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Crystal Structures and Thermal Properties of Two Transition-Metal Compounds $\{[\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_3][\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_4]\} \cdot 6\text{H}_2\text{O}$ and $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$ (DNI = 2,4-Dinitroimidazolate)

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Abstract

Two transition-metal compounds derived from 2,4-dinitroimidazole, $\{[\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_3][\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_4]\} \cdot 6\text{H}_2\text{O}$, **1**, and $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, **2**, were characterized by elemental analysis, FT-IR, TG-DSC and X-ray single-crystal diffraction analysis. Crystal data for **1**: monoclinic, space group C2/c, $a = 26.826(3)$, $b = 7.7199(10)$, $c = 18.579(2)$ Å, $\beta = 111.241(2)^\circ$ and $Z = 4$; **2**: monoclinic, space group C2/c, $a = 6.5347(6)$, $b = 17.1727(17)$, $c = 14.1011(14)$ Å, $\beta = 97.7248(10)$ and $Z = 4$. Compound **1** contains two isolated nickel centers in its structure, one being six-coordinate and another five-coordinate. The structure of **2** contains a lead (II) center surrounded by two chelating DNI ligands and four water molecules in distorted square-antiprism geometry. The abundant hydrogen bonds in two compounds link the molecules into three-dimensional network and stabilize the molecules. The TG-DSC analysis reveals that the first step is the loss of water molecules and the final residue is the corresponding metal oxides and carbon.

Keywords

2,4-Dinitroimidazole; Nickel(II) compound; Lead(II) compound; Crystal structure; Thermal decomposition behavior

Introduction

Modern high-energy density materials (HEDM), where energetic nitrogen rich salts are among the most recent and exciting developments, continue to attract considerable interest [1-7]. In

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Supplementary Material

X-ray crystallographic files in CIF format for complexes **1** and **2** have been deposited at the Cambridge Crystallographic Data Center, CCDC Nos. 694508 for **1** and 686971 for **2**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; (fax: +44-1223-336033; deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

last decades, more attention has been focused on 3-nitro-1,2,4-triazol-5-one (NTO) [8-14], 1,3,5-trinitrohexahydrotriazine (RDX) [1,2], octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazo-cine (HMX) [1,2], etc. Recently we have focused our attentions on the studies of molecular structures and thermal properties of metal salts of 3,5-dinitropyridone [15-20].

2,4-Dinitroimidazole (DNI), an energetic compound, is highly insensitive towards impact and its thermal stability is excellent. The calculated detonation properties results indicate that its performance is about 30% better than triaminotrinitrobenzene (TATB). It can be prepared easily with good yield starting from inexpensive starting materials. Results from impact sensitivity, friction sensitivity time-to-explosion temperature and vacuum stability tests indicate that 2,4-dinitroimidazole is less sensitive than both RDX and HMX [21,22]. On comparison with 2,4-dinitroimidazole itself, the studies on its metal salts are rare, particularly their molecular structures [23-26]. Only its copper salt $[\text{Cu}(\text{DNI})_2(\text{H}_2\text{O})_2] \cdot 3\text{H}_2\text{O}$ has been characterized by single-crystal X-ray diffraction analysis and revealed that it is a distorted four-coordinate planar geometry [26]. In this paper, we reported the molecular structures and thermal properties of nickel (II) and lead (II) salts derived from 2,4-dinitroimidazole.

Experimental

Materials and Physical Measurements

All chemicals were of analytical reagent grade and used directly without purification. 2,4-Dinitroimidazole and sodium 2,4-dinitroimidazolate ($\text{Na}(\text{DNI}) \cdot 4\text{H}_2\text{O}$) were synthesized according to the description in the literature [25]. Barium 2,4-dinitroimidazolate ($\text{Ba}(\text{DNI})_2 \cdot 4\text{H}_2\text{O}$) was synthesized similar to that of $\text{Na}(\text{DNI}) \cdot 4\text{H}_2\text{O}$ using $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ instead of NaOH [25]. The infrared spectra of the complexes were recorded on a Perkin Elmer FT-IR spectrophotometer in the $4,000\text{--}400\text{ cm}^{-1}$ region using KBr pellets method. Elemental contents of carbon, hydrogen and nitrogen were determined by a German Vario EL III analyzer. The contents of metal elements were determined by a JY-38 Plus ICP spectrometer. The DSC and TG-DTG measurements were done with a Thermoanalyzer Systems Q1000DSC + LNCS + FACS Q600SDT of TA company. The crystal structure was determined with a Bruker Smart-1000 CCD diffractometer.

Synthesis

$\{[\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_3][\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_4]\} \cdot 6\text{H}_2\text{O}$, **1**: To a 30 mL aqueous solution containing $\text{Ba}(\text{DNI})_2 \cdot 4\text{H}_2\text{O}$ (2.607 g, 0.005 mol) was added a 30 mL aqueous solution of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ (1.443 g, 0.005 mol) dropwise with the formation of white precipitate BaSO_4 . The suspension was stirred for another 2 h at 60°C , filtrated and washed twice with distilled water. The green filtrate was evaporated to dryness and recrystallized with ethanol. The green prism crystals suitable for single-crystal X-ray diffraction analysis were separated from the ethanol solution by slow evaporation at room temperature after two weeks. Yield: 4.523 g (92.31%). Elemental analysis [found (Calcd)] for $\text{C}_{12}\text{H}_{30}\text{N}_{16}\text{Ni}_2\text{O}_{29}$: C, 14.94 (14.71); H, 2.78 (3.09); N, 23.29 (22.87); Ni 12.42(11.98). FT-IR (KBr, cm^{-1}): 3577(vs), 3539(vs), 3393(br), 3152(vs), 1632(s), 1546(s), 1497(s), 1452(vs), 1380(m), 1317(s), 1225(m), 838(m), 764(m), 656(m).

$\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, **2**: to a 20 mL aqueous solution containing $\text{Na}(\text{DNI}) \cdot 4\text{H}_2\text{O}$ (2.511 g, 0.010 mol) was added a 30 mL solution of $\text{Pb}(\text{NO}_3)_2$ (1.656 g, 0.005 mol) dropwise with the formation of yellow precipitate. After completion of the addition, the suspended solution was stirred for another one hour, followed by filtration and then washing with distilled water twice. The precipitate was dried in air. The single crystals suitable for X-ray diffraction analysis were grown as follows: the aqueous solution of $\text{Na}(\text{DNI}) \cdot 4\text{H}_2\text{O}$ was carefully layered by aqueous solution of $\text{Pb}(\text{NO}_3)_2$. After one week yellowish block-like crystals were collected and washed by distilled water. Yield: 2.892 g (97.47%). Elemental analysis [found (Calcd)] for

C₆H₁₀N₈O₁₂Pb: C, 12.22 (12.14); H, 1.566 (1.70); N, 18.89 (18.88); Pb 35.03(34.92). FT-IR (KBr, cm⁻¹): 3565(vs), 3524(s), 3369(br), 3131(vs), 1655(s), 1593(s), 1505(s), 1486(s), 1434(vs), 1392(m), 1359(s), 1291(m), 850(m), 837(m), 758(m), 671(m).

X-ray Crystal Structure Determination

The determination of the unit cell and the data collection for the complexes **1** and **2** were performed on a Bruker Smart-1000 CCD diffractometer with graphite monochromated Mo K α radiation ($\gamma = 0.71073$ Å) using phi and omega scans technique. The structure was solved by direct methods and refined on F^2 by full matrix least-squares with the Bruker's SHELXL-97 program [27,28]. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were treated using a riding model. The crystals used for the diffraction study showed no decomposition during data collection. Data collection details and structure determination results are summarized in Table 1. Selected bond lengths and angles are presented in Table 2.

Results and Discussion

IR Spectra

The strong and broad bands in the range of 3,600–3,350 cm⁻¹ are the characteristic peaks of OH groups in H₂O. Imidazolyl ring C–H stretching and bending vibrations are located in the 3,130–3,155 cm⁻¹ and 650–770 cm⁻¹ regions, respectively. The NO₂ symmetric and asymmetric stretching vibrations and bending vibration are in the range of 1,310–1,500 cm⁻¹ and 830–850 cm⁻¹, respectively.

Structural Description

The structural analysis revealed that compound **1**, as shown in Fig. 1, consists of two isolated nickel centers 10.589(2) Å apart, one being six-coordinate and another five-coordinate.

The six-coordinate nickel center is occupied equatorially by oxygen atoms from four water molecules and axially by nitrogen atoms from two DNI ligands, thus exhibiting slightly distorted octahedral coordination geometry with an inversion center located on the central metal ion. The Ni–Ow bond lengths of 2.031(2) and 2.081(2) Å and Ni(1)–N(1) distance of 2.143(2) Å are comparable to the corresponding bond lengths in compound Ni(2DNPO)₂(H₂O)₄ and Ni(H₂O)₆(NTO)₂·2H₂O reported previously [14,16]. The Ni–N distance is longer than those of Ni–Ow bonds. Thus the coordination geometry around the nickel ion (Ni1) is of an elongated octahedron.

The five-coordinate nickel moiety in **1** has a two-fold axis that runs along the “axial” Ni2–O4w bond ensure that the NiO₂N₂ core geometry adopt either pyramidal or butterfly shape. The C2 symmetry also ensures that in this five-coordinate species the nitro groups adopt a Z-type conformation compared with the Ni1-defined six-coordinate species where the nitro groups are related by inversion ensuring they adopt an E-type conformation. In fact, the structure of the five-coordinate species could be described as an intermediate between the idealized square-pyramidal and trigonal-bipyramidal extremes. The geometric parameter $\tau = 0.235$, according to the definition of Addison [29], indicated that it can be described as a distorted square pyramid, with atom O(4w) at the apex and its base being formed by two O atoms of water molecules and two N atoms of DNI ligands. The bond lengths of Ni(2)–Ow (1.999(2) and 2.035(3) Å) and Ni(2)–N (2.088(2) Å) are slightly shorter than those in the six-coordinate nickel center. The angles between the imidazolyl rings and the nitro groups are less than 3.0(6)°, indicating that the nitro groups and the imidazolyl rings are nearly parallel to each other. As regards the crystal packing, the structure is stabilized by an extensive network of

hydrogen bonds, which involve the DNI ligands, through the nitro group O atoms, the imidazolyl ring N atoms, and the coordinated as well as the uncoordinated water O atoms.

The structure of compound $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, **2**, as shown in Fig. 2, could be described as a distorted square-antiprism geometry, with a twofold axis along the angle bisector of the angle $\text{O}(1)\text{--Pb}(1)\text{--O}(1)\#1$. The two chelating 2,4-dinitroimidazolato (DNI) units are therefore *cis* to one another, with $\text{Pb}(1)\text{--N}(2)$ and $\text{Pb}(1)\text{--O}(1)$ bond lengths of 2.538(3) and 3.045(4) Å, respectively. The bond angle of $\text{N}(2)\text{--Pb}(1)\text{--N}(2)\#1$ is $80.78(15)^\circ$ and of $\text{O}(1)\text{--Pb}(1)\text{--O}(1)\#1$ is $177.94(10)^\circ$. The remaining four sites on the Pb(II) center are occupied by four water molecules, with $\text{Pb}(1)\text{--O}(1\text{w})$ and $\text{Pb}(1)\text{--O}(2\text{w})$ bond lengths of 2.607(3) and 2.917(4) Å, respectively, which are substantially longer than that of Pb--Ow (2.466(5) Å) in $\text{Pb}(\text{NTO})_2(\text{H}_2\text{O})$ [14]. The arrangement of the ligands together with the spread variety of bond lengths suggests a gap or hole in the coordination geometry around the lead ion [the $\text{O}(1)\text{--Pb}(1)\text{--O}(1)\#1$ and $\text{O}(2\text{w})\text{--Pb}(1)\text{--O}(2\text{w})\#1$ angles are $177.94(10)^\circ$ and $126.74(14)^\circ$, respectively], occupied possibly by a stereoactive lone pair of electrons on lead (II) [30–34]. The possible stereochemical activity of the lone pair in divalent lead compounds has been discussed by Shimoni-Livny et al. [30]. They concluded that for coordination number 6–8 both holodirected and hemidirected arrangements are possible, depending on other factors such as the nature of the ligands, their capacity to transfer electrons to the metal and etc. [30–34]. There also exist a few hydrogen bonds between the uncoordinated nitrogen atoms of imidazolyl rings and oxygen atoms of water molecules and nitro groups. These weak interactions link the molecules into a 3D molecular network and seem to contribute to the stabilization of the crystal structure.

In order to evaluate the thermal stability of the synthesized compounds, TG-DTG and DSC experiments were employed under N_2 atmosphere. Typical TG-DTG curve for lead(II) compound **2** are displayed in Fig. 3. There are four main peaks (104.00, 259.32, 274.03 and 429.78 °C) on DTG curve, indicating that the decomposition process of $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$ can be divided into four stages. The first stage completes at 124.36 °C accompanying with 12.29% mass loss. It is in good agreement with the theoretical value of the mass loss of 12.14%, corresponding to the loss of $4\text{H}_2\text{O}$. The second and third stages cannot be distinguished clearly. These two stages ceased at 305.25 °C accompanied with 31.01% weight loss, which approaches to the theoretical value of the mass loss of 30.77% of all four nitro groups. The fourth stage ends at 442.28 °C accompanied with 27.27% mass loss. After the fourth stage the mass of the residue is 39.67% in accordance excellently with the mass summation (39.64%) of $\text{PbO} + \text{C}$. Similarly, the studies on the thermal decomposition behaviors of the nickel compound **1** showed that the decomposition process is similar: the first stage is the dehydration course and the final residue is the corresponding metal oxide and carbon.

Conclusions

We have successfully grown and determined the molecular structures of $\{[\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_3][\text{Ni}(\text{DNI})_2(\text{H}_2\text{O})_4]\} \cdot 6\text{H}_2\text{O}$ and $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, which represent few 2,4-dinitroimidazole-derived metal salts under structural elucidation by single-crystal X-ray diffraction analysis. It will be undoubtedly helpful for us to better understand their decomposition mechanisms.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Fig. 1.
The structure of the compound $[\text{Ni}_2(\text{DNI})_4(\text{H}_2\text{O})_7] \cdot 6\text{H}_2\text{O}$, **1**, showing 30% probability displacement ellipsoids

Fig. 2.
The structure of the compound $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, 2, showing 30% probability displacement ellipsoids

Fig. 3.
The TG-DTG curves for $\text{Pb}(\text{DNI})_2(\text{H}_2\text{O})_4$, **2**, at a heating rate of $10^\circ\text{C min}^{-1}$

Table 1Crystallographic data for **1** and **2**

	1	2
Empirical formula	C ₁₂ H ₃₀ N ₁₆ Ni ₂ O ₂₉	C ₆ H ₁₀ N ₈ O ₁₂ Pb
Formula weight	979.94	593.41
Temperature (K)	293(2)	291(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	C2/c
Unit cell dimensions (Å , °)	<i>a</i> = 26.826(3) <i>b</i> = 7.7199(10) <i>c</i> = 18.579(2) β = 111.241(2)	<i>a</i> = 6.5347(6) <i>b</i> = 17.1727(17) <i>c</i> = 14.1011(14) β = 97.7240(10)
Volume (Å ³), <i>Z</i>	3586.1(8), 4	1568.0(3), 4
<i>D</i> _{calcd} (g/cm ³)	1.815	2.514
Crystal size (mm ³)	0.30 × 0.23 × 0.10	0.49 × 0.34 × 0.30
θ Range for data collection (°)	2.3–27.5	2.78–27.50
Adsorption coefficient	1.177	10.847
Reflections collected	4078	1805
Independent reflections	2684	1761
Goodness-of-fit on <i>F</i> ²	0.963	1.009
Final <i>R</i> indices	<i>R</i> 1 = 0.0722,	<i>R</i> 1 = 0.0233,
[<i>I</i> > 2σ(<i>I</i>)]	w <i>R</i> 2 = 0.0997	w <i>R</i> 2 = 0.0508
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0389,	<i>R</i> 1 = 0.0262,
	w <i>R</i> 2 = 0.0837	w <i>R</i> 2 = 0.0552

Table 2Selected bond lengths (Å) and bond angles (°) for **1** and **2**

1		2	
Ni(1)–O(2w)	2.031(2)	Pb(1)–N(1)	2.538(3)
Ni(1)–O(1w)	2.081(2)	Pb(1)–O(1w)	2.600(3)
Ni(1)–N(1)	2.143(2)	Pb(1)–O(2w)	2.917(4)
Ni(2)–O(3w)	1.999(2)	Pb(1)–O(1)	3.045(4)
Ni(2)–O(4w)	2.035(3)		
Ni(2)–N(5)	2.088(2)		
O(2w)–Ni(1)–O(2w)#1	180	N(1)#1–Pb(1)–N(1)	80.82(16)
O(2w) #1–Ni(1)–O(1w)	94.0 (1)	N(1)–Pb(1)–O(1w)#1	79.31(12)
O(2w)–Ni(1)–O(1w)	86.0(1)	N(1)–Pb(1)–O(1w)	72.00(11)
O(1w)–Ni(1)–O(1w)#1	180	O(1w)#1–Pb(1)–O(1w)	142.11(16)
O(2w)–Ni(1)–N(1) #1	84.4 (1)	N(1)–Pb(1)–O(2w)#1	80.89(10)
O(1w)–Ni(1)–N(1)	88.7(1)	O(1w)–Pb(1)–O(2w)#1	69.32(10)
O(2w)–Ni(1)–N(1)	95.6(1)	N(1)–Pb(1)–O(2w)	147.25(11)
O(1w)#1–Ni(1)–N(1)	91.3 (1)	O(1w)–Pb(1)–O(2w)	130.10(9)
N(1)#1–Ni(1)–N(1)	180	O(2w)#1–Pb(1)–O(2w)	126.74(14)
O(3w)#2–Ni(2)–O(3w)	165.3(1)	N(2)#1–Pb(1)–O(1)	124.82(9)
O(3w)–Ni(2)–O(4w)	97.4(1)	N(2)–Pb(1)–O(1)	57.07(9)
O(3w)–Ni(2)–N(5)	89.2(1)	O(1w)#1–Pb(1)–O(1)	66.90(10)
O(3w)#2–Ni(2)–N(5)	90.7(1)	O(1w)–Pb(1)–O(1)	113.83(10)
O(4w)–Ni(2)–N(5)	90.3(1)	O(2w)#1–Pb(1)–O(1)	63.84(10)
N(5)–Ni(2)–N(5)#2	179.4(1)	O(2w)–Pb(1)–O(1)	115.13(9)
		O(1)–Pb(1)–O(1)#1	177.94(10)

Symmetry transformations used to generate equivalent atoms: (#1) in **1**: $x, -y, z + 1/2$; (#2) in **1**: $x + 1/2, y + 1/2, z$; (#1) in **2**: $-x, y, -z + 1/2$