**

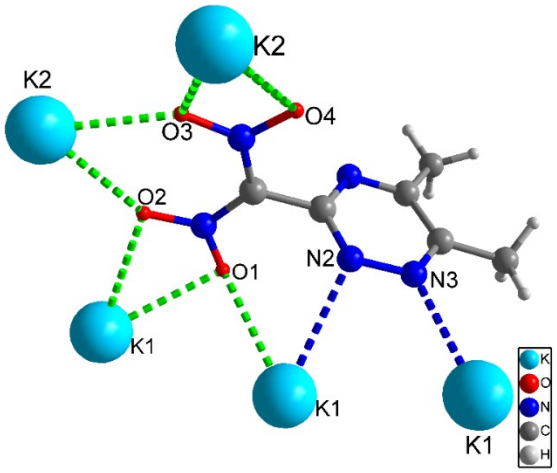


Figure S1: Thermal ellipsoids shown at 50% **5 (Form i)**.

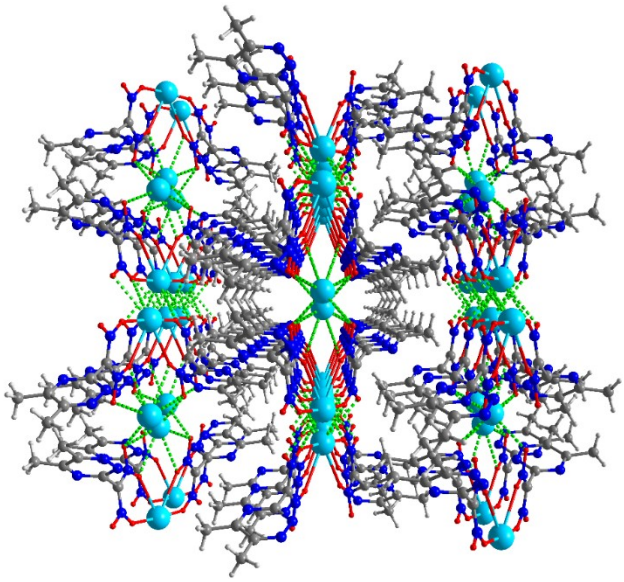


Figure S2: Ball-and-stick packing diagram of **5 (Form i)** viewed up the c axis

Table S3: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5 (Form i)**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
K1	5000	4644.6(3)	7500	14.45(13)
K2	5000	796.8(3)	7500	16.15(13)
O1	5869.4(6)	3979.1(8)	4897.8(12)	16.6(2)
O2	5512.8(6)	2635.9(8)	6218.5(12)	16.3(2)
O3	5620.5(7)	842.1(8)	4687.9(13)	21.4(2)
O4	6037.0(6)	969.5(8)	2308.3(13)	19.9(2)
N1	7055.1(7)	3001.3(9)	2455.8(14)	14.0(2)
N2	5828.0(7)	3357.1(10)	1217.8(14)	14.8(3)
N3	6113.0(7)	3810.2(10)	-56.1(14)	14.6(2)

Atom	x	y	z	U_{eq}
N4	5773.4(7)	3010.5(10)	5009.2(14)	13.5(2)
N5	5868.8(7)	1382.5(10)	3608.4(14)	14.9(2)
C1	5974.2(8)	2432.3(11)	3728.5(17)	13.8(3)
C2	6305.8(8)	2962.2(11)	2380.1(17)	13.2(3)
C3	7334.8(8)	3486.7(11)	1239.1(16)	13.1(3)
C4	6846.8(8)	3883.2(11)	-70.5(16)	14.1(3)
C5	8160.8(8)	3637.6(12)	1320.0(18)	16.9(3)
C6	7133.7(9)	4412.1(13)	-1492.3(18)	19.7(3)

Table S4: Anisotropic Displacement Parameters ($\times 10^4$) for **5 (Form i)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \ldots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
K1	15.6(2)	15.4(2)	12.3(2)	0	1.07(14)	0
K2	19.6(2)	13.5(2)	16.4(2)	0	6.99(16)	0
O1	21.3(5)	11.4(5)	17.5(5)	-0.9(4)	3.7(4)	-1.3(4)
O2	21.0(5)	16.4(5)	12.3(5)	2.4(4)	6.1(4)	0.5(4)
O3	32.8(6)	14.6(5)	19.0(5)	2.5(4)	13.0(5)	-4.0(5)
O4	29.3(6)	15.0(5)	16.9(5)	-2.9(4)	9.6(4)	-1.2(4)
N1	16.2(6)	12.3(6)	13.8(6)	-1.4(4)	2.0(4)	0.7(5)
N2	16.1(6)	14.6(6)	14.1(6)	1.0(5)	2.9(4)	-0.4(5)
N3	17.1(6)	13.2(6)	13.6(5)	0.8(4)	2.4(4)	0.2(4)
N4	13.8(5)	13.6(6)	13.0(5)	0.9(4)	1.0(4)	0.2(4)
N5	16.9(6)	13.4(6)	14.8(5)	0.5(4)	4.0(4)	0.3(5)
C1	16.1(7)	12.4(7)	13.3(6)	0.0(5)	3.1(5)	-0.1(5)
C2	16.0(6)	10.6(6)	13.2(6)	-2.2(5)	3.1(5)	-0.4(5)
C3	15.7(6)	10.1(6)	14.0(6)	-2.0(5)	3.2(5)	-0.4(5)
C4	17.4(6)	10.5(6)	14.6(6)	-0.7(5)	3.6(5)	0.9(5)
C5	14.3(7)	18.0(7)	18.8(7)	-0.4(5)	3.5(5)	0.0(5)
C6	21.2(7)	20.4(7)	18.3(7)	6.6(6)	5.4(6)	3.2(6)

Table S5: Bond Lengths in Å for **5 (Form i)**.

Atom	Atom	Length/Å
K1	$O1^1$	2.9104(10)
K1	$O1^2$	2.9898(11)
K1	$O1^3$	2.9898(11)
K1	O1	2.9104(10)
K1	O2	2.9834(11)
K1	$O2^1$	2.9834(11)
K1	$N2^2$	3.2127(13)
K1	$N2^3$	3.2127(13)
K1	$N3^4$	2.9113(12)
K1	$N3^5$	2.9113(12)
K1	$N4^1$	3.3455(12)
K1	N4	3.3455(12)
K2	O2	2.7949(11)

Atom	Atom	Length/Å
K2	O2[1]	2.7948(11)
K2	O3[6]	2.9343(12)
K2	O3[1]	2.6753(11)
K2	O3	2.6753(11)
K2	O3[7]	2.9343(12)
K2	O4[6]	2.9634(11)
K2	O4[7]	2.9634(11)
K2	N5[7]	3.3075(13)
K2	N5[6]	3.3075(13)
K2	C5[8]	3.4409(14)
K2	C5[9]	3.4409(14)
O1	N4	1.2679(16)
O2	N4	1.2427(16)
O3	N5	1.2493(16)
O4	N5	1.2625(16)
N1	C2	1.3465(19)
N1	C3	1.3253(18)
N2	N3	1.3489(17)
N2	C2	1.3279(18)
N3	C4	1.3268(19)
N4	C1	1.3724(18)
N5	C1	1.3719(19)
C1	C2	1.4820(19)
C3	C4	1.4219(19)
C3	C5	1.4959(19)
C4	C6	1.4941(19)

——[1]1-x,+y,3/2-z; [2]1-x,1-y,1-z; [3]+x,1-y,1/2+z; [4]1-x,+y,1/2-z; [5]+x,+y,1+z; [6]1-x,-y,1-z; [7]+x,-y,1/2+z; [8]-1/2+x,1/2-y,1/2+z; [9]3/2-x,1/2-y,1-z

Table S6: Bond Angles in ° for **5 (Form i)**.

Atom	Atom	Atom	Angle/°
O1[1]	K1	O1	145.63(4)
O1[2]	K1	O1[3]	107.01(4)
O1[1]	K1	O1[3]	112.36(4)
O1[1]	K1	O1[2]	88.34(3)
O1	K1	O1[2]	112.36(4)
O1	K1	O1[3]	88.34(3)
O1	K1	O2	43.39(3)
O1[1]	K1	O2	102.28(3)
O1[1]	K1	O2[1]	43.39(3)
O1	K1	O2[1]	102.28(3)
O1	K1	N2[3]	141.40(3)
O1[3]	K1	N2[2]	62.20(3)
O1[1]	K1	N2[3]	72.14(3)
O1[3]	K1	N2[3]	60.68(3)
O1[2]	K1	N2[3]	62.20(3)

Atom	Atom	Atom	Angle/°
O1[1]	K1	N2[2]	141.40(3)
O1	K1	N2[2]	72.14(3)
O1[2]	K1	N2[2]	60.68(3)
O1[1]	K1	N3[4]	91.10(3)
O1[1]	K1	N3[5]	76.23(3)
O1	K1	N3[4]	76.23(3)
O1	K1	N3[5]	91.10(3)
O1	K1	N4	22.00(3)
O1[3]	K1	N4[1]	124.08(3)
O1[1]	K1	N4	123.66(3)
O1[3]	K1	N4	100.97(3)
O1[2]	K1	N4	124.08(3)
O1	K1	N4[1]	123.66(3)
O1[2]	K1	N4[1]	100.97(3)
O1[1]	K1	N4[1]	22.00(3)
O2	K1	O1[3]	116.27(3)
O2[1]	K1	O1[2]	116.27(3)
O2[1]	K1	O1[3]	126.32(3)
O2	K1	O1[2]	126.31(3)
O2	K1	O2[1]	59.10(4)
O2[1]	K1	N2[3]	114.35(3)
O2	K1	N2[3]	170.37(3)
O2[1]	K1	N2[2]	170.37(3)
O2	K1	N2[2]	114.35(3)
O2[1]	K1	N4	80.28(3)
O2	K1	N4	21.69(3)
O2[1]	K1	N4[1]	21.69(3)
O2	K1	N4[1]	80.28(3)
N2[2]	K1	N2[3]	73.04(4)
N2[3]	K1	N4	160.83(3)
N2[2]	K1	N4	94.00(3)
N2[3]	K1	N4[1]	94.00(3)
N2[2]	K1	N4[1]	160.83(3)
N3[4]	K1	O1[3]	58.62(3)
N3[5]	K1	O1[2]	58.62(3)
N3[5]	K1	O1[3]	164.00(3)
N3[4]	K1	O1[2]	164.00(3)
N3[4]	K1	O2	69.38(3)
N3[5]	K1	O2	73.01(3)
N3[5]	K1	O2[1]	69.38(3)
N3[4]	K1	O2[1]	73.01(3)
N3[4]	K1	N2[2]	112.30(3)
N3[5]	K1	N2[3]	112.30(3)
N3[4]	K1	N2[3]	102.46(3)
N3[5]	K1	N2[2]	102.46(3)
N3[4]	K1	N3[5]	136.53(5)
N3[4]	K1	N4	68.87(3)

Atom	Atom	Atom	Angle/°
N3[5]	K1	N4	83.86(3)
N3[4]	K1	N4[1]	83.86(3)
N3[5]	K1	N4[1]	68.87(3)
N4[1]	K1	N4	101.74(4)
O2[1]	K2	O2	63.53(4)
O2	K2	O3[6]	120.02(3)
O2	K2	O3[7]	136.62(3)
O2[1]	K2	O3[6]	136.62(3)
O2[1]	K2	O3[7]	120.02(3)
O2	K2	O4[6]	155.28(3)
O2	K2	O4[7]	113.65(3)
O2[1]	K2	O4[7]	155.27(3)
O2[1]	K2	O4[6]	113.65(3)
O2	K2	N5[6]	141.61(3)
O2	K2	N5[7]	131.58(3)
O2[1]	K2	N5[7]	141.61(3)
O2[1]	K2	N5[6]	131.58(3)
O2[1]	K2	C5[8]	93.26(3)
O2	K2	C5[8]	65.30(3)
O2[1]	K2	C5[9]	65.30(3)
O2	K2	C5[9]	93.26(3)
O3[1]	K2	O2	119.47(3)
O3[1]	K2	O2[1]	58.06(3)
O3	K2	O2	58.06(3)
O3	K2	O2[1]	119.47(3)
O3	K2	O3[7]	112.62(3)
O3[6]	K2	O3[7]	87.62(5)
O3	K2	O3[1]	177.49(5)
O3[1]	K2	O3[6]	112.62(3)
O3	K2	O3[6]	69.33(4)
O3[1]	K2	O3[7]	69.33(4)
O3[6]	K2	O4[6]	43.34(3)
O3	K2	O4[6]	112.46(3)
O3[6]	K2	O4[7]	67.41(3)
O3[7]	K2	O4[6]	67.41(3)
O3[1]	K2	O4[6]	69.61(3)
O3[7]	K2	O4[7]	43.34(3)
O3[1]	K2	O4[7]	112.47(3)
O3	K2	O4[7]	69.61(3)
O3[6]	K2	N5[6]	22.06(3)
O3	K2	N5[7]	91.87(3)
O3[1]	K2	N5[6]	91.87(3)
O3[6]	K2	N5[7]	72.43(3)
O3[1]	K2	N5[7]	90.26(3)
O3[7]	K2	N5[7]	22.06(3)
O3[7]	K2	N5[6]	72.43(3)
O3	K2	N5[6]	90.26(3)

Atom	Atom	Atom	Angle/°
O3	K2	C5[8]	76.66(4)
O3	K2	C5[9]	102.79(4)
O3[6]	K2	C5[9]	71.32(3)
O3[1]	K2	C5[8]	102.79(4)
O3[1]	K2	C5[9]	76.66(4)
O3[7]	K2	C5[9]	128.81(4)
O3[7]	K2	C5[8]	71.32(3)
O3[6]	K2	C5[8]	128.81(3)
O4[6]	K2	O4[7]	79.28(4)
O4[7]	K2	N5[6]	67.27(3)
O4[7]	K2	N5[7]	22.37(3)
O4[6]	K2	N5[6]	22.37(3)
O4[6]	K2	N5[7]	67.27(3)
O4[6]	K2	C5[9]	65.40(3)
O4[7]	K2	C5[8]	65.40(3)
O4[6]	K2	C5[8]	138.01(4)
O4[7]	K2	C5[9]	138.01(4)
N5[6]	K2	N5[7]	63.29(4)
N5[7]	K2	C5[9]	132.55(3)
N5[7]	K2	C5[8]	71.66(3)
N5[6]	K2	C5[8]	132.55(3)
N5[6]	K2	C5[9]	71.66(3)
C5[8]	K2	C5[9]	155.48(5)
K1	O1	K1[3]	91.66(3)
N4	O1	K1	98.68(8)
N4	O1	K1[3]	124.67(8)
K2	O2	K1	118.68(4)
N4	O2	K1	95.79(8)
N4	O2	K2	143.17(9)
K2	O3	K2[6]	110.67(4)
N5	O3	K2	147.27(9)
N5	O3	K2[6]	96.02(8)
N5	O4	K2[6]	94.32(8)
C3	N1	C2	115.84(12)
N3	N2	K1[3]	97.63(8)
C2	N2	K1[3]	111.07(9)
C2	N2	N3	117.52(12)
N2	N3	K1[10]	114.41(8)
C4	N3	K1[10]	125.97(9)
C4	N3	N2	119.54(12)
O1	N4	K1	59.31(7)
O1	N4	C1	115.67(12)
O2	N4	K1	62.52(7)
O2	N4	O1	120.51(12)
O2	N4	C1	123.82(12)
C1	N4	K1	167.08(9)
O3	N5	K2[6]	61.92(7)

Atom	Atom	Atom	Angle/°
O3	N5	O4	120.24(12)
O3	N5	C1	123.89(12)
O4	N5	K2[6]	63.31(7)
O4	N5	C1	115.87(12)
C1	N5	K2[6]	156.96(9)
N4	C1	C2	118.95(13)
N5	C1	N4	123.29(13)
N5	C1	C2	117.74(12)
N1	C2	C1	117.25(12)
N2	C2	N1	126.58(13)
N2	C2	C1	116.16(12)
N1	C3	C4	119.68(13)
N1	C3	C5	118.53(12)
C4	C3	C5	121.74(13)
N3	C4	C3	120.72(13)
N3	C4	C6	117.43(12)
C3	C4	C6	121.84(13)
C3	C5	K2[8]	165.22(10)

——[1]1-x,+y,3/2-z; [2]+x,1-y,1/2+z; [3]1-x,1-y,1-z; [4]1-x,+y,1/2-z; [5]+x,+y,1+z; [6]1-x,-y,1-z; [7]+x,-y,1/2+z; [8]3/2-x,1/2-y,1-z; [9]-1/2+x,1/2-y,1/2+z; [10]+x,+y,-1+z

Table S7: Torsion Angles in ° for **5 (Form i)**.

Atom	Atom	Atom	Atom	Angle/°
K1[1]	O1	N4	K1	-98.12(8)
K1	O1	N4	O2	-13.51(13)
K1[1]	O1	N4	O2	-111.63(11)
K1	O1	N4	C1	166.48(10)
K1[1]	O1	N4	C1	68.36(14)
K1	O2	N4	O1	13.09(12)
K1	O2	N4	C1	-166.90(11)
K1[1]	N2	N3	K1[2]	-61.23(7)
K1[1]	N2	N3	C4	115.81(12)
K1[1]	N2	C2	N1	-108.35(13)
K1[1]	N2	C2	C1	71.25(13)
K1[2]	N3	C4	C3	176.85(9)
K1[2]	N3	C4	C6	-2.04(18)
K1	N4	C1	N5	-112.1(4)
K1	N4	C1	C2	66.4(5)
K2	O2	N4	K1	160.06(15)
K2	O2	N4	O1	173.14(10)
K2	O2	N4	C1	-6.8(2)
K2	O3	N5	K2[3]	-145.12(18)
K2	O3	N5	O4	-170.93(12)
K2[3]	O3	N5	O4	-25.81(13)
K2[3]	O3	N5	C1	153.91(12)
K2	O3	N5	C1	8.8(3)
K2[3]	O4	N5	O3	25.47(13)

Atom	Atom	Atom	Atom	Angle/°
K2[3]	O4	N5	C1	-154.27(10)
K2[3]	N5	C1	N4	93.3(2)
K2[3]	N5	C1	C2	-85.2(3)
O1	N4	C1	N5	-176.21(13)
O1	N4	C1	C2	2.28(18)
O2	N4	C1	N5	3.8(2)
O2	N4	C1	C2	-177.73(12)
O3	N5	C1	N4	-4.2(2)
O3	N5	C1	C2	177.27(13)
O4	N5	C1	N4	175.51(13)
O4	N5	C1	C2	-3.00(19)
N1	C3	C4	N3	2.8(2)
N1	C3	C4	C6	-178.38(13)
N1	C3	C5	K2[4]	-48.1(4)
N2	N3	C4	C3	0.2(2)
N2	N3	C4	C6	-178.71(13)
N3	N2	C2	N1	2.8(2)
N3	N2	C2	C1	-177.58(12)
N4	C1	C2	N1	94.45(16)
N4	C1	C2	N2	-85.20(16)
N5	C1	C2	N1	-86.98(16)
N5	C1	C2	N2	93.38(16)
C2	N1	C3	C4	-2.81(19)
C2	N1	C3	C5	174.81(12)
C2	N2	N3	K1[2]	-179.83(9)
C2	N2	N3	C4	-2.79(19)
C3	N1	C2	N2	0.1(2)
C3	N1	C2	C1	-179.52(12)
C4	C3	C5	K2[4]	129.5(3)
C5	C3	C4	N3	-174.77(13)
C5	C3	C4	C6	4.1(2)

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

Table S8: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5 (Form i)**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H5A	8282.49	4358.82	1601.13	25
H5B	8333.83	3474.1	261.14	25
H5C	8407.04	3178.85	2150.76	25
H6A	6714.92	4587.28	-2291.93	30
H6B	7477.27	3949.55	-1992	30
H6C	7397.06	5046.22	-1125.68	30

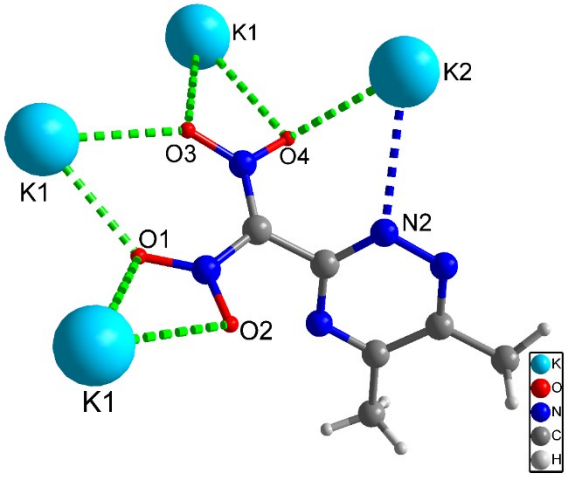


Figure S3: Thermal ellipsoids shown at 50% **5 (Form ii)**.

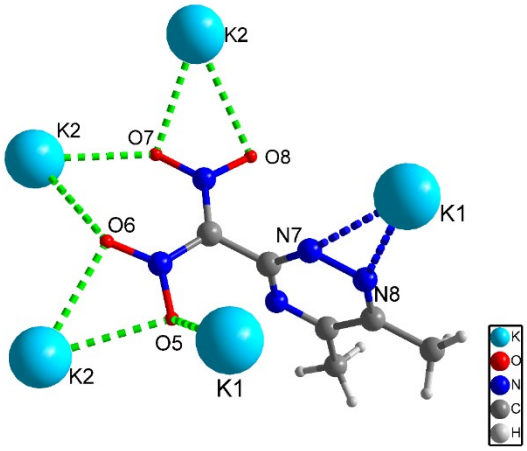


Figure S4: Thermal ellipsoids shown at 50% **5 (Form ii)**.

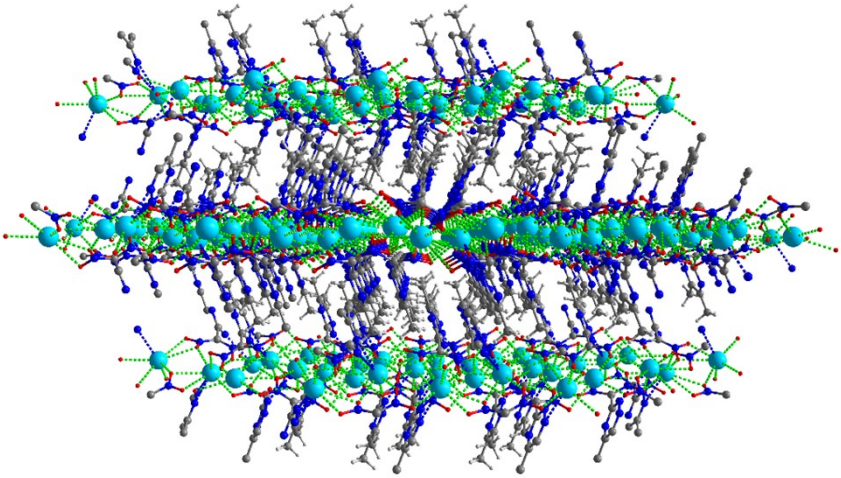


Figure S5: Ball-and-stick packing diagram of **5 (Form ii)** viewed up the b axis

Table S9: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5 (Form ii)**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
K1	8196.7(11)	6757.9(6)	5194.5(5)	20.4(2)
K2	5249.4(11)	1659.6(6)	5129.6(5)	21.3(2)
O1	9714(4)	8136(2)	4310.1(16)	23.6(6)
O2	11004(4)	9563(2)	4035.3(16)	23.1(6)
O3	6657(4)	8546(2)	4788.4(17)	25.4(6)
O4	5419(4)	9831(2)	4245.6(17)	23.8(6)
O5	7909(4)	4900(2)	4387.6(17)	23.6(6)
O6	6710(4)	3559(2)	4885.6(17)	22.8(6)
O7	3638(4)	3179(2)	4416.6(17)	25.5(6)
O8	2499(4)	4458(2)	3837.5(18)	28.1(7)
N1	8837(5)	10485(3)	2972.0(18)	19.3(7)
N2	7798(5)	11477(3)	3950(2)	25.9(8)
N3	7962(5)	12358(3)	3571(2)	27.0(8)
N4	9682(5)	9076(2)	4149.8(18)	18.6(7)
N5	6726(5)	9303(2)	4379(2)	20.5(7)
N6	5921(5)	5487(3)	3025.5(19)	20.2(7)
N7	4756(5)	6441(3)	3991(2)	26.3(8)
N8	4842(5)	7327(3)	3610(2)	27.2(8)
N9	6638(5)	4322(2)	4484(2)	20.4(7)
N10	3748(5)	4055(3)	4135(2)	20.8(7)
C1	8190(5)	9632(3)	4072(2)	19.7(8)
C2	8268(5)	10598(3)	3645(2)	20.3(8)
C3	9005(5)	11348(3)	2598(2)	19.9(8)
C4	8568(6)	12318(3)	2916(2)	22.5(8)
C5	9633(6)	11275(3)	1858(2)	24.5(9)
C6	8802(7)	13338(3)	2540(2)	28.5(9)
C7	5218(5)	4621(3)	4122(2)	19.8(8)
C8	5293(5)	5579(3)	3686(2)	19.1(8)
C9	6024(5)	6357(3)	2651(2)	19.8(8)
C10	5479(6)	7311(3)	2963(2)	21.3(8)
C11	5596(6)	8326(3)	2581(2)	28.1(9)
C31	6692(7)	6305(3)	1915(2)	28.1(9)

Table S10: Anisotropic Displacement Parameters ($\times 10^4$) for **5 (Form ii)**. 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}
K1	20.3(4)	15.9(3)	24.9(4)	2.2(3)	-1.3(3)	-1.4(3)
K2	20.9(4)	17.2(4)	25.9(4)	2.5(3)	0.3(3)	-0.2(3)
O1	29.8(15)	13.1(11)	27.8(14)	3.5(11)	4.8(13)	3.2(12)
O2	21.5(14)	21.8(13)	26.1(15)	6.3(12)	-1.1(12)	-1.5(12)
O3	24.7(14)	22.3(13)	29.3(15)	9.5(12)	2.2(13)	-0.1(12)
O4	19.9(14)	19.2(12)	32.4(15)	3.0(12)	-1.3(13)	2.4(12)
O5	23.7(15)	19.2(12)	27.9(15)	1.3(11)	-1.4(13)	-5.1(12)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O6	25.0(14)	14.9(12)	28.5(15)	5.5(11)	-0.1(13)	1.4(11)
O7	24.5(15)	17.8(13)	34.3(15)	7.0(12)	-0.9(13)	-4.1(12)
O8	21.4(15)	27.0(14)	35.8(16)	5.7(13)	-3.9(13)	1.5(13)
N1	21.1(17)	17.8(14)	18.9(15)	0.2(13)	-1.2(14)	0.2(13)
N2	37(2)	14.6(14)	26.1(17)	1.9(13)	7.4(16)	-1.7(15)
N3	37(2)	14.6(14)	29.7(18)	1.7(14)	9.0(17)	2.6(15)
N4	21.6(16)	17.2(14)	17.1(15)	1.7(12)	1.6(14)	0.9(14)
N5	20.5(16)	14.8(14)	26.1(17)	3.4(13)	-1.1(14)	1.1(13)
N6	21.0(17)	17.4(14)	22.4(15)	-1.3(13)	-1.0(14)	1.1(13)
N7	37(2)	16.9(15)	25.3(17)	0.7(14)	4.4(16)	2.3(15)
N8	35(2)	17.4(14)	28.7(18)	-0.9(15)	2.2(17)	1.9(15)
N9	22.3(17)	13.5(14)	25.4(17)	1.2(13)	0.9(14)	-2.1(13)
N10	19.2(17)	17.6(14)	25.7(17)	-1.3(13)	-1.0(14)	-1.7(14)
C1	18.2(19)	16.7(17)	24.3(19)	2.4(15)	1.4(16)	1.0(16)
C2	19.1(19)	18.2(16)	23.6(18)	-1.8(15)	0.9(17)	0.5(15)
C3	17.5(18)	18.3(16)	23.9(19)	-0.2(15)	-2.8(16)	1.8(15)
C4	24(2)	17.5(17)	26(2)	2.5(16)	2.2(17)	-0.8(16)
C5	27(2)	21.8(18)	24(2)	1.5(16)	-0.3(18)	0.1(18)
C6	39(2)	16.7(18)	30(2)	4.2(17)	7.8(19)	0.0(18)
C7	19.2(19)	17.6(16)	22.6(18)	3.1(15)	-0.6(16)	-1.5(16)
C8	18.1(19)	15.5(15)	23.7(18)	0.1(15)	-0.8(16)	0.3(15)
C9	19.1(18)	16.8(16)	23.6(18)	-0.9(15)	-2.8(16)	0.3(15)
C10	21.9(19)	16.6(17)	25.4(19)	2.0(16)	-1.1(17)	-1.2(15)
C11	35(2)	19.9(19)	29(2)	5.0(18)	-0.9(18)	2.3(18)
C31	33(2)	24.9(19)	27(2)	0.6(17)	1.4(19)	0.4(19)

Table S11: Bond Lengths in Å for **5 (Form ii)**.

Atom	Atom	Length/Å
K1	K1[1]	4.4754(7)
K1	O1	2.718(3)
K1	O1[1]	2.929(3)
K1	O2[1]	2.836(3)
K1	O3	2.718(3)
K1	O3[2]	2.782(3)
K1	O4[2]	2.903(3)
K1	O5	2.844(3)
K1	N4[1]	3.241(4)
K1	N5[2]	3.225(4)
K1	N7[2]	3.046(4)
K1	N8[2]	2.859(4)
K2	K2[3]	4.5565(7)
K2	O4[4]	2.887(3)
K2	O5[3]	2.886(3)
K2	O6[3]	2.832(3)
K2	O6	2.747(3)
K2	O7	2.698(3)

Atom	Atom	Length/Å
K2	O7[5]	2.838(3)
K2	O8[5]	3.011(3)
K2	N2[4]	3.019(4)
K2	N3[1]	3.303(4)
K2	N9[3]	3.224(4)
K2	N10[5]	3.244(4)
O1	N4	1.247(4)
O2	N4	1.244(5)
O3	N5	1.244(4)
O4	N5	1.268(5)
O5	N9	1.269(5)
O6	N9	1.242(4)
O7	N10	1.249(5)
O8	N10	1.254(5)
N1	C2	1.355(5)
N1	C3	1.324(5)
N2	N3	1.346(5)
N2	C2	1.323(5)
N3	C4	1.326(6)
N4	C1	1.395(5)
N5	C1	1.369(5)
N6	C8	1.347(6)
N6	C9	1.326(5)
N7	N8	1.349(5)
N7	C8	1.321(5)
N8	C10	1.321(6)
N9	C7	1.374(6)
N10	C7	1.379(5)
C1	C2	1.482(5)
C3	C4	1.429(6)
C3	C5	1.483(6)
C4	C6	1.504(6)
C7	C8	1.483(5)
C9	C10	1.429(5)
C9	C31	1.486(6)
C10	C11	1.496(5)

——[1]-1/2+x,3/2-y,1-z; [2]1/2+x,3/2-y,1-z; [3]-1/2+x,1/2-y,1-z; [4]+x,-1+y,+z; [5]1/2+x,1/2-y,1-z

Table S12: Bond Angles in ° for **5 (Form ii)**.

Atom	Atom	Atom	Angle/°
O1	K1	K1[1]	90.91(7)
O1[1]	K1	K1[1]	35.95(6)
O1	K1	O1[1]	125.85(8)
O1	K1	O2[1]	168.17(10)
O1	K1	O3[2]	70.06(9)
O1	K1	O3	58.30(9)

Atom	Atom	Atom	Angle/°
O1	K1	O4^{2}	114.36(9)
O1	K1	O5	104.92(9)
O1	K1	N4^{1}	146.46(9)
O1^{1}	K1	N4^{1}	22.59(8)
O1^{1}	K1	N5^{2}	139.89(9)
O1	K1	N5^{2}	92.43(9)
O1	K1	N7^{2}	68.33(10)
O1^{1}	K1	N7^{2}	100.94(10)
O1	K1	N8^{2}	90.60(11)
O2^{1}	K1	K1^{1}	78.03(6)
O2^{1}	K1	O1^{1}	44.13(8)
O2^{1}	K1	O4^{2}	76.49(9)
O2^{1}	K1	O5	73.71(9)
O2^{1}	K1	N4^{1}	22.37(8)
O2^{1}	K1	N5^{2}	98.89(9)
O2^{1}	K1	N7^{2}	116.70(10)
O2^{1}	K1	N8^{2}	97.23(11)
O3^{2}	K1	K1^{1}	160.44(7)
O3	K1	K1^{1}	36.02(7)
O3	K1	O1^{1}	67.88(9)
O3^{2}	K1	O1^{1}	160.06(9)
O3	K1	O2^{1}	111.98(9)
O3^{2}	K1	O2^{1}	121.27(9)
O3	K1	O3^{2}	124.67(8)
O3^{2}	K1	O4^{2}	45.06(9)
O3	K1	O4^{2}	166.92(9)
O3	K1	O5	121.72(10)
O3^{2}	K1	O5	88.15(9)
O3	K1	N4^{1}	89.91(9)
O3^{2}	K1	N4^{1}	143.47(9)
O3^{2}	K1	N5^{2}	22.38(9)
O3	K1	N5^{2}	145.25(9)
O3^{2}	K1	N7^{2}	72.40(10)
O3	K1	N7^{2}	71.39(10)
O3	K1	N8^{2}	94.56(10)
O3^{2}	K1	N8^{2}	66.13(11)
O4^{2}	K1	K1^{1}	154.45(7)
O4^{2}	K1	O1^{1}	119.58(9)
O4^{2}	K1	N4^{1}	98.85(9)
O4^{2}	K1	N5^{2}	23.13(8)
O4^{2}	K1	N7^{2}	96.04(10)
O5	K1	K1^{1}	101.54(7)
O5	K1	O1^{1}	97.66(9)
O5	K1	O4^{2}	69.53(9)
O5	K1	N4^{1}	81.70(9)
O5	K1	N5^{2}	81.21(9)
O5	K1	N7^{2}	160.54(11)

Atom	Atom	Atom	Angle/°
O5	K1	N8[2]	143.56(10)
N4[1]	K1	K1[1]	55.68(6)
N5[2]	K1	K1[1]	174.98(8)
N5[2]	K1	N4[1]	121.10(9)
N7[2]	K1	K1[1]	96.88(8)
N7[2]	K1	N4[1]	114.31(10)
N7[2]	K1	N5[2]	80.89(10)
N8[2]	K1	K1[1]	111.12(9)
N8[2]	K1	O1[1]	99.40(11)
N8[2]	K1	O4[2]	74.05(10)
N8[2]	K1	N4[1]	103.38(11)
N8[2]	K1	N5[2]	65.11(10)
N8[2]	K1	N7[2]	26.17(10)
O4[3]	K2	K2[4]	111.48(7)
O4[3]	K2	O8[5]	87.44(9)
O4[3]	K2	N2[3]	58.69(9)
O4[3]	K2	N3[1]	139.97(9)
O4[3]	K2	N9[4]	81.50(9)
O4[3]	K2	N10[5]	88.58(9)
O5[4]	K2	K2[4]	78.49(6)
O5[4]	K2	O4[3]	69.18(8)
O5[4]	K2	O8[5]	81.23(9)
O5[4]	K2	N2[3]	127.71(9)
O5[4]	K2	N3[1]	71.17(9)
O5[4]	K2	N9[4]	23.13(8)
O5[4]	K2	N10[5]	102.84(9)
O6	K2	K2[4]	85.91(7)
O6[4]	K2	K2[4]	34.63(5)
O6	K2	O4[3]	127.58(9)
O6[4]	K2	O4[3]	87.67(9)
O6	K2	O5[4]	160.98(9)
O6[4]	K2	O5[4]	44.90(8)
O6	K2	O6[4]	120.53(7)
O6[4]	K2	O7[5]	162.99(10)
O6	K2	O7[5]	65.35(9)
O6[4]	K2	O8[5]	123.42(9)
O6	K2	O8[5]	106.39(9)
O6[4]	K2	N2[3]	130.81(10)
O6	K2	N2[3]	70.23(9)
O6[4]	K2	N3[1]	59.81(9)
O6	K2	N3[1]	90.92(9)
O6	K2	N9[4]	139.85(9)
O6[4]	K2	N9[4]	22.48(8)
O6[4]	K2	N10[5]	146.13(9)
O6	K2	N10[5]	87.75(9)
O7[5]	K2	K2[4]	145.89(7)
O7	K2	K2[4]	35.60(7)

Atom	Atom	Atom	Angle/°
O7	K2	O4[3]	109.05(10)
O7[5]	K2	O4[3]	100.90(9)
O7	K2	O5[4]	110.78(9)
O7[5]	K2	O5[4]	124.72(9)
O7	K2	O6[4]	66.06(9)
O7	K2	O6	58.12(9)
O7	K2	O7[5]	123.29(7)
O7	K2	O8[5]	162.04(10)
O7[5]	K2	O8[5]	43.51(8)
O7	K2	N2[3]	90.54(10)
O7[5]	K2	N2[3]	65.70(10)
O7	K2	N3[1]	80.16(10)
O7[5]	K2	N3[1]	105.82(10)
O7	K2	N9[4]	88.46(9)
O7[5]	K2	N9[4]	143.94(9)
O7[5]	K2	N10[5]	22.45(9)
O7	K2	N10[5]	145.74(9)
O8[5]	K2	K2[4]	144.66(7)
O8[5]	K2	N2[3]	92.23(10)
O8[5]	K2	N3[1]	91.83(9)
O8[5]	K2	N9[4]	101.35(9)
O8[5]	K2	N10[5]	22.74(9)
N2[3]	K2	K2[4]	123.04(8)
N2[3]	K2	N3[1]	161.10(9)
N2[3]	K2	N9[4]	137.32(10)
N2[3]	K2	N10[5]	73.46(10)
N3[1]	K2	K2[4]	54.27(7)
N9[4]	K2	K2[4]	55.36(6)
N9[4]	K2	N3[1]	59.42(9)
N9[4]	K2	N10[5]	123.91(9)
N10[5]	K2	K2[4]	158.49(7)
N10[5]	K2	N3[1]	105.38(9)
K1	O1	K1[2]	104.80(10)
N4	O1	K1	140.6(3)
N4	O1	K1[2]	92.9(2)
N4	O2	K1[2]	97.4(2)
K1	O3	K1[1]	108.92(10)
N5	O3	K1	144.9(3)
N5	O3	K1[1]	99.3(2)
K2[6]	O4	K1[1]	109.68(11)
N5	O4	K1[1]	92.8(2)
N5	O4	K2[6]	111.2(2)
K1	O5	K2[5]	111.40(11)
N9	O5	K1	118.7(2)
N9	O5	K2[5]	93.5(2)
K2	O6	K2[5]	109.51(9)
N9	O6	K2[5]	96.8(2)

Atom	Atom	Atom	Angle/°
N9	O6	K2	141.9(2)
K2	O7	K2[4]	110.79(10)
N10	O7	K2	146.4(3)
N10	O7	K2[4]	97.4(2)
N10	O8	K2[4]	89.1(2)
C3	N1	C2	116.2(3)
N3	N2	K2[6]	112.9(3)
C2	N2	K2[6]	125.2(3)
C2	N2	N3	117.6(3)
N2	N3	K2[2]	89.0(3)
C4	N3	K2[2]	120.3(3)
C4	N3	N2	119.8(3)
O1	N4	K1[2]	64.5(2)
O1	N4	C1	122.8(4)
O2	N4	K1[2]	60.19(19)
O2	N4	O1	120.9(4)
O2	N4	C1	116.3(3)
C1	N4	K1[2]	160.9(3)
O3	N5	K1[1]	58.4(2)
O3	N5	O4	120.5(3)
O3	N5	C1	122.8(3)
O4	N5	K1[1]	64.0(2)
O4	N5	C1	116.6(3)
C1	N5	K1[1]	168.1(3)
C9	N6	C8	116.2(4)
N8	N7	K1[1]	69.2(2)
C8	N7	K1[1]	172.3(3)
C8	N7	N8	117.7(4)
N7	N8	K1[1]	84.7(2)
C10	N8	K1[1]	155.5(3)
C10	N8	N7	119.7(4)
O5	N9	K2[5]	63.3(2)
O5	N9	C7	114.9(3)
O6	N9	K2[5]	60.7(2)
O6	N9	O5	120.9(3)
O6	N9	C7	124.1(3)
C7	N9	K2[5]	162.7(3)
O7	N10	K2[4]	60.2(2)
O7	N10	O8	120.5(4)
O7	N10	C7	123.0(4)
O8	N10	K2[4]	68.1(2)
O8	N10	C7	116.5(3)
C7	N10	K2[4]	152.4(3)
N4	C1	C2	116.9(4)
N5	C1	N4	121.4(3)
N5	C1	C2	121.7(4)
N1	C2	C1	115.6(3)

Atom	Atom	Atom	Angle/°
N2	C2	N1	126.4(4)
N2	C2	C1	118.1(4)
N1	C3	C4	119.1(4)
N1	C3	C5	118.8(4)
C4	C3	C5	122.1(4)
N3	C4	C3	120.9(4)
N3	C4	C6	116.8(4)
C3	C4	C6	122.3(4)
N9	C7	N10	122.8(3)
N9	C7	C8	118.3(4)
N10	C7	C8	118.9(4)
N6	C8	C7	117.0(3)
N7	C8	N6	126.5(4)
N7	C8	C7	116.5(4)
N6	C9	C10	119.2(4)
N6	C9	C31	118.7(4)
C10	C9	C31	122.1(4)
N8	C10	C9	120.7(4)
N8	C10	C11	117.0(4)
C9	C10	C11	122.3(4)

——[1]-1/2+x,3/2-y,1-z; [2]1/2+x,3/2-y,1-z; [3]+x,-1+y,+z; [4]-1/2+x,1/2-y,1-z; [5]1/2+x,1/2-y,1-z; [6]+x,1+y,+z

Table S13: Torsion Angles in ° for **5 (Form ii)**.

Atom	Atom	Atom	Atom	Angle/°
K1	O1	N4	K1[1]	117.8(4)
K1	O1	N4	O2	139.6(4)
K1[1]	O1	N4	O2	21.8(4)
K1[1]	O1	N4	C1	-159.7(3)
K1	O1	N4	C1	-42.0(6)
K1[1]	O2	N4	O1	-22.7(4)
K1[1]	O2	N4	C1	158.7(3)
K1	O3	N5	K1[2]	143.4(5)
K1[2]	O3	N5	O4	16.5(4)
K1	O3	N5	O4	159.9(3)
K1[2]	O3	N5	C1	-166.0(3)
K1	O3	N5	C1	-22.5(7)
K1[2]	O4	N5	O3	-15.6(4)
K1[2]	O4	N5	C1	166.7(3)
K1	O5	N9	K2[3]	-116.8(2)
K1	O5	N9	O6	-96.8(4)
K1	O5	N9	C7	82.3(4)
K1[1]	N4	C1	N5	-85.6(8)
K1[1]	N4	C1	C2	94.1(8)
K1[2]	N5	C1	N4	-86.0(13)
K1[2]	N5	C1	C2	94.3(12)
K1[2]	N7	N8	C10	-177.3(5)

Atom	Atom	Atom	Atom	Angle/°
K1[2]	N8	C10	C9	-171.3(5)
K1[2]	N8	C10	C11	8.7(10)
K2[4]	O4	N5	K1[2]	112.39(18)
K2[4]	O4	N5	O3	96.8(3)
K2[4]	O4	N5	C1	-80.9(4)
K2[3]	O5	N9	O6	20.0(4)
K2[3]	O5	N9	C7	-160.9(3)
K2	O6	N9	K2[3]	-134.2(4)
K2	O6	N9	O5	-154.8(3)
K2[3]	O6	N9	O5	-20.5(4)
K2	O6	N9	C7	26.2(6)
K2[3]	O6	N9	C7	160.5(3)
K2	O7	N10	K2[5]	-147.3(5)
K2	O7	N10	O8	179.5(3)
K2[5]	O7	N10	O8	-33.2(4)
K2	O7	N10	C7	0.3(7)
K2[5]	O7	N10	C7	147.7(4)
K2[5]	O8	N10	O7	30.8(4)
K2[5]	O8	N10	C7	-150.1(3)
K2[4]	N2	N3	K2[1]	77.55(19)
K2[4]	N2	N3	C4	-157.6(4)
K2[4]	N2	C2	N1	151.9(3)
K2[4]	N2	C2	C1	-28.6(6)
K2[1]	N3	C4	C3	110.0(4)
K2[1]	N3	C4	C6	-68.7(5)
K2[3]	N9	C7	N10	96.6(9)
K2[3]	N9	C7	C8	-81.8(9)
K2[5]	N10	C7	N9	80.5(7)
K2[5]	N10	C7	C8	-101.2(6)
O1	N4	C1	N5	21.4(6)
O1	N4	C1	C2	-158.9(4)
O2	N4	C1	N5	-160.1(4)
O2	N4	C1	C2	19.6(5)
O3	N5	C1	N4	6.0(6)
O3	N5	C1	C2	-173.7(4)
O4	N5	C1	N4	-176.3(4)
O4	N5	C1	C2	4.0(6)
O5	N9	C7	N10	176.5(4)
O5	N9	C7	C8	-1.9(5)
O6	N9	C7	N10	-4.4(7)
O6	N9	C7	C8	177.2(4)
O7	N10	C7	N9	-7.2(7)
O7	N10	C7	C8	171.1(4)
O8	N10	C7	N9	173.6(4)
O8	N10	C7	C8	-8.0(6)
N1	C3	C4	N3	-1.6(7)
N1	C3	C4	C6	177.0(4)

Atom	Atom	Atom	Atom	Angle/°
N2	N3	C4	C3	1.8(7)
N2	N3	C4	C6	-176.8(4)
N3	N2	C2	N1	-3.1(7)
N3	N2	C2	C1	176.4(4)
N4	C1	C2	N1	57.1(5)
N4	C1	C2	N2	-122.4(4)
N5	C1	C2	N1	-123.2(4)
N5	C1	C2	N2	57.3(6)
N6	C9	C10	N8	-1.6(7)
N6	C9	C10	C11	178.4(4)
N7	N8	C10	C9	2.3(7)
N7	N8	C10	C11	-177.8(4)
N8	N7	C8	N6	-0.3(7)
N8	N7	C8	C7	178.7(4)
N9	C7	C8	N6	83.7(5)
N9	C7	C8	N7	-95.5(5)
N10	C7	C8	N6	-94.8(5)
N10	C7	C8	N7	86.1(5)
C2	N1	C3	C4	-0.8(6)
C2	N1	C3	C5	179.6(4)
C2	N2	N3	K2[1]	-124.5(4)
C2	N2	N3	C4	0.4(7)
C3	N1	C2	N2	3.2(7)
C3	N1	C2	C1	-176.2(4)
C5	C3	C4	N3	178.0(4)
C5	C3	C4	C6	-3.4(7)
C8	N6	C9	C10	0.0(6)
C8	N6	C9	C31	-179.4(4)
C8	N7	N8	K1[2]	176.0(4)
C8	N7	N8	C10	-1.3(6)
C9	N6	C8	N7	0.9(6)
C9	N6	C8	C7	-178.1(4)
C31	C9	C10	N8	177.9(4)
C31	C9	C10	C11	-2.1(7)

——[1]1/2+x,3/2-y,1-z; [2]-1/2+x,3/2-y,1-z; [3]1/2+x,1/2-y,1-z; [4]+x,1+y,+z; [5]-1/2+x,1/2-y,1-z

Table S14: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5 (Form ii)**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H5A	10102.36	10581.61	1776.71	37
H5B	10507.8	11798.9	1781.91	37
H5C	8703.74	11396.38	1527.21	37
H6A	8298.65	13896.06	2822.09	43
H6B	8255.16	13308.23	2073.96	43
H6C	10004.24	13473.84	2477.19	43

Atom	x	y	z	U_{eq}
H11A	5141.4	8879.62	2881.68	42
H11B	4949.64	8289.02	2138.75	42
H11C	6775.29	8475.9	2470.79	42
H31A	7215.39	5625.76	1836.37	42
H31B	7529.92	6853.21	1846.87	42
H31C	5769.63	6401.31	1576.67	42

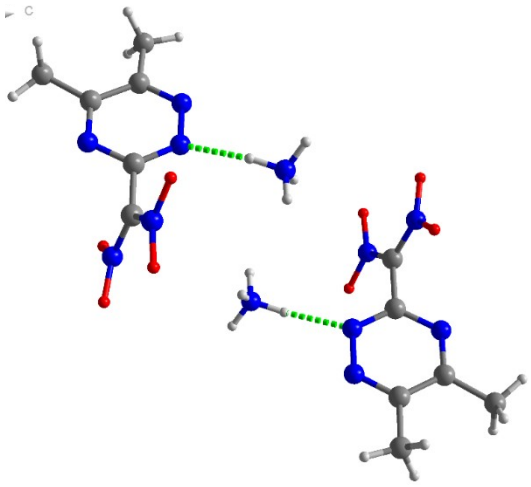


Figure S6: Thermal ellipsoids shown at 50% **5c**.

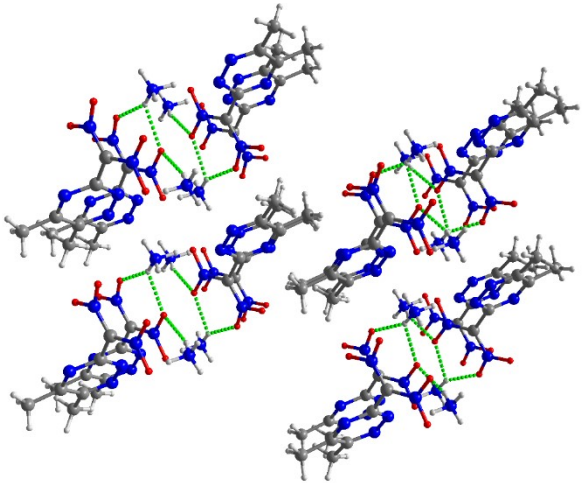


Figure S7: Ball-and-stick packing diagram of **5c** viewed up the a axis.

Table S15: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5c**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
O1	357.1(17)	5721.6(13)	7272.6(8)	27.7(2)
O2	-2414.0(16)	4278.5(13)	8327.8(8)	23.8(2)

Atom	x	y	z	U_{eq}
O3	2233.1(14)	4214.0(11)	5485.6(7)	18.0(2)
O4	1292.4(14)	1436.8(12)	5505.0(8)	19.8(2)
N1	-3288.7(17)	1005.5(14)	6790.9(9)	17.6(2)
N2	-4730.4(16)	-301.2(14)	7272.8(9)	17.0(2)
N3	-1436.1(16)	275.2(13)	8503.4(8)	14.9(2)
N4	-800.2(17)	4369.2(14)	7518.1(9)	17.2(2)
N5	1097.1(16)	2865.6(13)	5948.8(8)	14.4(2)
C1	-1793.5(19)	1288.6(16)	7435.0(10)	15.0(2)
C2	-4595.1(18)	-1260.9(16)	8350.4(10)	14.5(2)
C3	-2814.6(18)	-1033.5(15)	8966.9(10)	14.1(2)
C4	-409.4(19)	2875.2(16)	6944.8(10)	15.3(2)
C5	-6358.2(19)	-2565.1(17)	8878.7(11)	17.8(2)
C6	-2429.3(19)	-2265.2(16)	10122.3(10)	17.9(2)
N6	3704.7(17)	7622.8(14)	5668.7(9)	15.5(2)

Table S16: Anisotropic Displacement Parameters ($\times 10^4$) for **5c**. 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	36.1(5)	24.1(5)	26.5(5)	-12.2(4)	10.9(4)	-19.9(4)
O2	25.7(4)	21.8(4)	22.5(5)	-7.3(4)	10.3(4)	-7.5(3)
O3	19.0(4)	15.6(4)	18.9(4)	-2.8(3)	3.2(3)	-7.5(3)
O4	25.0(4)	15.8(4)	20.6(4)	-8.3(3)	0.7(3)	-5.0(3)
N1	19.4(5)	19.0(5)	15.4(5)	-2.3(4)	-2.6(4)	-7.1(4)
N2	18.7(5)	18.1(5)	15.4(5)	-2.8(4)	-2.3(4)	-6.6(4)
N3	16.7(4)	14.6(5)	13.8(5)	-2.7(4)	-1.3(3)	-3.7(4)
N4	20.3(5)	16.0(5)	15.2(5)	-3.0(4)	1.3(4)	-6.0(4)
N5	15.9(4)	13.3(4)	14.3(5)	-2.1(3)	-2.2(4)	-3.1(3)
C1	16.5(5)	14.7(5)	13.9(5)	-2.9(4)	-0.2(4)	-3.3(4)
C2	15.5(5)	13.5(5)	15.1(5)	-4.4(4)	-0.8(4)	-2.4(4)
C3	14.8(5)	12.4(5)	15.3(5)	-4.5(4)	-0.1(4)	-1.8(4)
C4	17.7(5)	14.7(5)	14.2(5)	-3.6(4)	-0.7(4)	-4.7(4)
C5	15.8(5)	18.8(5)	19.5(6)	-3.5(4)	-0.9(4)	-6.6(4)
C6	18.7(5)	18.4(5)	16.6(6)	0.2(4)	-4.0(4)	-5.7(4)
N6	18.7(5)	14.6(5)	14.1(5)	-3.6(4)	0.4(4)	-6.1(4)

Table S17: Bond Lengths in Å for **5c**.

Atom	Atom	Length/Å
O1	N4	1.2446(13)
O2	N4	1.2507(13)
O3	N5	1.2545(12)
O4	N5	1.2559(13)
N1	N2	1.3513(13)
N1	C1	1.3241(15)
N2	C2	1.3233(15)
N3	C1	1.3497(15)

Atom	Atom	Length/Å
N3	C3	1.3235(14)
N4	C4	1.3870(15)
N5	C4	1.3728(15)
C1	C4	1.4829(15)
C2	C3	1.4253(15)
C2	C5	1.4944(15)
C3	C6	1.4942(15)

Table S18: Bond Angles in ° for **5c**.

Atom	Atom	Atom	Angle/°
C1	N1	N2	117.82(10)
C2	N2	N1	119.80(9)
C3	N3	C1	116.25(9)
O1	N4	O2	120.70(10)
O1	N4	C4	123.79(10)
O2	N4	C4	115.51(9)
O3	N5	O4	120.57(9)
O3	N5	C4	122.43(10)
O4	N5	C4	117.00(9)
N1	C1	N3	125.80(10)
N1	C1	C4	117.07(10)
N3	C1	C4	117.13(9)
N2	C2	C3	120.14(10)
N2	C2	C5	117.63(10)
C3	C2	C5	122.23(10)
N3	C3	C2	119.69(10)
N3	C3	C6	118.71(10)
C2	C3	C6	121.59(10)
N4	C4	C1	116.37(10)
N5	C4	N4	122.90(10)
N5	C4	C1	120.68(10)

Table S19: Torsion Angles in ° for **5c**.

Atom	Atom	Atom	Atom	Angle/°
O1	N4	C4	N5	-9.02(18)
O1	N4	C4	C1	173.62(10)
O2	N4	C4	N5	171.62(10)
O2	N4	C4	C1	-5.74(15)
O3	N5	C4	N4	0.06(17)
O3	N5	C4	C1	177.30(9)
O4	N5	C4	N4	-179.53(10)
O4	N5	C4	C1	-2.28(15)
N1	N2	C2	C3	-5.26(16)
N1	N2	C2	C5	174.72(10)
N1	C1	C4	N4	113.45(12)

Atom	Atom	Atom	Atom	Angle/°
N1	C1	C4	N5	-63.97(14)
N2	N1	C1	N3	6.08(17)
N2	N1	C1	C4	-173.11(10)
N2	C2	C3	N3	6.72(16)
N2	C2	C3	C6	-172.13(10)
N3	C1	C4	N4	-65.81(14)
N3	C1	C4	N5	116.77(12)
C1	N1	N2	C2	-0.77(16)
C1	N3	C3	C2	-1.88(15)
C1	N3	C3	C6	177.00(10)
C3	N3	C1	N1	-4.58(17)
C3	N3	C1	C4	174.61(9)
C5	C2	C3	N3	-173.27(10)
C5	C2	C3	C6	7.88(17)

Table S20: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5c**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H5A	-7197.06	-2181.22	9580.53	27
H5B	-5611.31	-3854.98	9085.66	27
H5C	-7422.48	-2514.2	8316.44	27
H6A	-3893.85	-2380.17	10618.73	27
H6B	-1416.05	-1706.22	10494.58	27
H6C	-1725.28	-3516.99	10011.51	27
H6D	3820(30)	8510(20)	6074(15)	26(4)
H6E	2770(30)	6750(20)	6023(15)	27(4)
H6F	3270(30)	8230(30)	4931(17)	33(4)
H6G	5100(40)	7030(30)	5503(17)	36(5)

Table S21: Hydrogen Bond information for **5c**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N6	H6E	O1	0.900(18)	2.000(18)	2.8312(13)	152.8(16)
N6	H6E	O3	0.900(18)	2.171(17)	2.8242(12)	128.9(15)

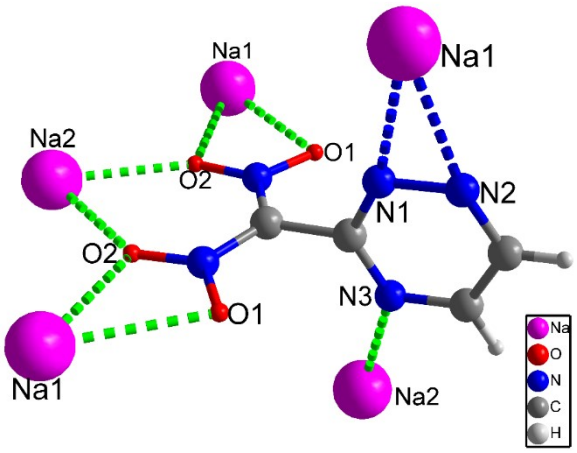


Figure S8: Thermal ellipsoids shown at 50% **5e**.

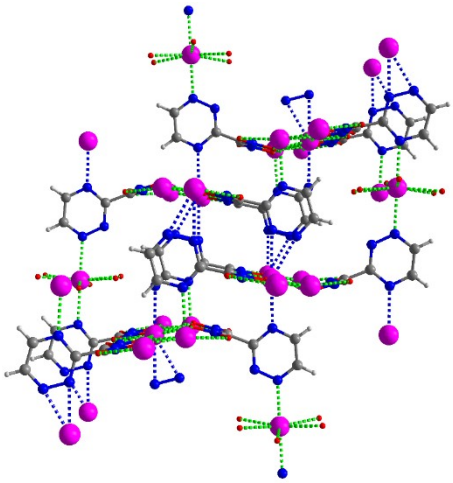


Figure S9: Ball-and-stick packing diagram of **5e** viewed up the b axis.

Table S22: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5e**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
Na1	6599.1(6)	7500	6525.9(4)	15.87(18)
Na2	11948.0(6)	7500	3343.7(4)	15.67(18)
O1	4741.0(8)	5473.5(12)	6278.8(6)	21.1(2)
O2	6299.4(7)	4243.1(12)	6499.7(6)	19.6(2)
O3	8234.2(7)	5741.5(12)	6880.8(6)	16.0(2)
O4	9766.2(7)	4528.2(12)	6549.7(6)	19.5(2)
N1	3385.5(13)	2500	5188.3(10)	18.8(3)
N2	2332.8(13)	2500	4967.1(10)	20.8(3)
N3	2936.3(12)	2500	6709.7(9)	15.0(3)
N4	5314.3(8)	4105.8(14)	6345.8(7)	15.2(2)
N5	10872.6(12)	7500	5326.4(10)	18.3(3)

Atom	x	y	z	U_{eq}
N6	11855.8(12)	7500	4955.3(10)	18.4(3)
N7	11638.0(12)	7500	6765.6(9)	15.1(3)
N8	9198.8(8)	5891.1(14)	6666.0(6)	13.8(2)
C1	3638.3(14)	2500	6036.9(11)	14.1(3)
C2	1605.6(15)	2500	5596.4(12)	19.0(4)
C3	1909.7(15)	2500	6480.4(12)	17.7(4)
C4	4800.2(14)	2500	6241.8(12)	15.4(4)
C5	10812.8(14)	7500	6198.9(11)	13.5(3)
C6	12705.1(15)	7500	5477.0(12)	18.1(4)
C7	12601.5(15)	7500	6390.5(11)	16.4(4)
C8	9704.1(14)	7500	6548.4(11)	13.3(3)

Table S23: Anisotropic Displacement Parameters ($\times 10^4$) for **5e**. 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}
Na1	17.1(4)	14.8(3)	15.7(3)	0	0.9(3)	0
Na2	18.5(4)	14.0(3)	14.5(3)	0	0.5(3)	0
O1	18.2(5)	11.7(4)	33.5(5)	1.1(4)	1.6(4)	4.2(4)
O2	11.4(4)	17.4(4)	30.0(5)	-0.8(3)	-1.7(3)	-2.3(3)
O3	11.1(4)	16.1(4)	20.7(4)	1.0(3)	1.6(3)	-1.6(3)
O4	17.3(4)	12.3(4)	29.0(5)	-1.8(3)	1.9(3)	3.8(4)
N1	16.7(7)	23.2(8)	16.6(7)	0	1.0(6)	0
N2	19.4(8)	25.3(8)	17.6(7)	0	-3.3(6)	0
N3	14.6(7)	15.0(7)	15.4(7)	0	0.2(5)	0
N4	12.5(5)	14.1(5)	19.1(5)	-0.4(4)	1.2(4)	0.2(4)
N5	14.8(7)	23.9(8)	16.3(7)	0	-0.3(6)	0
N6	14.3(7)	24.7(8)	16.1(7)	0	1.2(6)	0
N7	13.6(7)	15.8(7)	16.1(7)	0	-1.2(5)	0
N8	13.3(5)	13.8(5)	14.2(5)	-0.6(4)	-1.1(4)	0.9(4)
C1	13.7(8)	11.3(8)	17.2(8)	0	0.3(6)	0
C2	15.2(9)	22.8(9)	18.9(8)	0	-3.1(7)	0
C3	13.9(9)	20.8(9)	18.5(8)	0	1.8(7)	0
C4	13.2(8)	11.0(8)	21.8(8)	0	0.1(6)	0
C5	12.4(8)	11.8(8)	16.2(8)	0	0.5(6)	0
C6	13.9(8)	22.3(9)	18.1(8)	0	1.9(7)	0
C7	12.4(8)	19.3(8)	17.5(8)	0	-1.4(7)	0
C8	11.5(8)	12.2(8)	16.1(8)	0	-0.3(6)	0

Table S24: Bond Lengths in Å for **5e**.

Atom	Atom	Length/Å
Na1	Na2[1]	4.1732(4)
Na1	O1	2.7940(11)
Na1	O1[2]	2.7940(11)
Na1	O2	2.4751(9)
Na1	O2[2]	2.4751(9)

Atom	Atom	Length/Å
Na1	O3[2]	2.4856(10)
Na1	O3	2.4856(10)
Na1	N1[3]	2.6132(16)
Na1	N2[3]	2.6358(17)
Na1	N4[2]	3.0220(11)
Na1	N4	3.0220(11)
Na1	N7[4]	2.6048(16)
Na2	O2[5]	2.5550(11)
Na2	O2[6]	2.5550(11)
Na2	O3[6]	2.4693(9)
Na2	O3[5]	2.4693(9)
Na2	O4[6]	2.6267(11)
Na2	O4[5]	2.6267(11)
Na2	N3[7]	2.4949(15)
Na2	N6	2.4593(16)
Na2	N8[5]	2.9199(11)
Na2	N8[6]	2.9199(11)
O1	N4	1.2550(13)
O2	N4	1.2525(14)
O3	N8	1.2494(14)
O4	N8	1.2563(13)
N1	N2	1.353(2)
N1	C1	1.331(2)
N2	C2	1.319(3)
N3	C1	1.347(2)
N3	C3	1.325(2)
N4	C4	1.3747(13)
N5	N6	1.348(2)
N5	C5	1.332(2)
N6	C6	1.323(2)
N7	C5	1.342(2)
N7	C7	1.328(2)
N8	C8	1.3742(13)
C1	C4	1.479(2)
C2	C3	1.400(3)
C5	C8	1.479(2)
C6	C7	1.398(2)

——[1]2-x,2-y,1-z; [2]+x,3/2-y,+z; [3]1-x,1-y,1-z; [4]-1/2+x,+y,3/2-z; [5]2-x,1-y,1-z; [6]2-x,1/2+y,1-z; [7]3/2-x,1-y,-1/2+z

Table S25: Bond Angles in ° for **5e**.

Atom	Atom	Atom	Angle/°
O1[1]	Na1	Na2[2]	82.80(2)
O1	Na1	Na2[2]	148.80(3)
O1	Na1	O1[1]	66.03(4)
O1	Na1	N4	24.52(3)
O1[1]	Na1	N4	90.55(3)

Atom	Atom	Atom	Angle/°
O1[1]	Na1	N4[1]	24.52(3)
O1	Na1	N4[1]	90.55(3)
O2[1]	Na1	Na2[2]	34.56(2)
O2	Na1	Na2[2]	162.87(3)
O2[1]	Na1	O1[1]	48.29(3)
O2	Na1	O1	48.28(3)
O2[1]	Na1	O1	114.31(4)
O2	Na1	O1[1]	114.31(4)
O2	Na1	O2[1]	162.57(5)
O2[1]	Na1	O3[1]	66.52(3)
O2	Na1	O3	66.52(3)
O2	Na1	O3[1]	130.70(4)
O2[1]	Na1	O3	130.70(4)
O2[1]	Na1	N1[3]	89.14(3)
O2	Na1	N1[3]	89.14(3)
O2[1]	Na1	N2[3]	93.56(3)
O2	Na1	N2[3]	93.56(3)
O2[1]	Na1	N4	138.83(4)
O2	Na1	N4[1]	138.83(4)
O2	Na1	N4	23.78(3)
O2[1]	Na1	N4[1]	23.78(3)
O2	Na1	N7[4]	91.09(3)
O2[1]	Na1	N7[4]	91.09(3)
O3	Na1	Na2[2]	96.50(3)
O3[1]	Na1	Na2[2]	32.50(2)
O3	Na1	O1	114.67(3)
O3[1]	Na1	O1	175.17(4)
O3	Na1	O1[1]	175.16(4)
O3[1]	Na1	O1[1]	114.67(3)
O3[1]	Na1	O3	64.21(4)
O3[1]	Na1	N1[3]	102.21(4)
O3	Na1	N1[3]	102.21(4)
O3[1]	Na1	N2[3]	77.00(4)
O3	Na1	N2[3]	77.00(4)
O3	Na1	N4[1]	154.33(3)
O3[1]	Na1	N4	154.33(3)
O3[1]	Na1	N4[1]	90.26(3)
O3	Na1	N4	90.26(3)
O3	Na1	N7[4]	76.54(4)
O3[1]	Na1	N7[4]	76.54(4)
N1[3]	Na1	Na2[2]	92.55(2)
N1[3]	Na1	O1	82.60(4)
N1[3]	Na1	O1[1]	82.61(4)
N1[3]	Na1	N2[3]	29.87(5)
N1[3]	Na1	N4	85.01(3)
N1[3]	Na1	N4[1]	85.01(3)
N2[3]	Na1	Na2[2]	79.78(2)

Atom	Atom	Atom	Angle/°
N2[3]	Na1	O1[1]	107.52(4)
N2[3]	Na1	O1	107.52(4)
N2[3]	Na1	N4	100.86(3)
N2[3]	Na1	N4[1]	100.86(3)
N4	Na1	Na2[2]	173.16(3)
N4[1]	Na1	Na2[2]	58.28(2)
N4[1]	Na1	N4	115.07(5)
N7[4]	Na1	Na2[2]	86.81(2)
N7[4]	Na1	O1[1]	98.64(4)
N7[4]	Na1	O1	98.64(4)
N7[4]	Na1	N1[3]	178.51(6)
N7[4]	Na1	N2[3]	148.64(6)
N7[4]	Na1	N4[1]	95.78(3)
N7[4]	Na1	N4	95.78(3)
Na1[2]	Na2	Na1[5]	128.32(2)
O2[6]	Na2	Na1[5]	94.99(3)
O2[5]	Na2	Na1[5]	33.33(2)
O2[6]	Na2	Na1[2]	33.33(2)
O2[5]	Na2	Na1[2]	94.99(3)
O2[6]	Na2	O2[5]	61.66(4)
O2[6]	Na2	O4[5]	170.00(4)
O2[5]	Na2	O4[5]	112.97(3)
O2[6]	Na2	O4[6]	112.97(3)
O2[5]	Na2	O4[6]	170.00(4)
O2[5]	Na2	N8[5]	88.32(3)
O2[6]	Na2	N8[5]	149.86(3)
O2[6]	Na2	N8[6]	88.32(3)
O2[5]	Na2	N8[6]	149.86(3)
O3[5]	Na2	Na1[5]	32.74(2)
O3[6]	Na2	Na1[5]	159.05(3)
O3[5]	Na2	Na1[2]	159.05(3)
O3[6]	Na2	Na1[2]	32.74(2)
O3[5]	Na2	O2[6]	126.63(4)
O3[5]	Na2	O2[5]	65.54(3)
O3[6]	Na2	O2[5]	126.63(4)
O3[6]	Na2	O2[6]	65.54(3)
O3[5]	Na2	O3[6]	160.87(5)
O3[5]	Na2	O4[6]	120.40(4)
O3[6]	Na2	O4[5]	120.40(4)
O3[6]	Na2	O4[6]	50.36(3)
O3[5]	Na2	O4[5]	50.36(3)
O3[6]	Na2	N3[7]	82.36(3)
O3[5]	Na2	N3[7]	82.36(3)
O3[5]	Na2	N8[6]	144.56(4)
O3[6]	Na2	N8[6]	25.06(3)
O3[6]	Na2	N8[5]	144.56(4)
O3[5]	Na2	N8[5]	25.06(3)

Atom	Atom	Atom	Angle/°
O4[5]	Na2	Na1[5]	80.03(2)
O4[6]	Na2	Na1[2]	80.03(2)
O4[5]	Na2	Na1[2]	150.58(3)
O4[6]	Na2	Na1[5]	150.58(3)
O4[5]	Na2	O4[6]	70.91(4)
O4[6]	Na2	N8[5]	96.28(4)
O4[6]	Na2	N8[6]	25.48(3)
O4[5]	Na2	N8[6]	96.28(4)
O4[5]	Na2	N8[5]	25.48(3)
N3[7]	Na2	Na1[5]	91.29(2)
N3[7]	Na2	Na1[2]	91.29(2)
N3[7]	Na2	O2[5]	92.52(4)
N3[7]	Na2	O2[6]	92.52(4)
N3[7]	Na2	O4[5]	96.24(4)
N3[7]	Na2	O4[6]	96.24(4)
N3[7]	Na2	N8[6]	91.33(3)
N3[7]	Na2	N8[5]	91.33(3)
N6	Na2	Na1[2]	88.43(2)
N6	Na2	Na1[5]	88.43(2)
N6	Na2	O2[5]	86.93(4)
N6	Na2	O2[6]	86.93(4)
N6	Na2	O3[5]	97.71(3)
N6	Na2	O3[6]	97.71(3)
N6	Na2	O4[5]	84.28(4)
N6	Na2	O4[6]	84.28(4)
N6	Na2	N3[7]	179.37(6)
N6	Na2	N8[5]	88.98(3)
N6	Na2	N8[6]	88.98(3)
N8[6]	Na2	Na1[5]	175.69(3)
N8[5]	Na2	Na1[2]	175.69(3)
N8[5]	Na2	Na1[5]	55.03(2)
N8[6]	Na2	Na1[2]	55.03(2)
N8[5]	Na2	N8[6]	121.48(5)
N4	O1	Na1	87.96(7)
Na1	O2	Na2[5]	112.11(4)
N4	O2	Na1	103.41(7)
N4	O2	Na2[5]	144.18(8)
Na2[5]	O3	Na1	114.75(4)
N8	O3	Na1	132.99(7)
N8	O3	Na2[5]	98.08(7)
N8	O4	Na2[5]	90.42(7)
N2	N1	Na1[3]	75.99(9)
C1	N1	Na1[3]	165.91(13)
C1	N1	N2	118.11(15)
N1	N2	Na1[3]	74.15(9)
C2	N2	Na1[3]	166.96(13)
C2	N2	N1	118.90(15)

Atom	Atom	Atom	Angle/°
C1	N3	Na2[8]	136.27(12)
C3	N3	Na2[8]	108.61(11)
C3	N3	C1	115.12(15)
O1	N4	Na1	67.51(6)
O1	N4	C4	116.38(11)
O2	N4	Na1	52.82(6)
O2	N4	O1	120.29(10)
O2	N4	C4	123.33(11)
C4	N4	Na1	175.64(9)
C5	N5	N6	118.02(15)
N5	N6	Na2	117.49(11)
C6	N6	Na2	124.29(12)
C6	N6	N5	118.22(15)
C5	N7	Na1[9]	129.00(12)
C7	N7	Na1[9]	116.56(11)
C7	N7	C5	114.44(15)
O3	N8	Na2[5]	56.85(6)
O3	N8	O4	120.26(10)
O3	N8	C8	123.57(11)
O4	N8	Na2[5]	64.10(6)
O4	N8	C8	116.17(11)
C8	N8	Na2[5]	172.01(9)
N1	C1	N3	125.90(16)
N1	C1	C4	115.86(15)
N3	C1	C4	118.24(15)
N2	C2	C3	120.98(17)
N3	C3	C2	120.99(17)
N4	C4	N4[10]	122.69(16)
N4	C4	C1	118.64(8)
N4[10]	C4	C1	118.64(8)
N5	C5	N7	126.86(16)
N5	C5	C8	114.31(15)
N7	C5	C8	118.83(15)
N6	C6	C7	121.67(17)
N7	C7	C6	120.79(16)
N8	C8	N8[1]	123.16(15)
N8[1]	C8	C5	118.29(8)
N8	C8	C5	118.29(8)

——[1]+x,3/2-y,+z; [2]2-x,2-y,1-z; [3]1-x,1-y,1-z; [4]-1/2+x,+y,3/2-z; [5]2-x,1-y,1-z; [6]2-x,1/2+y,1-z; [7]3/2-x,1-y,-1/2+z; [8]3/2-x,1-y,1/2+z; [9]1/2+x,+y,3/2-z; [10]+x,1/2-y,+z

Table S26: Torsion Angles in ° for **5e**.

Atom	Atom	Atom	Atom	Angle/°
Na1	O1	N4	O2	2.17(11)
Na1	O1	N4	C4	-177.84(11)
Na1	O2	N4	O1	-2.52(13)

Atom	Atom	Atom	Atom	Angle/°
Na1	O2	N4	C4	177.49(12)
Na1	O3	N8	Na2[1]	-135.27(10)
Na1	O3	N8	O4	-145.23(9)
Na1	O3	N8	C8	35.17(17)
Na1[2]	N1	N2	C2	180.000(0)
Na1[2]	N1	C1	N3	180.000(1)
Na1[2]	N1	C1	C4	0.000(1)
Na1[2]	N2	C2	C3	180.000(1)
Na1[3]	N7	C5	N5	180.000(0)
Na1[3]	N7	C5	C8	0.000(0)
Na1[3]	N7	C7	C6	180.000(0)
Na2[1]	O2	N4	Na1	-172.53(16)
Na2[1]	O2	N4	O1	-175.05(9)
Na2[1]	O2	N4	C4	5.0(2)
Na2[1]	O3	N8	O4	-9.95(11)
Na2[1]	O3	N8	C8	170.45(11)
Na2[1]	O4	N8	O3	9.26(10)
Na2[1]	O4	N8	C8	-171.11(10)
Na2[4]	N3	C1	N1	180.000(0)
Na2[4]	N3	C1	C4	0.000(0)
Na2[4]	N3	C3	C2	180.000(0)
Na2	N6	C6	C7	180.000(0)
O1	N4	C4	N4[5]	-176.30(11)
O1	N4	C4	C1	1.7(2)
O2	N4	C4	N4[5]	3.7(3)
O2	N4	C4	C1	-178.29(13)
O3	N8	C8	N8[6]	0.8(2)
O3	N8	C8	C5	-173.31(12)
O4	N8	C8	N8[6]	-178.82(11)
O4	N8	C8	C5	7.07(19)
N1	N2	C2	C3	0.000(0)
N1	C1	C4	N4[5]	-90.95(14)
N1	C1	C4	N4	90.95(14)
N2	N1	C1	N3	0.000(0)
N2	N1	C1	C4	180.000(0)
N2	C2	C3	N3	0.000(0)
N3	C1	C4	N4	-89.05(14)
N3	C1	C4	N4[5]	89.05(14)
N5	N6	C6	C7	0.000(0)
N5	C5	C8	N8	87.20(13)
N5	C5	C8	N8[6]	-87.20(13)
N6	N5	C5	N7	0.000(0)
N6	N5	C5	C8	180.000(0)
N6	C6	C7	N7	0.000(0)
N7	C5	C8	N8	-92.80(13)
N7	C5	C8	N8[6]	92.80(13)
C1	N1	N2	Na1[2]	180.000(0)

Atom	Atom	Atom	Atom	Angle/°
C1	N1	N2	C2	0.000(0)
C1	N3	C3	C2	0.000(0)
C3	N3	C1	N1	0.000(0)
C3	N3	C1	C4	180.000(0)
C5	N5	N6	Na2	180.000(0)
C5	N5	N6	C6	0.000(0)
C5	N7	C7	C6	0.000(0)
C7	N7	C5	N5	0.000(0)
C7	N7	C5	C8	180.000(0)

——[1]2-x,1-y,1-z; [2]1-x,1-y,1-z; [3]1/2+x,+y,3/2-z; [4]3/2-x,1-y,1/2+z; [5]+x,1/2-y,+z; [6]+x,3/2-y,+z

Table S27: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **5e**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H2	880(20)	2500	5441(16)	26(6)
H3	1380(20)	2500	6954(16)	27(6)
H6	13402(19)	7500	5208(14)	17(5)
H7	13211(19)	7500	6744(15)	18(6)

Table S28. Crystallographic data and structure refinement details for **9**, **11** and **12a**

Compound	**9**	**11**	**12a**
Formula	$C_9H_4F_5N_5O_4$	$C_5H_7N_{11}O_4$	$C_7H_8F_3N_5O_5$
$D_{calc.}$/ g cm^{-3}	1.952	1.669	1.714
μ/mm^{-1}	1.854	1.259	1.557
Formula Weight	341.17	285.22	299.18
Color	yellow	yellow	colorless
Shape	plate	plate	plate
Size/mm^3	0.26×0.17×0.08	0.31×0.24×0.07	0.48×0.34×0.08
T/K	173(1)	172.99	173(1)
Crystal System	monoclinic	monoclinic	triclinic
Space Group	$P2_1/c$	$C2/c$	P-1
a/Å	14.2809(2)	27.0867(4)	7.6530(4)
b/Å	6.21890(10)	6.36110(10)	8.3737(4)
c/Å	14.2905(2)	13.7829(2)	10.1818(6)
α/°	90	90	108.537(4)
β/°	113.8050(10)	107.1240(10)	105.840(4)
γ/°	90	90	96.862(3)
V/Å^3	1161.19(3)	2269.53(6)	579.73(6)
Z	4	8	2
Z'	1	1	1
Wavelength/Å	1.54178	1.54178	1.54178
Radiation type	CuK$_a$	CuK$_a$	CuK□
θ_{min}/°	6.228	3.415	4.853
θ_{max}/°	72.357	70.052	68.151
Measured Refl's.	7683	7818	7520
Ind't Refl's	2246	2144	2067
Refl's with I > 2(I)	1966	1862	1816
R_{int}	0.0298	0.0292	0.0501
Parameters	221	193	213
Restraints	0	0	0
Largest Peak	0.321	0.207	0.290
Deepest Hole	-0.243	-0.228	-0.314
GooF	1.103	1.022	1.037
wR_2 (all data)	0.1100	0.1100	0.1142
wR_2	0.0962	0.1051	0.1105
R_1 (all data)	0.0411	0.0445	0.0457
R_1	0.0352	0.0389	0.0415
CCDC	**1975379**	**1975378**	**2154059**

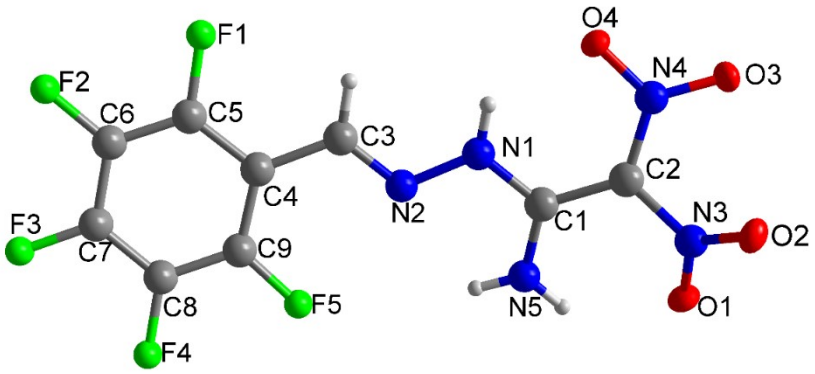


Figure S10: Thermal ellipsoids shown at 50% **9**.

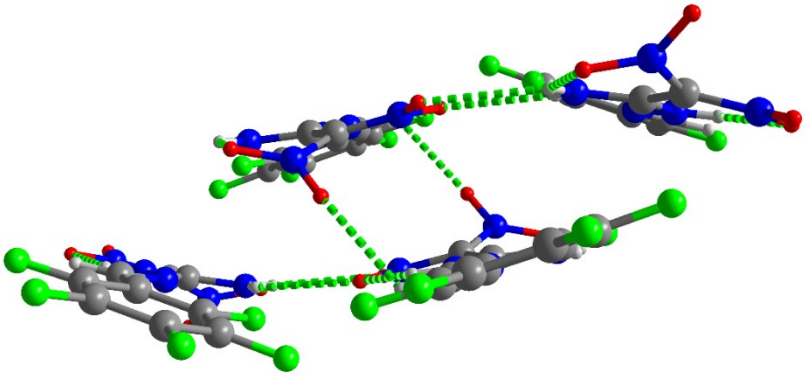


Figure S11: Ball-and-stick packing diagram of **9** viewed up the b axis

Table S29: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **9**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
F1	9495.0(8)	1963.0(18)	5453.1(8)	29.1(3)
F2	10962.8(8)	2684.2(18)	4777.0(9)	32.4(3)
F3	10457.6(9)	4105(2)	2824.7(9)	38.0(3)
F4	8466.3(9)	4778.8(19)	1578.1(8)	33.7(3)
F5	6994.9(7)	3915.1(17)	2197.3(7)	26.8(3)
O1	2658.2(9)	3566(2)	2486.1(9)	28.7(3)
O2	2658.1(10)	5042(2)	3856.8(10)	29.2(3)
O3	3529.4(10)	2145(2)	5267.3(10)	28.5(3)
O4	5155.4(9)	2127(2)	5624.5(9)	24.4(3)
N1	5790.8(11)	2645(2)	4169.3(11)	20.5(3)
N2	6461.3(11)	2810(2)	3700.3(11)	22.0(3)
N3	3057.9(10)	3902(2)	3418.6(11)	20.6(3)
N4	4241.0(10)	2432(2)	5005.1(11)	19.2(3)
N5	4539.7(12)	3194(3)	2579.5(11)	24.7(3)
C1	4798.0(13)	2930(2)	3568.9(12)	18.9(3)
C2	4046.8(12)	3013(3)	4003.8(12)	19.0(3)
C3	7410.8(13)	2584(3)	4275.9(13)	21.4(4)

Atom	x	y	z	U_e
C4	8177.6(13)	2906(2)	3857.5(13)	20.2(4)
C5	9217.8(13)	2600(3)	4478.6(13)	21.9(4)
C6	9982.2(13)	2979(3)	4150.1(14)	23.8(4)
C7	9726.7(13)	3713(3)	3164.3(14)	24.7(4)
C8	8719.7(14)	4043(3)	2530.7(13)	23.9(4)
C9	7958.4(13)	3618(3)	2865.5(13)	20.9(3)

Table S30: Anisotropic Displacement Parameters ($\times 10^4$) for **9**. 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}
F1	24.7(6)	36.2(6)	22.4(5)	4.6(4)	5.4(4)	1.8(4)
F2	16.4(6)	38.8(6)	37.2(6)	-3.1(5)	5.9(4)	0.4(4)
F3	26.7(6)	50.5(8)	44.9(7)	2.4(5)	22.8(5)	-4.5(5)
F4	34.8(6)	45.2(7)	24.3(5)	3.4(5)	15.1(4)	-3.3(5)
F5	17.4(5)	36.1(6)	22.9(5)	1.3(4)	4.1(4)	0.6(4)
O1	23.6(7)	35.7(7)	21.0(6)	2.0(5)	3.1(5)	3.4(5)
O2	23.5(7)	31.8(7)	32.9(7)	0.2(5)	12.0(5)	8.6(5)
O3	21.7(7)	39.2(7)	27.9(6)	6.1(5)	13.5(5)	-0.2(5)
O4	18.6(6)	31.1(7)	20.1(6)	2.1(5)	4.3(5)	1.5(5)
N1	17.2(7)	24.6(7)	20.6(7)	0.0(6)	8.6(5)	0.2(5)
N2	20.6(8)	22.9(7)	25.1(7)	-1.8(6)	12.0(6)	-1.5(5)
N3	16.0(7)	20.4(7)	23.7(7)	2.7(5)	6.2(5)	-0.1(5)
N4	16.2(7)	18.8(7)	21.2(7)	-0.6(5)	6.1(5)	-0.2(5)
N5	21.8(8)	32.1(8)	20.7(7)	1.5(6)	9.0(6)	1.3(6)
C1	19.7(8)	13.4(7)	22.1(8)	-0.7(6)	7.0(6)	-0.5(6)
C2	16.0(8)	18.8(8)	20.5(8)	0.4(6)	5.5(6)	0.3(6)
C3	19.9(9)	22.3(8)	22.9(8)	-1.7(6)	9.6(7)	1.0(6)
C4	19.9(9)	17.1(8)	23.0(8)	-3.2(6)	7.9(6)	-1.2(6)
C5	22.1(9)	20.1(8)	20.9(8)	-2.1(6)	6.1(7)	0.2(6)
C6	15.3(9)	21.8(8)	30.8(9)	-4.8(7)	5.6(7)	-1.1(6)
C7	22.3(9)	24.5(8)	32.6(9)	-4.5(7)	16.5(7)	-4.5(6)
C8	26.5(9)	24.4(8)	22.6(8)	-2.8(7)	11.7(7)	-2.8(6)
C9	18.8(8)	19.3(8)	22.2(8)	-3.5(6)	5.8(6)	-0.2(6)

Table S31: Bond Lengths in Å for **9**.

Atom	Atom	Length/Å
F1	C5	1.345(2)
F2	C6	1.336(2)
F3	C7	1.3392(19)
F4	C8	1.340(2)
F5	C9	1.3335(19)
O1	N3	1.2374(18)
O2	N3	1.2290(19)
O3	N4	1.2298(19)
O4	N4	1.2616(19)

Atom	Atom	Length/Å
N1	N2	1.376(2)
N1	C1	1.340(2)
N2	C3	1.279(2)
N3	C2	1.430(2)
N4	C2	1.391(2)
N5	C1	1.320(2)
C1	C2	1.442(2)
C3	C4	1.458(2)
C4	C5	1.402(2)
C4	C9	1.395(2)
C5	C6	1.372(2)
C6	C7	1.382(3)
C7	C8	1.371(3)
C8	C9	1.379(2)

Table S32: Bond Angles in ° for **9.**

Atom	Atom	Atom	Angle/°
C1	N1	N2	116.27(14)
C3	N2	N1	116.37(15)
O1	N3	C2	118.82(14)
O2	N3	O1	122.61(14)
O2	N3	C2	118.50(14)
O3	N4	O4	120.86(14)
O3	N4	C2	120.34(13)
O4	N4	C2	118.74(13)
N1	C1	C2	120.52(15)
N5	C1	N1	117.93(15)
N5	C1	C2	121.52(15)
N3	C2	C1	119.51(14)
N4	C2	N3	116.21(14)
N4	C2	C1	124.07(14)
N2	C3	C4	119.88(16)
C5	C4	C3	120.06(16)
C9	C4	C3	124.25(16)
C9	C4	C5	115.61(15)
F1	C5	C4	119.29(15)
F1	C5	C6	117.58(15)
C6	C5	C4	123.10(16)
F2	C6	C5	120.70(16)
F2	C6	C7	120.18(15)
C5	C6	C7	119.11(16)
F3	C7	C6	120.38(16)
F3	C7	C8	119.78(16)
C8	C7	C6	119.84(16)
F4	C8	C7	120.22(15)
F4	C8	C9	119.37(15)

Atom	Atom	Atom	Angle/°
C7	C8	C9	120.40(16)
F5	C9	C4	121.00(15)
F5	C9	C8	117.09(15)
C8	C9	C4	121.91(16)

Table S33: Torsion Angles in ° for **9**.

Atom	Atom	Atom	Atom	Angle/°
F1	C5	C6	F2	1.2(2)
F1	C5	C6	C7	-177.60(15)
F2	C6	C7	F3	1.0(3)
F2	C6	C7	C8	-178.98(15)
F3	C7	C8	F4	-0.7(3)
F3	C7	C8	C9	179.10(15)
F4	C8	C9	F5	1.8(2)
F4	C8	C9	C4	-178.25(15)
O1	N3	C2	N4	-147.79(15)
O1	N3	C2	C1	37.1(2)
O2	N3	C2	N4	35.2(2)
O2	N3	C2	C1	-139.91(16)
O3	N4	C2	N3	19.3(2)
O3	N4	C2	C1	-165.91(15)
O4	N4	C2	N3	-163.44(14)
O4	N4	C2	C1	11.4(2)
N1	N2	C3	C4	-175.07(13)
N1	C1	C2	N3	162.52(14)
N1	C1	C2	N4	-12.1(2)
N2	N1	C1	N5	4.5(2)
N2	N1	C1	C2	-173.49(13)
N2	C3	C4	C5	-178.18(15)
N2	C3	C4	C9	5.1(3)
N5	C1	C2	N3	-15.4(2)
N5	C1	C2	N4	169.94(15)
C1	N1	N2	C3	178.57(14)
C3	C4	C5	F1	1.4(2)
C3	C4	C5	C6	-176.61(15)
C3	C4	C9	F5	-4.8(2)
C3	C4	C9	C8	175.28(15)
C4	C5	C6	F2	179.24(14)
C4	C5	C6	C7	0.5(3)
C5	C4	C9	F5	178.32(14)
C5	C4	C9	C8	-1.6(2)
C5	C6	C7	F3	179.73(15)
C5	C6	C7	C8	-0.2(3)
C6	C7	C8	F4	179.21(15)
C6	C7	C8	C9	-0.9(3)
C7	C8	C9	F5	-178.00(14)

Atom	Atom	Atom	Atom	Angle/°
C7	C8	C9	C4	1.9(3)
C9	C4	C5	F1	178.45(14)
C9	C4	C5	C6	0.4(2)

Table S34: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **9**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1	5976(16)	2490(30)	4813(17)	23(5)
H5A	3900(20)	3240(50)	2160(20)	61(9)
H5B	5004(19)	3180(40)	2354(17)	28(5)
H3	7616.34	2206.58	4974.94	26

Table S35: Hydrogen Bond information for **9**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N1	H1	O4	0.85(2)	1.97(2)	2.6028(19)	130.4(19)
N5	H5A	O1	0.87(3)	2.01(3)	2.646(2)	129(3)
N5	H5A	O3[1]	0.87(3)	2.55(3)	3.032(2)	116(2)

[1]+x,1/2-y,-1/2+z

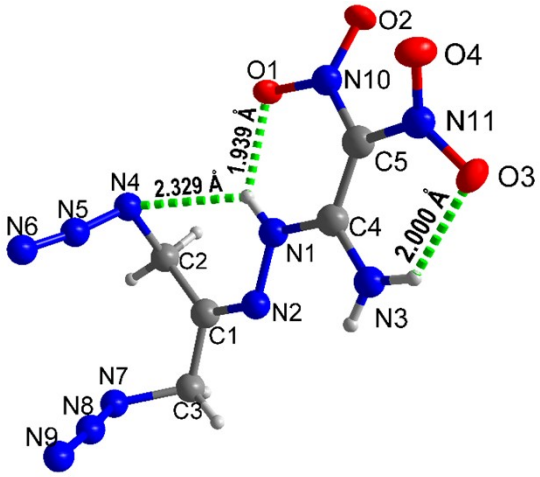


Figure S12: Thermal ellipsoids shown at 50% **11**.

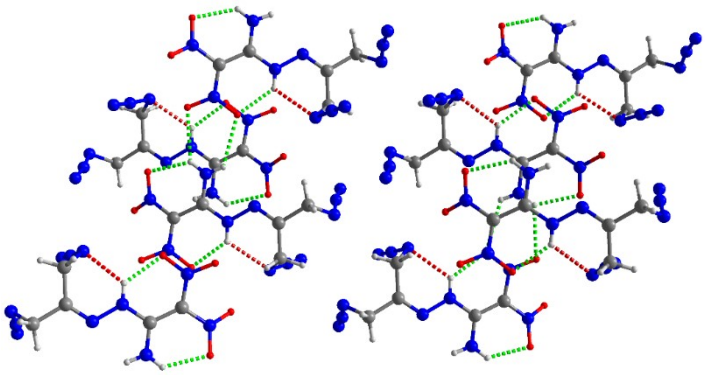


Figure S13: Ball-and-stick packing diagram of **11** viewed up the b axis. Dashed lines indicate strong hydrogen bonding.

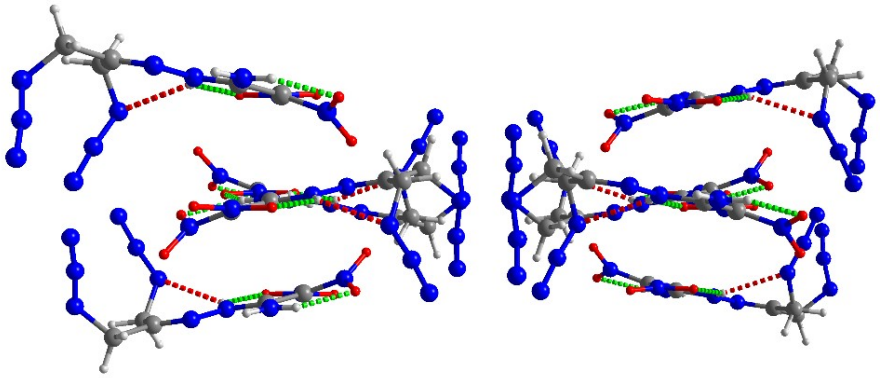


Figure S14: Ball-and-stick packing diagram of **11** viewed up the c axis. Dashed lines indicate strong hydrogen bonding.

Table S36: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **11**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
O1	7316.3(4)	5326(2)	2245.0(8)	29.4(3)
O2	8143.2(4)	5280.4(19)	2484.3(9)	30.0(3)
O3	8492.2(5)	5705(2)	5379.8(9)	33.8(3)
O4	8576.8(5)	7460(2)	4094.1(9)	38.0(3)
N1	6950.8(5)	4782(2)	3748.0(10)	22.9(3)
N2	6591.0(5)	4376(2)	4258.9(10)	24.4(3)
N3	7535.1(6)	4976(2)	5318.4(10)	24.5(3)
N4	6054.8(6)	6132(2)	2132.7(11)	32.2(3)
N5	5818.3(6)	7686(2)	2309.6(11)	33.4(3)
N6	5626.9(7)	9207(3)	2410.0(16)	51.3(5)
N7	5293.5(6)	5051(3)	3980.1(13)	39.1(4)
N8	5344.4(5)	6723(3)	4453.7(13)	39.0(4)
N9	5334.0(8)	8284(3)	4822(2)	69.9(6)
N10	7770.7(5)	5383.0(19)	2823.9(10)	22.2(3)

Atom	x	y	z	U_{eq}
N11	8336.4(5)	6231(2)	4472.1(10)	26.2(3)
C1	6124.5(6)	4089(2)	3718.7(12)	24.4(3)
C2	5928.3(6)	4141(2)	2573.2(12)	27.6(4)
C3	5743.9(6)	3607(3)	4298.0(13)	31.6(4)
C4	7440.8(6)	5073(2)	4325.1(11)	20.6(3)
C5	7843.4(6)	5485(2)	3870.2(11)	21.6(3)

Table S37: Anisotropic Displacement Parameters ($\times 10^4$) for **11**. 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	22.9(5)	48.5(7)	16.6(5)	-1.1(5)	5.4(4)	-2.5(5)
O2	25.4(6)	46.0(7)	22.2(6)	-3.9(5)	12.5(5)	-1.2(5)
O3	33.0(6)	44.9(7)	18.6(6)	3.2(5)	-0.1(5)	-2.4(5)
O4	34.8(6)	52.7(7)	26.8(6)	0.1(6)	9.7(5)	-15.9(6)
N1	24.0(6)	30.2(7)	16.7(7)	0.3(5)	9.6(5)	0.3(5)
N2	28.5(7)	25.2(6)	23.9(7)	1.9(5)	14.4(6)	0.9(5)
N3	30.0(7)	27.3(7)	17.2(7)	0.8(5)	8.4(6)	-0.8(6)
N4	33.6(7)	37.9(8)	27.3(7)	5.6(6)	12.3(6)	1.8(6)
N5	33.7(8)	34.8(8)	29.4(8)	5.7(6)	5.8(6)	-2.0(6)
N6	55.4(11)	35.3(9)	64.1(12)	5.3(8)	19.0(9)	4.7(8)
N7	26.0(7)	54.5(9)	38.3(9)	-10.6(7)	11.9(6)	1.2(7)
N8	23.7(7)	46.0(9)	50.1(10)	-1.2(8)	14.9(7)	2.6(6)
N9	48.2(11)	51.2(11)	110.7(19)	-19.6(12)	24.1(11)	8.7(9)
N10	24.3(6)	25.2(6)	18.3(6)	-0.7(5)	8.3(5)	-0.4(5)
N11	25.8(7)	33.0(7)	19.6(6)	-1.1(5)	6.4(5)	-0.4(5)
C1	26.9(8)	21.7(7)	27.4(8)	0.2(6)	12.5(6)	1.4(6)
C2	27.5(8)	29.4(8)	27.3(8)	-0.8(7)	10.2(7)	-0.8(6)
C3	29.9(8)	35.4(8)	34.1(9)	0.8(7)	16.6(7)	-2.9(7)
C4	27.0(8)	17.5(6)	18.1(7)	-1.0(5)	8.1(6)	1.3(5)
C5	24.4(7)	24.9(7)	15.6(7)	-0.8(6)	6.1(6)	0.8(6)

Table S38: Bond Lengths in Å for **11**.

Atom	Atom	Length/Å
O1	N10	1.2550(17)
O2	N10	1.2326(17)
O3	N11	1.2427(18)
O4	N11	1.2275(18)
N1	N2	1.3843(18)
N1	C4	1.344(2)
N2	C1	1.277(2)
N3	C4	1.318(2)
N4	N5	1.241(2)
N4	C2	1.487(2)
N5	N6	1.126(2)
N7	N8	1.234(2)

Atom	Atom	Length/Å
N7	C3	1.486(2)
N8	N9	1.119(3)
N10	C5	1.3984(19)
N11	C5	1.430(2)
C1	C2	1.511(2)
C1	C3	1.510(2)
C4	C5	1.433(2)

Table S39: Bond Angles in ° for **11**.

Atom	Atom	Atom	Angle/°
C4	N1	N2	116.42(13)
C1	N2	N1	117.00(13)
N5	N4	C2	113.82(13)
N6	N5	N4	173.00(18)
N8	N7	C3	114.99(15)
N9	N8	N7	172.1(2)
O1	N10	C5	118.09(12)
O2	N10	O1	121.09(12)
O2	N10	C5	120.78(13)
O3	N11	C5	118.50(13)
O4	N11	O3	122.23(14)
O4	N11	C5	119.12(13)
N2	C1	C2	126.13(14)
N2	C1	C3	115.68(15)
C3	C1	C2	118.18(14)
N4	C2	C1	113.13(13)
N7	C3	C1	110.35(14)
N1	C4	C5	120.82(14)
N3	C4	N1	117.64(14)
N3	C4	C5	121.54(15)
N10	C5	N11	115.95(13)
N10	C5	C4	123.26(14)
N11	C5	C4	120.53(13)

Table S40: Torsion Angles in ° for **11**.

Atom	Atom	Atom	Atom	Angle/°
O1	N10	C5	N11	-160.17(13)
O1	N10	C5	C4	14.0(2)
O2	N10	C5	N11	22.23(19)
O2	N10	C5	C4	-163.61(14)
O3	N11	C5	N10	-155.64(13)
O3	N11	C5	C4	30.0(2)
O4	N11	C5	N10	28.6(2)
O4	N11	C5	C4	-145.76(15)
N1	N2	C1	C2	0.3(2)

Atom	Atom	Atom	Atom	Angle/°
N1	N2	C1	C3	178.81(13)
N1	C4	C5	N10	-7.3(2)
N1	C4	C5	N11	166.63(13)
N2	N1	C4	N3	-0.8(2)
N2	N1	C4	C5	179.48(12)
N2	C1	C2	N4	-55.3(2)
N2	C1	C3	N7	127.85(15)
N3	C4	C5	N10	173.06(13)
N3	C4	C5	N11	-13.0(2)
N5	N4	C2	C1	-68.86(18)
N8	N7	C3	C1	-88.02(19)
C2	C1	C3	N7	-53.50(19)
C3	C1	C2	N4	126.19(15)
C4	N1	N2	C1	-179.77(13)

Table S41: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **11**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1	6868(9)	5000(30)	3103(18)	39(6)
H3A	7287(8)	4780(30)	5538(15)	26(5)
H3B	7838(9)	5020(30)	5706(17)	31(5)
H2A	6080.18	2948.34	2298.62	33
H2B	5548.63	3953.06	2358.4	33
H3C	5624.38	2134.72	4168.95	38
H3D	5915.16	3767.05	5035.05	38

Table S42: Hydrogen Bond information for **11**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N1	H1	O1	0.86(2)	1.94(2)	2.5688(17)	129(2)
N1	H1	N4	0.86(2)	2.33(2)	2.899(2)	123.9(18)
N3	H3A	O1[1]	0.83(2)	2.33(2)	2.8926(17)	125.9(17)
N3	H3B	O2[1]	0.84(2)	2.35(2)	2.9590(19)	129.9(18)
N3	H3B	O3	0.84(2)	2.00(2)	2.610(2)	128.9(19)

[1]+x,1-y,1/2+z

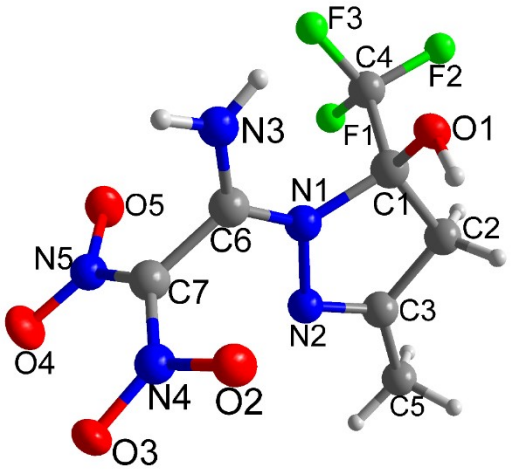


Figure S15: Thermal ellipsoids shown at 50% **12a**

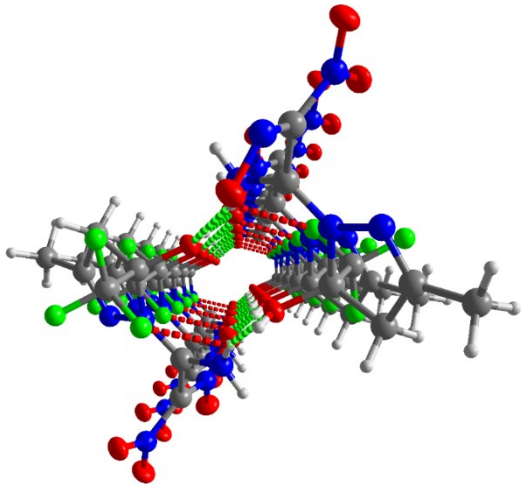


Figure S16: Ball-and-stick packing diagram of **12a** viewed up the a axis. Dashed lines indicate strong hydrogen bonding.

Table S43: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **12a**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
F1	6668.0(17)	5814.0(17)	4281.3(12)	43.5(3)
F2	7637.8(15)	7354.4(17)	3173.2(16)	47.4(4)
F3	6724.5(15)	4594.1(16)	2109.6(12)	38.0(3)
O1	4252.2(17)	6325.4(16)	1040.6(13)	24.6(3)
O2	-977.9(18)	3122.3(17)	46.9(15)	31.9(3)
O3	-2295.7(17)	1172.0(17)	704.5(16)	32.3(3)
O4	176(2)	73.0(19)	2448.2(16)	37.3(4)
O5	3074.3(18)	1332.0(18)	2947.6(15)	34.1(3)
N1	3014(2)	4753.8(18)	2298.2(15)	23.0(3)
N2	2047(2)	5246(2)	3311.5(16)	26.5(4)

Atom	x	y	z	U_{eq}
N3	3070(2)	2602(2)	211.1(17)	26.4(4)
N4	-877(2)	2056.7(19)	709.6(16)	24.9(3)
N5	1368(2)	1076.1(19)	2302.3(16)	26.3(4)
C1	4412(2)	6236(2)	2394.0(19)	23.5(4)
C2	4034(3)	7746(2)	3525(2)	28.6(4)
C3	2634(3)	6884(2)	4002.6(19)	27.8(4)
C4	6387(3)	5980(2)	2994(2)	29.5(4)
C5	1936(3)	7823(3)	5189(2)	38.5(5)
C6	2415(2)	3167(2)	1282.9(19)	23.6(4)
C7	894(2)	1978(2)	1370.4(19)	23.6(4)

Table S44: Anisotropic Displacement Parameters ($\times 10^4$) for **12a**. 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}
F1	45.7(7)	57.2(8)	24.3(6)	17.3(5)	0.2(5)	20.0(6)
F2	21.4(6)	45.3(7)	61.0(9)	19.0(6)	-3.2(5)	-4.0(5)
F3	29.9(6)	45.3(7)	30.6(6)	6.9(5)	2.7(5)	15.8(5)
O1	20.6(6)	31.0(7)	23.1(7)	13.5(5)	4.6(5)	4.9(5)
O2	27.6(7)	37.9(8)	34.3(8)	21.0(6)	6.8(6)	9.0(5)
O3	21.5(6)	29.8(7)	42.6(8)	11.1(6)	10.3(6)	1.1(5)
O4	39.8(8)	37.8(8)	37.7(8)	21.7(6)	12.3(6)	0.7(6)
O5	30.2(7)	37.2(7)	29.2(7)	14.8(6)	-1.3(6)	5.7(5)
N1	23.2(7)	24.7(8)	19.9(7)	8.4(6)	5.9(6)	2.9(6)
N2	26.0(7)	31.9(8)	19.3(8)	8.5(6)	6.0(6)	4.6(6)
N3	25.3(8)	24.6(8)	24.7(8)	4.9(6)	8.5(7)	-0.6(6)
N4	24.3(7)	24.6(7)	22.3(8)	5.5(6)	6.4(6)	4.1(6)
N5	30.5(8)	24.4(8)	20.7(8)	7.1(6)	6.1(7)	3.6(6)
C1	22.4(8)	23.2(8)	20.7(9)	8.3(6)	2.0(7)	1.3(6)
C2	30.8(9)	24.7(9)	24.2(9)	6.6(7)	3.1(8)	4.7(7)
C3	27.2(9)	31.7(9)	19.5(9)	7.7(7)	1.6(7)	7.5(7)
C4	26.7(9)	31.6(10)	22.8(9)	9.0(7)	-0.6(8)	3.7(7)
C5	37.2(11)	40.2(12)	27.6(11)	0.9(8)	8.8(9)	6.9(9)
C6	22.9(8)	24.8(8)	21.5(9)	10.3(7)	3.1(7)	4.3(6)
C7	23.8(8)	24.3(9)	21.5(9)	9.0(7)	5.5(7)	3.7(7)

Table S45: Bond Lengths in Å for **12a**.

Atom	Atom	Length/Å
F1	C4	1.324(2)
F2	C4	1.335(2)
F3	C4	1.326(2)
O1	C1	1.377(2)
O2	N4	1.276(2)
O3	N4	1.237(2)
O4	N5	1.237(2)
O5	N5	1.248(2)

Atom	Atom	Length/Å
N1	N2	1.415(2)
N1	C1	1.498(2)
N1	C6	1.329(2)
N2	C3	1.283(2)
N3	C6	1.301(2)
N4	C7	1.359(2)
N5	C7	1.391(2)
C1	C2	1.533(2)
C1	C4	1.537(2)
C2	C3	1.493(3)
C3	C5	1.487(3)
C6	C7	1.480(2)

Table S46: Bond Angles in ° for **12a**.

Atom	Atom	Atom	Angle/°
N2	N1	C1	112.89(13)
C6	N1	N2	117.84(14)
C6	N1	C1	128.51(14)
C3	N2	N1	107.04(14)
O2	N4	C7	113.97(14)
O3	N4	O2	121.37(15)
O3	N4	C7	124.65(15)
O4	N5	O5	123.11(15)
O4	N5	C7	121.88(15)
O5	N5	C7	114.99(14)
O1	C1	N1	112.32(13)
O1	C1	C2	117.65(15)
O1	C1	C4	105.88(14)
N1	C1	C2	100.82(13)
N1	C1	C4	109.30(14)
C2	C1	C4	110.76(15)
C3	C2	C1	103.61(14)
N2	C3	C2	115.31(16)
N2	C3	C5	121.14(18)
C5	C3	C2	123.55(17)
F1	C4	F2	108.25(15)
F1	C4	F3	107.42(15)
F1	C4	C1	110.90(15)
F2	C4	C1	109.42(15)
F3	C4	F2	107.64(16)
F3	C4	C1	113.05(15)
N1	C6	C7	117.82(15)
N3	C6	N1	124.09(16)
N3	C6	C7	118.09(16)
N4	C7	N5	124.43(15)
N4	C7	C6	116.73(15)

Atom	Atom	Atom	Angle/°
N5	C7	C6	118.01(15)

Table S47: Torsion Angles in ° for **12a**.

Atom	Atom	Atom	Atom	Angle/°
O1	C1	C2	C3	127.80(15)
O1	C1	C4	F1	-77.75(14)
O1	C1	C4	F2	62.91(18)
O1	C1	C4	F3	-57.03(19)
O2	N4	C7	N5	171.86(15)
O2	N4	C7	C6	2.5(2)
O3	N4	C7	N5	-9.4(3)
O3	N4	C7	C6	-78.77(15)
O4	N5	C7	N4	10.0(3)
O4	N5	C7	C6	179.20(15)
O5	N5	C7	N4	-71.35(16)
O5	N5	C7	C6	-2.2(2)
N1	N2	C3	C2	1.9(2)
N1	N2	C3	C5	-78.26(17)
N1	C1	C2	C3	5.34(17)
N1	C1	C4	F1	-56.55(19)
N1	C1	C4	F2	-75.89(14)
N1	C1	C4	F3	64.17(19)
N1	C6	C7	N4	86.3(2)
N1	C6	C7	N5	-83.7(2)
N2	N1	C1	O1	-30.98(15)
N2	N1	C1	C2	-4.88(18)
N2	N1	C1	C4	111.82(15)
N2	N1	C6	N3	173.73(16)
N2	N1	C6	C7	-5.5(2)
N3	C6	C7	N4	-93.0(2)
N3	C6	C7	N5	97.0(2)
C1	N1	N2	C3	2.15(19)
C1	N1	C6	N3	4.5(3)
C1	N1	C6	C7	-74.75(15)
C1	C2	C3	N2	-4.9(2)
C1	C2	C3	C5	175.23(18)
C2	C1	C4	F1	53.7(2)
C2	C1	C4	F2	-65.7(2)
C2	C1	C4	F3	174.38(15)
C4	C1	C2	C3	-10.28(16)
C6	N1	N2	C3	-68.71(15)
C6	N1	C1	O1	38.7(2)
C6	N1	C1	C2	164.78(16)
C6	N1	C1	C4	-78.5(2)

Table S48: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **12a**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H2A	3510(30)	8520(30)	3080(20)	28(5)
H2B	5180(40)	8410(30)	4380(30)	43(6)
H5A	910(30)	7090(30)	5320(30)	35(6)
H5B	2990(40)	8350(40)	6080(30)	53(7)
H5C	1440(40)	8800(40)	4980(30)	45(7)
H3A	3880(40)	3120(30)	30(30)	32(6)
H3B	2640(40)	1580(40)	-320(30)	37(6)
H1	3230(40)	6550(30)	670(30)	43(7)

Table S49: Hydrogen Bond information for **12a**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N3	H3A	F3	0.81(3)	2.43(2)	2.8682(19)	115(2)
N3	H3A	O1[1]	0.81(3)	2.11(3)	2.889(2)	161(2)
N3	H3A	O1	0.81(3)	2.50(2)	2.913(2)	113.2(19)
N3	H3B	O3[2]	0.82(3)	2.18(3)	2.930(2)	152(2)
O1	H1	O2[3]	0.85(3)	1.77(3)	2.6090(18)	174(2)

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

4. NMR Spectra

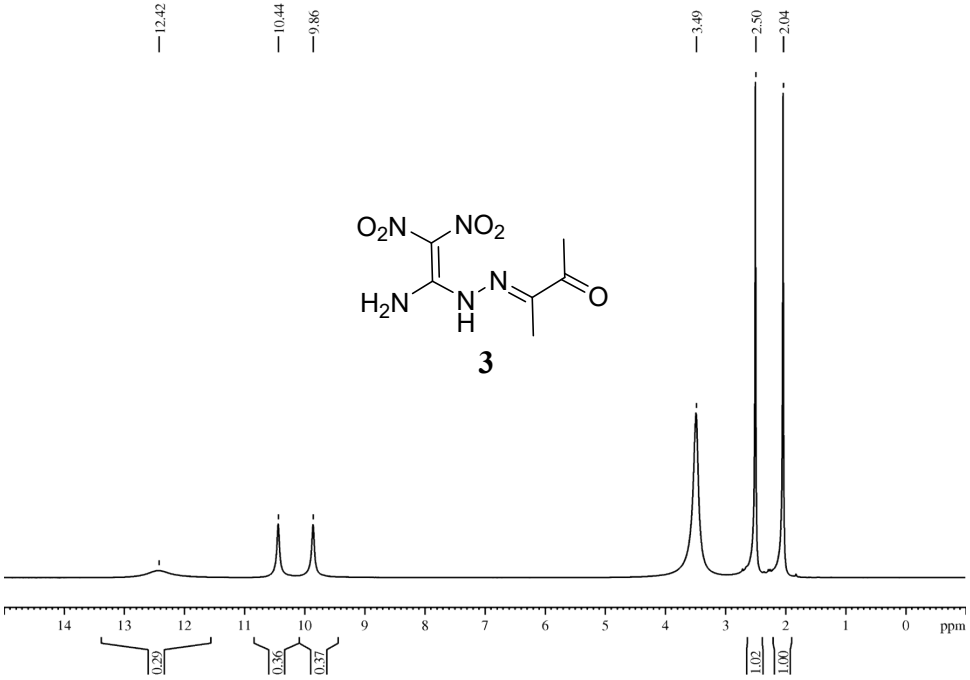


Figure S17: ^{1}H NMR-Compound **3**

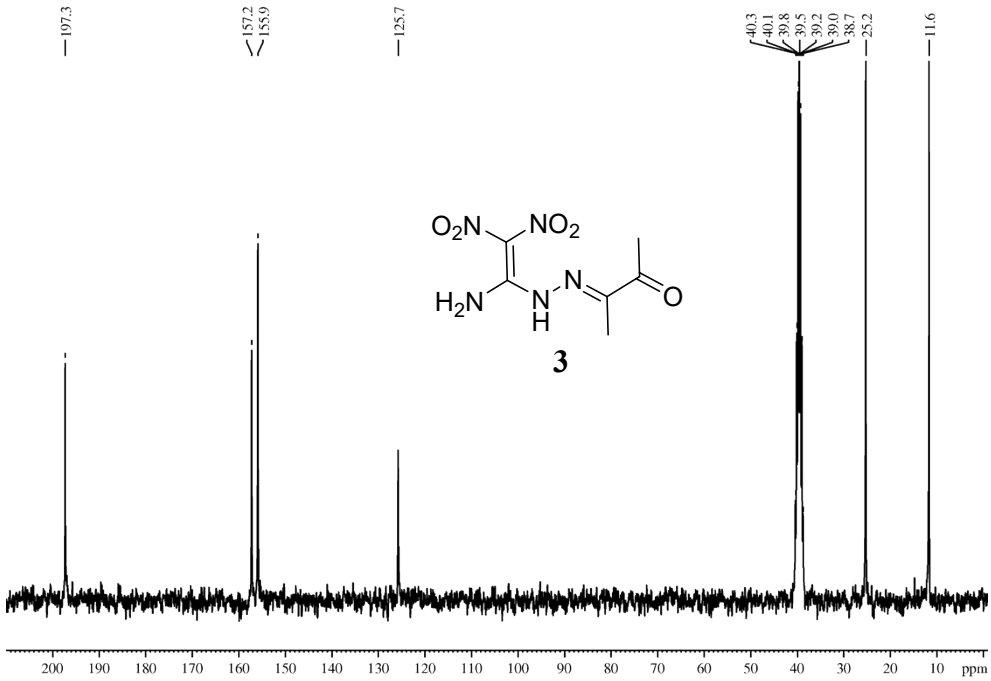


Figure S18: ^{13}C NMR-Compound **3**

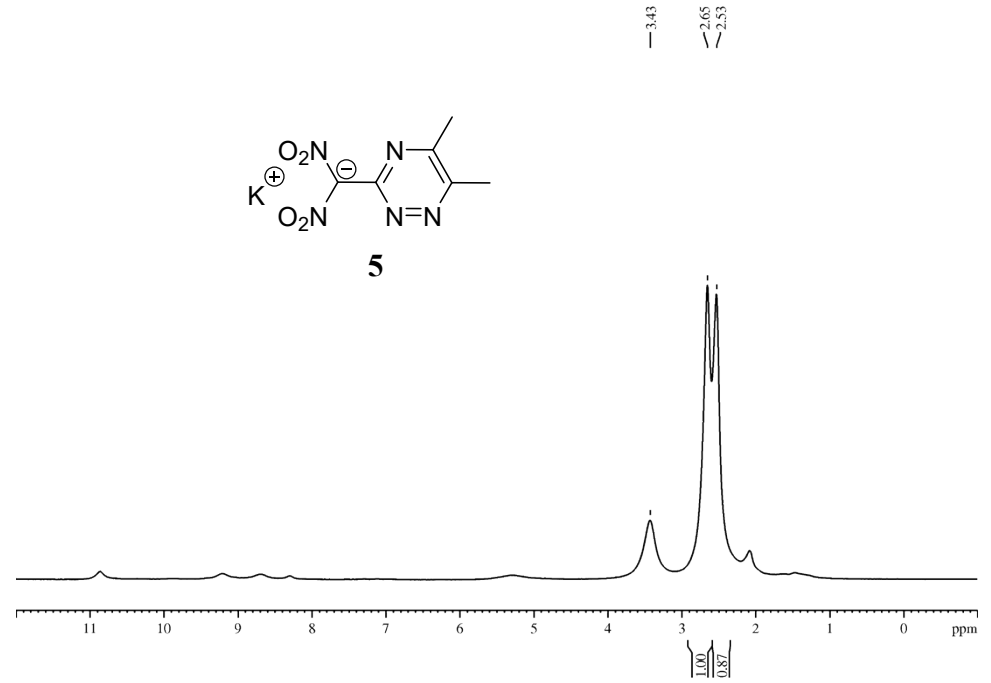


Figure S19: ^{1}H NMR-Compound **5**

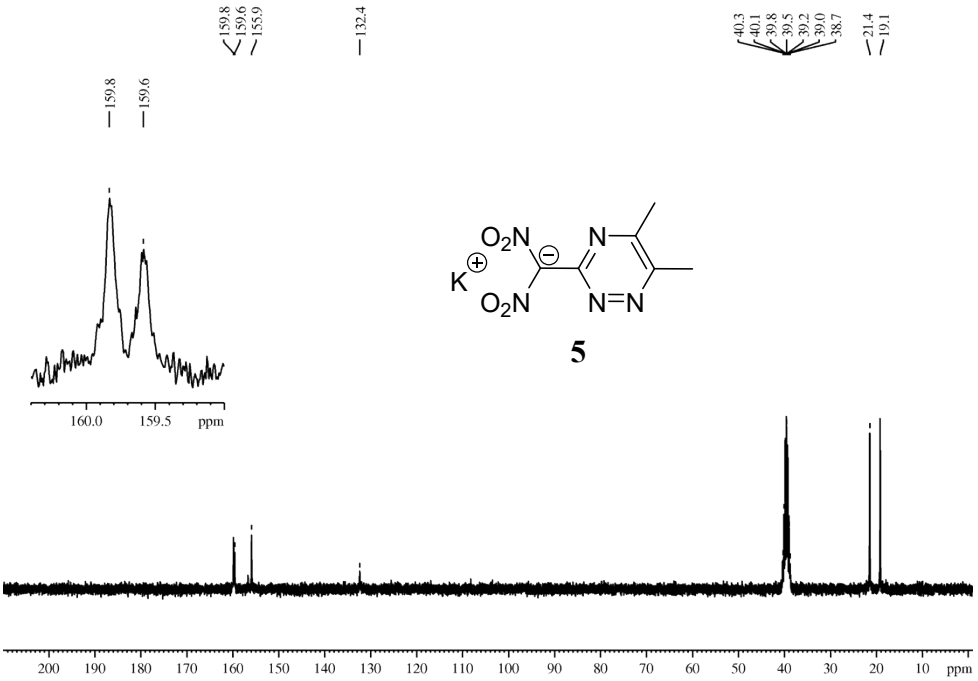


Figure S20: ^{13}C NMR-Compound **5**

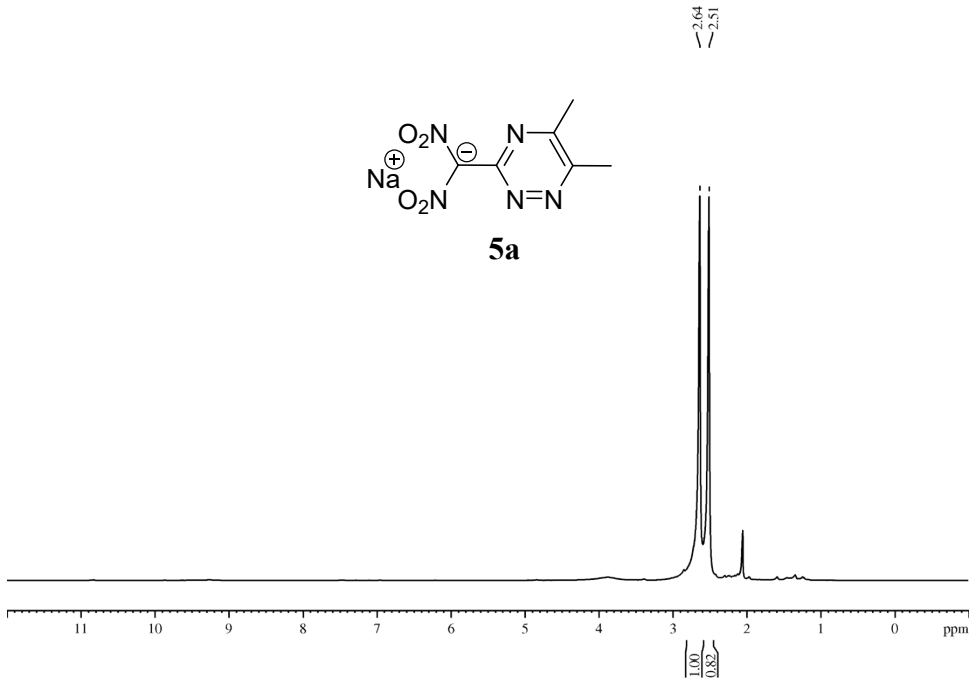


Figure S21: ^{1}H NMR-Compound **5a**

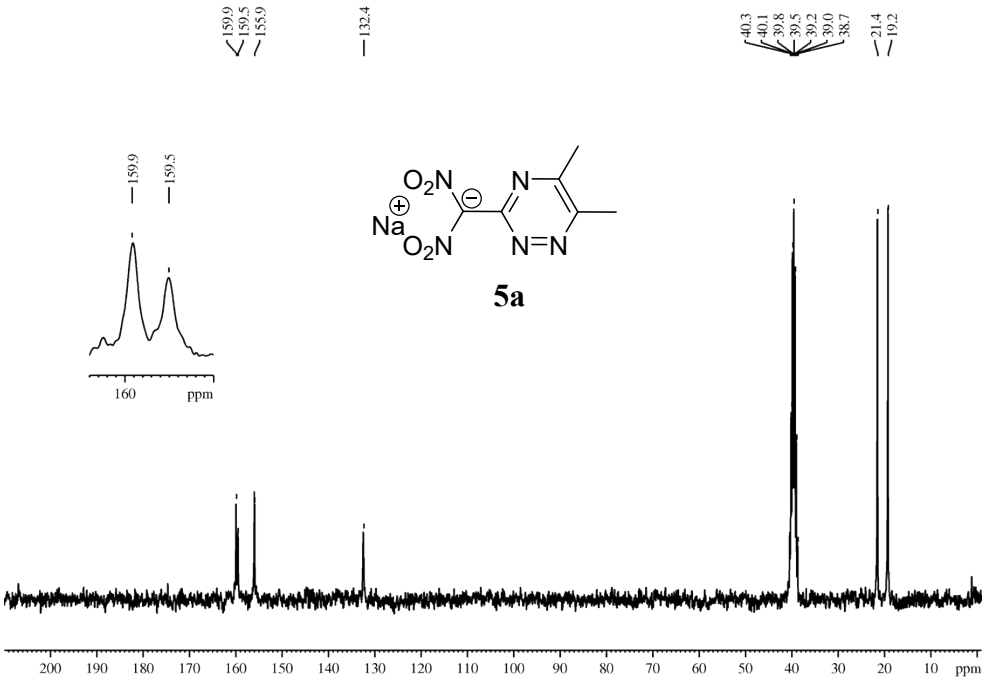


Figure S22: ^{13}C NMR-Compound **5a**

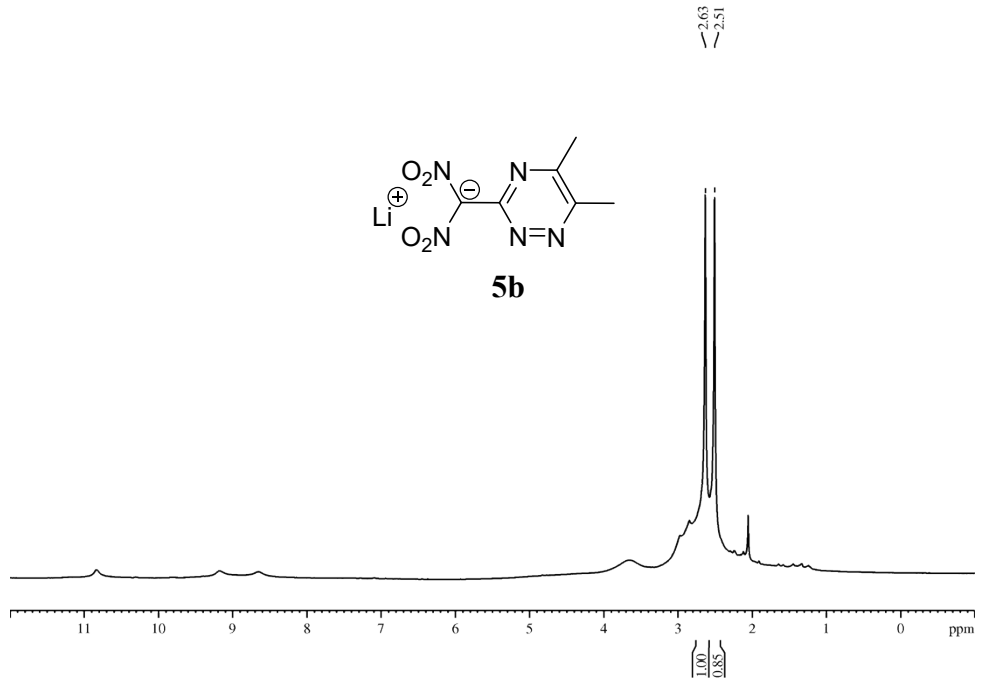


Figure S23: ^{1}H NMR-Compound **5b**

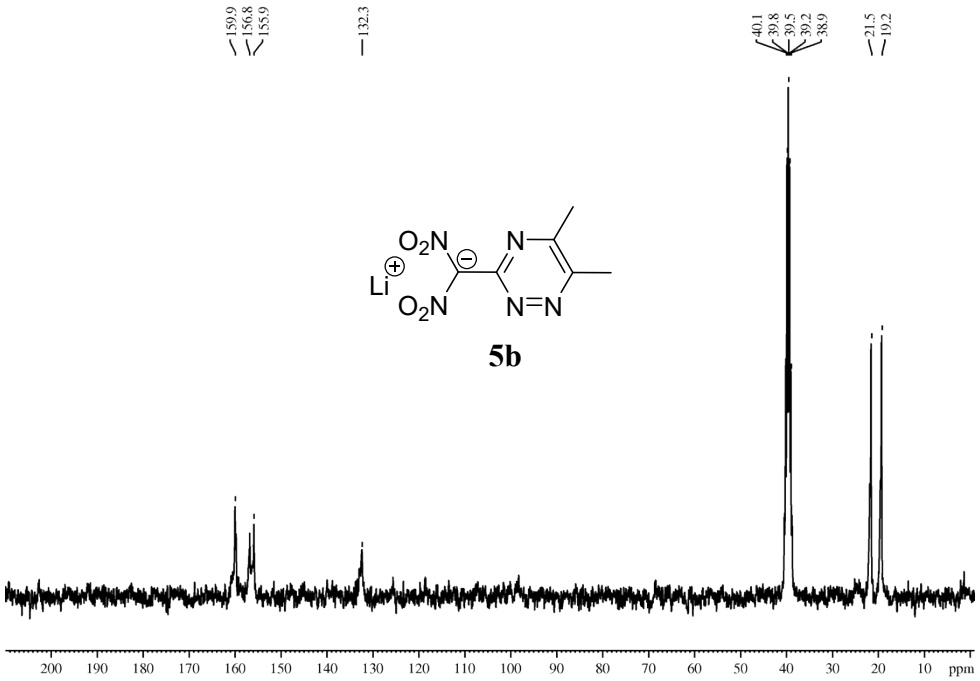


Figure S24: ^{13}C NMR-Compound **5b**

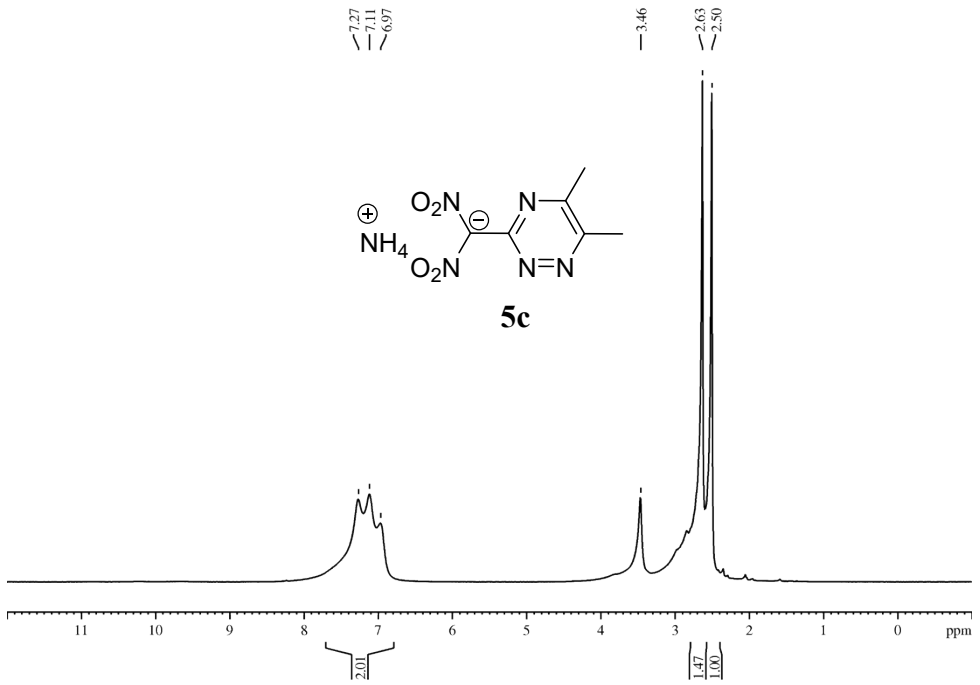


Figure S25: ^{1}H NMR-Compound **5c**

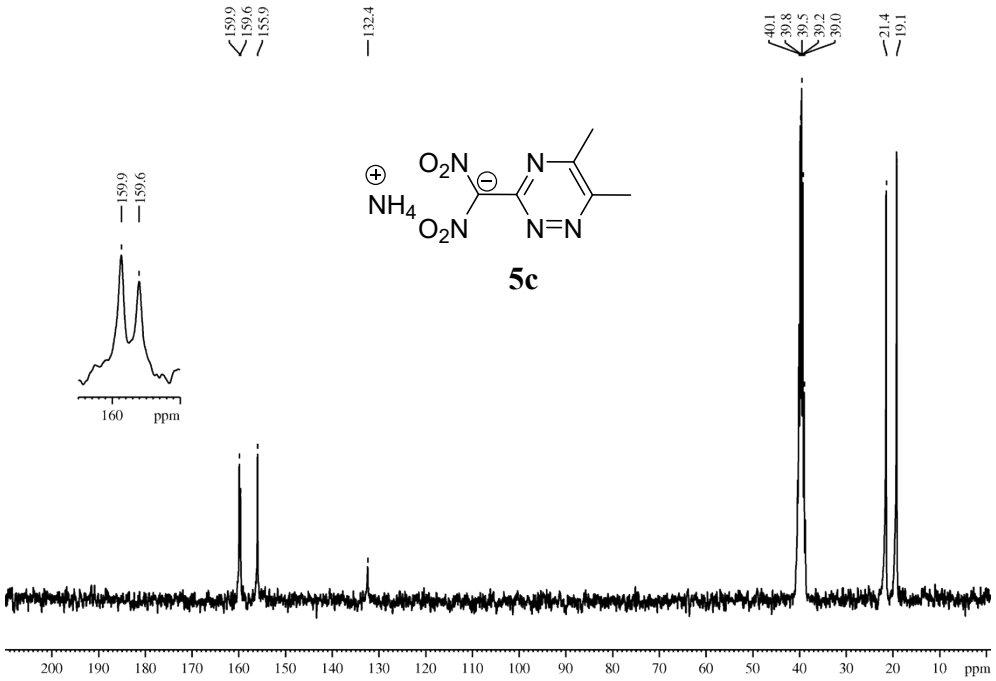


Figure S26: ^{13}C NMR-Compound **5c**

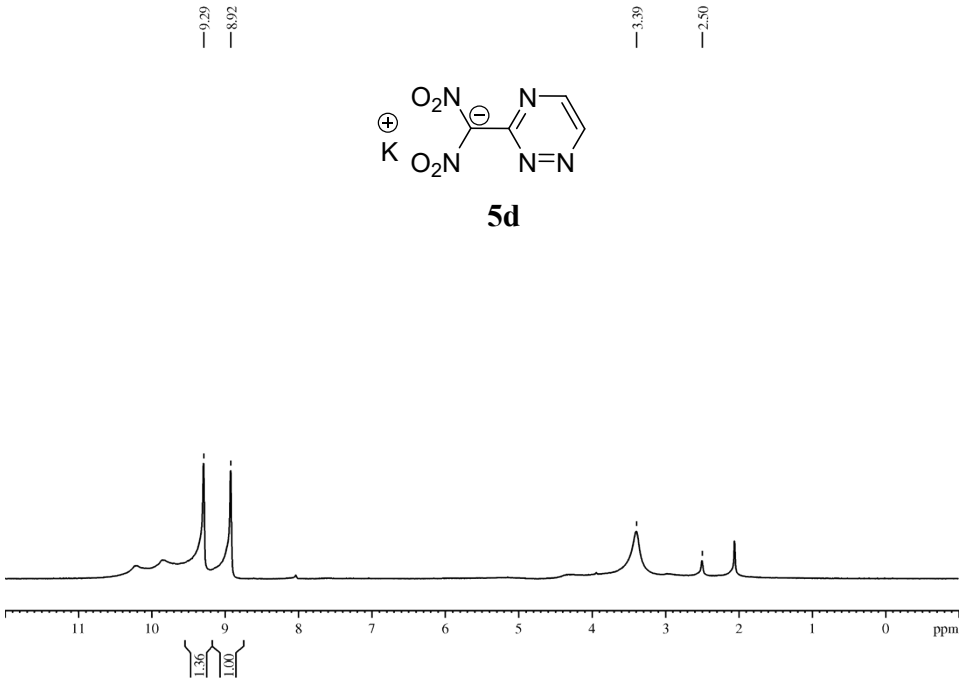
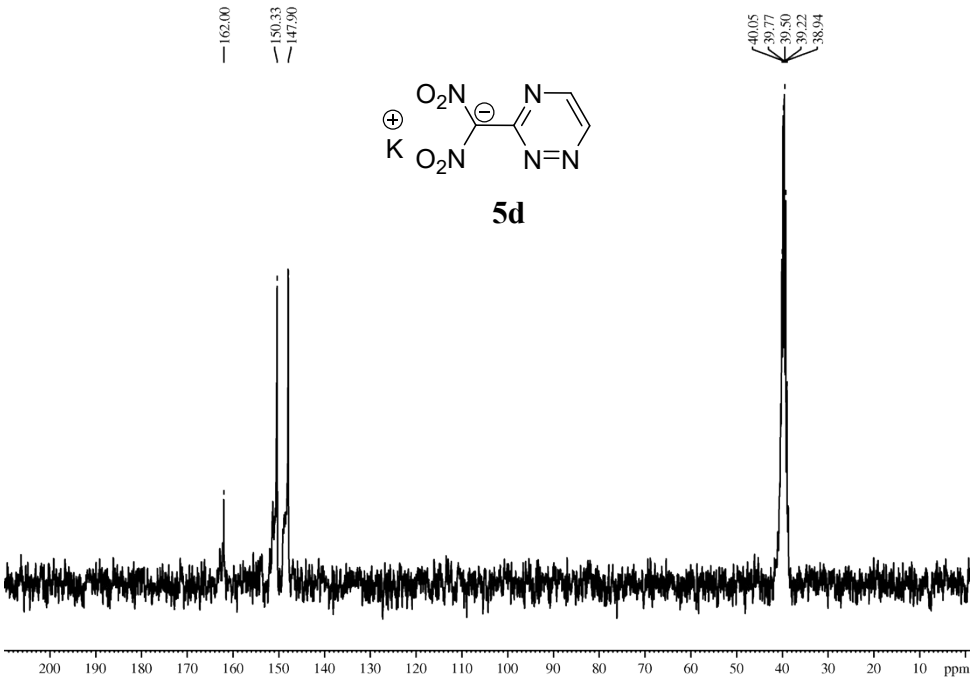
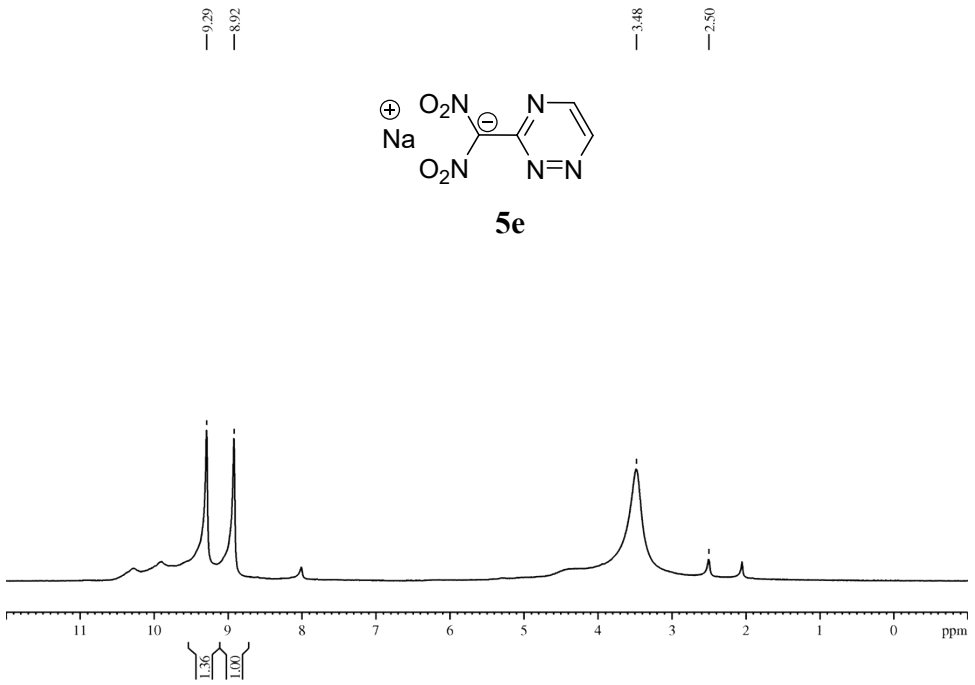


Figure S27: ^{1}H NMR-Compound **5d**



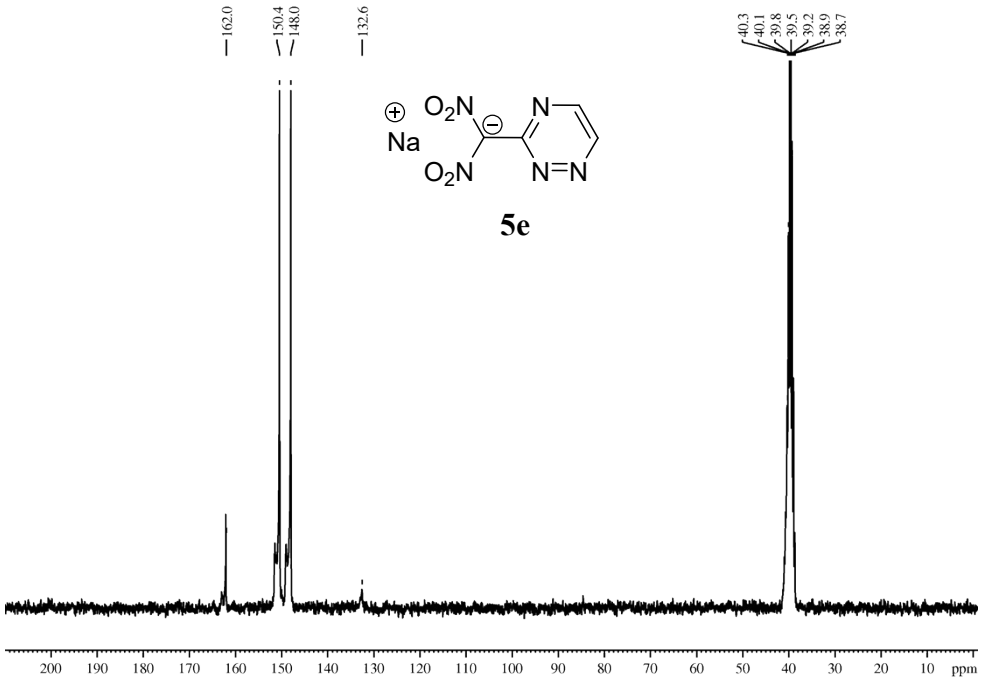
5d

Figure S28: ^{13}C NMR-Compound **5d**



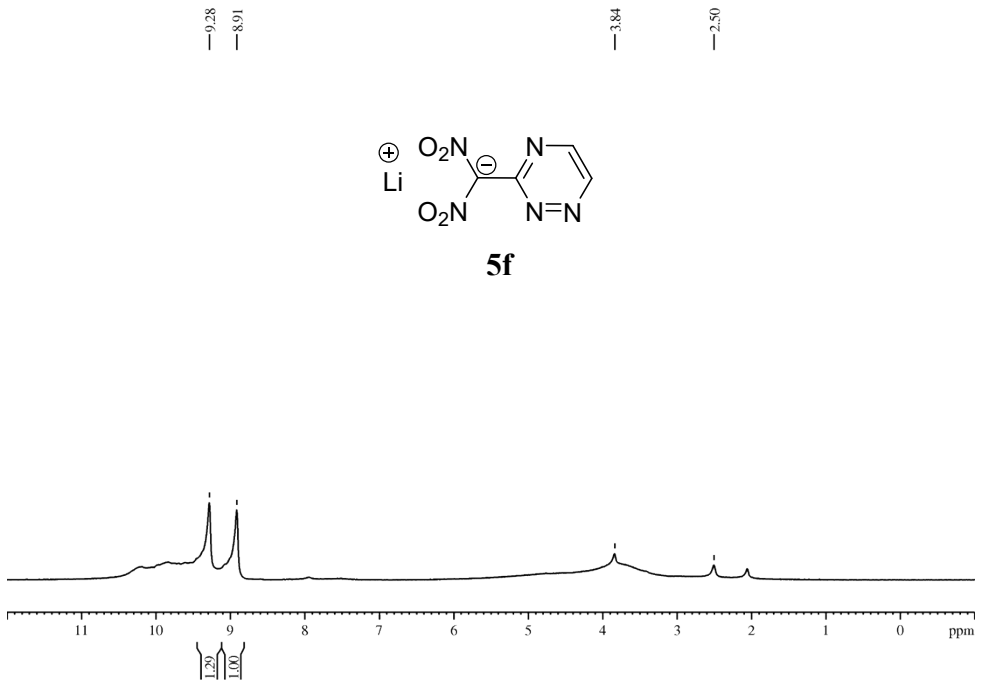
5e

Figure S29: ^{1}H NMR-Compound **5e**



5e

Figure S30: ^{13}C NMR-Compound **5e**



5f

Figure S31: ^{1}H NMR-Compound **5f**

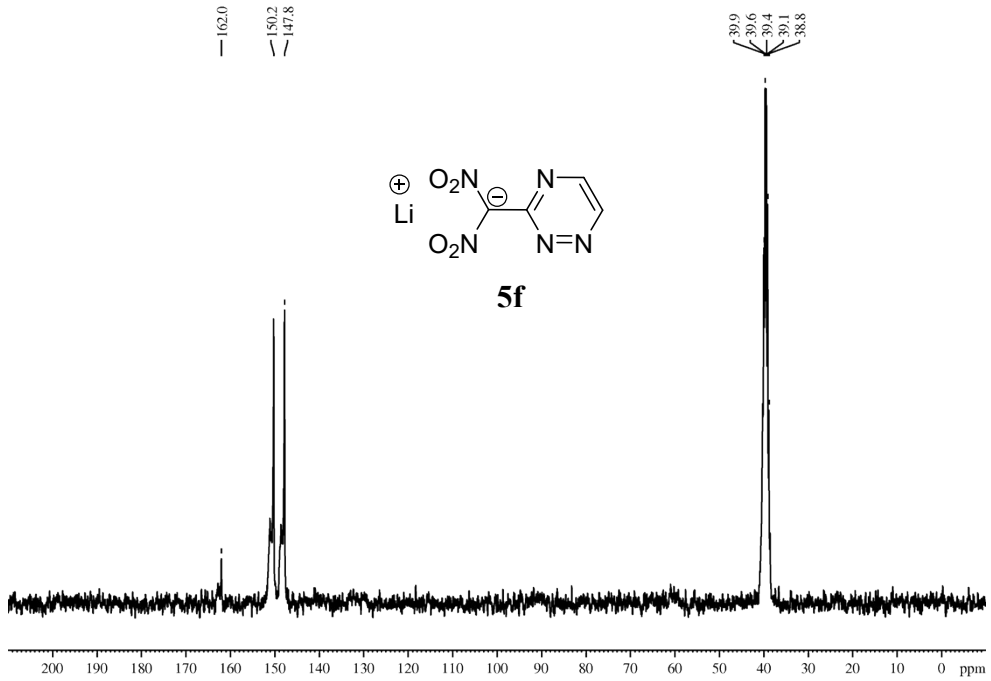


Figure S32: ^{13}C NMR-Compound **5f**

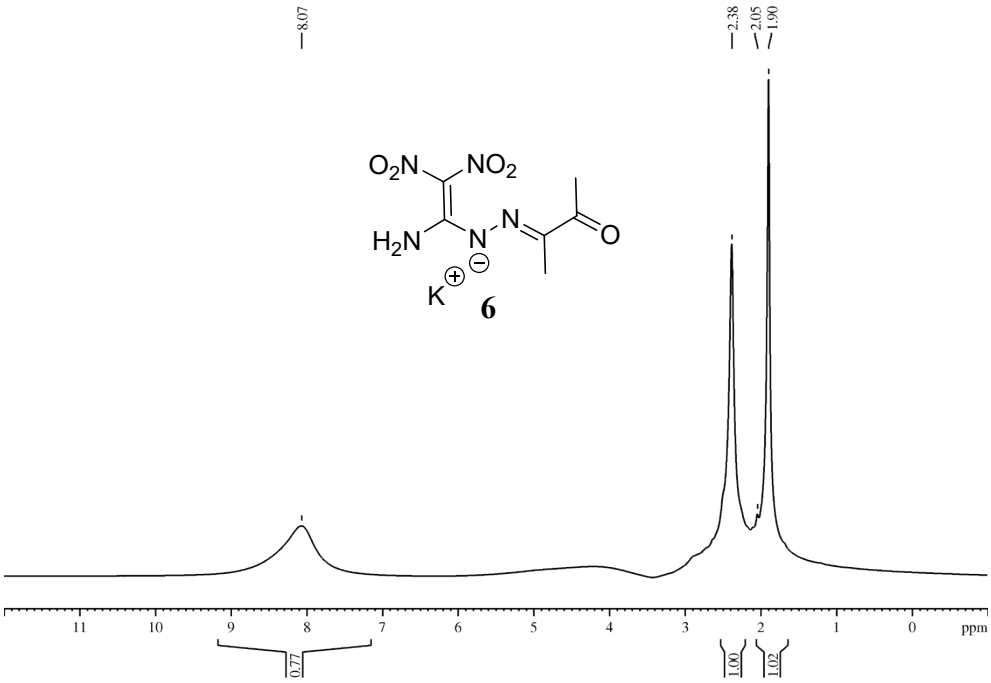


Figure S33: ^{1}H NMR-Compound **6**

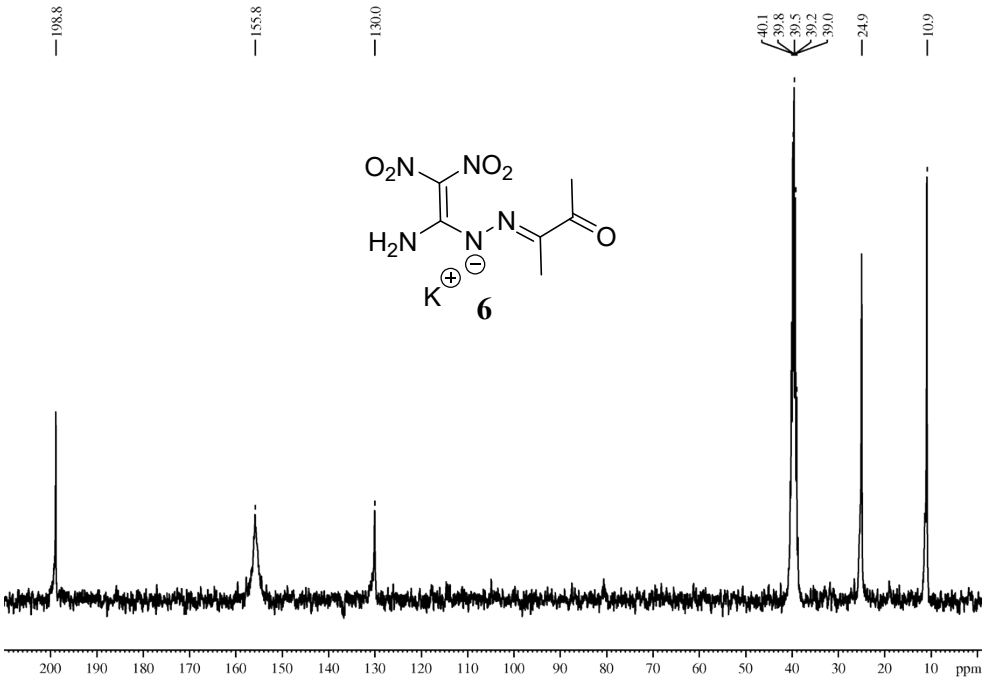


Figure S34: ^{13}C NMR-Compound **6**

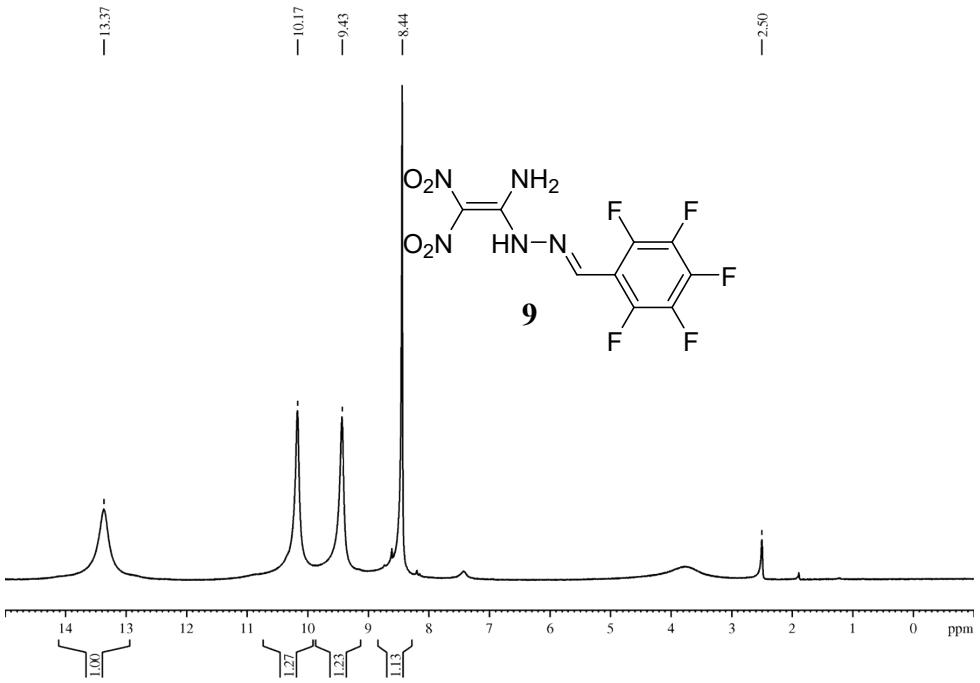


Figure S35: ^{1}H NMR-Compound **9**

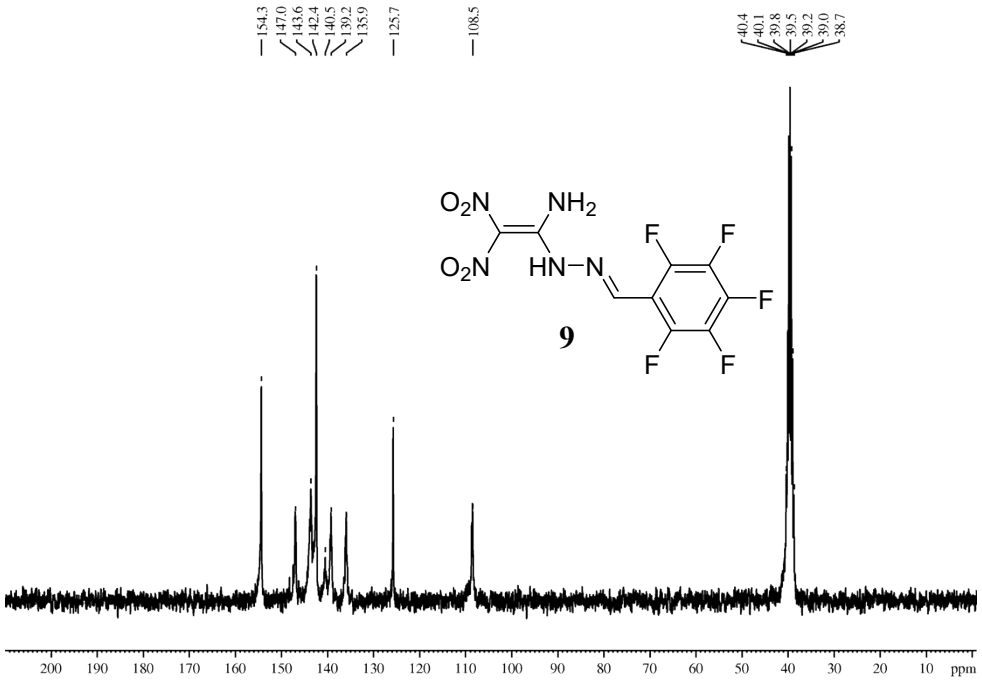


Figure S36: ^{13}C NMR-Compound **9**

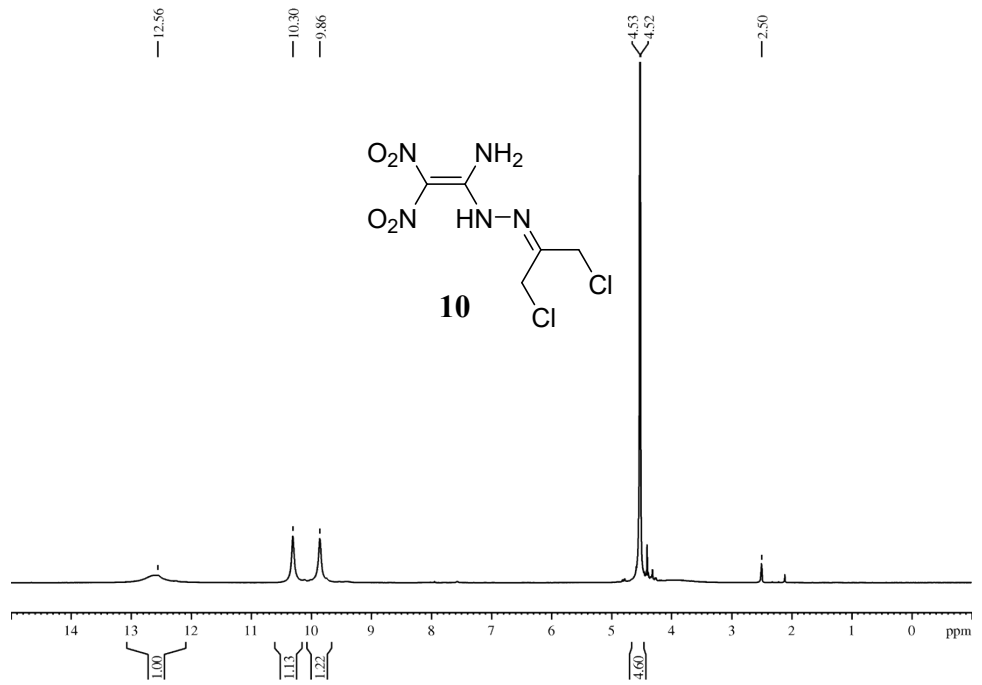


Figure S37: ^{1}H NMR-Compound **10**

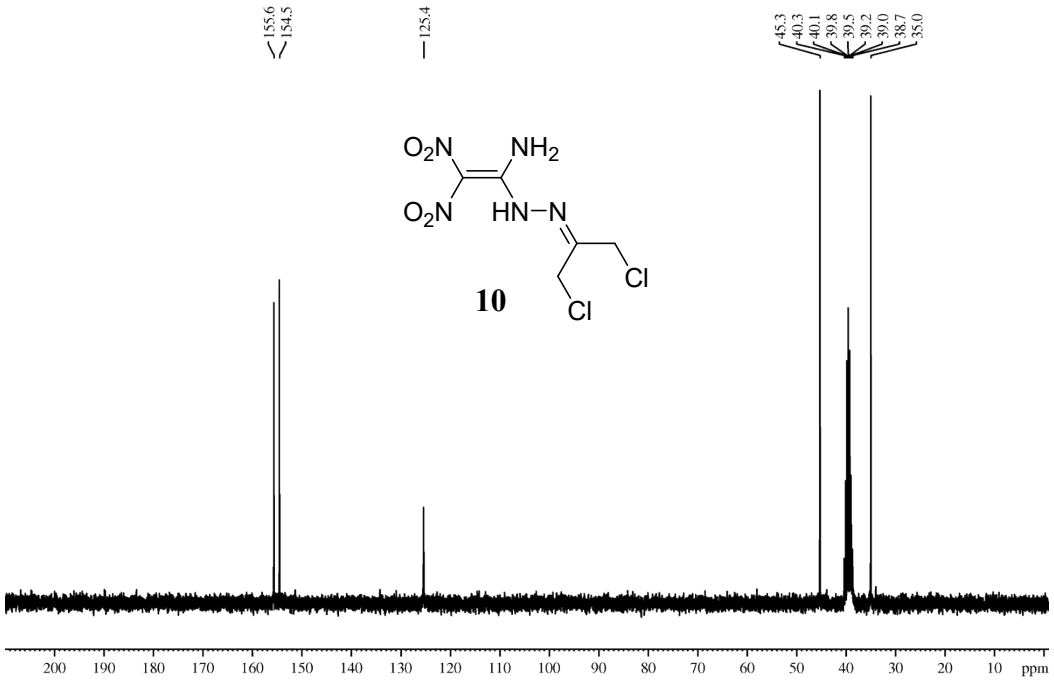


Figure S38: ^{13}C NMR-Compound **10**

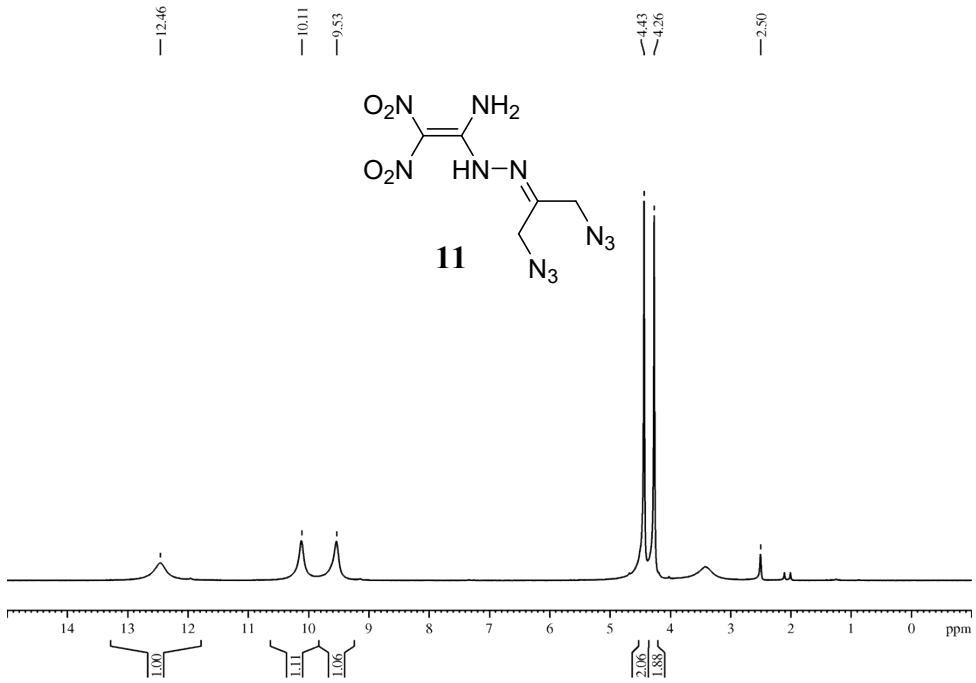


Figure S39: ^{1}H NMR-Compound **11**

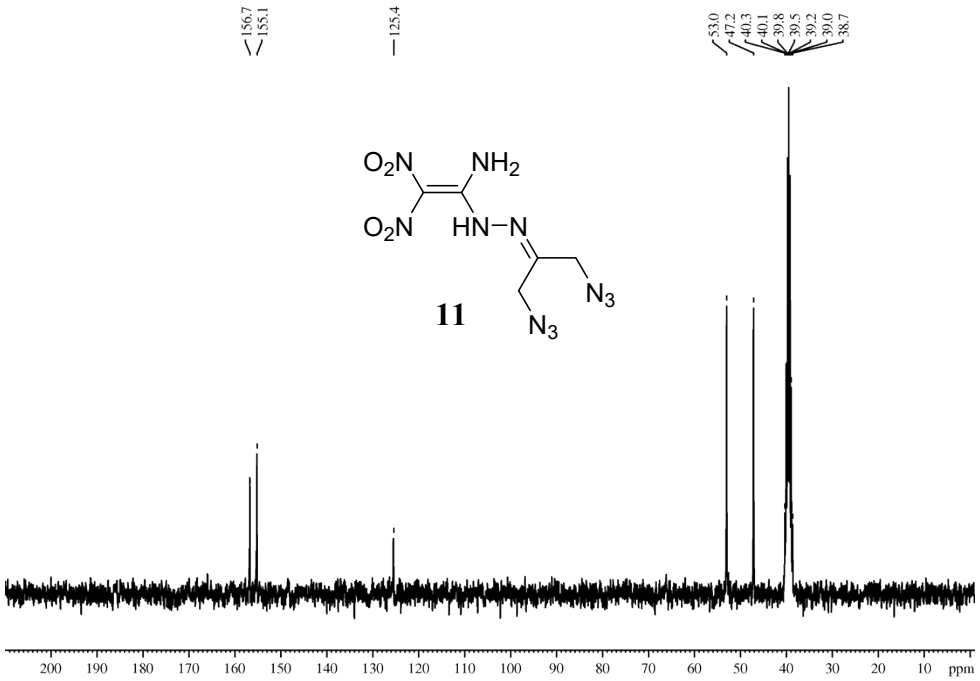


Figure S40: ^{13}C NMR-Compound **11**

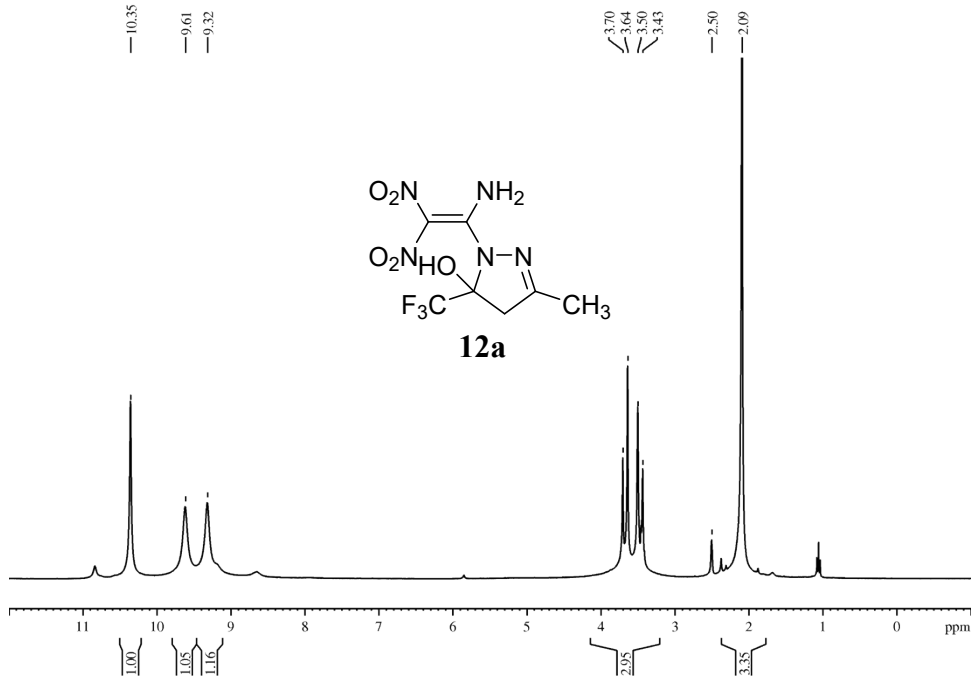


Figure S41: ^{1}H NMR-Compound **12a**

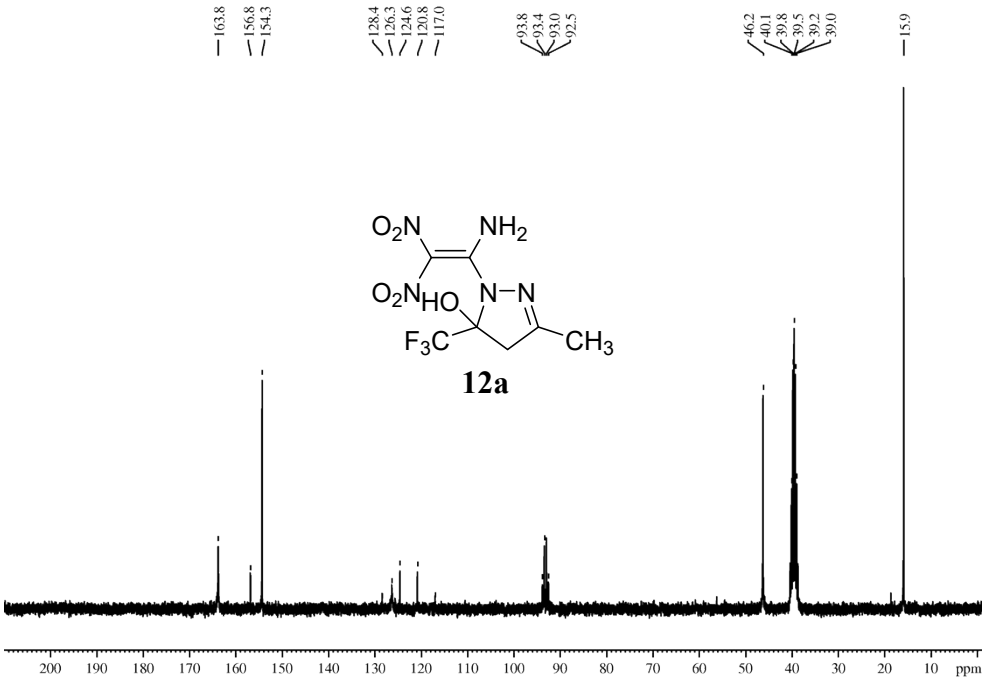


Figure S42: ^{13}C NMR-Compound **12a**

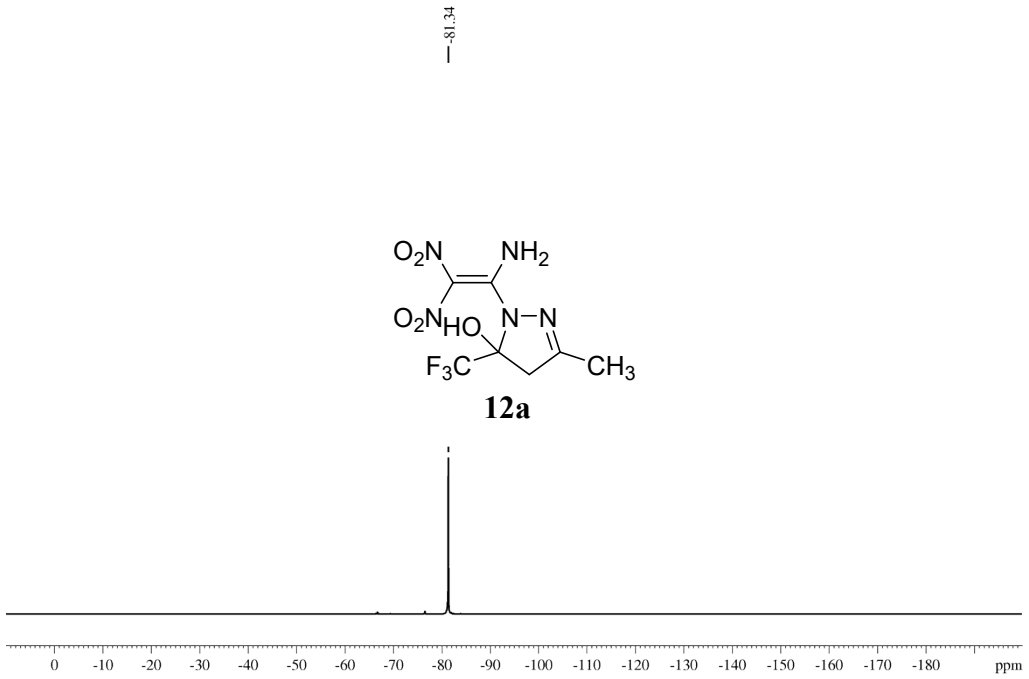


Figure S43: ^{19}F NMR-Compound **12a**

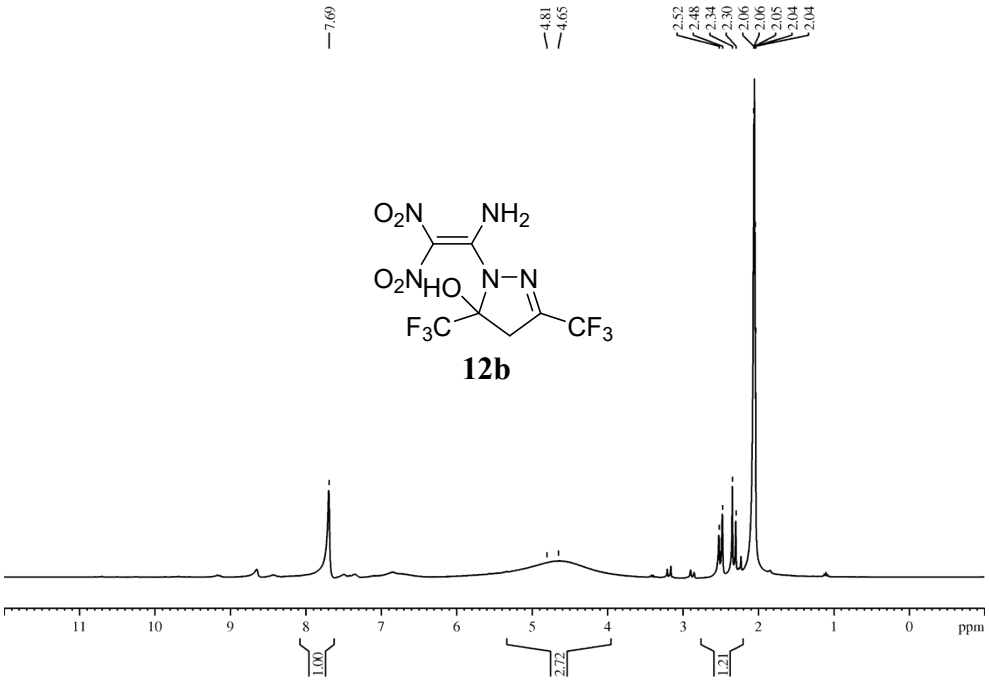


Figure S44: ^{1}H NMR-Compound **12b** in Acetone-d_6

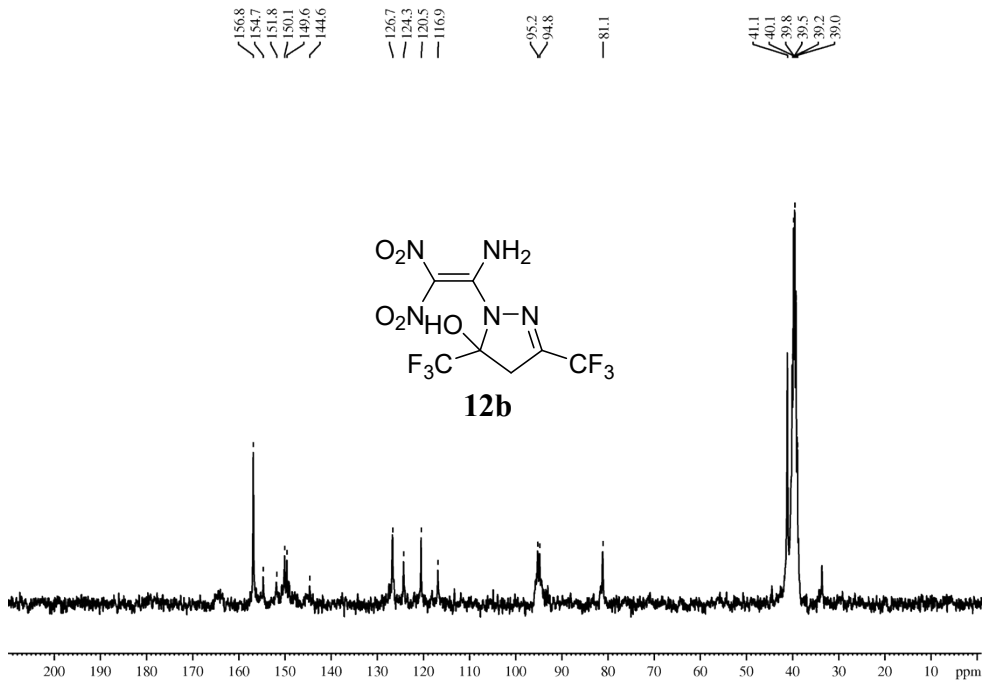


Figure S45: ^{13}C NMR-Compound **12b** in DMSO-d_6

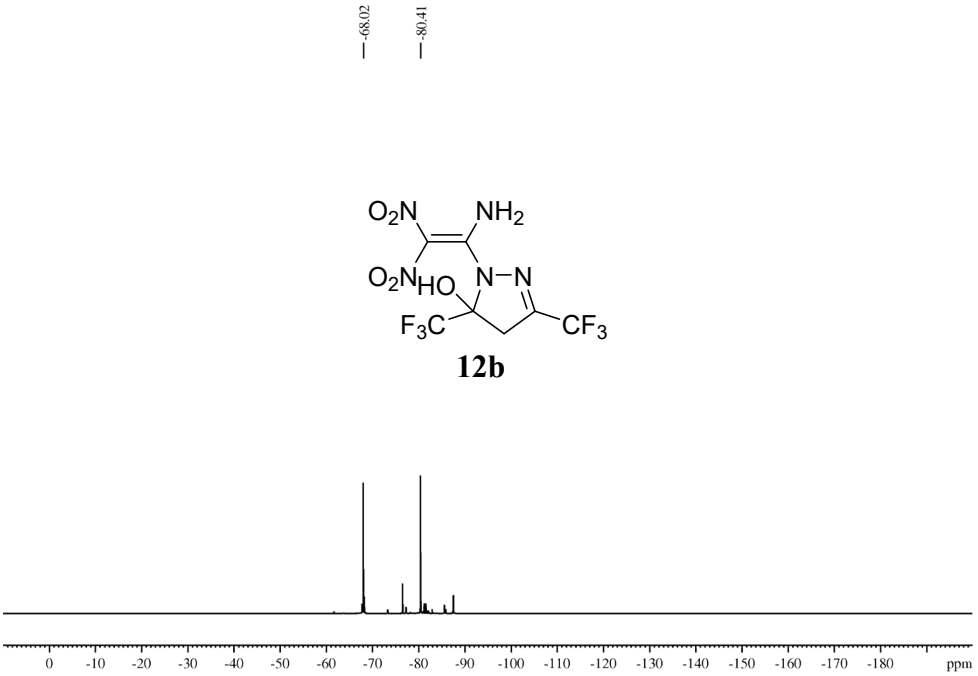


Figure S46: ^{19}F NMR-Compound **12b** in DMSO-d_6

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