Supporting Information

Pushing the Boundaries of the Oxygen Balance of a Pyrazole Ring

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1. General Information

Caution! All compounds prepared herein are extremely dangerous. Although we have encountered no difficulties in preparing these compounds in this work, manipulations must be carried out by using appropriate standard safety precautions. Eye protection must be worn. Mechanical actions of these energetic materials involving scratching or scraping must be avoided.

Solvents were dried and distilled using standard methods. Unless otherwise noted, all reagents were purchased at the highest commercial quality and used without further purification. ^{1}H spectra were recorded on a 400 MHz (JEOL ECZ400S spectrometer) nuclear magnetic resonance spectrometer operating at 400 MHz. ^{13}C spectra were recorded on a 400 MHz (JEOL ECZ400S spectrometer) nuclear magnetic resonance spectrometer operating at 100 MHz and a 600 MHz (JEOL ECZ600R spectrometer) nuclear magnetic resonance spectrometer operating at 150 MHz. DMSO-d_6 was used as solvent. Chemical shifts in the ^{1}H and ^{13}C spectra are reported relative to Me_4Si. High Resolution Mass spectra (HRMS) were performed on Bruker Daltonics Micro Tof-Q II mass spectrometer. The melting and decomposition (onset temperature) points were obtained on a differential scanning calorimeter (Mettler Toledo DSC 3) at a heating rate of 5 ℃ min^{-1}. IR spectra were recorded using KBr pellets with a FTIR spectrometer (Bruker INVENIO R instrument). X-ray intensity data were collected on a Bruker APEX-II CCD diffractometer. Density was determined from single crystal X-ray diffraction. Elemental analyses (C, H, N) were performed on a Euro Vector EA3000 Elementar Analyser. Impact and friction sensitivity measurements were made using a standard BAM Fall hammer and a BAM friction tester.

2. Computational Details

Computations were carried out by using the Gaussian 09 suite of programs.[1] The elementary geometric optimization and the frequency analysis were performed at the level of Becke three Lee-Yan-Parr (B3LYP) Functionals[2-3] with 6-31+G** basis set.[4] All of the optimized structures were characterized to be local energy minima on the

potential surface without any imaginary frequencies. Then, the single-point energies of optimized structures were accessed under the level of MP2/6-311++G**. The predictions of heats of formation (HOF) were implemented via designed isodesmic reactions. The isodesmic reaction processes, i.e., the number of each kind of formal bond is conserved, are used with application of the bond separation reaction (BSR) rules. The isodesmic reactions used to derive the HOF of these compounds are in Fig. S1.

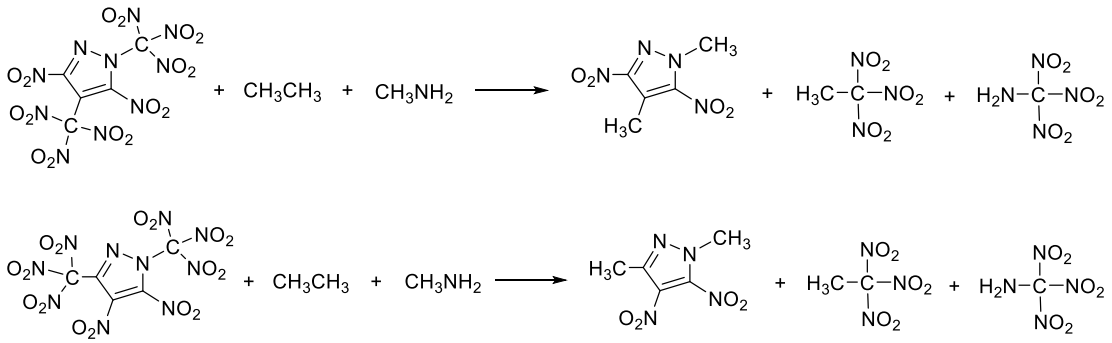


Fig. S1 Isodemic reaction for computing the HOF.

The change of enthalpy for the reactions at 298 K can be expressed as:

$\Delta H_{298} = \sum \Delta_f H_P - \sum \Delta_f H_R$ (eq. 1)

Where $\Delta_f H_P$ and $\Delta_f H_R$ are the HOF of reactants and products at 298 K, respectively, and $\Delta H298$ can be calculated using the following expression:

$\Delta H_{298} = \Delta E_{298} + \Delta(PV) = \Delta E_0 + \Delta ZPE + \Delta H_T + \Delta nRT$ (eq. 2)

Where E_0 is the change in total energy between the products and the reactants at 0 K; ΔZPE is the difference between the zero-point energies (ZPE) of the products and the reactants at 0 K; ΔH_T is thermal correction from 0 to 298 K. The $\Delta(PV)$ value in eq (2) is the PV work term. It equals ΔnRT for the reactions of ideal gas. For the isodesmic reactions, $\Delta n = 0$, so $\Delta(PV) = 0$. On the left side of Eq. (1), apart from target compound, all the others are called reference compounds. The HOF of reference compounds are available either from the experiments[5-8] or from the high level computing like G4(MP2)-6x.[9]

For neutral compounds with calculated gas state heat of formation, the solid state heat of formation was calculated by the following equation (3). The heat of sublimation ΔH_{sub} was estimated by the following equation (4). T is the melting point or decomposition temperature in Kelvin.

$\Delta H_f\,(s) = \Delta H_f\,(g) - \Delta H_{sub}$ (eq. 3)

$\Delta H_{sub} = 0.188 * T$ (eq. 4)

3. Synthetic Procedures

3,5-Dinitro-1*H*-pyrazole-4-carbonitrile (3a).

3,5-Dinitro-1*H*-pyrazole-4-carboxamide[10] (5.0 mmol, 1.0 g) was dissolved in anhydrous CH_3CN (25.0 mL), $POCl_3$ (20.0 mmol, 1.8 mL) was added. The mixture was refluxed for 24 hours. After cooled to room temperature, the mixture was adjusted to neutral by aqueous ammonia. The mixture was filtered and the filtrate was removed under reduced pressure and used without further purification (3.3 mmol, 603.9 mg, 66% yield). **HRMS** (ESI) exact mass calculated for $[M-H]^-$ ($C_4N_5O_4$) requires *m/z* 181.9956; found *m/z* 181.9956.

N'-Hydroxy-3,5-dinitro-1*H*-pyrazole-4-carboximidamide (4a).

3a (3.3 mmol, 603.9 mg) was dissolved in absolute ethanol (10.0 mL), then a solution of hydroxylamine hydrochloride (4.95 mmol, 344.0 mg) and Na_2CO_3 (2.48 mmol, 262.8 mg) in water (20.0 mL) was added. The mixture was then refluxed for 24 hours. After cooled to room temperature, the solvent was removed under reduced pressure. Ethanol was added to the remaining solid, and filtered. The filtrate was evaporated to dryness and washed with ethyl acetate to obtain the product (Yellow solid, 2.64 mmol, 570.2 mg, 80% yield). T_d (onset): 162.7 ^oC. ^1H NMR (400 MHz, DMSO-d_6) δ 7.59 (br, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.8, 152.8, 145.3, 111.4. IR (KBr pellet): $\tilde{\upsilon}$ = 3370, 3177, 1659, 1514, 1405, 1354, 1318, 1126, 1091, 1031, 999, 941, 851, 772, 705, 648, 560, 412 cm^{-1}. **HRMS** (ESI) exact mass calculated for $[M-H]^-$ ($C_4H_3N_6O_5$) requires *m/z* 215.0170; found *m/z* 215.0166.

N-Hydroxy-3,5-dinitro-1*H*-pyrazole-4-carbimidoyl chloride (5a).

To an ice-cold solution of **4a** (2.7 mmol, 583.2 mg) in semi-HCl (15.0 mL), was added dropwise a solution of $NaNO_2$ (4.0 mmol, 276.0 mg) in water (2.0 mL). The reaction was gradually warmed to room temperature and stirred overnight. The reaction was extracted by ethyl acetate and washed with water, dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure to obtain a yellow hygroscopic solid (1.89 mmol, 445.2 mg, 70% yield). **HRMS** (ESI) exact mass calculated for $[M-H]^-$ ($C_4HClN_5O_5$) requires *m/z* 233.9672; found *m/z* 233.9672.

Potassium (3,5-dinitro-1-(2-oxopropyl)-1*H*-pyrazol-4-yl)dinitromethanide (8a).

To an ice-cold solution of fuming HNO_3 (2.0 mL) and trifluoroacetic anhydride (4.6 mL), a solution of **5a** (2.0 mmol, 471.1 mg) in anhydrous dichloromethane (5.0 mL) was added dropwise. The reaction was then stirred for 2 hours at 0 ^oC and 24 hours at room temperature. The reaction was poured into ice water, extracted by dichloromethane for 3 times, washed with water, dried over anhydrous Na_2SO_4. The solvent was removed by air to get the crude product **6a**. Then ethyl acetate (6.0 mL) was added, and the solution was cooled to 0 ^oC. A solution of diazoacetone (4.0 mmol, 336.0 mg) in ethyl acetate (3.0 mL) was added and the reaction was stirred at room temperature for 24 hours. The solvent was removed by air and methanol (6.0 mL) and KI (5.0 mmol, 830.0 mg) were added. The reaction mixture was protected from light and reacted overnight at room temperature. Then the mixture was filtered, washed with methanol and ether to obtain a yellow solid **8a** (1.1 mmol, 391.9 mg, 55% yield). T_d (onset): 218.1 ^oC. ^1H NMR (400 MHz, DMSO-d_6) δ 5.78 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 200.0, 148.9, 141.4, 123.6, 109.0, 63.6, 27.1. IR (KBr pellet): $\tilde{\upsilon}$ = 3458, 3027, 2965, 2557, 2362, 1737, 1607, 1552, 1535, 1486, 1441, 1400, 1367, 1334, 1287, 1256, 1203, 1133, 898, 852, 836, 812, 789, 755, 689, 655, 508, 460 cm^{-1}. **HRMS** (ESI) exact mass calculated for $[M-K]^-$ ($C_7H_6N_6O_9$) requires *m/z* 317.0123; found *m/z* 317.0120.

3,5-Dinitro-1,4-bis(trinitromethyl)-1*H*-pyrazole (1).

To an ice-cold suspension of **8a** (0.5 mmol, 178.1 mg) in 98% H_2SO_4 (0.92 mL), fuming HNO_3 (0.6 mL) was added. The reaction was stirred for 3 days at room temperature. The mixture was filtered and washed by trifluoroacetic acid to get white solid (0.15 mmol, 68.4 mg, 30% yield). T_d (onset): 147.0 ^oC. ^{13}C NMR (150 MHz, DMSO-d_6) δ 169.3, 163.2, 161.0, 124.3, 112.7. IR (KBr pellet): $\tilde{\upsilon}$ = 3438, 2978, 2892, 2619, 2362, 2335, 1642, 1613, 1548, 1423, 1381, 1318, 1267, 1193, 998, 849, 828, 786, 723 cm^{-1}. Elemental analysis (%) $C_5N_{10}O_{16}$ (455.95) calcd C, 13.17, N, 30.71; found C, 12.72, N, 30.13.

N'-Hydroxy-3,4-dinitro-1*H*-pyrazole-5-carboximidamide (4b).

3,4-Dinitro-1*H*-pyrazole-5-carbonitrile (**3b**) was synthesized according to a

literature procedure.[11] To a solution of **3b** (7.1 mmol, 1.3 g) in absolute ethanol (12.0 mL), was added a solution of hydroxylamine hydrochloride (11.44 mmol, 795.1 mg) in water (5.0 mL), and a solution of Na_2CO_3 (8.3 mmol, 879.7 mg) in water (25.0 mL). The mixture was then reacted for 16 hours at room temperature. The solvent was removed under reduced pressure. Ethanol was added to the remaining solid and filtered. The filtrate was evaporated to dryness and washed with ether to obtain the product **4b** (Yellow solid, 6.75 mmol, 1.46 g, 95% yield). M.p. = 94.9 ^{o}C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.54 (br, 1H), 5.57 (br, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.1, 146.2, 138.4, 125.6. IR (KBr pellet): $\tilde{\upsilon}$ = 3509, 3410, 2243, 1645, 1531, 1500, 1387, 1368, 1345, 1256, 1128, 1091, 930, 855, 842, 737, 718, 654, 465 cm^{-1}. **HRMS** (ESI) exact mass calculated for $[M-H]^-$ ($C_4H_3N_6O_5$) requires *m/z* 215.0170; found *m/z* 215.0172.

N-Hydroxy-3,4-dinitro-1*H*-pyrazole-5-carbimidoyl chloride (5b).

To an ice-cold solution of **4b** (6.9 mmol, 1.5 g) in semi-HCl (38.0 mL), was added a solution of $NaNO_2$ (8.75 mmol, 603.8 mg) in water (7.0 mL). The reaction was then stirred at the same temperature for an hour and filtered. The cake was washed with water and dried by air to give a pale white solid (5.4 mmol, 1.27 g, 77% yield). T_d (onset): 185.7 ^{o}C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.46 (br, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 146.6, 133.4, 124.9, 122.7. IR (KBr pellet): $\tilde{\upsilon}$ = 3636, 3567, 3240, 2721, 1786, 1619, 1549, 1513, 1432, 1377, 1338, 1305, 1223, 1175, 1073, 1032, 913, 850, 815, 757, 717, 504 cm^{-1}. **HRMS** (ESI) exact mass calculated for $[M-H]^-$ ($C_4HClN_5O_5$) requires *m/z* 233.9672; found *m/z* 233.9672.

Potassium (4,5-dinitro-1-(2-oxopropyl)-1*H*-pyrazol-3-yl)dinitromethanide (8b).

To an ice-cold solution of fuming HNO_3 (7.0 mL) and trifluoroacetic anhydride (9.28 mL), a solution of **5b** (5.4 mmol, 1.27 g) in anhydrous dichloromethane (14.0 mL) was added. The reaction was then stirred for 3 hours at the same temperature, and poured into ice water. The mixture was extracted by dichloromethane for 3 times, washed with water, dried over anhydrous Na_2SO_4. The solvent was removed by air to get the crude product **6b**. Then ethyl acetate (20.0 mL) was added, and the solution was cooled to 0 ^{o}C. A solution of diazoacetone (10.8 mmol, 907.2 mg) in ethyl acetate

(10.0 mL) was added and the reaction was stirred at room temperature for 3 hours. The solvent was removed by air. Methanol (15.0 mL) and KI (15.0 mmol, 2.5 g) were added. The reaction mixture was protected from light and reacted overnight at room temperature. Then the mixture was filtered, washed with methanol and ether to obtain **8b** (Yellow solid 4.32 mmol, 1.54 g, 80% yield). T_d (onset): 208.5 ^oC. ^1H NMR (400 MHz, DMSO-d_6) δ 5.70 (s, 2H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 199.9, 139.6, 139.5, 125.6, 124.0, 62.4, 27.0. IR (KBr pellet): $\tilde{\upsilon}$ = 3441, 3000, 2958, 1726, 1532, 1460, 1409, 1383, 1358, 1337, 1278, 1229, 1207, 1173, 1137, 1043, 885, 843, 808, 789, 755, 698, 657, 585, 527, 478 cm^{-1}. **HRMS** (ESI) exact mass calculated for $[M-K]^-$ ($C_7H_6N_6O_9$) requires *m/z* 317.0123; found *m/z* 317.0122.

4,5-Dinitro-1,3-bis(trinitromethyl)-1*H*-pyrazole (2).

To an ice-cold suspension of **8b** (0.5 mmol, 178.0 mg) in 98% H_2SO_4 (0.92 mL), fuming HNO_3 (0.6 mL) was added dropwise. The reaction was stirred for 2 days at room temperature. The mixture was filtered and washed by trifluoroacetic acid to give compound **2** (White solid, 0.21 mmol, 95.7 mg, 42% yield). T_d (onset): 145.5 ^oC. ^{13}C NMR (150 MHz, DMSO-d_6) δ 156.4, 138.5, 123.6, 120.9, 111.9. IR (KBr pellet): $\tilde{\upsilon}$ = 3447, 2598, 2490, 2362, 2337, 1733, 1628, 1557, 1327, 1240, 1206, 1068, 997, 850, 791, 614, 571 cm^{-1}. Elemental analysis (%) $C_5N_{10}O_{16}$ (455.95) calcd C, 13.17, N, 30.71; found C, 13.33, N, 30.63.

4. X-ray Crystallographic Data

The single crystal of **1** and **2** were obtained by slow evaporation from fuming nitric acid at room temperature.

The obtained diffraction data was processed by the Bruker APEX6 program suite. The structure was solved with dual-space method using SHELXT program[12] and refined with non-linear least square method using SHELXL program[13] embedded in the program suite OLEX2.[14]

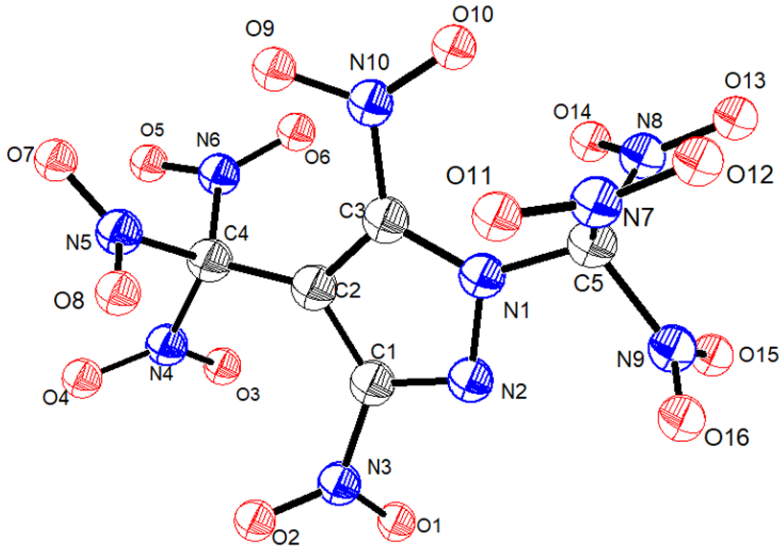


Fig. S2 Thermal ellipsoid plot of 3,5-dinitro-1,4-bis(trinitromethyl)-1H-pyrazole (**1**) (ellipsoid contour probability levels:50%).

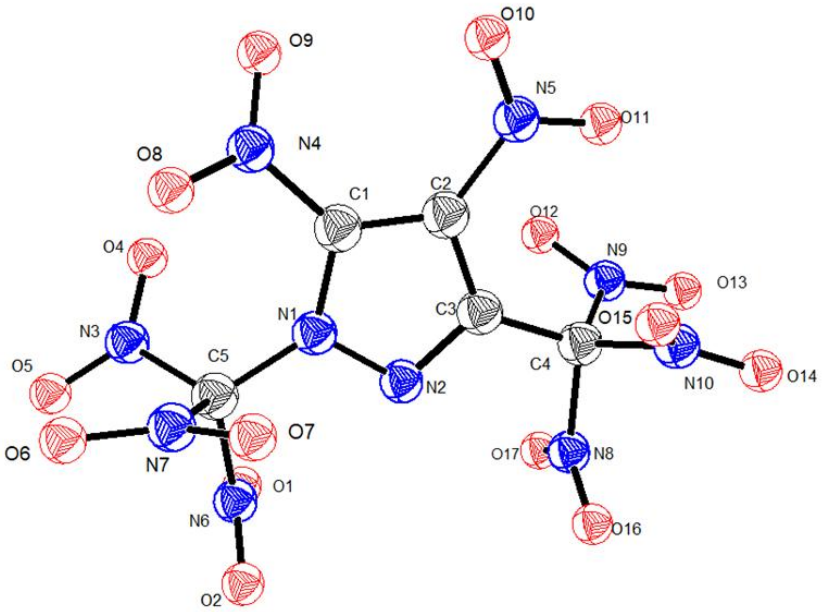


Fig. S3 Thermal ellipsoid plot of 4,5-dinitro-1,3-bis(trinitromethyl)-1H-pyrazole (**2**) (ellipsoid contour probability levels:50%).

Table S1 Crystal data of **1** and **2**

Compound	**1**	**2**
Empirical formula	$C_5N_{10}O_{16}$	$C_5N_{10}O_{16}$
CCDC number	2445620	2387892
Formula weight	456.15	456.15
Temperature/K	298.00	301.5
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	C2/c
a/Å	8.9631(5)	26.9816(15)
b/Å	19.6803(11)	6.5292(4)
c/Å	9.1908(5)	19.4184(12)
α/°	90	90
β/°	105.758(2)	111.089(2)
γ/°	90	90
Volume/$Å^3$	1560.29(15)	3191.8(3)
Z	4	8
$\rho_{calc}g/cm^3$	1.942	1.899
μ/mm^{-1}	1.148	0.194
F(000)	912.0	1824.0
Radiation	GaKα (λ = 1.34139)	MoKα (λ = 0.71073)
2Θ range for data collection/ °	7.816 to 113.972	4.496 to 55. 052
Index ranges	$-11 \le h \le 11$, $-23 \le k \le 24$, $-11 \le l \le 11$	$-32 \le h \le 31$, $-6 \le k \le 7$, $-22 \le l \le 23$
Reflections collected	13919	11091
Independent reflections	3153 [R_{int} = 0.0840, R_{sigma} = 0.0849]	2808 [R_{int} = 0.0515, R_{sigma} = 0.0432]
Data/restraints/parameters	3153/0/280	2808/21/290
Goodness-of-fit on F^2	1.083	1.076

Final R indexes [I>=2σ (I)]	R_1 = 0.0635, wR_2 = 0.1767	R_1 = 0.0708, wR_2 = 0.1729
Final R indexes [all data]	R_1 = 0.0912, wR_2 = 0.2025	R_1 = 0.1013, wR_2 = 0.1957
Largest diff. peak/hole / e Å^{-3}	0.26/-0.43	0.38/-0.36

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **1**

Atom	***x***	***y***	***z***	**U(eq)**
O1	3448(3)	3661.3(19)	6609(3)	84.7(9)
O2	3144(3)	4418.7(16)	4838(3)	77.4(8)
O3	2957(3)	3185.5(13)	3065(3)	72.4(8)
O4	2571(3)	4052.2(15)	1545(3)	74.9(8)
O5	4667(4)	2856.1(16)	750(3)	97.5(11)
O6	6531(3)	2669.2(12)	2737(3)	74.0(8)
O7	5525(4)	4153.7(17)	403(3)	84.8(9)
O8	5457(3)	4821.9(12)	2264(3)	76.7(8)
O9	8390(3)	3817.5(13)	2767(2)	60.0(7)
O10	10076(2)	3597.0(17)	4877(3)	75.1(9)
O11	9767(4)	4811.1(14)	6446(4)	94.0(11)
O12	11585(3)	4437.5(13)	8316(3)	60.9(6)
O13	11384(3)	3102.5(14)	8628(4)	88.5(10)
O14	9450(3)	2672.3(11)	6942(3)	72.0(8)
O15	8661(3)	3389.6(14)	9714(3)	72.7(8)
O16	8802(3)	4481.8(13)	9485(3)	64.4(7)
N1	7868(2)	3807.1(11)	6413(2)	31.3(5)
N2	6531(3)	3876.7(11)	6829(3)	35.5(5)
N3	3863(3)	3981.0(16)	5665(3)	53.6(7)
N4	3362(3)	3646.9(14)	2387(3)	51.5(7)
N5	5414(3)	4275.8(14)	1651(3)	53.5(7)
N6	5491(3)	2999.8(13)	1978(3)	54.4(7)
N7	10339(3)	4410.1(12)	7431(3)	41.1(6)
N8	10141(3)	3122.8(12)	7715(3)	45.5(6)
N9	8890(3)	3906.5(13)	9107(3)	42.7(6)
N10	8783(3)	3715.5(13)	4119(3)	41.1(6)

C1	5440(3)	3857.9(13)	5553(3)	34.2(6)
C2	5983(3)	3774.3(12)	4258(3)	32.0(6)
C3	7561(3)	3753.7(12)	4875(3)	31.6(6)
C4	5122(3)	3684.1(13)	2640(3)	36.3(6)
C5	9284(3)	3810.8(12)	7581(3)	30.0(6)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Table S3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for **1**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	47.8(15)	146(3)	66.8(17)	-1.5(18)	26.8(13)	-8.4(16)
O2	49.3(15)	87(2)	83.4(19)	-9.8(16)	-3.0(13)	28.0(14)
O3	53.6(15)	65.0(16)	89.3(19)	5.5(14)	3.9(13)	-21.5(12)
O4	56.7(15)	88.6(19)	61.0(16)	11.2(14)	-15.5(12)	21.8(14)
O5	115(2)	84(2)	63.2(17)	-42.9(16)	-27.0(16)	16.7(18)
O6	69.4(17)	46.6(13)	86.0(19)	-22.2(13)	-12.7(14)	19.7(12)
O7	105(2)	111(2)	34.4(14)	19.2(14)	13.5(14)	16.9(18)
O8	111(2)	37.1(13)	68.8(16)	12.0(12)	2.9(15)	-11.1(13)
O9	63.7(16)	89.7(18)	28.8(11)	-1.5(10)	16.4(11)	-5.6(12)
O10	32.3(13)	147(3)	46.0(13)	-0.6(15)	10.6(11)	3.0(14)
O11	89(2)	63.3(16)	101(2)	42.4(16)	-22.5(17)	-30.0(15)
O12	38.0(12)	71.8(16)	64.1(14)	-5.9(12)	-1.1(11)	-17.3(10)
O13	63.9(17)	57.8(16)	108(2)	-12.2(15)	-37.2(16)	26.9(13)
O14	76.9(18)	36.9(12)	86.5(18)	-19.9(12)	-4.9(14)	2.4(11)
O15	114(2)	67.7(16)	38.9(13)	16.3(11)	24.5(13)	1.6(15)
O16	74.1(17)	58.4(15)	64.5(15)	-29.9(12)	25.5(13)	-8.6(12)
N1	28.6(11)	38.5(12)	24.5(11)	-1.4(8)	3.2(9)	0.7(8)
N2	32.1(12)	41.4(12)	32.6(12)	-2.2(9)	7.9(10)	0.6(9)
N3	36.0(14)	72.1(18)	50.1(16)	-13.5(14)	7.4(13)	4.8(13)
N4	43.1(15)	52.5(15)	47.2(15)	-4.4(13)	-7.7(12)	0.0(12)
N5	63.2(18)	51.6(16)	36.8(15)	13.4(12)	-1.3(12)	1.9(13)
N6	62.6(18)	43.1(14)	47.4(15)	-15.1(12)	-2.0(13)	4.1(13)
N7	40.1(14)	37.2(13)	41.8(13)	-4.5(10)	4.1(11)	-6.9(10)
N8	47.9(15)	33.7(13)	47.8(14)	-2.1(11)	0.9(12)	9.2(10)
N9	47.2(14)	49.5(15)	27.8(12)	-4.5(10)	4.2(11)	-0.6(11)
N10	38.4(14)	56.7(15)	28.6(12)	-4.4(10)	10.0(11)	-6.1(10)
C1	29.4(14)	36.0(13)	34.6(14)	-3.4(10)	4.6(11)	1.5(10)

C2	34.4(14)	28.0(12)	27.7(13)	0.3(10)	-1.6(11)	-0.9(10)
C3	32.9(14)	35.3(13)	25.0(12)	-1.9(10)	5.2(11)	-0.3(10)
C4	41.9(16)	29.7(13)	29.1(13)	-0.5(10)	-4.2(11)	0.3(11)
C5	29.5(14)	30.6(12)	26.0(12)	-1.7(10)	0.7(10)	0.9(9)

The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Table S4 Bond Lengths for **1**

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
O1	N3	1.209(4)	N1	N2	1.360(3)
O2	N3	1.212(4)	N1	C3	1.369(3)
O3	N4	1.212(3)	N1	C5	1.422(3)
O4	N4	1.200(3)	N2	C1	1.308(3)
O5	N6	1.203(4)	N3	C1	1.466(4)
O6	N6	1.194(3)	N4	C4	1.533(4)
O7	N5	1.202(4)	N5	C4	1.542(4)
O8	N5	1.209(4)	N6	C4	1.550(3)
O9	N10	1.213(3)	N7	C5	1.541(3)
O10	N10	1.202(3)	N8	C5	1.545(3)
O11	N7	1.205(3)	N9	C5	1.549(3)
O12	N7	1.191(3)	N10	C3	1.450(3)
O13	N8	1.200(3)	C1	C2	1.413(4)
O14	N8	1.198(3)	C2	C3	1.373(4)
O15	N9	1.204(3)	C2	C4	1.489(3)
O16	N9	1.193(3)			

Table S5 Bond Angles for **1**

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N2	N1	C3	110.6(2)	O9	N10	C3	115.8(2)
N2	N1	C5	117.5(2)	O10	N10	O9	126.3(2)
C3	N1	C5	131.9(2)	O10	N10	C3	117.9(2)
C1	N2	N1	104.3(2)	N2	C1	N3	115.6(2)
O1	N3	O2	127.3(3)	N2	C1	C2	114.5(2)
O1	N3	C1	117.1(3)	C2	C1	N3	129.7(2)
O2	N3	C1	115.6(3)	C1	C2	C4	130.7(2)
O3	N4	C4	113.4(2)	C3	C2	C1	102.0(2)
O4	N4	O3	128.5(3)	C3	C2	C4	127.2(2)
O4	N4	C4	118.0(3)	N1	C3	N10	122.2(2)
O7	N5	O8	128.4(3)	N1	C3	C2	108.7(2)
O7	N5	C4	118.8(3)	C2	C3	N10	129.1(2)
O8	N5	C4	112.7(2)	N4	C4	N5	106.2(2)
O5	N6	C4	115.5(3)	N4	C4	N6	102.7(2)
O6	N6	O5	127.1(3)	N5	C4	N6	109.7(2)
O6	N6	C4	117.4(2)	C2	C4	N4	112.9(2)
O11	N7	C5	114.9(2)	C2	C4	N5	111.9(2)
O12	N7	O11	127.7(3)	C2	C4	N6	112.8(2)
O12	N7	C5	117.3(2)	N1	C5	N7	112.3(2)
O13	N8	C5	115.6(2)	N1	C5	N8	112.2(2)
O14	N8	O13	128.5(3)	N1	C5	N9	107.92(19)
O14	N8	C5	115.8(2)	N7	C5	N8	112.0(2)
O15	N9	C5	115.3(2)	N7	C5	N9	106.52(18)
O16	N9	O15	129.3(3)	N8	C5	N9	105.37(19)
O16	N9	C5	115.4(2)				

Table S6 Torsion Angles for **1**

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	N3	C1	N2	-49.2(4)	O14	N8	C5	N7	-134.2(3)
O1	N3	C1	C2	136.5(3)	O14	N8	C5	N9	110.4(3)
O2	N3	C1	N2	127.6(3)	O15	N9	C5	N1	88.9(3)
O2	N3	C1	C2	-46.8(4)	O15	N9	C5	N7	-150.2(3)
O3	N4	C4	N5	177.8(3)	O15	N9	C5	N8	-31.1(3)
O3	N4	C4	N6	62.7(3)	O16	N9	C5	N1	-88.4(3)
O3	N4	C4	C2	-59.1(3)	O16	N9	C5	N7	32.4(3)
O4	N4	C4	N5	-0.5(3)	O16	N9	C5	N8	151.5(2)
O4	N4	C4	N6	-115.7(3)	N1	N2	C1	N3	-175.0(2)
O4	N4	C4	C2	122.5(3)	N1	N2	C1	C2	0.2(3)
O5	N6	C4	N4	47.7(4)	N2	N1	C3	N10	176.2(2)
O5	N6	C4	N5	-64.9(4)	N2	N1	C3	C2	-1.3(3)
O5	N6	C4	C2	169.6(3)	N2	N1	C5	N7	-117.7(2)
O6	N6	C4	N4	-131.1(3)	N2	N1	C5	N8	115.1(2)
O6	N6	C4	N5	116.3(3)	N2	N1	C5	N9	-0.6(3)
O6	N6	C4	C2	-9.2(4)	N2	C1	C2	C3	-0.9(3)
O7	N5	C4	N4	-94.1(3)	N2	C1	C2	C4	175.4(2)
O7	N5	C4	N6	16.2(4)	N3	C1	C2	C3	173.5(3)
O7	N5	C4	C2	142.2(3)	N3	C1	C2	C4	-10.1(5)
O8	N5	C4	N4	83.9(3)	C1	C2	C3	N1	1.3(3)
O8	N5	C4	N6	-165.8(3)	C1	C2	C3	N10	-176.0(2)
O8	N5	C4	C2	-39.8(3)	C1	C2	C4	N4	-3.0(4)
O9	N10	C3	N1	-164.6(3)	C1	C2	C4	N5	116.8(3)
O9	N10	C3	C2	12.3(4)	C1	C2	C4	N6	-118.9(3)
O10	N10	C3	N1	14.4(4)	C3	N1	N2	C1	0.6(3)
O10	N10	C3	C2	-168.7(3)	C3	N1	C5	N7	61.3(3)
O11	N7	C5	N1	6.0(3)	C3	N1	C5	N8	-65.9(3)

O11	N7	C5	N8	133.4(3)	C3	N1	C5	N9	178.4(2)
O11	N7	C5	N9	-112.0(3)	C3	C2	C4	N4	172.5(2)
O12	N7	C5	N1	-176.4(2)	C3	C2	C4	N5	-67.7(3)
O12	N7	C5	N8	-49.1(3)	C3	C2	C4	N6	56.6(3)
O12	N7	C5	N9	65.6(3)	C4	C2	C3	N1	-175.3(2)
O13	N8	C5	N1	176.4(3)	C4	C2	C3	N10	7.5(4)
O13	N8	C5	N7	48.9(3)	C5	N1	N2	C1	179.8(2)
O13	N8	C5	N9	-66.5(3)	C5	N1	C3	N10	-2.8(4)
O14	N8	C5	N1	-6.8(3)	C5	N1	C3	C2	179.7(2)

Table S7 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **2**

Atom	***x***	***y***	***z***	**U(eq)**
O8	2595.4(13)	8200(6)	4305.3(19)	84.1(10)
N1	3619.4(12)	7662(5)	4282.9(17)	58.6(8)
O7	3280.1(17)	11444(6)	4040(2)	98.8(12)
N2	4006.7(11)	7442(5)	4004.6(18)	57.0(8)
N5	2936.7(16)	4567(6)	2689(2)	71.8(10)
O10	2471.6(14)	4551(6)	2560(2)	103.0(13)
N4	2690.1(13)	6614(7)	4047(2)	64.3(9)
O4	3461.0(16)	6285(7)	5467.4(19)	96.6(11)
C3	3797.1(13)	6271(5)	3409.0(18)	44.7(8)
N3	3641.2(17)	7985(7)	5567(2)	75.8(11)
C2	3276.1(14)	5745(6)	3304.3(19)	49.7(9)
O9	2431.8(13)	5084(6)	3934(2)	104.2(13)
N9	4245.4(16)	3467(6)	2987(2)	76.9(11)
N10	3910.7(18)	6624(8)	2195(2)	83.0(12)
N7	3385.4(17)	10974(6)	4680(2)	80.5(11)
O16	4744.1(17)	8394(7)	3167(3)	113.7(14)
O12	4133(2)	2507(6)	3430(3)	136.0(19)
O5	3768(2)	8967(7)	6117(2)	123.5(16)
C1	3167.3(13)	6630(6)	3862(2)	48.7(9)
N8	4692.1(16)	6750(8)	3366(3)	76.9(11)
O11	3154.5(18)	3613(9)	2341(2)	141(2)
O13	4451.9(19)	2873(7)	2585(2)	120.6(15)
O14	4152(2)	6287(7)	1816(2)	119.9(15)
O6	3255(2)	11810(6)	5147(2)	127.4(17)
C5	3720.2(16)	9023(6)	4907(2)	59.1(10)
C4	4135.7(16)	5758(6)	2989(2)	58.0(10)

N6	4311(2)	9682(10)	5195(3)	94.1(14)
O17	5001.1(16)	5717(8)	3833(3)	126.8(16)
O2	4394(2)	11377(9)	5040(3)	145(2)
O1	4609.2(17)	8369(10)	5537(3)	142(2)
O15	3611(6)	8100(30)	2192(9)	111(4)
O15A	3448(4)	6990(30)	1940(6)	97(4)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Table S8 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for **2**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O8	72(2)	105(3)	90(2)	2(2)	47.6(18)	11.8(19)
N1	52.8(18)	66(2)	58.0(19)	-8.7(16)	20.7(16)	-0.2(15)
O7	134(3)	81(2)	86(2)	26(2)	46(2)	12(2)
N2	44.3(17)	67(2)	63(2)	-12.1(16)	22.7(15)	-4.6(15)
N5	66(2)	78(3)	61(2)	-9.1(19)	11.2(19)	-13.9(19)
O10	57(2)	113(3)	114(3)	-34(2)	1.6(18)	-17.4(19)
N4	44.1(19)	82(3)	65(2)	17.2(19)	18.0(16)	-0.7(19)
O4	118(3)	93(3)	74(2)	26(2)	29(2)	5(2)
C3	41.7(18)	47(2)	45.1(19)	-5.9(15)	15.6(15)	-1.7(15)
N3	89(3)	84(3)	52(2)	5(2)	22(2)	19(2)
C2	47(2)	50(2)	46(2)	0.3(16)	10.2(16)	-3.3(16)
O9	65(2)	109(3)	142(3)	9(3)	42(2)	-28(2)
N9	86(3)	70(3)	90(3)	-7(2)	51(2)	6(2)
N10	74(3)	105(3)	84(3)	17(2)	45(2)	4(2)
N7	105(3)	64(2)	76(3)	-1(2)	37(2)	10(2)
O16	120(3)	88(3)	157(4)	-20(3)	79(3)	-36(2)
O12	185(5)	83(3)	200(5)	35(3)	141(4)	34(3)
O5	181(4)	132(4)	57(2)	-13(2)	42(2)	15(3)
C1	38.6(18)	52(2)	53(2)	4.9(17)	12.8(16)	0.8(15)
N8	64(2)	86(3)	90(3)	-22(2)	39(2)	-3(2)
O11	106(3)	216(5)	104(3)	-96(4)	41(3)	-53(3)
O13	161(4)	111(3)	122(3)	-10(3)	90(3)	41(3)
O14	154(4)	143(4)	83(2)	9(2)	68(3)	6(3)
O6	190(5)	95(3)	102(3)	-15(2)	58(3)	48(3)
C5	61(2)	63(3)	53(2)	-5.2(19)	19.5(19)	1.9(19)
C4	61(2)	62(3)	56(2)	-5.5(19)	26.5(19)	-4.5(19)
N6	81(3)	121(4)	74(3)	-39(3)	20(2)	-21(3)

O17	74(2)	144(4)	134(4)	-9(3)	3(2)	10(3)
O2	148(4)	164(5)	130(4)	-49(4)	57(3)	-90(4)
O1	68(2)	190(5)	134(4)	-51(4)	-4(2)	17(3)
O15	92(7)	146(9)	105(8)	65(7)	46(6)	21(7)
O15A	89(6)	123(9)	81(6)	26(6)	34(5)	9(6)

The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Table S9 Bond Lengths for **2**

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O8	N4	1.217(5)	C2	C1	1.350(5)
N1	N2	1.346(4)	N9	O12	1.189(5)
N1	C1	1.376(5)	N9	O13	1.175(5)
N1	C5	1.448(5)	N9	C4	1.526(6)
O7	N7	1.210(5)	N10	O14	1.167(5)
N2	C3	1.331(4)	N10	C4	1.545(6)
N5	O10	1.188(5)	N10	O15	1.256(11)
N5	C2	1.439(5)	N10	O15A	1.190(9)
N5	O11	1.215(5)	N7	O6	1.213(5)
N4	O9	1.193(5)	N7	C5	1.532(6)
N4	C1	1.456(5)	O16	N8	1.167(5)
O4	N3	1.199(5)	N8	C4	1.555(6)
C3	C2	1.389(5)	N8	O17	1.195(6)
C3	C4	1.466(5)	C5	N6	1.549(6)
N3	O5	1.185(5)	N6	O2	1.188(7)
N3	C5	1.531(6)	N6	O1	1.199(7)

Table S10 Bond Angles for **2**

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N2	N1	C1	111.1(3)	O15A	N10	C4	117.7(6)
N2	N1	C5	117.6(3)	O7	N7	O6	128.9(4)
C1	N1	C5	131.1(3)	O7	N7	C5	113.7(4)
C3	N2	N1	105.2(3)	O6	N7	C5	117.3(4)
O10	N5	C2	119.2(4)	N1	C1	N4	122.0(3)
O10	N5	O11	124.3(4)	C2	C1	N1	106.5(3)
O11	N5	C2	116.4(4)	C2	C1	N4	131.5(3)
O8	N4	C1	115.5(4)	O16	N8	C4	115.9(5)
O9	N4	O8	126.4(4)	O16	N8	O17	129.6(5)
O9	N4	C1	118.0(4)	O17	N8	C4	114.5(4)
N2	C3	C2	111.1(3)	N1	C5	N3	112.9(3)
N2	C3	C4	117.4(3)	N1	C5	N7	110.9(3)
C2	C3	C4	131.5(3)	N1	C5	N6	109.4(3)
O4	N3	C5	116.4(4)	N3	C5	N7	111.3(3)
O5	N3	O4	128.0(5)	N3	C5	N6	104.9(4)
O5	N3	C5	115.6(5)	N7	C5	N6	107.3(4)
C3	C2	N5	124.2(3)	C3	C4	N9	112.7(3)
C1	C2	N5	129.6(3)	C3	C4	N10	111.8(3)
C1	C2	C3	106.1(3)	C3	C4	N8	109.4(3)
O12	N9	C4	114.5(4)	N9	C4	N10	111.3(4)
O13	N9	O12	127.8(5)	N9	C4	N8	104.7(3)
O13	N9	C4	117.6(4)	N10	C4	N8	106.5(3)
O14	N10	C4	116.9(4)	O2	N6	C5	115.4(6)
O14	N10	O15	130.3(6)	O2	N6	O1	131.1(6)
O14	N10	O15A	121.0(8)	O1	N6	C5	113.5(5)
O15	N10	C4	107.8(7)				

Table S11 Torsion Angles for **2**

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O8	N4	C1	N1	36.2(5)	O16	N8	C4	N9	148.7(4)
O8	N4	C1	C2	-144.1(4)	O16	N8	C4	N10	30.7(5)
N1	N2	C3	C2	0.2(4)	O12	N9	C4	C3	-13.8(6)
N1	N2	C3	C4	-179.7(3)	O12	N9	C4	N10	-140.3(5)
N1	C5	N6	O2	104.5(5)	O12	N9	C4	N8	105.0(5)
N1	C5	N6	O1	-75.2(5)	O5	N3	C5	N1	172.9(4)
O7	N7	C5	N1	-28.6(5)	O5	N3	C5	N7	-61.7(5)
O7	N7	C5	N3	-155.1(4)	O5	N3	C5	N6	54.0(5)
O7	N7	C5	N6	90.7(5)	C1	N1	N2	C3	0.0(4)
N2	N1	C1	N4	179.6(3)	C1	N1	C5	N3	61.1(5)
N2	N1	C1	C2	-0.1(4)	C1	N1	C5	N7	-64.6(5)
N2	N1	C5	N3	-125.2(4)	C1	N1	C5	N6	177.4(4)
N2	N1	C5	N7	109.1(4)	O11	N5	C2	C3	17.0(6)
N2	N1	C5	N6	-8.9(5)	O11	N5	C2	C1	-166.4(5)
N2	C3	C2	N5	177.1(4)	O13	N9	C4	C3	170.0(4)
N2	C3	C2	C1	-0.2(4)	O13	N9	C4	N10	43.5(6)
N2	C3	C4	N9	116.6(4)	O13	N9	C4	N8	-71.3(5)
N2	C3	C4	N10	-117.2(4)	O14	N10	C4	C3	-179.7(4)
N2	C3	C4	N8	0.5(5)	O14	N10	C4	N9	-52.7(6)
N5	C2	C1	N1	-176.9(4)	O14	N10	C4	N8	60.9(6)
N5	C2	C1	N4	3.4(7)	O6	N7	C5	N1	150.2(5)
O10	N5	C2	C3	-164.4(4)	O6	N7	C5	N3	23.7(6)
O10	N5	C2	C1	12.2(7)	O6	N7	C5	N6	-90.5(5)
O4	N3	C5	N1	-6.9(5)	C5	N1	N2	C3	-174.9(3)
O4	N3	C5	N7	118.5(4)	C5	N1	C1	N4	-6.4(6)
O4	N3	C5	N6	-125.8(4)	C5	N1	C1	C2	173.9(4)
C3	C2	C1	N1	0.2(4)	C4	C3	C2	N5	-3.1(6)

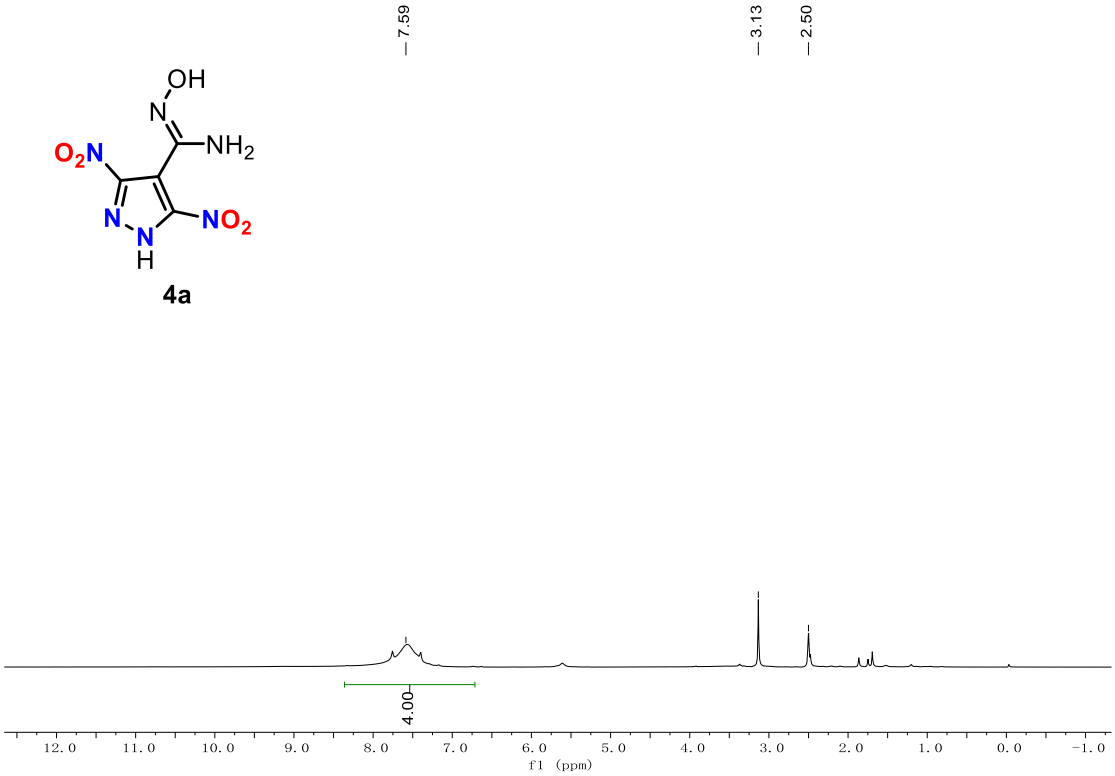
C3	C2	C1	N4	-179.5(4)	C4	C3	C2	C1	179.6(4)
N3	C5	N6	O2	-134.2(5)	O17	N8	C4	C3	88.3(5)
N3	C5	N6	O1	46.0(5)	O17	N8	C4	N9	-32.7(5)
C2	C3	C4	N9	-63.2(5)	O17	N8	C4	N10	-150.7(4)
C2	C3	C4	N10	63.0(5)	O15	N10	C4	C3	22.6(11)
C2	C3	C4	N8	-179.3(4)	O15	N10	C4	N9	149.6(10)
O9	N4	C1	N1	-144.0(4)	O15	N10	C4	N8	-96.8(10)
O9	N4	C1	C2	35.8(6)	O15A	N10	C4	C3	-22.9(12)
N7	C5	N6	O2	-15.7(6)	O15A	N10	C4	N9	104.1(12)
N7	C5	N6	O1	164.5(4)	O15A	N10	C4	N8	-142.3(11)
O16	N8	C4	C3	-90.3(5)					

Table S12 Atomic Occupancy for 2

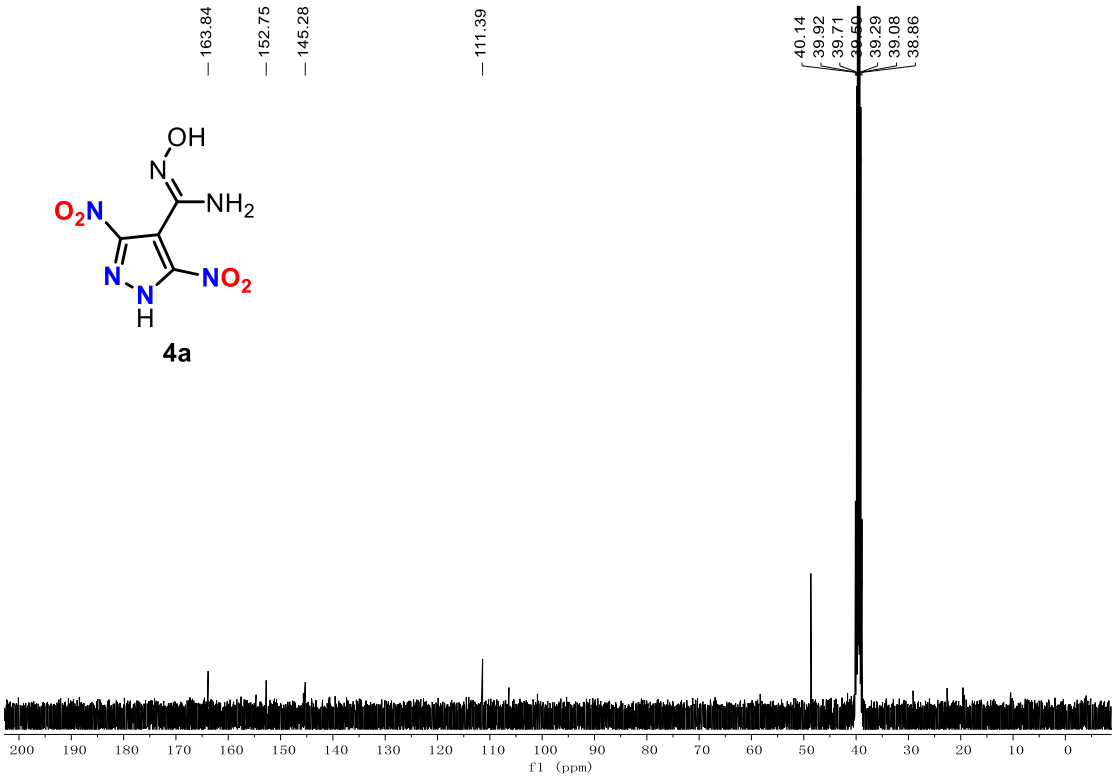
Atom	Occupancy	Atom	Occupancy
O15	0.51(2)	O15A	0.49(2)

5. NMR Spectra

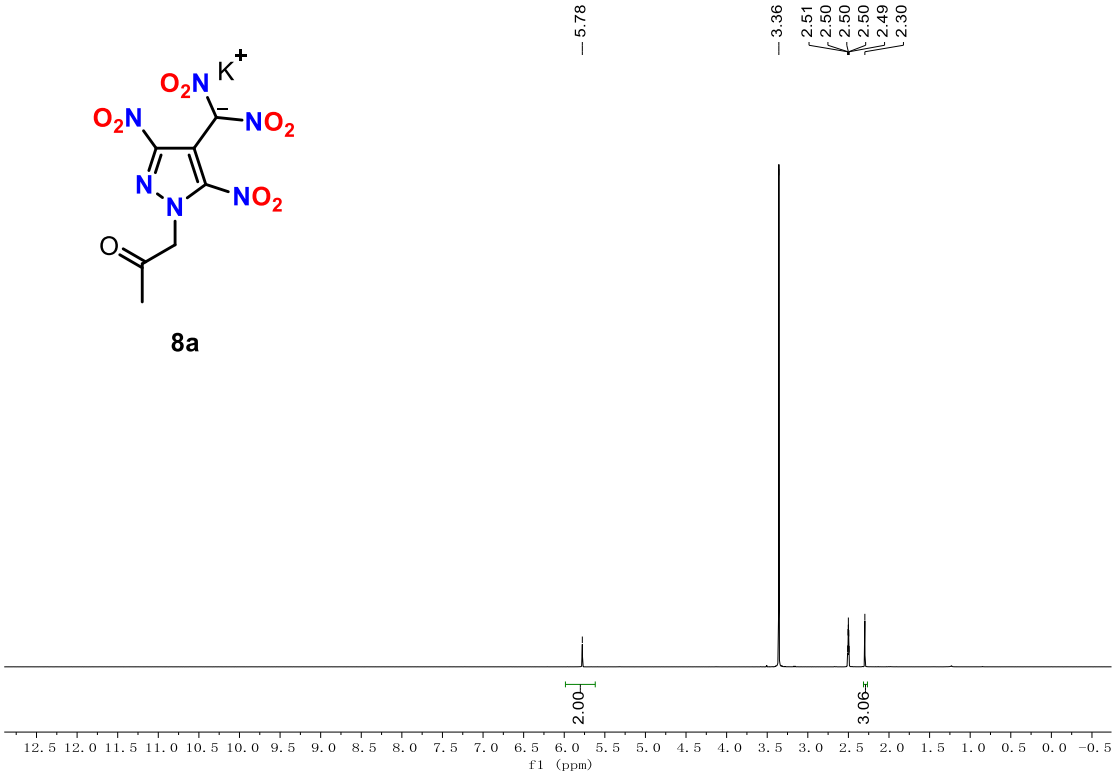
^{1}H NMR spectrum of compound **4a** (400 MHz, DMSO-d_6)



^{13}C NMR spectrum of compound **4a** (100 MHz, DMSO-d_6)

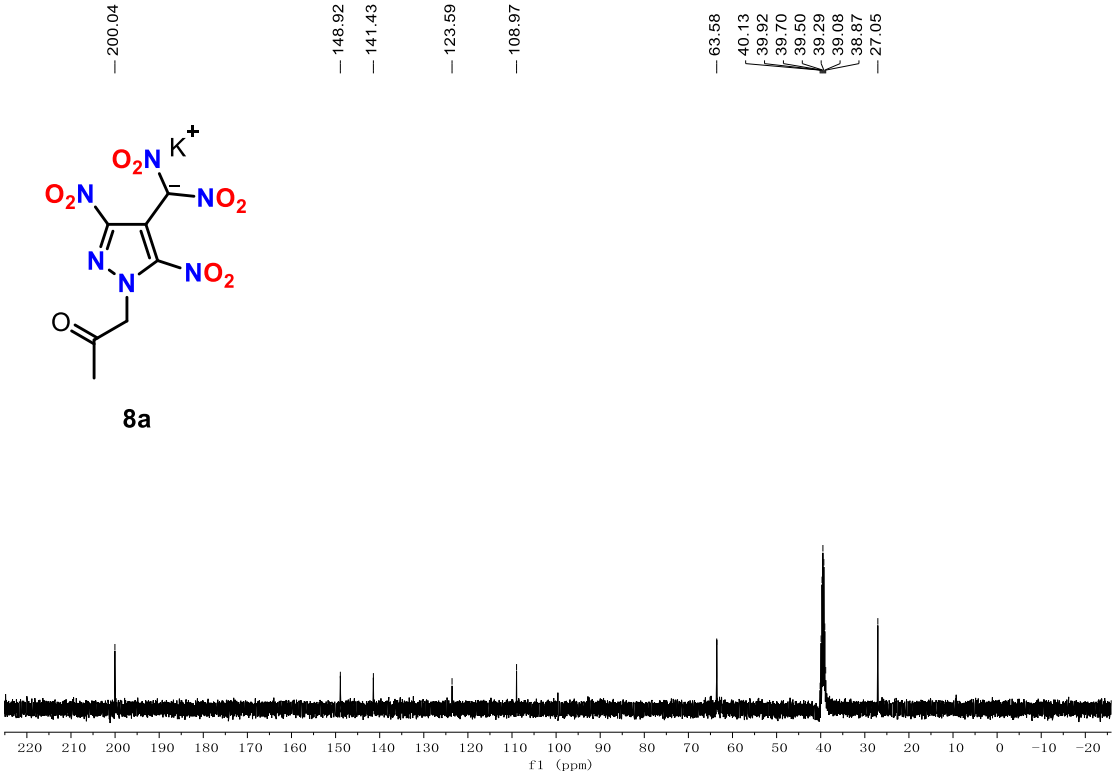


^{1}H NMR spectrum of compound **8a** (400 MHz, DMSO-d_6)



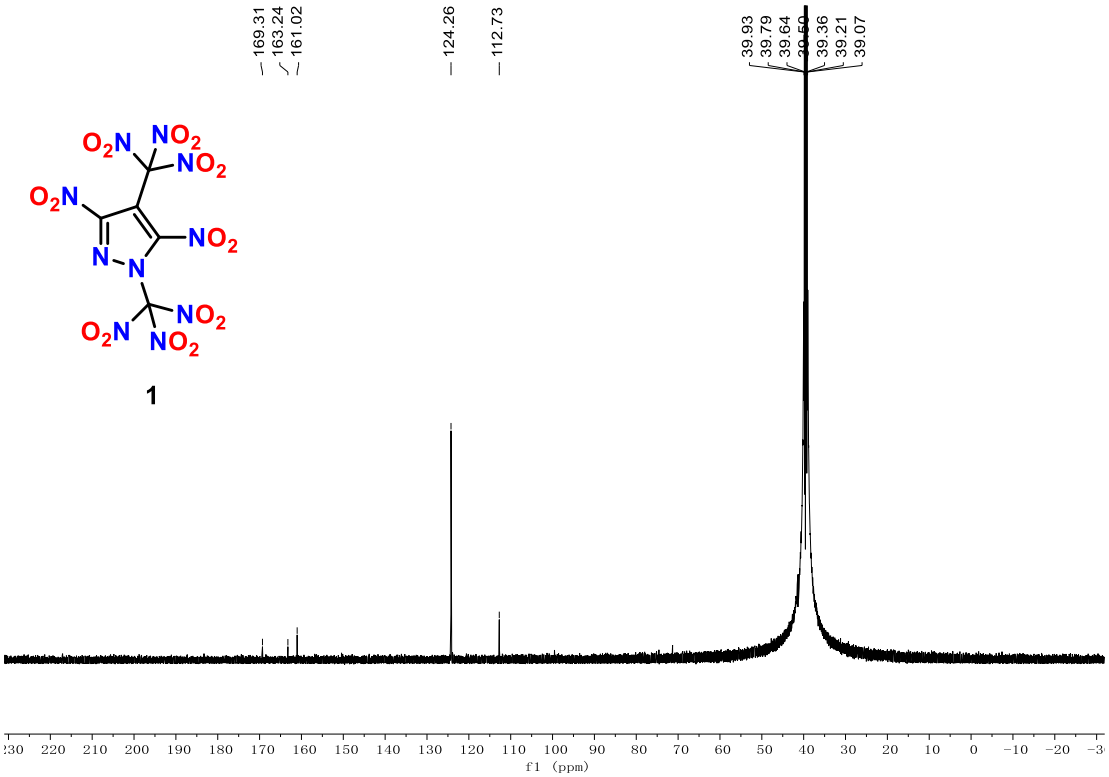
8a

^{13}C NMR spectrum of compound **8a** (100 MHz, DMSO-d_6)

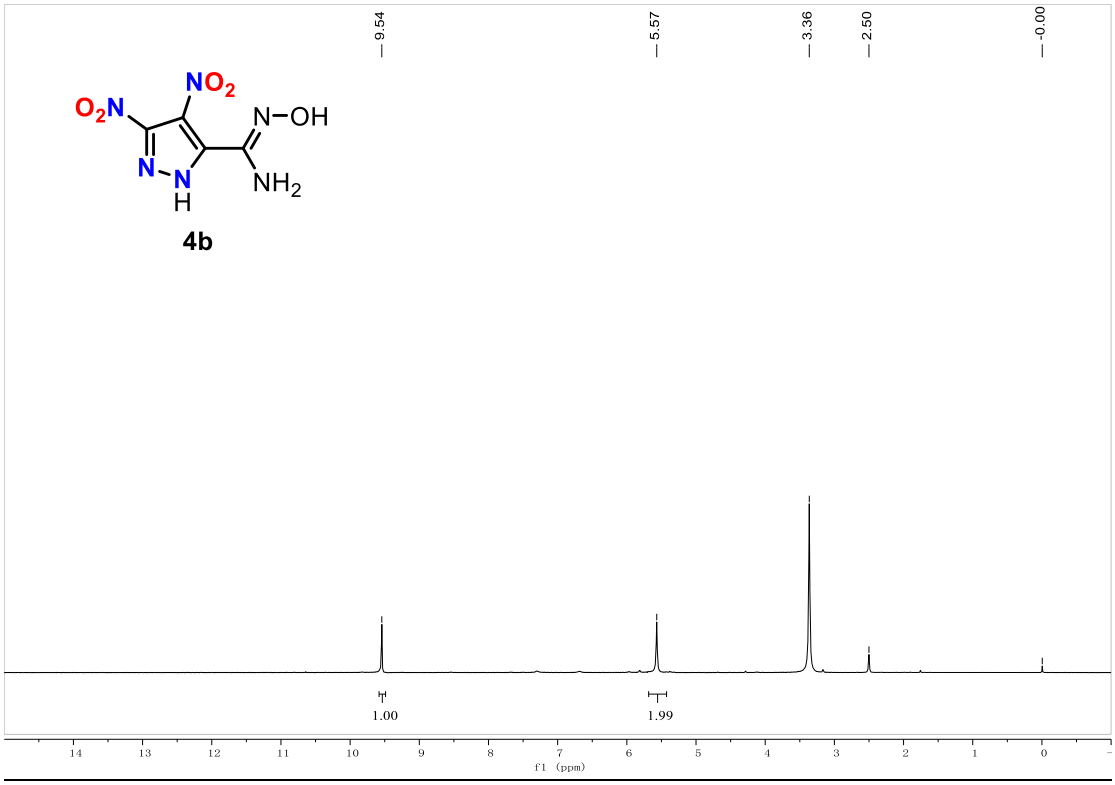


8a

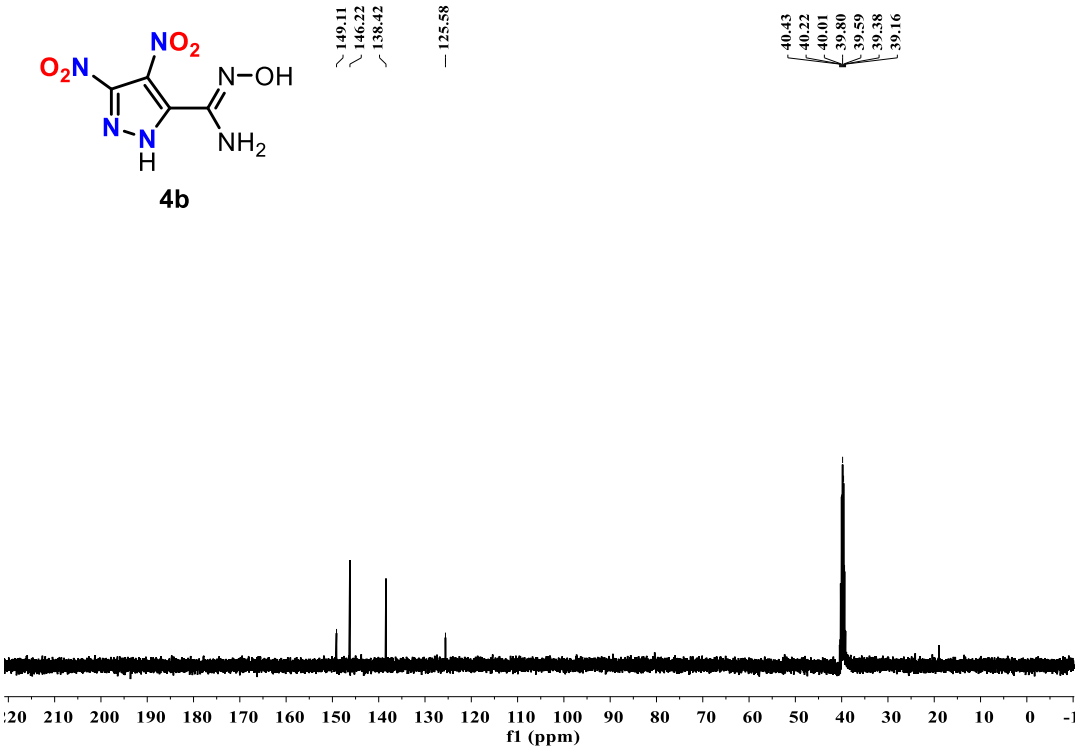
^{13}C NMR spectrum of compound **1** (150 MHz, DMSO-d_6)



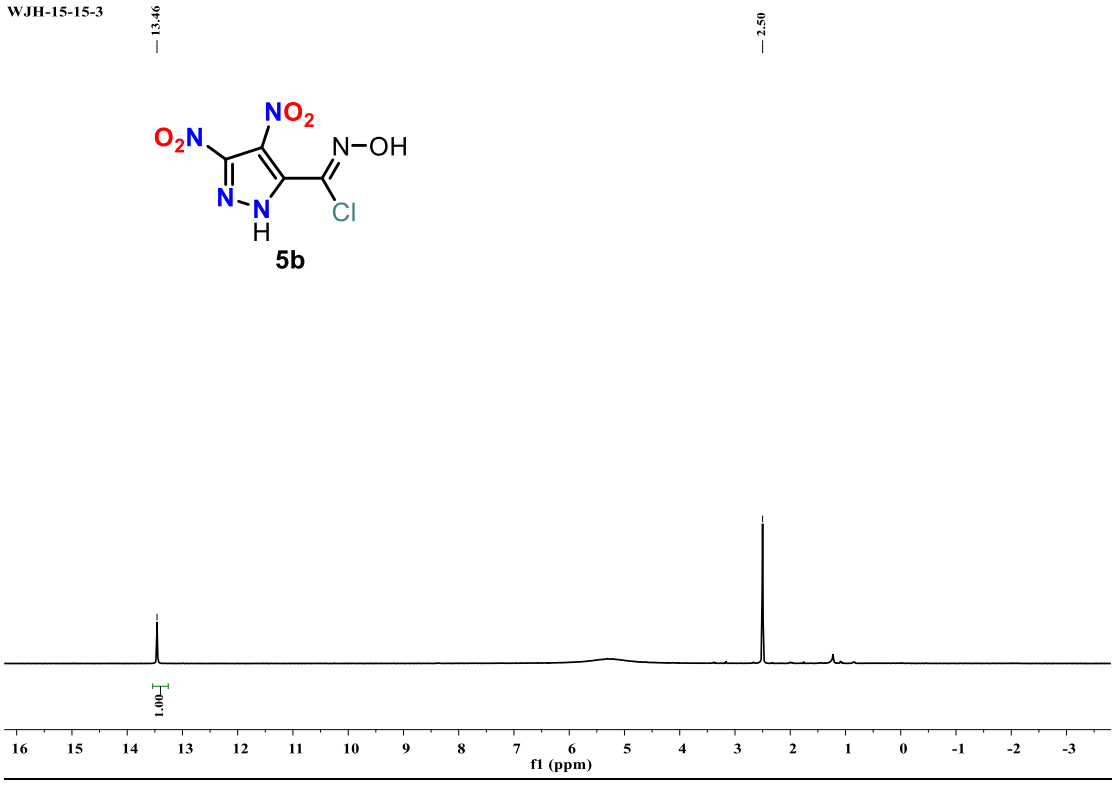
^{1}H NMR spectrum of compound **4b** (400 MHz, DMSO-d_6)



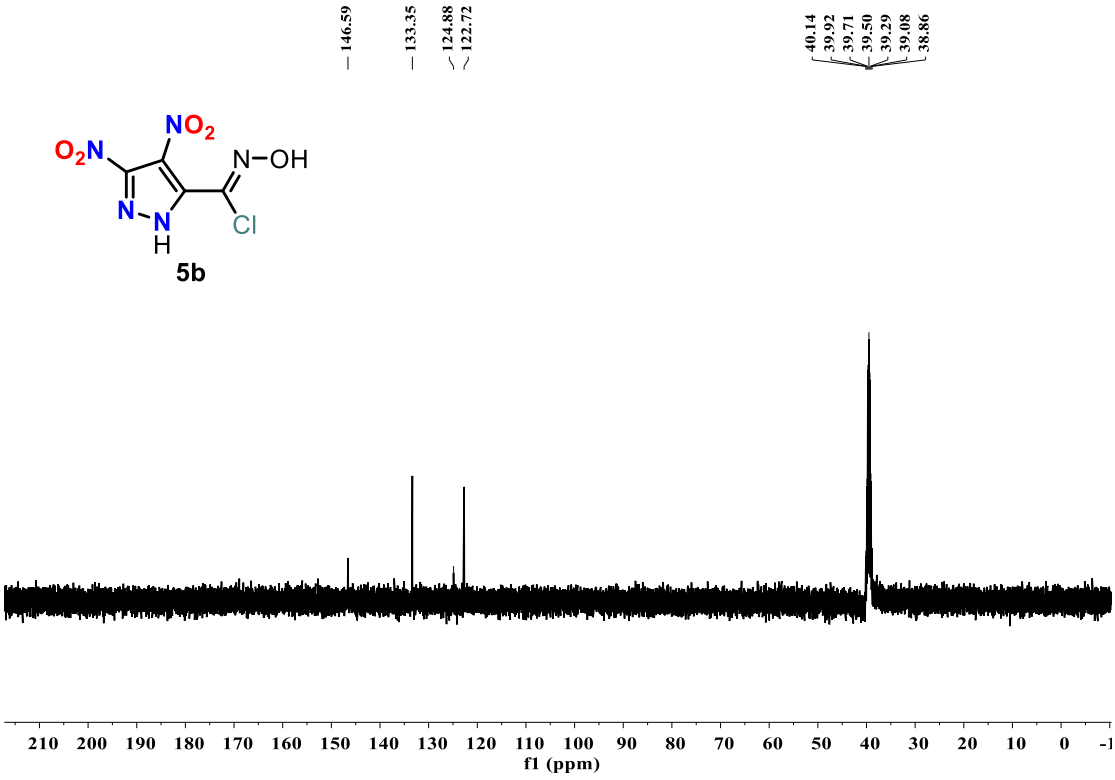
^{13}C NMR spectrum of compound **4b** (100 MHz, DMSO-d_6)



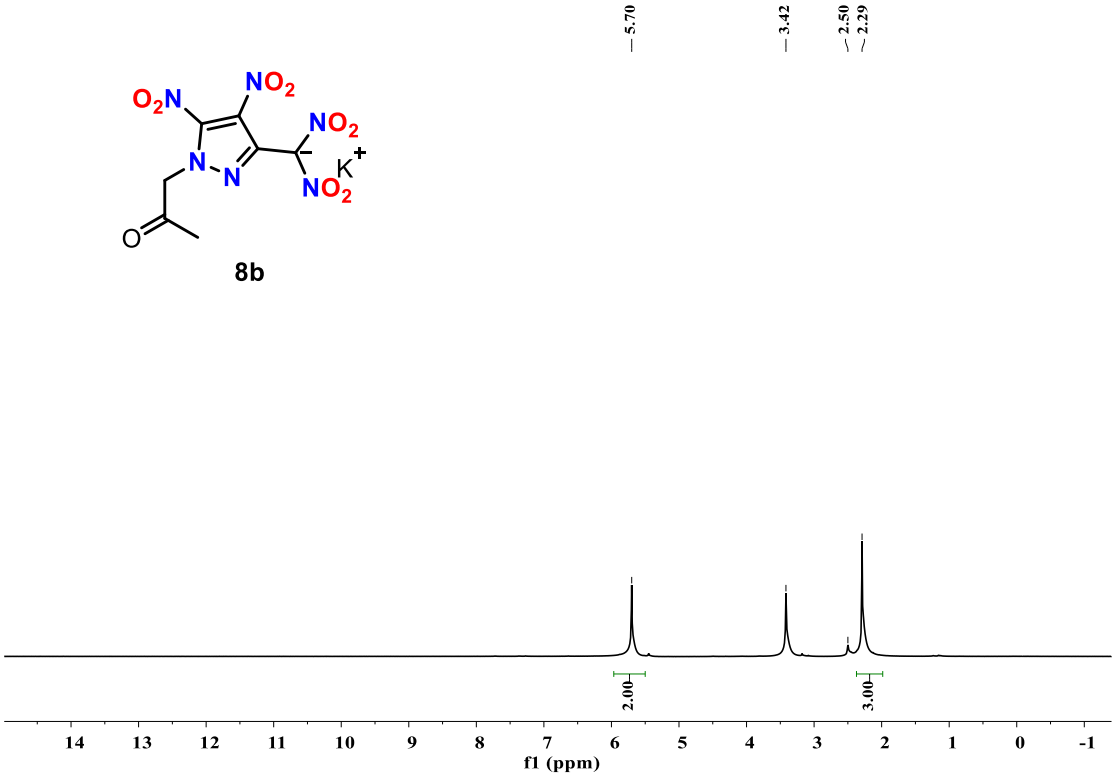
^{1}H NMR spectrum of compound **5b** (400 MHz, DMSO-d_6)



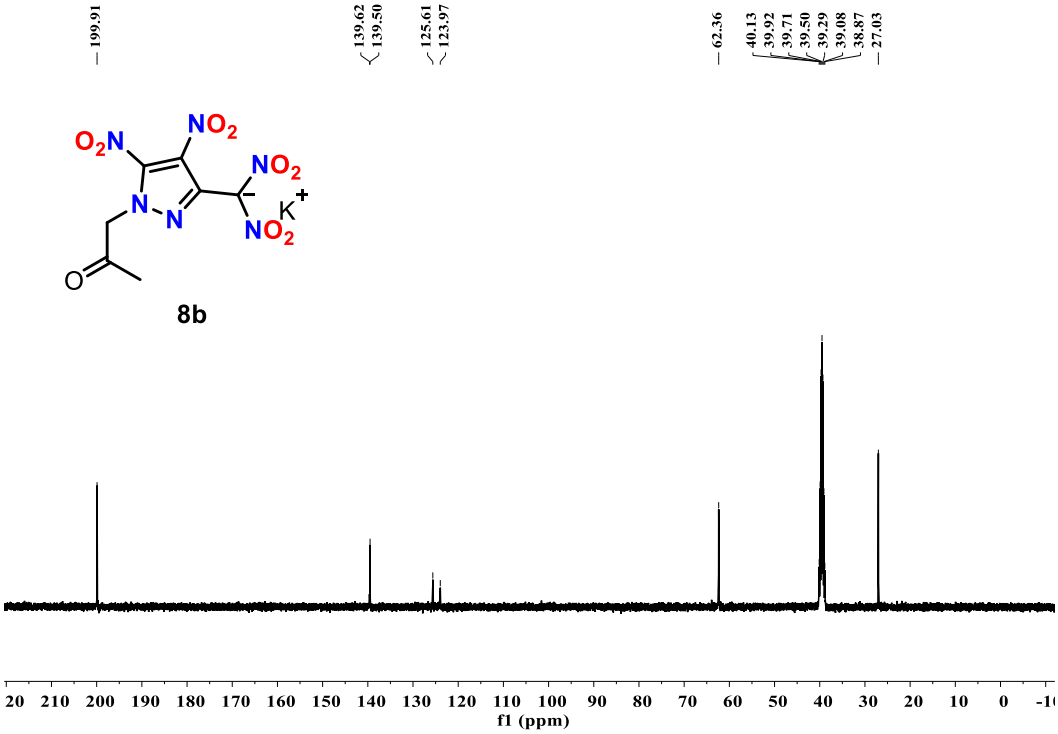
^{13}C NMR spectrum of compound **5b** (100 MHz, DMSO-d_6)



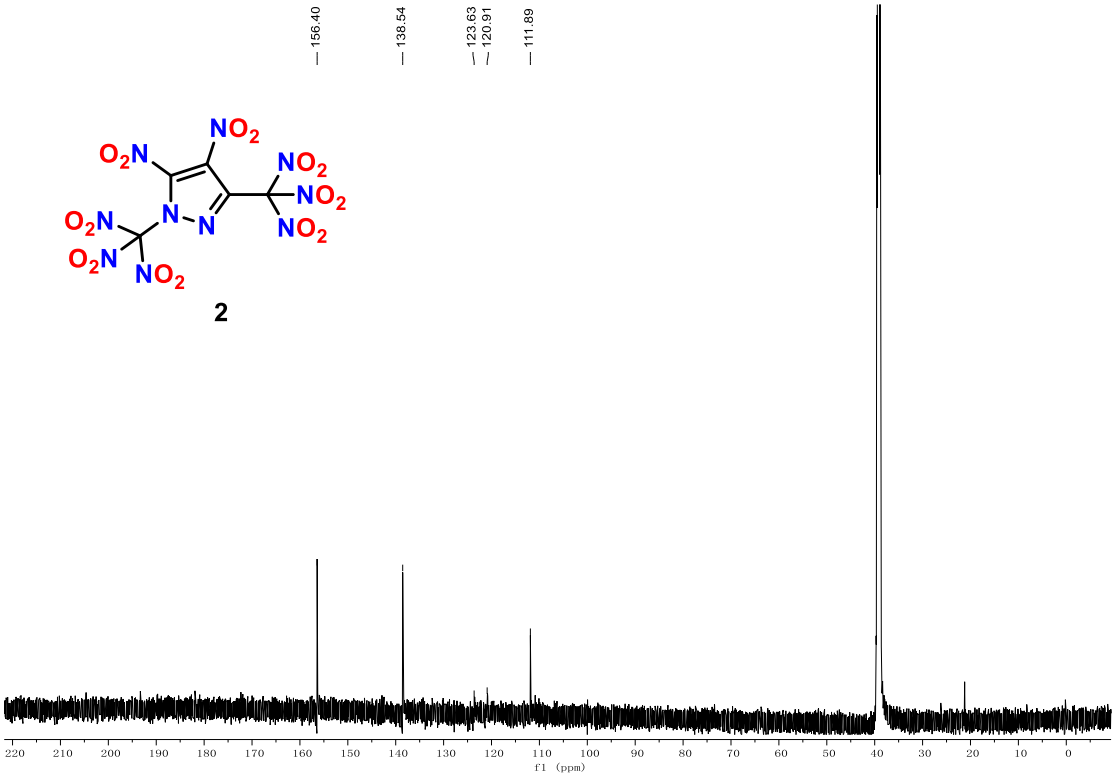
^{1}H NMR spectrum of compound **8b** (400 MHz, DMSO-d_6)



^{13}C NMR spectrum of compound **8b** (100 MHz, DMSO-d_6)



^{13}C NMR spectrum of compound **2** (150 MHz, DMSO-d_6)



6. References

1 M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, J. J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski and D. J. Fox, *Gaussian 09, Revision E.01*, Gaussian, Inc., Wallingford, CT, 2013.

2 A. D. Becke, *J. Chem. Phys.*, 1993, **98**, 5648–5652.

3 P. J. Stephens, F. J. Devlin, C. F. Chabalowski and M. J. Frisch, *J. Phys. Chem.*, 1994, **98**, 11623–11627.

4 P. C. Hariharan and J. A. Pople, *Theor. Chim. Acta*, 1973, **28**, 213–222.

5 M. W. Schmidt, M. S. Gordon and J. A. Boatz, *J. Phys. Chem. A*, 2005, **109**, 7285–7295.

6 H. Gao, C. Ye, C. M. Piekarski and J. M. Shreeve, *J. Phys. Chem. C*, 2007, **111**, 10718–10731.

7 M. Jaidann, S. Roy, H. Abou-Rachid and L.-S. Lussier, *J. Hazard. Mater.*, 2010, **176**, 165–173.

8 J. A. Pople, J. S. Binkley and R. Seeger, *Int. J. Quantum Chem.*, 1976, **10**, 1–19.

9 B. Chan, J. Deng and L. Radom, *J. Chem. Theory Comput.*, 2011, **7**, 112–120.

10 W. Xie, X. Jiang, M. Sun, X. Zhang, P. Yin and Q. Lai, *Org. Lett.*, 2024, **26**, 6591–6596.

11 V. Thaltiri, R. J. Staples and J. M. Shreeve, *J. Mater. Chem. A*, 2024, **12**, 16729–16734.

12 G.M. Sheldrick, *Acta Crystallogr A Found Adv.* 2015, **71**, 3–8.

13 C.B. Hübschle, G.M. Sheldrick, B. Dittrich,*J. Appl. Crystallogr.* 2011, **44,** 128 1–1284.

14 O.V. Dolomanov, L.J. Bourhis, R.J. Gildea, J.A.K. Howard, H. Puschmann, *J. Appl. Crystallogr.* **2009**, 42, 339–341.