Electronic Supplementary Information

A new oxygen-rich energetic salt dihydrazine tetranitroethide: a promising explosive alternative with high density and good performance

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1. Experimental procedure

Safety precautions: Dipotassium tetranitroethide is sensitive to friction and must be manipulated using appropriate safety precautions. Although safe in the preparation and handling of these compounds, the mechanical action of these energetic materials, including scratching or scraping, must be avoided.

General Methods: The chemical reagents and solvents were obtained from Sinopharm Chemical Reagent Co., Ltd. Elemental analysis (C, H, N) was performed with an Elementar Vario El III analyzer. Infrared spectra were recorded on a Bruker Equinox 55 spectrometer. The structure of compound (5) was solved by using direct methods and successive Fourier difference syntheses (SHELXS-97)[1], and were refined by using full-matrix least-squares on F2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97)[2]. All non-hydrogen atoms were obtained from the difference Fourier map and refined anisotropically. Hydrogen atoms were generated geometrically, assigned appropriated isotropic thermal parameters, and included in structure factor calculations. Impact and friction sensitivities were performed on a BAM fall hammer BFH-10 and a BAM friction apparatus FSKM-10, respectively. ^{1}H and ^{13}C NMR spectra were recorded using a Bruker AVANCE Ⅲ instrument, and using D_2O as solvent and locking solvent.

Dipotassium tetranitroethide(3)

Tetraiodoethylene powder (20.0 g, 0.04 mol) was added to 50 ml of 96% HNO_3 in -40 ℃, with stirring. Then the solution heated quickly to 70 ℃. After 20 min, the mixture was poured onto 200 g of ice, and extracted with dichloromethane (100 mL). The solution was washed with $NaHSO_3$ solution (50 mL), brine (100 mL) and dried ($MgSO_4$). Removal of solvent gave 1,1-diiododinitroethylene(2), 8 g (50%) of a yellow solid. The solution of 1,1-diiododinitroethylene (8.0 g) in 50 mL of 60% aqueous methanol was added to a solution of KNO_2 (8.0 g) in 50mL of 60% aqueous methanol. After 18h, the product,5.5 g (90%), was gave by filtered and washed with methanol. Dipotassium tetranitroethide is a bright yellow solid, mp 280 ℃

2. Thermal Analysis

The thermal decomposition behavior of the salt was tested using differential scanning calorimetry (DSC) and thermogravimetric analysis (TG) technologies at heating rate of 5 ℃ min^{-1}, under N_2 gas.

DSC plot:

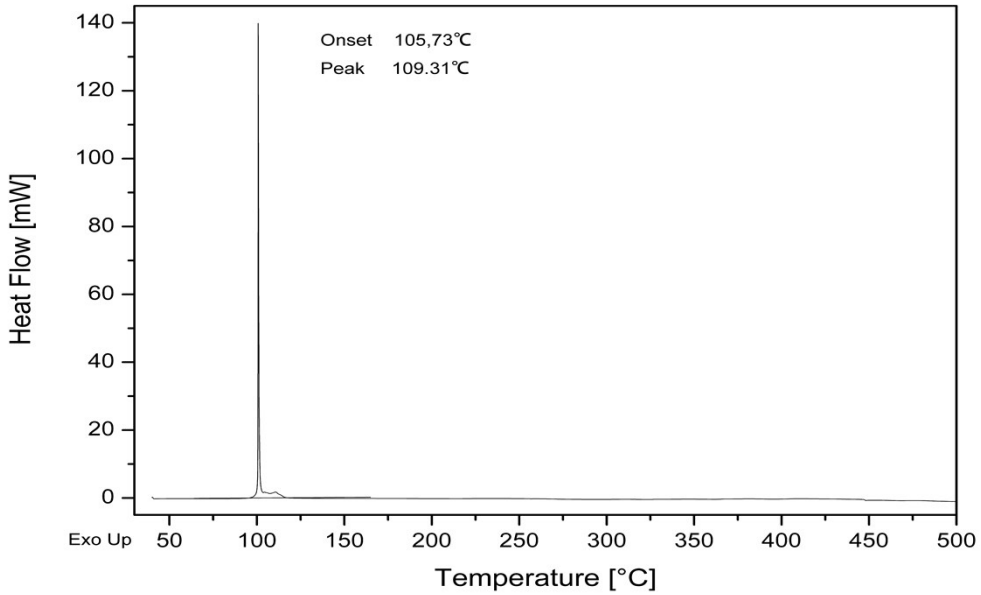

Fig S1a The DSC plot of compound 5

TG plot:

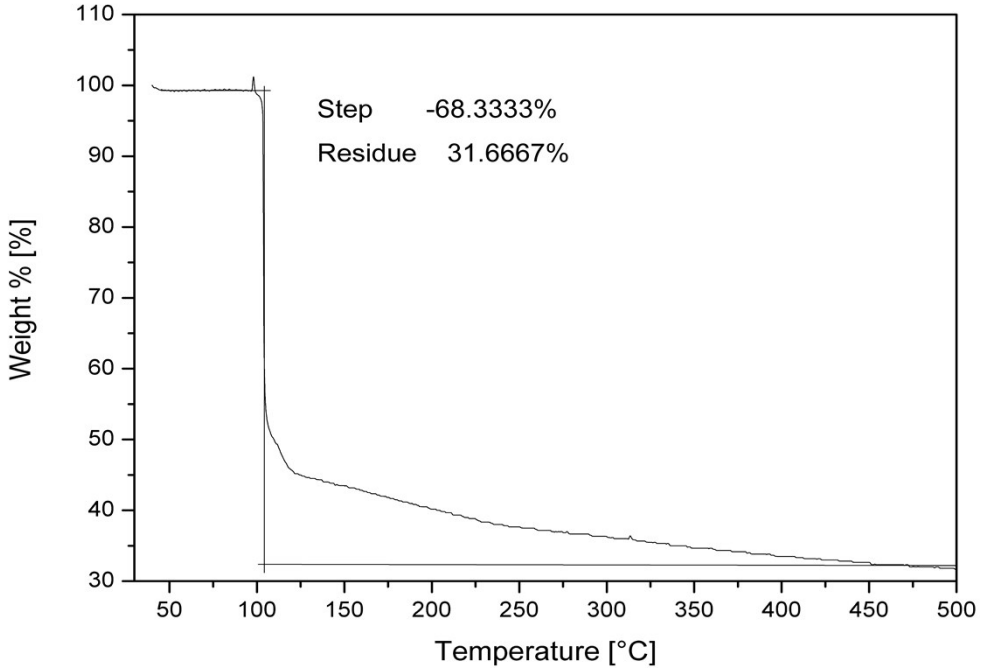

Fig S1b The TG plot of compound 5

3. NMR Spectra

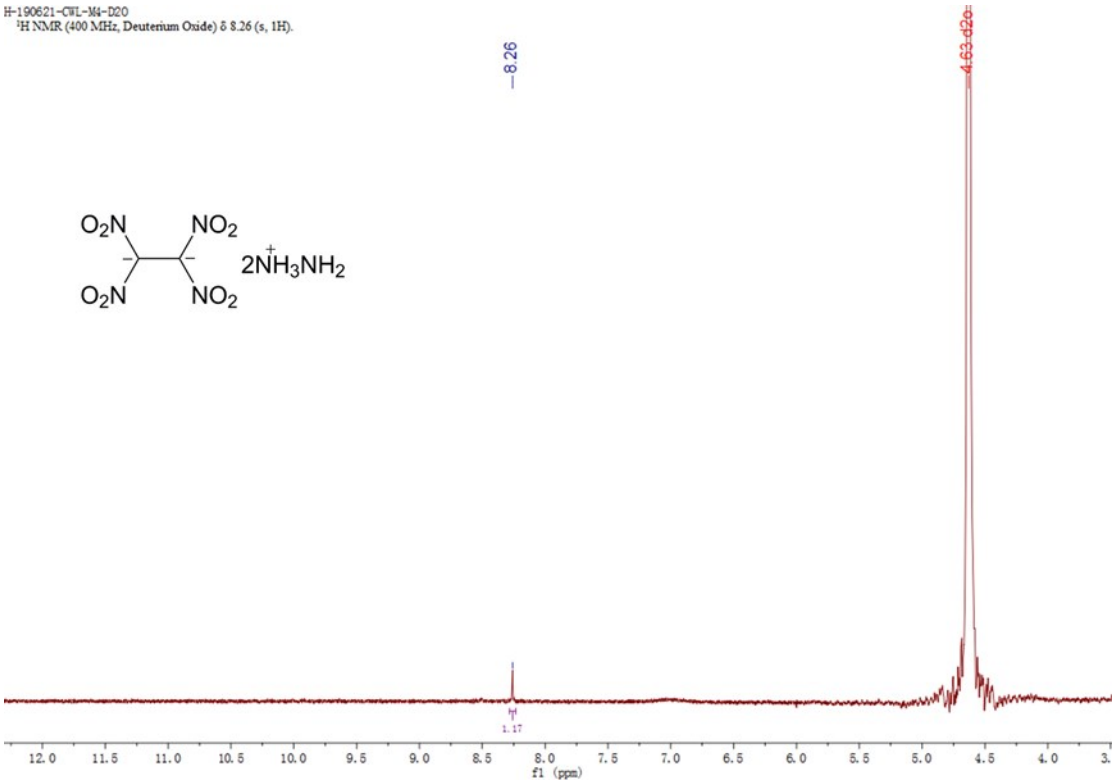

Fig S2a The ^{1}H-NMR of compound 5

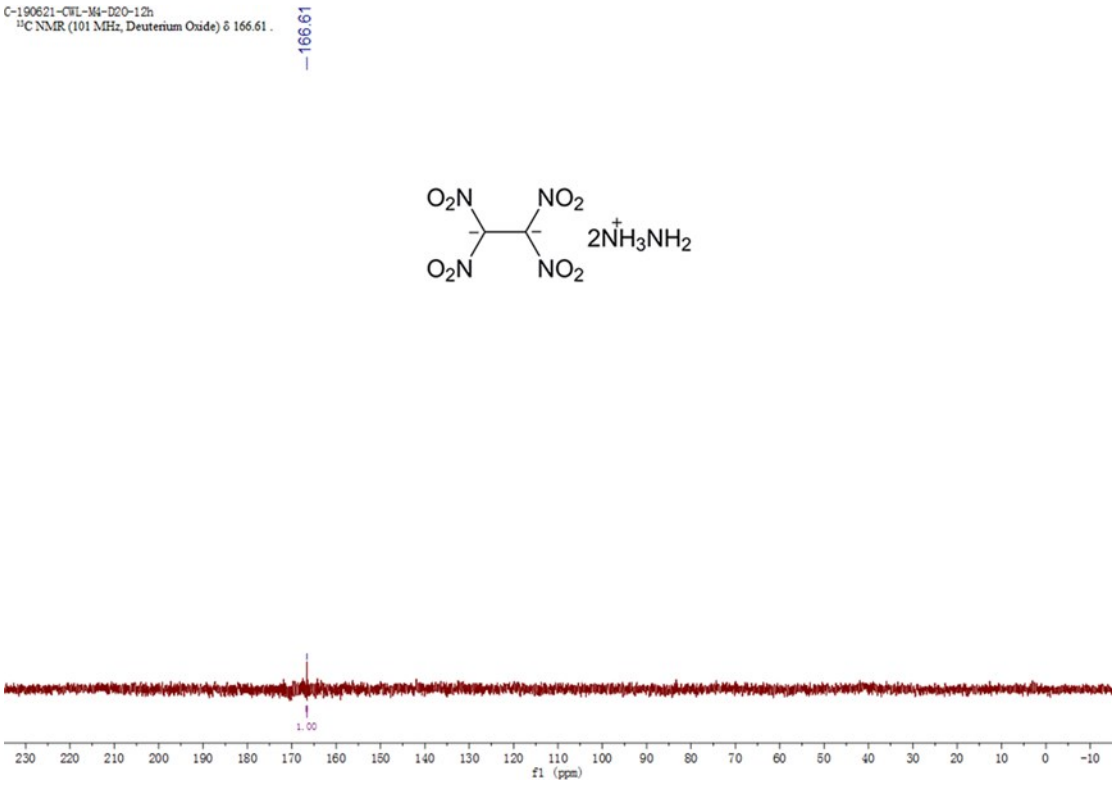

Fig S2b The ^{13}C-NMR of compound 5

4. Crystal Structure Analysis

Table S1 Crystallographic data and structure determination details for 5

Compound	5
Chemical formula	$C_2H_{10}N_8O_8$
Formula weight	274.18
Crystal size [mm3]	0.24×0.21×0.2
Temperature [K]	153(2)
Crystal system	monoclinic
Space group	C2/c
a [Å]	16.946(3)
b [Å]	9.3354(19)
c [Å]	14.414(3)
α [°]	90
β [°]	120.28(3)
γ [°]	90
Volume [Å]^3	1969.1(9)
Z	8
ρ [g·cm-3]	1.85
F(000)	1136
μ [mm-1]	0.181
θ range (°)	2.588-27.473
Reflections collected	5853 (R_{int}=0.0394)
Independent reflections	2248
Data/restraints/parameters	2248/0/163
Index ranges	-21≤h≤18, -10≤k≤12, -13≤l≤18
Final R indexes [I>2σ(I)]	R_1=0.0511, wR_2=0.1236
Final R indexes (all data)	R_1=0.0595, wR_2=0.1176
CCDC number	1946324

Table S2a Bond lengths for compound 5

Bond length (Å)							
C1-C1	1.433(4)	N3-C2	1.392(2)	O1-N1	1.247(2)	O5-N3	1.256(2)
C2-C2	1.431(4)	N4-C2	1.376(3)	O2-N1	1.254(2)	O6-N3	1.240(2)
N1-C1	1.385(2)	N5-N6	1.446(3)	O3-N2	1.270(2)	O7-N4	1.257(2)
N2-C1	1.376(3)	N7-N8	1.423(3)	O4-N2	1.243(2)	O8-N4	1.261(2)

Table S2b Bond angles for compound 5

Bond angle(°)					
N1-C1-C1	118.28(18)	O1-N1-O2	120.62(16)	O6-N3-O5	120.59(17)
N2-C1-C1	119.53(18)	O2-N1-C1	115.72(16)	O6-N3-C2	124.16(17)
N2-C1-N1	121.89(17)	O3-N2-C1	115.70(16)	O7-N4-C2	122.93(17)
N3-C2-C2	118.03(17)	O4-N2-C1	123.98(16)	O7-N4-O8	119.84(16)
N4-C2-N3	121.55(17)	O4-N2-O3	120.29(17)	O8-N4-C2	117.22(17)
O1-N1-C1	123.65(17)	O5-N3-C2	115.25(17)		

References

1. G. M. Sheldrick, SHELXS-97, Program for the Solution of Crystal Structure, University of Göttingen, Göttingen (Germany), 1997.

2. G. M. Sheldrick, SHELXL-97, Program for Crystal Structure Refinement from Diffraction Data, University of Göttingen, Göttingen (Germany), 1997.