Highly Stable Energetic Coordination Polymer Assembled with Co(II) and Tetrazole Derivatives

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Supporting Information

ABSTRACT: A solvothermal reaction of Co(CH₃COO)₂·4H₂O and 5-mercapto-1-methyl-tetrazole (Hmmtz) in methanol (MeOH) yielded a one-dimensional solvent-free energetic coordination polymer, namely, [Co(mmtz)]ₙ 1, which was structurally characterized. The enthalpy of formation (ΔH°) of 1 (907 kJ mol⁻¹) is much larger than that of commercial 2,4,6-trinitrotoluene (~59 kJ mol⁻¹). The impact sensitivity and the friction sensitivity are greater than 40 J and 360 N, respectively, indicating that compound 1 exhibits a potential application as a safe explosive. Temperature-dependent molar magnetic susceptibilities show that weak antiferromagnetic behavior exists in 1.

INTRODUCTION

Energetic materials (EMs) are compounds or mixtures that react sustainably and release a large amount of energy within a short time period, including explosives, gunpowder, and pyrotechnic agents.¹⁻⁵ Traditional EMs like 2,4,6-trinitrotoluene (TNT), 1,3,5-trinitro-1,3,5-triazine (RDX), and 1,3,5,7-tetranitro-1,3,5,7-tetrazacyclooctane (HMX) are commonly used for military and commercial blasting.⁴ However, the most prominent problem of traditional EMs is high sensitivity toward friction and shock, which creates serious safety hazard and significant economic losses in their applications.⁶ To acquire less sensitive CPs, two two-dimensional (2D) materials, [Zn₂(N₂H₄)₃(N₂H₄CO₂)₂(ClO₄)₂·H₂O]ₙ (ZnHHP) and [Co₂(N₂H₄)₄(N₂H₃CO₂)₂(ClO₄)₂·H₂O]ₙ (CHHP), were synthesized.⁶ The results show that the sensitivity have significantly been reduced through ligand modification. Afterward, Pang et al. first designed two three-dimensional (3D) 4,4'-azo-1,2,4-triazole-based coordination polymers [Ag(atrz)₁.₅(NO₃)]ₙ and [Cu(atrz)₃(NO₃)₂]ₙ with excellent thermal stability, high heats of detonation, and less sensitivity.⁷ These reported results indicate that coordination polymers, rationally designed with different structural dimensionalities, can serve as new-generation energetic material candidates. The design strategies of ECPs are focused on the diverse structural types realized by strong coordination bonds between the metal centers and the ligands.¹⁸ On the other hand, intermolecular interaction in EMs, such as π–π stacking, which can effectively buffer against mechanical stimulation to decrease the sensitivity, is also an effective way to improve the stability of EMs.²⁻⁴ Additional, highly energetic covalent bonds of the ligand can offer the primary energetic source to enhance energetic performances of ECPs.¹,¹²,¹⁷

In this study, a 1D solvent-free ECP [Co(mmtz)]ₙ 1 was obtained by the solvothermal method (Scheme 1). The constant-volume combustion heat, thermogravimetric analysis-differential scanning calorimetry (TGA-DSC) curves, and sensitivities of 1 were measured. The values of ΔH° (907 kJ
Scheme 1. Synthesis of 1

Energetic Properties. The enthalpy of formation ($\Delta H^\circ$) is a crucial parameter to assess the performance of ECPs. The constant-volume combustion heat ($Q_v$) measurement of 1 was carried out, and the test value of $Q_v$ is 14.438 kJ g$^{-1}$. According to the gas volume correction: $\Delta H = Q_v + \Delta nRT$ ($\Delta n$ is determined by the change in the number of gas constituents before and after the reaction, $T = 298.15$ K and $R = 8.314$ J mol$^{-1}$ K$^{-1}$) and the ideal combustion reaction eq 1, the enthalphy of combustion ($\Delta_l H$), can be calculated. The ideal combustion reaction equation is given as follows

$$\begin{align*}
\text{C}_2\text{H}_6\text{Co}_2\text{S}_2\text{(s)} + 8\text{O}_2\text{(g)} & \rightarrow \text{CoO}\text{(s)} + 4\text{CO}_2\text{(g)} + 3\text{H}_2\text{O}\text{(l)} + 4\text{N}_2\text{(g)} \\
& + 2\text{SO}_2\text{(g)} \\
\Delta_l H^\circ[1, s] &= \Delta_l H^\circ[\text{CoO, s}] + 4\Delta_l H^\circ[\text{CO}_2, \text{gl}] + 3\Delta_l H^\circ[\text{H}_2\text{O, I}] + 2\Delta_l H^\circ[\text{SO}_2, \text{gl}] - \Delta_l H^\circ[1, s] \tag{1}
\end{align*}$$

The $\Delta_l H$ value of 1 was calculated as $-14.421$ kJ g$^{-1}$. According to eq 2, the $\Delta H^\circ$ value of 1 can be deduced using Hess’s law and inferred as $907$ kJ mol$^{-1}$ at $101.325$ kPa and $298.15$ K, with the known enthalpies of CoO (s, $-237.9$ kJ mol$^{-1}$), $\text{H}_2\text{O}$ (l, $-285.83$ kJ mol$^{-1}$), $\text{CO}_2$ (g, $-393.51$ kJ mol$^{-1}$), and $\text{SO}_2$ (g, $-296.8$ kJ mol$^{-1}$). Compound 1 has a high nitrogen content leading to the relatively high positive $\Delta_l H^\circ$. Compared to that of the commercial TNT ($-59$ kJ mol$^{-1}$), the $\Delta H^\circ$ value of 1 is higher. In addition, compared to other reported ECPs like Co-BTA $15$ ($\Delta H^\circ = 8.314$ J mol$^{-1}$; $\text{Co}_9\text{(BTA)}_{10}\text{(HBTA)}_2\text{(H}_2\text{O})_{10}$), $\text{H}_2\text{BTA} = \text{N,N-bis}(1\text{-H-tetrazole-5-yl})$-amine), $\text{Cd-BTA}5$ ($\text{[Cd(BTA)(H}_2\text{O})_4]$), and $\text{Cu-DNS}^1$ ($\text{Cu}_2\text{(DNS)}\text{(tetrazole)(H}_2\text{O})$, $\text{H}_2\text{DNS} = 3\text{-dinitrosalicylic acid}$) (Table 1), the $\Delta H^\circ$ value of 1 has obvious advantages.

Sensitivity Test. The impact and friction sensitivity test is an indispensable measurement of EMs for the risk assessment. The experimental data of 1 as well as those of the commercial explosive TNT and some selected ECPs were also given for comparison in Table 1. For compound 1, the experimental value of IS is greater than $40$ J and FS is larger than $360$ N. Compound 1 possesses low impact and friction sensitivity, showing that 1 has an excellent mechanical stability than TNT and the selected ECPs, such as Co-BTA $15$ (IS = 27 J, FS > 360 N). These results denoted that compound 1 is an insensitive energetic material in accordance with the U.N. Standard. $31$ The introduction of electron-donating substituents like methyl ($-\text{CH}_3$), hydroxyl ($-\text{OH}$), or sulphydryl ($-\text{SH}$) can also reduce the sensitivity of compounds. $32$ The good thermal stability, low mechanical sensitivity, and high $\Delta_H^\circ$ value of 1

![Figure 1](image-url)
Magnetic Properties. The temperature-dependent molar magnetic susceptibilities were obtained in an external field of 1 kOe at temperatures ranging from 2 to 300 K. The plots of $\chi_M$ and $\chi_M T$ vs $T$ for 1 are shown in Figure 3. The measured

Figure 3. $\chi_M$ and $\chi_M T$ vs $T$ plots for 1. The red solid line represents the best fit of the Curie–Weiss law within 70–300 K.

$\chi_M T$ value is 2.26 emu K mol$^{-1}$, which is slightly higher than that of the theoretical value of a magnetic isolated Co(II) (1.875 emu K mol$^{-1}$, $g = 2.00$ and $S = 3/2$) at 300 K because of the orbital contribution of Co(II) in a tetrahedral surrounding. The $\chi_M T$ values reduce gradually to 1.90 emu K mol$^{-1}$ at 100 K when the temperature decreases and then reduces rapidly to 0.078 em mol$^{-1}$ K$^{-1}$ at 2 K. The results indicate that compound 1 has antiferromagnetic interactions. The magnetic susceptibility within 70–300 K was fitted well to the Curie–Weiss law $\chi_M = C/(T - \theta)$ with $C = 2.516(1)$ emu mol$^{-1}$ K$^{-1}$ and $\theta = -29.985(0)$ K. The effective magnetic moment ($\mu_{\text{eff}}$ = $(8C)^{1/2} \mu_B$) per Co$^{2+}$ ion in 1 is 4.5 $\mu_B$, which approached the value of 5.1 $\mu_B$ for a free Co(II) ion. The negative Weiss constant also denotes the antiferromagnetic interactions in 1 (the distance between the nearest Co$^{2+}$ ions is 3.908(4) Å). The measured field-dependent isothermal magnetization curve at 10 K shows that the magnetization decreases almost linearly at an external field ranging from 80 to 0 kOe (Figure S1, Supporting Information). When the magnetic field $H = 0$, the magnetization value of 1 is zero, which indicates that compound 1 does not generate magnetism without the external magnetic field. The magnetic influence of 1 on the electronic instrument signals of transport vehicles can be neglected.

Table 1. Energetic Performance of Compound 1, Traditional Explosive TNT, and Some Selected ECPs

|               | Co-BTA$^{15}$ | Cd-BTA$^{30}$ | Co-DNS$^{1}$ | TNT$^{32}$ |
|---------------|---------------|---------------|--------------|------------|
| IS$^a$/[J]   | >40           | 27            | >40          | 15         |
| FS$^b$/[N]   | >360          | >360          | >360         | >360       |
| $\Delta H^m$/$[kJ \text{ mol}^{-1}]$ | 907           | 860           | 212          | 304        |
| $N^c$/[N]    | 38.72         | 59.85         | 44.76        | 19.08      |
| $T_d^d$/$[^\circ C]$ | 304           | 253           | 356          | 317        |

$^a$Impact sensitivity. $^b$Friction sensitivity. $^c$Enthalpy of formation. $^d$Nitrogen content. $^e$Decomposition temperature.

In summary, a new nitrogen-rich Co(II) coordination polymer 1 was successfully obtained and structurally characterized. The energetic properties of 1 have been thoroughly investigated. Compound 1 displays good thermal stability ($T_d = 304 ^\circ C$), a high $\Delta H^m$ value (907 kJ mol$^{-1}$), and low sensitivity (IS > 40 J and FS > 360 N), which are superior to the traditional TNT ($T_d = 295 ^\circ C$, $\Delta H^m = -59$ kJ mol$^{-1}$, IS = 15 J, FS > 353 N). In contrast to the ligand Hmmtz with the $T_d$ value of 125 $^\circ C$, the thermal stability of 1 remarkably increases through utilizing coordination polymerization and $\pi-\pi$ interactions. The temperature-dependent molar magnetic susceptibilities show the existence of weak antiferromagnetic interactions in 1. These results imply that compound 1 can act as a safe energetic material candidate. This work can provide valuable references for the rational design of new safe EMs.

**CONCLUSIONS**

**EXPERIMENTAL SECTION**

**Safety Precautions.** Caution! Hmmtz and 1 are potential EMs. Dealing with these materials requires appropriate protective measures like safety glasses and leather gloves.

**Materials and Methods.** All chemicals can be commercially obtained and used without further purification. TGA and DSC data were collected using a Mettler Toledo instrument under a N$_2$ atmosphere with 2.7 mg power of the samples heated in an Al$_2$O$_3$ crucible at a linear heating rate of 5 K min$^{-1}$. Elemental analyses were performed on an Elementar Vario EL III microanalyzer. The Fourier transform infrared...
spectra were recorded on a PerkinElmer Spectrum using KBr pellets. The temperature-dependent molar magnetic susceptibilities were calculated on a Quantum Design MPMS-XL SQUID magnetometer using crushed samples (in the powdered form). IS and FS were measured on a BAM fall hammer BFH-12 and a BAM friction apparatus FSKM-10, respectively, calibrated by manufacturers using traditional energetic materials such as TNT, RDX, and HMX. Powdered X-ray diffraction (PXRD) patterns were recorded on a Rigaku MiniFlex 600 diffractometer using Cu Kα radiation in the 2θ range of 5° < 2θ < 65° at room temperature. The Mercury software can produce the simulated patterns of compound 1.

**Synthesis of [Co(mmtz)2].** A mixture of Hmmtz (174 mg, 1.5 mmol) and Co(CH3COO)2·4H2O (125 mg, 0.5 mmol) in MeOH (10 mL) was sealed into a 25 mL Teflon-lined autoclave after 15 min of ultrasonic treatment, then heated to 120 °C for 8 h, and kept for 3 days at 120 °C. Finally, it was cooled to 30 °C at a linear rate of 3 °C h⁻¹. Dark blue block crystals of 1 were formed (yield: 60%, based on Co). Calcd for C14H8CoN8S2: C, 56.96; H, 2.56; N, 38.48%. Found: C, 56.72; H, 2.38; N, 38.57%. IR (KBr pellet, cm⁻¹): 2949 w, 1717 w, 1466 m, 1385 s, 1310 m, 1182 m, 1086 w, 1032 w, 989 w, 831 w, 712 m (Figure S2, Supporting Information). The peak of experimental PXRD pattern (Figure S3, Supporting Information) was in accordance with the simulation, showing the pure phase of the as-synthesized 1.

**X-ray Crystallography.** Diffraction data of 1 were measured on a Rigaku PILATUS CCD diffractometer at 293 K using Mo Kα radiation (λ = 0.71073 Å). The intensity data sets were measured on the ω-scan technique and found to decrease on CrystalClear software. The direct methods were used to resolve the structure and the subsequent successive difference Fourier yielded in other nonhydrogen atoms. The hydrogen atoms were calculated and added to the idealized positions. A full-matrix least-squares refinement based on F² refined the final structure. Table 2 summarizes the relevant crystal data and structure refinement results of 1.

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**Table 2. Crystal Data and Structure Refinements for 1**

| Parameter | Value |
|-----------|-------|
| CCDC      | 1558432 |
| formula   | C14H8CoN8S2 |
| formula weight | 578.22 |
| crystal system | monoclinic |
| space group | C2/c |
| a [Å]     | 14.902(14) |
| b [Å]     | 13.529(12) |
| c [Å]     | 12.197(11) |
| α [deg]   | 90 |
| β [deg]   | 121.158(11) |
| γ [deg]   | 90 |
| V [Å³]    | 2104(3) |
| Z         | 8 |
| DcCAL [g cm⁻³] | 1.826 |
| F(000)    | 1160 |
| 2θ [deg]  | 6.886–54.974 |
| refnls    | 8757 |
| ∑ond²     | 0.992 |
| R₁ (I>2σ(I)) | 0.0588 |
| wR₁ (I>2σ(I)) | 0.1026 |
| R₁ (all data) | 0.1423 |
| wR₁ (all data) | 0.1298 |

"R₁ = \sum(Fo - Fc)/\sumFc, wR₂ = \left(\sum(w(Fo² - Fc²)^2)/\sum(w(Fc²))\right)^{1/2}."

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