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Research paper

Preparation and Characterization of a Novel Coordination Compound [K(18-crown-6)][N(NO₂)₂]

Zhimeng Lu¹, Xiaoting Ren^{2,*}, Jinxuan He²

¹ South China University of Technology Key Laboratory of Polymer Processing Engineering, Ministry of Education, Hubei Institute of Aerospace Chemotechnology, Guangzhou, Guangdong, 510640, China

² Science and Technology on Aerospace Chemical Power Laboratory, Hubei Institute of Aerospace Chemotechnology, Xiangyang, Hubei, 441003, China

* E-mail: xiaot_ren@126.com

Abstract: Low-frequency resonance technology is a promising technology for mixing or manufacturing energetic materials, and has been a hot topic in the field of material preparation. In this paper, we describe the first use of low-frequency resonance technology for the synthesis of new compounds and for performing innovative research. The coordination compound, [K(18-crown-6)][N(NO₂)₂]₂ ([K(18C6)][N(NO₂)₂]₂), was first prepared by using a conventional slow solvent evaporation method over 10 days. The crystal structure of this compound was characterized by single crystal X-ray diffraction (SXD) and revealed that [K(18C6)][N(NO₂)₂] belongs to the monoclinic system, in which the space group is P2₁/c. The main intermolecular interactions in [K(18C6)][N(NO₂)₂] are the K–O coordination bonds and C–H···O hydrogen bonds. The crystallographic parameters are $a = 8.5032(2)$ Å, $b = 14.4060(4)$ Å, $c = 8.1708(2)$ Å, $\beta = 101.896(2)$, $V = 979.40(4)$ Å³, $Z = 2$, $\rho = 1.388$ g·cm⁻³. The coordination compound [K(18C6)][N(NO₂)₂] was then prepared by low-frequency resonance technology in only 65 min. The results of X-ray powder diffraction (PXRD), differential scanning calorimetry (DSC) and infrared spectroscopy (FTIR) showed that low-frequency resonance technology can successfully and efficiently prepare [K(18C6)]

[N(NO₂)₂]. Compared to potassium dinitramide (KDN), [K(18C6)][N(NO₂)₂] has unique characteristics.

Keywords: low-frequency resonance technology, [K(18C6)][N(NO₂)₂], coordination compound structure

Supporting Information (SI) is available at:

<https://ipo.lukasiewicz.gov.pl/wydawnictwa/cejem-woluminy/vol-18-nr-2/>

1 Introduction

Low-frequency resonance technology has the characteristics of high resonance energy efficiency and phase invariance, which can accelerate the interaction between molecular-level sized species, and which directly couples the energy source to the molecular material being affected. By directly coupling the molecular materials being mixed to the low-frequency acoustic source, the application becomes significantly more efficient and intense. In recent years, low-frequency resonance technology has been widely used in fields such as co-crystal preparation, material mixing, and material grinding [1-5], and this technology will lead to new innovative attempts to develop new compounds. A novel compound refers to a compound that is newly emerging or is under research and has excellent properties and special functions that traditional compounds do not have [6]. A new compound can potentially improve the formulation density, thermal stability, hygroscopicity, solubility, and response to stimuli, *etc.* [7-13]. Potassium dinitramide (KDN) [14-16] is a halogen-free, environmentally friendly oxidant with a density of 2.17 g·cm⁻³ and an enthalpy of formation of -250 kJ·mol⁻¹. It can be used as a phase stabilizer for ammonium nitrate and as an energetic pyrotechnic or smoke suppressant instead of potassium nitrate. It can also be used as an intermediate for the preparation of ammonium dinitramide or other dinitramide salts.

However, the hygroscopicity of KDN severely limits its wide application in the field of energetic materials. In order to reduce the strong hygroscopicity of KDN, it may be modified at the molecular level by coordination technology. This is expected to relax the stringent requirements of KDN in storage and use, and to provide a new method for the modification of highly hygroscopic energetic compounds.

In the present study, the coordination compound $[K(18\text{-crown-}6)] [N(\text{NO}_2)_2]$ ($[K(18\text{C}6)][N(\text{NO}_2)_2]$) was prepared by a conventional slow solvent evaporation method, and its crystal structure was determined by X-ray single crystal diffractometry. At the same time, in order to overcome the problems of a long preparation period, poor repeatability, low success rate, low synthesis efficiency, and difficulty in achieving scale-up, a low-frequency resonance technique [17] with a short preparation period, high safety and efficiency, and a simple and stable process was used to prepare $[K(18\text{C}6)][N(\text{NO}_2)_2]$. The formation of the coordination compound was established. By combining $[N(\text{NO}_2)_2]^-$ from KDN through coordination technology, it was anticipated that new compounds with low hygroscopicity relative to KDN could be obtained.

2 Materials and Test Methods

The following materials were used:

- KDN, purity 99.30% (Hubei Institute of Aerospace Chemotechnology) [18, 19],
- [18-crown-6] (18C6), analytical purity (Sinopharm Chemical Reagent Co., Ltd),
- anhydrous methanol, analytical purity (Shanghai Zhenxing Chemical Factory No. 1).

The following instruments were used:

- Ultra High Efficient Mixer (Hubei Hangpeng Chemical Power Technology Co., Ltd) model: Lab, mixing capacity 1 kg, maximum acceleration 980 m/s^2 , rated power 0.45 kW, acceleration sensor range 1626.8 m/s^2 ,
- Single crystal X-ray diffractometer (Germany Bruker AXS company), model: SMART-APEX2, CCD face inspection,
- Fourier Transform Infrared Spectrometer (Germany Bruker company), model: EQUINOX 55, KBr compressed discs, wavelength range $4000\sim 400 \text{ cm}^{-1}$, resolution 4 cm^{-1} ,
- high pressure hot re-scanning calorimeter (American TA company), with method 502.1 of GJB772A-1997 [20], in a nitrogen atmosphere, heating rate $10 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$, gas flow rate $40 \text{ mL}\cdot\text{min}^{-1}$, temperature range of $10\sim 600 \text{ }^\circ\text{C}$, open aluminum crucible, sample approx. 0.7 mg,
- X-ray powder diffractometer (Germany Bruker company), model: D8 ADVANCE, range $5\sim 50^\circ$, scanning speed $10^\circ/\text{min}$, step size 0.02° , Cu target, tube pressure 40 kV, tube flow 40 mA.

Hygroscopicity measurements were carried out according to the following: a chosen amount of completely dried KDN, or $[K(18\text{C}6)][N(\text{NO}_2)_2]$ crystals,

were placed in a wide-mouth observation bottle, and completely exposed to air at a temperature of 30 °C and a relative humidity of 75%. The weight was measured once every 24 h, and the state of the crystals before and after the test was recorded. The moisture absorption rate was calculated by Equation 1:

$$w = \frac{m_2 - m_1}{m_1} \times 100\% \quad (1)$$

where m_1 is the initial mass of the material and m_2 is the mass after absorbing moisture.

3 Preparation of [K(18C6)][N(NO₂)₂]

3.1 Preparation by the solvent evaporation method and structure determination

KDN (145 mg) and 18C6 (264 mg) were dissolved in anhydrous methanol (100 mL), and filtered, then 50 mL of the solution was placed in a 30 °C constant temperature incubator and slowly evaporated for about 10 days. Pale yellow transparent block crystals separated from the solution.

A single crystal of suitable size, 0.36×0.36×0.35 mm, was selected and a single crystal X-ray diffraction (SXD) experiment was performed on a Rigaku XtaLAB P300DS diffractometer with graphite-monochromated Mo-K α radiation ($\lambda = 0.154178$ nm) at 298(2) K. The structure was solved with the SHELXS structure solution program [18] using direct methods and refined with the ShELXL refinement package [19] using full-matrix least-squares methods. Anisotropic displacement parameters were applied to all non-hydrogen atoms. All hydrogen atoms were generated geometrically.

3.2 Preparation using low-frequency resonance technology

3.2.1 Low-frequency resonance technology

Low-frequency resonance technology is the core of ultra high efficient mixers [21], and the principle of the mixer is shown in Figure 1.

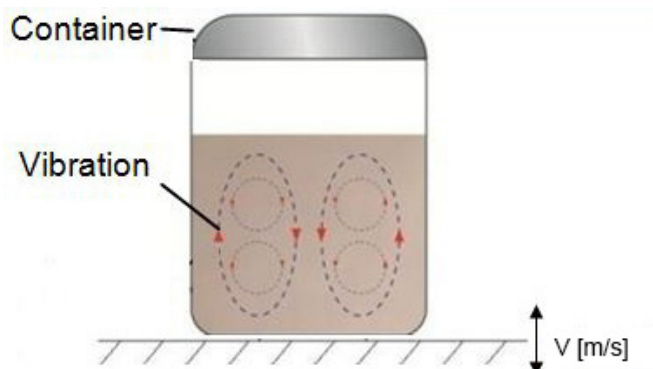


Figure 1. Principle of the Ultra High Efficient Mixer

Low-frequency resonance technology mainly uses the principle of mechanical resonance. The mechanical system has two parts, the driving unit and the load unit. Its aim is to efficiently create high amplitude oscillations within the load unit. By matching the mass, damping coefficient and stiffness of the two-degrees-of-freedom vibration system, the natural frequency of the system is about 60 Hz. The harmonic excitation force comes from the eccentric block driven by the explosion-proof servo motor. When the frequency corresponding to the motor speed is consistent with the natural frequency of the system, the system reaches a resonance state. At this point, the minimum force being applied to the driving unit, the load unit can receive high acceleration, and the mixing system's mechanical energy can be transferred into the material with virtually no loss.

The inverse of time is a frequency-like quantity. When the servo motor's speed is 3600 r/min, the driving frequency is a sinusoidal signal of 60 Hz. In the resonance system, the vibration direction is consistent with the direction of sound wave propagation. The longitudinal wave travels at a speed of 5790 m/s in stainless steel. Because the main material of the mixing container and connecting parts is 304 stainless steel, the sound waves of the vibration are mainly introduced into the material through the metal, so the longitudinal wavelength is 96.5 m. In the energy conversion process, the 96.5 m wave will be transmitted into the material through the container, while the container and the contained material together receive the high acceleration vibration. The acoustic currents and vortices accelerate the intermolecular motion of the material.

Based on the principle of vibrational chemical effect [22], the dynamic balance between two molecules is broken at a fixed frequency and a certain acceleration condition, which promotes non-covalent bonding and accelerates

the spontaneous assembly of molecules into specific physical and chemical structures. Crystals with uniform morphology and particle size can be obtained by optimizing the process parameters, such as acceleration and time.

3.2.2 Preparation of $[K(18C6)][N(NO_2)_2]$

The coordination compound test was carried out using the ultra high efficient mixer. The experiment was carried out as follows: KDN (1.45 g) was placed in a special stainless steel container followed by 18C6 (2.64 g) in a 1:1 molar ratio. The ultra high efficient mixer process parameters were as follows: resonance frequency 60 Hz, acceleration 196 m/s² for 5 min, then anhydrous methanol (1.0 mL) was added to the vial using a calibrated pipette. The ultra high efficient mixer was adjusted to an acceleration of 686 m/s², resonance frequency 60 Hz. After 60 min of mixing, the material was removed from the stainless steel container. Evaporation of the residual solvent by drying in a vacuum oven at 50 °C for 2 h gave a pure yellow powder, compound $[K(18C6)][N(NO_2)_2]$.

4 Results and Discussion

4.1 Structure analysis of $[K(18C6)][N(NO_2)_2]$

The X-ray diffraction data were analyzed by the SHELXTL program to obtain the crystallographic parameters of $[K(18C6)][N(NO_2)_2]$. The results are listed in Table 1. The crystallographic data for $[K(18C6)][N(NO_2)_2]$ have been saved in the Cambridge Crystal Structure Data Center (CCDC1935256). Selected bond lengths and angles are listed in Tables S1 and S2 (see SI).

Table 1. Crystallographic parameters of [K(18C6)][N(NO₂)₂]

Parameter	Values
Empirical formula	C ₁₂ H ₂₄ KN ₃ O ₁₀
Formula weight	409.44
Stoichiometry	1:1
Temperature [K]	298(2)
Crystal system	Monoclinic
Space group	P2 ₁ /c
<i>a</i> [Å]	8.5032(2)
<i>b</i> [Å]	14.4060(4)
<i>c</i> [Å]	8.1708(2)
α [°]	90.0
β [°]	101.896(2)
γ [°]	90.0
Volume [Å ³]	979.40(4)
<i>Z</i>	2
Calculated density [g·cm ⁻³]	1.388
<i>F</i> (000)	432
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0652, <i>wR</i> ₂ = 0.2054
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0722, <i>wR</i> ₂ = 0.2155

4.2 Analysis and discussion of the crystal structure

The crystal structure of the coordination compound [K(18C6)][N(NO₂)₂] belongs to the monoclinic system and the space group is P2₁/c. A single crystal of [K(18C6)][N(NO₂)₂] is shown in Figure 2. The molar ratio of KDN to 18C6 is 1:1. The three-dimensional unit cell stacking diagram of [K(18C6)][N(NO₂)₂] is shown in Figure 3. It can be seen from Figure 3 that the coordination compound is connected by K–O coordination bonds and hydrogen bonds. The formation of the coordination compound is mainly through K–O coordination bonds formed by K atoms in the KDN molecule and eight O atoms in the crown ether ring. C–H···O type weak hydrogen bonds are formed between the O atoms in N(NO₂)₂⁻ and the H atoms in the crown ether ring (Figure 4). The coordination bond and the intermolecular hydrogen bond interaction forces in the compound cause KDN and 18C6 to form a 3D network structure extended infinitely.

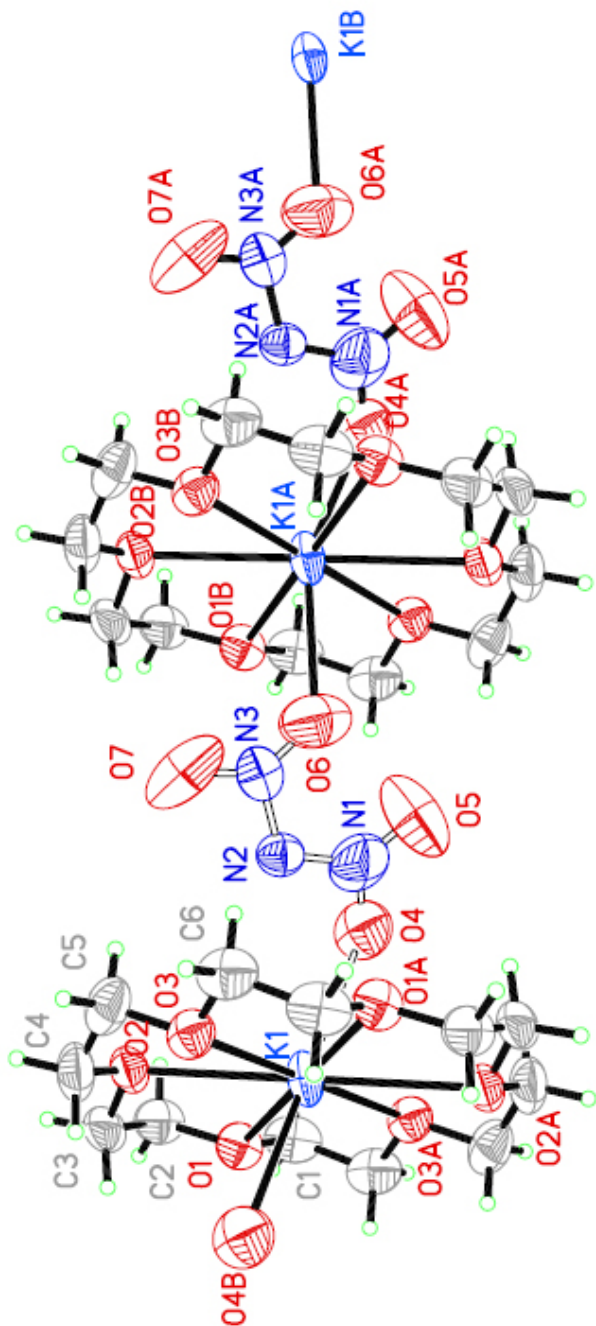


Figure 2. Crystal structure of $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$

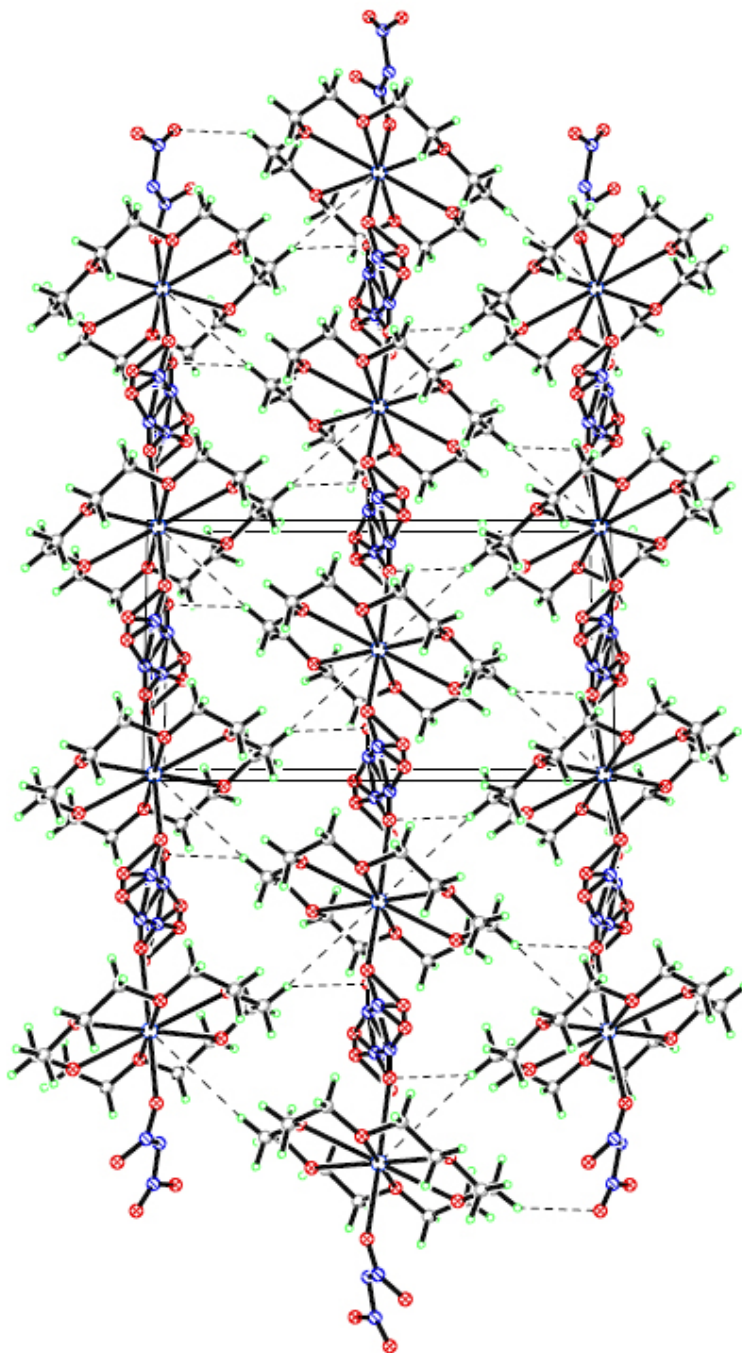


Figure 3. Packing diagram of [K(18C6)][N(NO₂)₂]

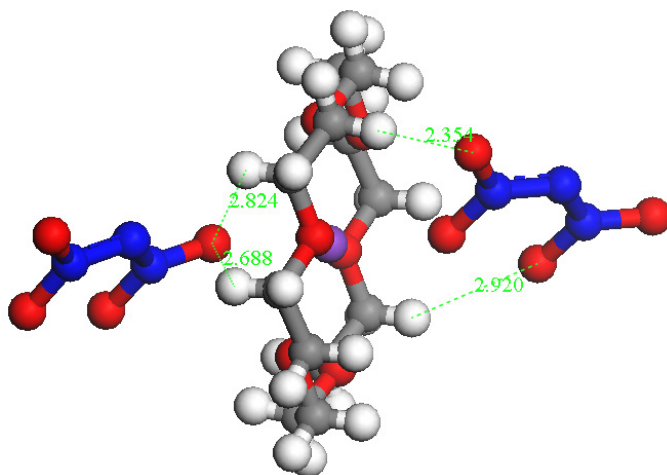
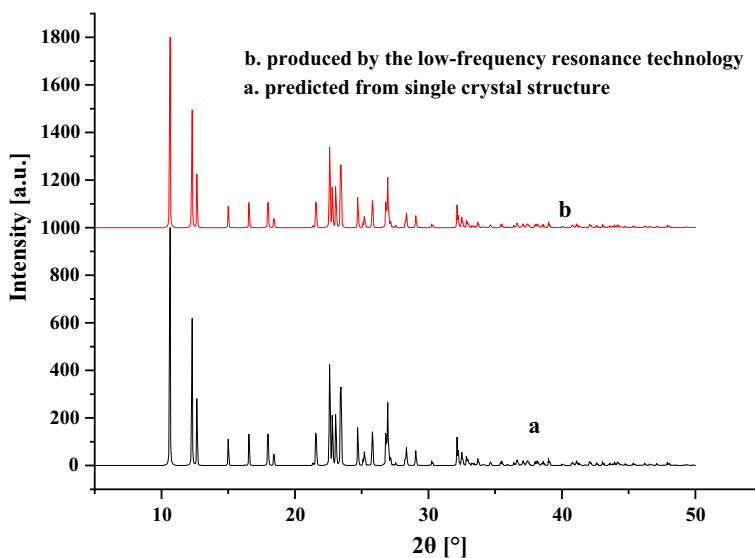


Figure 4. The intermolecular hydrogen bonding interactions of $[\text{K}(18\text{C}6)]$ $[\text{N}(\text{NO}_2)_2]$ (lengths in Å)

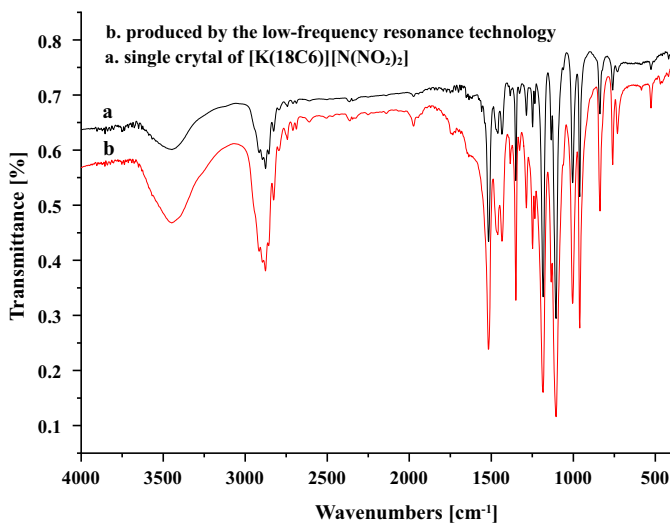
4.3 Properties of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$

4.3.1 PXRD and FT-IR analyses

Figure 5(a) shows the powder X-ray diffraction (PXRD) pattern of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$ produced by low-frequency resonance technology (top) vs. the diffraction pattern predicted from the single crystal structure. Figure 5(b) shows the FT-IR spectrum of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$ produced by low-frequency resonance technology (bottom) vs. the FT-IR spectrum of the single crystal of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$.



(a)



(b)

Figure 5. Powder X-ray diffraction pattern of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$ produced by low-frequency resonance technology (top) vs. the diffraction pattern predicted from the single crystal structure (5a), FT-IR spectrum of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$ produced by low-frequency resonance technology (bottom) vs. the FT-IR spectrum of the single crystal of $[\text{K}(18\text{C}6)][\text{N}(\text{NO}_2)_2]$ (5b)

Comparison of the reflections of the solids obtained by low-frequency resonance technology with the PXRD pattern calculated from the single crystal structure confirmed the production of the desired coordination compound $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$. The FT-IR spectrum of the $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$ single crystal prepared by slow evaporation showed a close match with the experimental FT-IR spectrum of the solid prepared by low-frequency resonance technology. Similar patterns proved that low-frequency resonance technology can efficiently prepare $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$.

4.3.2 Thermal analysis

The thermal decomposition properties of $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$, KDN and 18C6 were tested using differential scanning calorimetry (DSC), and the results are shown in Figure 6. It can be seen that there are two endothermic peaks (135.6 and 341.3 °C) on the DSC curve of KDN, and a single exothermic decomposition peak at 232.9 °C. The first endothermic peak at 135.6 °C is the melting peak of KDN, and the second endothermic peak at 341.3 °C is the melting endothermic peak of KNO_3 . There are two endothermic peaks and two exothermic peaks on the DSC curve of $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$. The endothermic peak temperatures are 166.0 and 179.6 °C, the first peak temperature being 30.4 °C higher than the melting peak temperature of KDN. The analytical data shows that $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$ has better thermal stability than KDN. As the temperature is increased, the molecular structure of the coordination compound is destroyed and becomes unstable. Two concentrated exothermic decomposition processes occur between 200 and 300 °C. The peak temperatures were 232.3 and 287.2 °C, respectively. It can be inferred that when the coordination compound decomposes, two single components are separately decomposed with liberation of heat. The above analysis shows that the formation of the coordination compound can produce unique thermal decomposition behaviour.

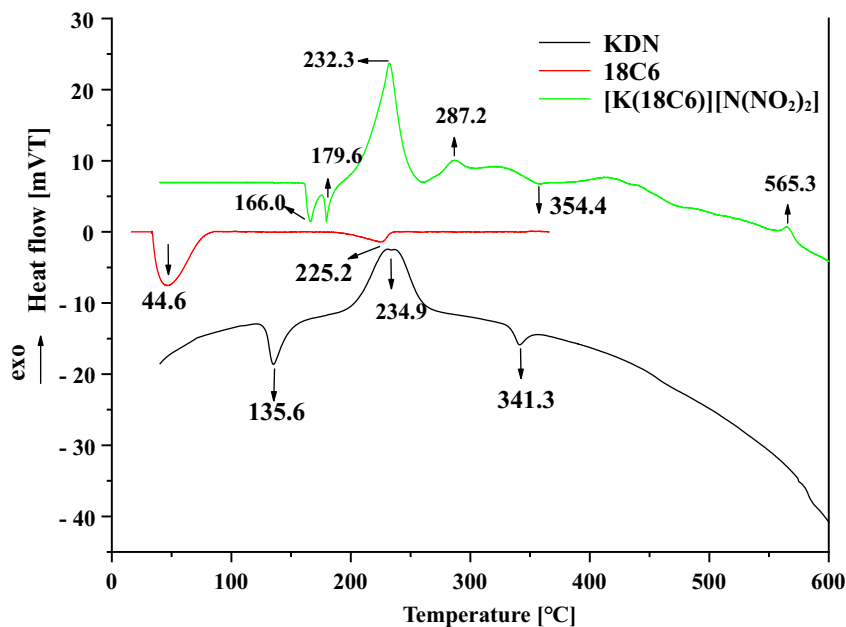


Figure 6. DSC curves of the explosive samples

4.3.3 Hygroscopicity analysis

The moisture absorption of KDN and $[K(18C6)][N(NO_2)_2]$ was measured under constant temperature and constant humidity conditions of 30 °C and 75% relative humidity, respectively. The results are listed in Table 2.

Table 2. Moisture absorption of KDN and $[K(18C6)][N(NO_2)_2]$ at 30 °C and 75% relative humidity

Sample	Moisture absorption [%] after [h]						
	24	48	72	96	120	144	168
KDN	1.428	2.112	3.507	4.885	6.150	7.745	8.968
$[K(18C6)][N(NO_2)_2]$	0.142	0.189	0.331	0.662	0.662	0.662	0.662

The hygroscopicity of the coordination compound was investigated by using the weight gain method. The formation of the coordination compound structure greatly reduces the hygroscopicity of KDN, which may be due to the formation of a variety of weak hydrogen bonds between the $[N(NO_2)_2]^-$ ion and the crown ether ring in the coordination compound molecule. The weak hydrogen bonds prevent the water molecules in the air from binding to $[N(NO_2)_2]^-$, thus the binding of KDN to water molecules is essentially reduced.

5 Conclusions

- ◆ A novel coordination compound $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$ was prepared by using the slow solvent evaporation method.
- ◆ This coordination compound belongs to the monoclinic system, in which the space group is $\text{P}2_1/\text{c}$. The formation of the coordination compound is mainly due to K–O coordination bonds formed by K atoms in the KDN molecule and eight O atoms in the crown ether ring. The C–H...O type weak hydrogen bonds are formed between the O atoms in $[\text{N}(\text{NO}_2)_2]^-$ and the H atoms in the crown ether ring.
- ◆ $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$ increased the melting point of KDN (166.0 vs. 135.6 °C).
- ◆ The novel method of low-frequency resonance technology can be used to successfully and efficiently prepare the coordination compound $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$.
- ◆ In the hygroscopicity experiments, it was found that the coordination compound $[\text{K}(\text{18C6})][\text{N}(\text{NO}_2)_2]$ can prevent the binding of water molecules in the air to $[\text{N}(\text{NO}_2)_2]^-$ and thus comprehensively tune the hygroscopicity.

Acknowledgements

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References

- [1] Zhan, X.; He, Y.; Sun, Z.; Shen, B.; Li, X. Mixing Characteristics of High-viscosity Fluids under Forced Vertical Vibration. *Chem. Eng. Technol.* **2020**, *43*(7): 1327-1335.
- [2] Park, J.H.; Bae, K.T.; Kim, K.J.; Joh, D.W.; Kim, D.; Myung, J.-h.; Lee, K.T. Ultra-fast Fabrication of Tape-cast Anode Supports for Solid Oxide Fuel Cells via Resonant Acoustic Mixing Technology. *Ceram. Int.* **2019**, *45*(9): 12154-12161.
- [3] Ren, X.-t.; Lu, Y.-h.; Lu, Z.-m.; Wang, Q.-s.; He, J.-x.; Ding, N. Preparation, Characterization of Ultrafine CL-20/HMX Cocrystal Explosive and Its Compatibility with the Components of Propellants. (in Chinese) *Chin. J. Energ. Mater. (Hanneng Cailiao)* **2020**, *28*(2): 137-144.
- [4] Michalchuk, A.A.L.; Hope, K.S.; Kennedy, S.R.; Blanco, M.V.; Boldyreva, E.V.; Pulham, C.R. Ball-free Mechanochemistry: *in situ* Real-Time Monitoring of

- Pharmaceutical co-Crystal Formation by Resonant Acoustic Mixing. *ChemComm.* **2018**, 54(32): 4033-4036.
- [5] Kotter, L.N.; Groven, L.J. Milling of Energetic Crystals with the LabRAM. *Propellants Explos., Pyrotech.* **2019**, 44(7): 908-914.
- [6] Liu, J.; Zeng, X.; Li, H. Synthesis, Structure and Spectroscopic Properties of a Novel Compound *bis*(Benzylamino)silver(I) Benzylcarbamate. *Acta Crystallogr., Sect. B* **2012**, 68(4): 401-406.
- [7] Sun, Q.; Zhang, H.; Yuan, Y.; Cao, X.; Sun, L. Polyethylene Glycol-CaCl₂ Coordination Compounds as a Novel Form-Stable Phase Change Material with Excellent Thermophysical Properties. *Adv. Eng. Mater.* **2018**, 20(3), paper 1700643: 1-8.
- [8] Cheng, M.; Liu, X.; Luo, Q.; Duan, X.; Pei, C. Cocrystals of Ammonium Perchlorate with a Series of Crown Ethers: Preparation, Structures, and Properties. *CrystEngComm* **2016**, 18: 8487-8496.
- [9] Hausmann, D.; Feldmann, C. Bromine-rich Zinc Bromides: Zn₆Br₁₂ (18-crown-6)₂×(Br₂)₅, Zn₄Br₈(18-crown-6)₂×(Br₂)₃, and Zn₆Br₁₂ (18-crown-6)₂×(Br₂)₂. *Inorg. Chem.* **2016**, 55: 6141-6147.
- [10] Wu, B.; Lai, Y.; Qi, X.; Du, H.; Pei, C. Facile Synthesis and Accelerated Combustion Effect of Micro-/Nanostructured Amorphous and Crystalline Metal Coordination Compounds Based on *N,N*-bis[1*H*-tetrazol-5-yl]amine. *ACS Appl. Energy Mater.* **2019**, 2(11): 8319-8327.
- [11] Chen, D.; Huang, S.; Zhang, Q.; Yu, Q.; Zhou, X.; Li, H.; Li, J. Two Nitrogen-rich Ni(II) Coordination Compounds based on 5,5'-Azotetrazole: Synthesis, Characterization and Effect on Thermal Decomposition for RDX, HMX and AP. *RSC Adv.* **2015**, 5: 32872-32879.
- [12] Yang, Q.; Ge, J.; Gong, Q.; Song, X.; Zhao J.; Wei, Q.; Xie, G.; Chen, S.; Gao, S. Two Energetic Complexes Incorporating 3,5-Dinitrobenzoic Acid and Azole Ligands: Microwave-assisted Synthesis, Favorable Detonation Properties, Insensitivity and Effects on the Thermal Decomposition of RDX. *New J. Chem.* **2016**, 40: 7779-7786.
- [13] Wang, H.-j.; Ma, Y.; Li, H.-z.; Yu, Y.-w.; Yang, Z.-w. Preparation and Characterization of ADN/18C6 Cocrystal. (in Chinese) *Chin. J. Energ. Mater. (Hanneng Cailiao)* **2018**, 26(6): 545-548.
- [14] Highsmith, T.K.; Hinshaw, C.J.; Wardle, R.B. Phase-stabilized Ammonium Nitrate and Method of Making Same. Patent US 5292387, **1994**.
- [15] Weiser, V.; Lity, A.; Kelzenberg, S.; Koleczko, A.; Roth, E.; Schaller, U.; Walschburger, E. Burning Behavior of B/KDN-mixtures Compared with B/KNO₃. *Proc. 45th Int. Annual Conf. ICT*, Karlsruhe, Germany, **2014**, 111/1-6.
- [16] Berger, B.; Bircher, H.; Studer, M.; Wälchli, M. Alkali Dinitramide Salts. Part 1: Synthesis and Characterization. *Propellants, Explos. Pyrotech.* **2005**, 30(3): 184-190.
- [17] Anderson, S.R.; am Ende, D.J.; Salan, S.J.; Samuels, P. Preparation of an Energetic-Energetic Cocrystal using Resonant Acoustic Mixing. *Propellants Explos.,*

- Pyrotech.* **2014**, 39(5): 637-640.
- [18] Sheldrick, G.M. *SHELXTL-2014, Structure Determination Software Suite*. Bruker AXS, Madison WI, **2015**.
- [19] Sheldrick, G.M. *SHELXS-2014, Program for the Refining of Crystal Structure*. University of Göttingen, Germany, **2015**.
- [20] *National Military Standard of China, Experimental Methods of Sensitivity and Safety*. (in Chinese) GJB/772A-97, **1997**.
- [21] Lu, Z.; Sun, T.; Wang, Q.; Zeng, Q.; Wen, C.; Du, T.; Ren, X.; Zhang, J. Sound Wave Mixing Device Based on Three-Degree-of-Freedom Resonance System. Patent EP 3459619, **2018**.
- [22] am Ende, D.J.; Anderson, S.R.; Salan, J.S. Development and Scale-Up of Cocrystals Using Resonant Acoustic Mixing. *Org. Process Res. Dev.* **2014**, 18: 331-341.

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