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CRYSTAL STRUCTURE AND THERMAL PROPERTIES OF NOVEL Co(II) COMPLEXES WITH 2,6-PYRIDINEDICARBOXYLATE CONTAINING INFINITE ONE-DIMENSIONAL CHAIN: $[\text{CoL}^1(\text{H}_2\text{O})_4][\text{Co}(\text{pydc})_2] \cdot 2\text{H}_2\text{O}$ AND $[\text{Co}(\text{H}_2\text{O})_6][\text{Co}(\text{pydc})_2]\text{L}^2 \cdot \text{H}_2\text{O}$ ($\text{L}^1 = 1,3\text{-BIS}(4\text{-PYRIDYL})\text{PROPANE}$, $\text{L}^2 = 3\text{-AMINO-1H-1,2,4-TRIAZOLE}$, $\text{PYDC} = 2,6\text{-PYRIDINEDICARBOXYLIC ACID}$)

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The two title compounds were prepared from the ligand pydc with cobalt(II) acetate in the presence of L^1 and L^2 ($\text{L}^1 = 1,3\text{-bis}(4\text{-pyridyl})\text{propane}$, $\text{L}^2 = 3\text{-amino-1H-1,2,4-triazole}$, $\text{pydc} = 2,6\text{-pyridinedicarboxylic acid}$). The complexes were characterized by elemental analysis, IR spectrum and single crystal X-ray diffraction. Single crystal analysis shows that in two complexes coordination number around Co atom is six with distorted octahedral geometry, and both two complexes consist of ion pairs containing cationic $[\text{Co}(\text{H}_2\text{O})_n]^{2+}$ and anionic $[\text{Co}(\text{pydc})_2]^{2-}$ units.

Keywords: Co(II) complexes; one-dimensional complexes; crystal structure; hydrogen bonding; 2,6-pyridinedicarboxylic acid.

INTRODUCTION

The design of crystal structures and control of molecular arrangement of coordination networks have provided exciting new prospects for chemists in recent years [1–6]. One-dimensional chain compounds consisting of metal ions connected together by bidentate ligands are of current interesting area due to their ability to show novel magnetic [7], nonlinear optica [8] and conductive properties [9]. Flexible dipyriddy ligand 1,3-bis(4-pyridyl)propane is good candidate for molecular building blocks because of its rodlike rigidity and length, and it was widely used to construct 1-D chain compounds [10, 11]. However the design and synthesis of transition metal complexes containing 2,6-pyridindicarboxylic acid has attracted considerable attention due to the versatile bidentate, tridentate, and bridging coordination modes of the ligand [12–14]. The ligand pydc is widely used in coordination chemistry forming stable complexes with most metal ions adopting tridentate coordination mode [15]. A few examples of poly-dimensional coordination networks of 2,6-pyridinedicarboxylic acid ligand have been studied [16–23]. In this paper, we report two novel one-dimensional chain cobalt (II) complexes containing the ligand pydc in the presence of 1,3-bis(4-pyridyl)propane and 3-amino-1H-1,2,4-triazole, their syntheses, crystal structures, and thermal properties.

EXPERIMENTAL

All chemicals and solvents purchased were of reagent grade and used without further purification. Elemental analyses for carbon, hydrogen and nitrogen atoms were performed on a Vario EL III elemental analyzer. The infrared spectra ($4000\text{--}600\text{ cm}^{-1}$) were recorded by using KBr pellet on an Ava-

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tar™ 360 E.S.P. IR spectrometer. Thermogravimetric analysis was carried out with a TA 4000 unit, at a heating rate 10 °C/min.

Synthesis of $[\text{CoL}^1(\text{H}_2\text{O})_4][\text{Co}(\text{pydc})_2] \cdot 2\text{H}_2\text{O}$ complex **1**: a MeOH/H₂O (1:1) solution (5 ml) of 1,3-di(4-pyridyl)propane (0.02301 g, 0.116 mmol) was added to a MeOH/H₂O solution (15 ml) containing $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (0.03314 g, 0.133 mmol) and pydc (0.0365 g, 0.184 mmol) under continuous stirring for 15 min. The mixed solution was filtered off and X-ray quality light brown block shaped of **1** were grown from the resulting solution by slow evaporation at room temperature. Yield 42.3 %. Anal. calcd for $\text{C}_{27}\text{H}_{32}\text{Co}_2\text{N}_4\text{O}_{14}$: C, 43.00; H, 4.27; N, 7.43. found: C, 43.05; H, 4.31; N, 7.45. Main IR absorption bands (KBr, cm^{-1}) ν : 3137(w), 1627(vs), 1396(vs), 1280(s), 1073(m), 810(m), 770(s) and 728(s).

The compound $[\text{Co}(\text{H}_2\text{O})_6][\text{Co}(\text{pydc})_2]\text{L}^2 \cdot \text{H}_2\text{O}$ **2** was prepared by procedure similar to that employed for complex **1**, except for 3-amine-1H-1,2,4-triazole instead of 1,3-di(4-pyridyl)propane. Yield 50 %. Anal. calcd. for $\text{C}_{16}\text{H}_{36}\text{Co}_2\text{N}_6\text{O}_{15}$: C, 28.67; H, 5.41; N, 12.54. found: C, 28.72; H, 5.60; N, 12.57. Main IR absorption bands (KBr, cm^{-1}) ν : 3117(w), 1704(vs), 1619(m), 1394(m), 1280(s), 1186(s), 1257(vs), 1068(vs), 946(s), 760(m) and 683(s).

All measurements were made on a Bruker SMART APEX II CCD diffractometer equipped with a graphite-monochromatized MoK_α radiation ($\lambda = 0.071073$ nm). Brown block single crystals of complex **1** (0.24 mm \times 0.22 mm \times 0.20 mm) and **2** (0.25 mm \times 0.23 mm \times 0.20 mm) were mounted on glass fibers. Diffraction data were collected at using an ω -2 θ scan technique at room temperature. A total of 15159 reflections were collected for **1** and 13050 for **2**, of which 2753 ($R_{\text{int}} = 0.0151$) and 3099 ($R_{\text{int}} = 0.0171$) were independent for **1** and **2** in the ω -2 θ range of 2.26 \sim 28.20° and 2.12 \sim 28.21°, and 6420 and 4534 observed reflections with $I > 2\sigma(I)$ were employed for structure refinements for **1** and **2**, respectively. The structures were solved by direct methods with SHELXS-97 [24]. The hydrogen atoms were assigned with common isotropic displacement factors and included in the final refinement by use of geometrical restraints. A full-matrix least-squares refinement on F^2 was carried out using SHELXL-97 [25]. The final agreement factor values are $R = 0.0230$ and $wR = 0.0635$ ($w = 1/[s^2(F_o^2) + (0.0421P)^2 + 0.1554P]$) where $P = (F_o^2 + 2F_c^2)/3$, $S = 1.059$ for complex **1**. The final agreement factor values are $R = 0.0278$ and $wR = 0.0783$ ($w = 1/[s^2(F_o^2) + (0.0447P)^2 + 0.4124P]$) where $P = (F_o^2 + 2F_c^2)/3$, $S = 1.068$ for complex **2**. A summary of the crystallographic data and refinement parameters are listed in Table 1. Selected bond distances and bond angles are listed in Table 2 for complex **1** and in Table 3 for complex **2**.

DESCRIPTION OF CRYSTAL STRUCTURES OF **1** AND **2**

Determination of the two title compounds by X-ray crystallography showed that in the two Co(II) complexes exist ionic complexes consisting of $[\text{Co}(\text{H}_2\text{O})_n]^{2+}$ and $[\text{Co}(\text{pydc})_2]^{2-}$ units. The each cobalt atom is in a distorted octahedral coordination geometry and the ligand pydc adopts tridentate coordination mode.

The compound **1** consists of $[\text{Co}(\text{pydc})_2]^{2-}$ anion units, $[\text{CoL}^1(\text{H}_2\text{O})_4]^{2+}$ cation chain units and lattice water (Figure 1). In the each anionic $[\text{Co}(\text{pydc})_2]^{2-}$ unit, the cobalt atom is in a distorted octahedral coordination geometry with six donor atoms of two tridentate ligand of $[\text{pydc}]^{2-}$. The Co1—N bond distance is 2.0320(11) Å and the Co1—O bonds rang from 2.1479(9) to 2.1771(10) Å, with the bond angles around Co1 atom deviating from 165.72(6) up to 75.86(4)°, respectively. The values of those bond lengths and angles around Co are close to the values reported Co(II) complexes containing pydc ligand [26]. The dihedral angle of two pyridine rings of pydc is 83.29 (0.13)°, a small deviation (i.e., 6.71°) from perpendicular being observed. In each cation chain of $[\text{CoL}^1(\text{H}_2\text{O})_4]^{2+}$ unit, the Co atom is also in a distorted octahedral geometry being bound to four water molecules and two nitrogen atoms of pyridyl of 1,3-di(4-pyridyl)propane. The Co2—O (aqua) bonds vary in the range of 2.0512(10)—2.0888(11) Å. O1w and O2w occupy the axial positions around the cobalt(II) ion and are nearly perpendicular with an O1w—Co2—O2w angle of 86.14(4)°, as well as N2 also occupying axial

Table 1

Crystal Data and Structure Definition for **1** and **2**

Compound	1	2
CCDC deposit number	CCDC-614556	CCDC-614557
Empirical formula	C ₂₇ H ₃₂ Co ₂ N ₄ O ₁₄	C ₃₂ H ₃₆ Co ₃ N ₁₂ O ₂₄
Molecular weight	754.43	1149.52
Crystal system	Monoclinic	Triclinic
Space group	<i>P</i> 2/ <i>c</i>	<i>P</i> -1
<i>a</i> , <i>b</i> , <i>c</i> , nm	0.97424(6), 0.89653(6), 1.8229(11)	0.89037(18), 0.9826(2), 1.2529(3)
α , β , γ , deg.	90.00, 99.41(3), 90.00	102.40(3), 93.06(3), 90.53(3)
<i>Z</i>	2	1
<i>V</i> , nm ³	1.5707(5)	1.0689(4)
<i>D</i> _c , mg/cm ³	1.595	1.786
μ , mm ⁻¹	1.131	1.257
<i>T</i> , K	293(2)	293(2)
Crystal shape, color	Block, brown block	Block, brown block
2 θ range, deg.	2.26–28.20	2.12–28.21
<i>hkl</i> range	$-12 \leq h \leq 12, -11 \leq k \leq 11, -24 \leq l \leq 23$	$-11 \leq h \leq 11, -13 \leq k \leq 13, -16 \leq l \leq 16$
<i>F</i> (000)	776	585
Reflns measured	7025	5167
Unique reflns, <i>R</i> _{int}	6420	4534
<i>R</i> 1, <i>wR</i> 2 (<i>I</i> > 2 σ (<i>I</i>))	0.0245, 0.0695	0.0278, 0.0783
<i>R</i> 1, <i>wR</i> 2 (all data)	0.0265, 0.0705	0.0324, 0.0805
GOOF on <i>F</i> ²	1.074	1.068
Largest residual peaks and holes/e. nm ⁻³	3.55 and -2.89	6.70 and -6.11

positions with O1w—Co2—N2 angle of 90.99(15)° and O2w—Co2—N2 angle of 88.39(14)°. The dihedral angle between two pyridine rings is 1.34(0.29)° which indicates that two pyridine rings are almost coplanar.

As shown in Figure 2, there are extensive network of hydrogen bonds involving the coordinated and uncoordinated water molecules, coordinated water molecules and pydc-oxygen atoms which pro-

Table 2

Selected Bond lengths (Å) and Bond Angles (deg.) of Complex **1**

Bond	Dist	Angle	(deg.)	Angle	(deg.)
Co1—N1	2.0320(11)	N(1) ¹ —Co(1)—N(1)	165.72(6)	O(2)—Co(1)—O(3) ¹	151.37(4)
Co1—O2	2.1479(9)	N(1) ¹ —Co(1)—O(2) ¹	75.86(4)	O(3) ¹ —Co(1)—O(3)	101.66(6)
Co1—O3	2.1771(10)	N(1)—Co(1)—O(2) ¹	94.63(4)	O(2W) ² —Co(2)—O(1W)	93.86(4)
Co2—N2	2.2440(11)	O(2) ¹ —Co(1)—O(2)	97.71(6)	O(2W)—Co(2)—O(1W)	86.14(4)
Co2—O2w	2.0512(10)	N(1) ¹ —Co(1)—O(3) ¹	113.89(4)	O(2W) ² —Co(2)—N(2) ²	88.39(4)
Co2—O4w	2.095(3)	N(1)—Co(1)—O(3) ¹	75.65(4)	O(2W)—Co(2)—N(2) ²	91.61(4)
		O(2) ¹ —Co(1)—O(3) ¹	87.33(4)	O(1W)—Co(2)—N(2) ²	89.01(5)

Symmetry codes: ¹ $-x+1, y, -z+3/2$; ² $-x+1, -y+1, -z+1$.

Table 3

Selected Bond lengths (Å) and Bond Angles (deg.) of Complex 2

Bond	Dist	Angle	(deg.)	Angle	(deg.)
Co1—O1w	2.0558(15)	O1w ⁱ —Co1—O2w	88.40(7)	O8—Co2—O4	90.03(6)
Co1—O2w	2.0975(14)	O1w—Co1—O2w	91.60(7)	N2—Co2—O2	97.01(6)
Co1—O3w	2.1001(15)	O1w ⁱ —Co1—O3w	89.46(6)	N1—Co2—O2	75.94(6)
Co2—N2	2.0243(14)	O1w—Co1—O3w	90.54(6)	O8—Co2—O2	99.48(6)
Co2—N1	2.0250(14)	O2w ⁱ —Co1—O3w	93.51(6)	O4—Co2—O2	151.88(5)
Co2—O8	2.1357(15)	O2w—Co1—O3w	86.49(6)	N2—Co2—O6	74.74(6)
Co2—O4	2.1728(14)	N2—Co2—N1	172.93(5)	N1—Co2—O6	104.67(6)
Co2—O2	2.1764(14)	N2—Co2—O8	76.93(6)	O8—Co2—O6	150.71(5)
Co2—O6	2.1935(13)	N1—Co2—O8	104.36(6)	O4—Co2—O6	93.15(5)
		N2—Co2—O4	110.95(6)	O2—Co2—O6	91.27(5)
		N1—Co2—O4	76.08(6)		

Symmetry codes: ⁱ 1-x, -y, -z.

vide additional stability to the structure, and leads to infinite three-dimensional framework structure. Hydrogen-bond lengths and angles are listed in Table 4.

The structure of the compound of **2** is similar to the compound **1**, existing ion pairs complex consisting of $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ anion and $[\text{Co}(\text{pydc})_2]^{2-}$ cation units, lattice water as well as free ligand 3-amine-1H-1,2,4-triazole (Figure 3). In cationic $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ unit, the cobalt atom has a distorted octahedral environment of six water molecules, whereas in anionic $[\text{Co}(\text{pydc})_2]^{2-}$ unit the cobalt is bonded to four oxygen atoms of carboxylate and two nitrogen atoms of pyridine in an octahedral geometry. The bond lengths and angles around Co in cationic $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ rang from 2.0538(15) to 2.1001(15) Å, and vary from 88.40(7) to 93.51(6)°, respectively. Whereas the bond lengths and angles around Co atom in anionic $[\text{Co}(\text{pydc})_2]^{2-}$ are very close to those observed in the previous structure. The dihedral angle of two pyridine rings is 84.14(0.05)° which is equivalent to the compound **1** with a small deviation (i.e., 0.85°).

An extensive network of hydrogen bonds (Figure 4) involving the coordinated and uncoordinated water molecules, the free ligand triazole and lattice water and pydc-oxygen atoms, coordinated water and pydc-oxygen atoms, pydc-hydrogen atoms and adjacent pydc-oxygen atoms contribute to the stabilization of the structure, resulting in one-dimensional network structure. Hydrogen-bond lengths and angles are listed in Table 5.

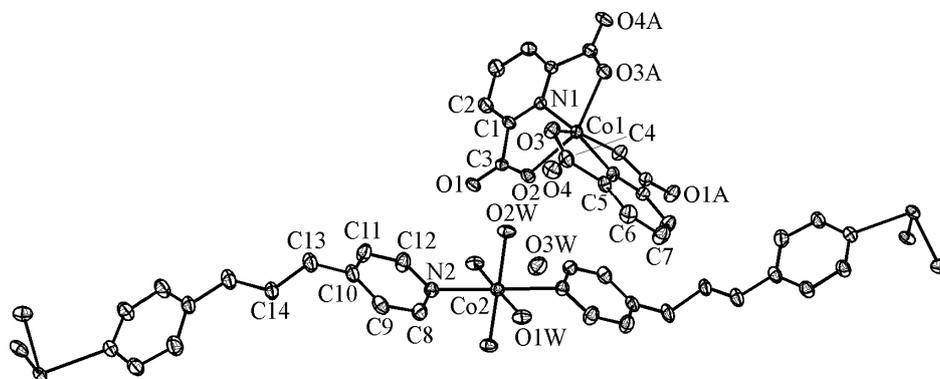


Fig. 1. The perspective view of $[\text{CoL}^1(\text{H}_2\text{O})_4][\text{Co}(\text{pydc})_2] \cdot 2\text{H}_2\text{O}$ with 30 % thermal ellipsoids, where hydrogen atoms are omitted for clarity

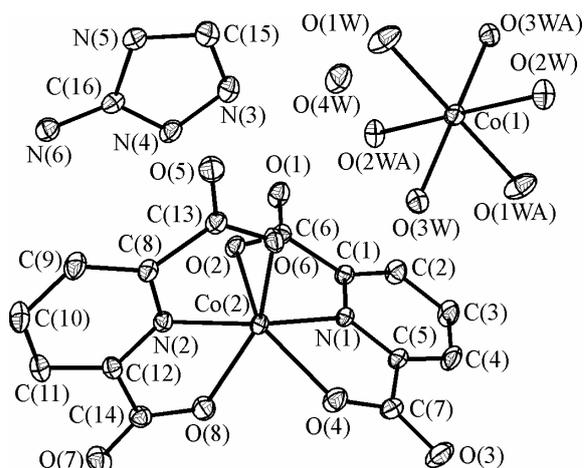
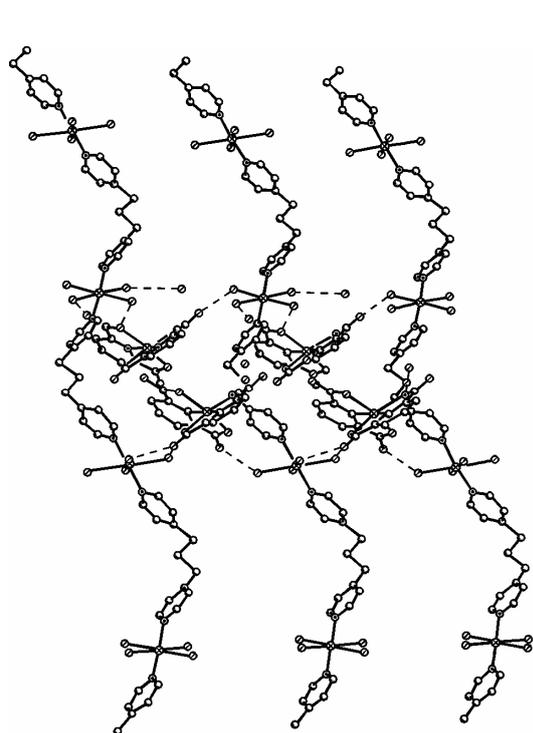


Fig. 2 (left). View of the crystal cell and hydrogen bonds of the crystal structure of $[\text{CoL}^1(\text{H}_2\text{O})_4] \times [\text{Co}(\text{pydc})_2] \cdot 2\text{H}_2\text{O}$ (shown broken lines)

Fig. 3 (right). The perspective view of $[\text{Co}(\text{H}_2\text{O})_6] \times [\text{Co}(\text{pydc})_2]\text{L}^2 \cdot \text{H}_2\text{O}$ with 30% thermal ellipsoids, where hydrogen atoms are omitted for clarity

Table 4

Hydrogen-bond lengths (\AA) and angles ($^\circ$) for structure 1

D—H...A	$d(\text{H}\dots\text{A})$	$d(\text{D}\dots\text{A})$	$\angle(\text{DHA})$	D—H...A	$d(\text{H}\dots\text{A})$	$d(\text{D}\dots\text{A})$	$\angle(\text{DHA})$
O3w—H1...O4 ¹	2.17(2)	3.0168(18)	170(3)	O2w—H2Aw...O2 ⁴	1.831(11)	2.6716(13)	168.6(17)
O1w—H1Aw...O1 ²	1.829(17)	2.6737(15)	171.4(17)	O1w—H1Bw...O3W	1.929(11)	2.7746(17)	173.8(18)
O3w—H2...O3 ³	1.98(2)	2.794(7)	157(2)	O2w—H2Bw...O4 ³	1.882(17)	2.7303(15)	174.6(18)

Symmetry codes: ¹ $1-x, 1-y, -z$; ² $1-x, y, 0.5-z$; ³ $+x, 1+y, +z$; ⁴ $+x, 1-y, -0.5+z$.

THERMOGRAVIMETRIC ANALYSIS

A TGA diagram of complex **1** shows three major weight losses. The first step is the loss of two lattice water molecules, completed at 153.4 °C. The weight loss of 4.61% (Cal. 4.77%). The second step is the loss of coordinated water molecules, completed at 200 °C. The weight loss of 9.60% (Cal. 9.54%). The third step corresponds to the burning of the organic group, completed at about 403 °C. Above this temperature the compound **1** continue to decompose in the continuous fashion. Complex **2** is stable up to 272 °C. The TGA diagram for complex **2** shows two step of major weight loss. The first step is the loss of lattice water, coordinated water molecules and free or-

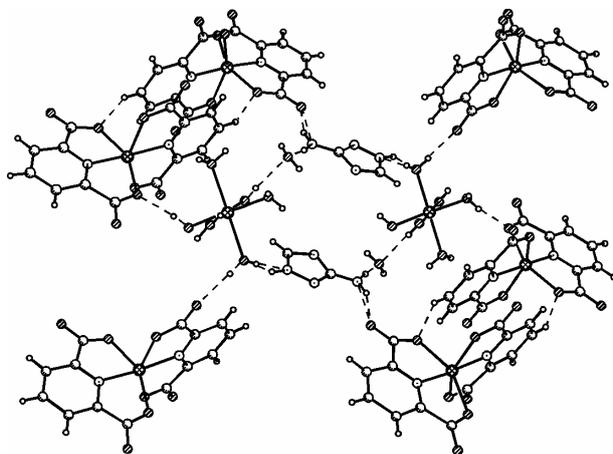


Fig. 4. View of the crystal cell and hydrogen bonds of the crystal structure of $[\text{Co}(\text{H}_2\text{O})_6][\text{Co}(\text{pydc})_2]\text{L}^2 \cdot \text{H}_2\text{O}$ (broken lines)

Table 5

Hydrogen-bond lengths (Å) and angles (deg.) for structure 2

D—H...A	d(H...A)	d(D...A)	∠(DHA)
C3—H3A...O8 ¹	2.5028	3.3216 (0.0024)	147.00
C11—H11A...O2 ²	2.6624 (0.0220)	3.5683 (0.0025)	161.38 (1.71)
O3w—H5...O3 ³	2.0464 (0.0290)	2.8023 (0.0021)	157.24 (2.79)
N3—H3B...O2w ⁴	2.1466	2.9515 (0.0022)	155.65
O4w—H7...O5	2.0542 (0.0228)	2.7988 (0.0022)	157.25 (3.15)
O2w—H4...O1 ⁵	1.9667	2.7865 (0.0021)	178.46
O1w—H1...O4w	1.9622 (0.0299)	2.6929 (0.0024)	162.98 (3.03)
O3w—H6...O6	1.8563	2.6663 (0.0019)	169.24
O1w—H2...O3 ⁶	1.8547	2.6696 (0.0023)	172.35
C2—H2A...O2w ⁷	2.9777	3.8209 (0.0026)	151.50

Symmetry codes: ¹ 2-x, -1-y, -z; ² 2-x, -1-y, -1-z; ³ 1-x, -1-y, -z; ⁴ 1-x, -y, -z; ⁵ x-1, +y, +z; ⁶ +x, 1+y, +z; ⁷ 1+x, +y, +z.

ganic ligand, completed at about 272 °C. The weight loss of 37.74 % (Cal. 37.62 %). Up to 416 °C, the second step corresponds to the burning of the organic group. Above this temperature the compound **2** continue to discompose in the continuous fashion.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis of **1** and **2** have been deposited at the Cambridge Crystallographic Data Centre, CCDC Nos. 614556 for **1**, 614557 for **2**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EQ, UK (fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk).

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