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Journal of Molecular Structure 733 (2005) 119-124

Journal of MOLECULAR STRUCTURE

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An ion-pair compound based on [Pd(mnt)₂]⁻ with strong antiferromagnetically coupled interaction: synthesis, crystal structure, magnetic property

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> Received 29 May 2004; revised 13 August 2004; accepted 16 August 2004 Available online 22 September 2004

Abstract

An ion-pair compound, which constructs from the 1-(4'-F-benzyl)pyridinium with Λ -shaped (abbreviated as [FBzPy]⁺) and planar bis(maleonitrileditholato)palladium(III) (abbreviated as [Pd(mnt)₂]⁻), has been synthesized and structurally characterized. The cations and anions in this compound stack into well-separated columns in the solid state, the adjacent anions exist the strong trend of dimerization within an anionic column, and the contacts between the neighbor Pd(III) ions alternate as 3.39 and 4.06 Å, respectively. The weak $\pi \cdots \pi$ stacking interactions were observed between adjacent benzene rings within a cationic column. The temperature dependence of magnetic susceptibility measured in the temperature range of 1.8–330 K; indicates this compound is almost diamagnetic. The magnetic susceptibility was successfully modeled by a spin dimer magnetic exchange scheme with $s = \frac{1}{2}$ to give rise to g = 2.1, and the energy gap between singlet ground state and triplet excited state is about 1310 K.

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Keywords: Bis(maleonitrileditholato)palladium(III) compound; Crystal structure; Magnetic property

1. Introduction

Recently, lots of research interests have been paying attentions to the low-dimensional spin systems [1–5] due to more and more new quantum effects were found, such as, slow relaxation of magnetization and hysteresis effects which are not associated with 3-D ordering [6a–d], distinct plateaus in the magnetization [6e], these findings also stimulated theoretical investigations in the low-dimensional spin systems [7].

It is possible that the molecular building blocks with planar extended electronic structure, $[M(mnt)_2]^-$

(M represents Ni(III), Pd(III) and Pt(III) ions; mnt^{2-} is maleonitriledithiolate), were constructed into the columnar stack structures, in which intermolecular $d_{r}2$ or π orbital interactions give rise to 1-D electronic nature of these types of compounds [8-11]. To build quasi-one-dimensional molecular magnets based on the blacks of $[M(mnt)_2]^-$, the key problem had to be solved is how to control $[M(mnt)_2]^-$ anions stacking into the columns. It is noted that the counter cationic topology and size in $[M(mnt)_2]^{-1}$ compounds play an important role in controlling the stacking pattern of anions and cations, for instance, the counter cations with bigger molecular size and approximate sphere shape lead to form isolated dimers of anions, while the planar cations induce to form the mixed stacking columns with alternative overlapping of anions and cations. In our current researches, the benzylpyridinium derivatives ([RBzPy]⁺) with Λ -shaped molecular configuration were selected as the counter cations for [Ni(mnt)₂]⁻

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^{0022-2860/\$ -} see front matter © 2004 Elsevier B.V. All rights reserved. doi:10.1016/j.molstruc.2004.08.009



and $[Pt(mnt)_2]^-$ anions, respectively. A series of ion-pair compounds (Scheme1) with quasi-one-dimensional structural feature, which exhibit magnetic diversity, were prepared by using this strategy. The investigations revealed also the nature of both the substituent groups in the benzylpyridinium derivatives and metal ions influence on the overlap patterns of the anions within an anionic column, and further tune the magnetic properties of these types of compounds [12]. To extend our research and understand the relationship between the magnetic properties of these types of compounds and their stacking structures, we are pursuing the study of the $[RBzPy]^+[Pd(mnt)_2]^-$, and $[Pd(mnt)_2]^-$ anion is similar to both $[Ni(mnt)_2]^-$ and $[Pt(mnt)_2]^-$ anions in the aspects of the molecular structure and the electronic configuration. Herein we report synthesis, crystal structure, magnetic property for the compound, [FBzPy][Pd(mnt)₂].

2. Experiments

2.1. Chemicals and reagents

All manipulations were carried out in air unless otherwise noted. All chemicals and solvents were reagent grade, and were used without further purification. 4-fluorobenzyl bromide is the product of Synica Chemicals, 1-(4'-fluorobenzyl)pyridinium bromide ([FBzPy]Br), Disodium maleonitriledithiolate (Na₂mnt) were synthesized following the published procedures [13].

2.2. Physical measurements

Elemental analyses were performed with a Perkin-Elmer 240 analytical instrument. IR spectra were recorded on a Fourier Transform Infrared Spectrometer (170SX) (KBr pellet). Magnetic susceptibility data on powder-sample were collected over the temperature range of 1.8–330 K using a Quantum Design MPMS7 superconducting quantum interference device (SQUID) magnetometer.

2.3. Synthesis of $[FBzPy]_2[Pd(mnt)_2](1)$

This compound was prepared by the direct combination of 1:2:2 mol equiv. of PdCl₂, Na₂mnt and [FBzPy]Br in warm solution of water (~50 °C). A green precipitate given was filtered off, washed by water and dried under vacuum. Yield: 86% (Found: C, 50.3; H, 2.96; N, 10.8%. Calc. for $C_{32}H_{22}F_{2}N_{6}PdS_{4}$: C, 50.4; H, 2.91; N, 11.0%). IR spectra data (cm⁻¹): ν (CN) of mnt²⁻ 2196.5(vs) and 2229(sh).

2.4. Synthesis of $[FBzPy][Pd(mnt)_2]$ (2)

A MeCN solution (10 cm^3) of I₂ (205 mg, 0.80 mmol) was slowly added to a MeCN solution (25 cm^3) of [FBzPy]₂[Pd(mnt)₂] (870 mg, 1.0 mmol), and stirred for 25 min. MeOH (150 cm³) was then added, and the mixture allowed to stand overnight. The microcrystals formed were filtered off, washed with MeOH and dried in vacuum. Yield: 76% (Found: C, 41.6; H, 1.95; N, 11.9%. Calc. for C₂₀H₁₁FN₅PdS₄: C, 41.8; H, 1.93; N, 12.2%). IR(cm⁻¹): ν (CN) of mnt²⁻ 2201 (sh) and 2214.8(vs).

The single crystals suitable for X-ray analysis was obtained by dispersing Et_2O into MeCN solution of **2** about 4 days.

2.5. X-ray crystallography

Diffraction data of **2** were collected at 293 K on a CCD area detector (Bruker-SMART) equipped with graphite-monochromated Mo–K α radiation. All computations were carried out in a PC-586 computer using the SHELXTL-PC

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Τa

Summary of crystal and structure refinement data for 2

Empirical formula	$C_{20}H_{10}FN_5PdS_4$
Formula weight	574.98
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystalline system	Monoclinic
Space group	$P2_{1}/c$
<i>a</i> (Å)	26.021(7)
<i>b</i> (Å)	12.607(3)
<i>c</i> (Å)	14.451(3)
β (deg)	103.42(2)
$V(Å^3), Z$	4611.3(19), 8
Density (calc) (mg/m ³)	1.656
Abs coeff. (mm^{-1})	1.193
F(000)	2280
θ range for data collection	1.80-25.04
Index ranges	$-21 \le h \le 30, -15 \le k \le 13,$
	$-17 \le l \le 16$
Reflections collected	8090
Independent refins	$6013 [R_{int} = 0.0322]$
Refinement method on F^2	Full-matrix least-squares
Data/restraints/params	6013/0/559
Goodness-of-fit on F^2	0.963
Final <i>R</i> indices $[I > 2\sigma(I)]$	R1 = 0.0348, wR2 = 0.0813
<i>R</i> indices (all data)	R1 = 0.0521, wR2 = 0.0897
Largest diff peak and hole, (e $Å^{-3}$)	0.37 and -0.41

program package [14]. The structure of **2** was solved by direct method and refined on F^2 by full-matrix least-squares method. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in their calculated positions and refined following the riding model. Crystallographic details for **2** are summarized in Table 1.

3. Results and discussion

3.1. Descriptions of crystal structure

An ORTEP drawing of **2** in an asymmetric unit is shown in Fig. 1a. Each Pd(III) ion in two crystallographic independence $[Pd(mnt)_2]^-$ moieties is coordinated by four sulfur atoms, and exhibit square-planar coordination geometry. The least-square planes with four sulfur atoms were calculated for each of $[Pd(mnt)_2]^-$ moieties, the results are summarized in Table 2. The average S-Pd-S bond angle within the chelated ring is 90.09° for the moiety of Pd(1)-contained (89.77° for the moiety of Pd(2)contained), and the Pd-S bond distances are from 2.2637(10) to 2.2783(10) Å for the entry of Pd(1)-contained (from 2.2618(10) to 2.2786(11) Å for the entry of Pd(2)contained), these values are in agreement with that of $[Pd(mnt)_2]^-$ complexes reported [15]. The CN groups of mnt² ligand are out of the coordinating plane as the serials of Ni(III)- and Pt(III)-based complexes with the deviations of 0.25 Å of N(1), 0.14 Å of N(2), 0.04 Å of N(3) and 0.21 Å of N(4) in the moiety of Pd(1)-contained, respectively (0.32 Å of N(1), 0.30 Å of N(2), 0.36 Å of N(3) and 0.19 Å of N(4) in the moiety of Pd(2)-contained). The two crystallographic independence [Pd(mnt)2]⁻ moieties are almost parallel to each other (the angle between the two coordination planes is only 2.2°). The bond lengths and angles of the two crystallographic independence [FBzPy]⁺



(b)

Fig. 1. (a) ORTEP view of 2 with 30% probability thermal ellipsoids. (b) Completely separated columnar stacking of 2 along the *c*-axis.

Table 2 Details of molecular planes (x, y, z are fractional atomic coordinates)

Distances from the plane (Å)	
-0.0052^{a} (4) S(1)	0.0068^{a} (5) S(5)
0.0050 ^a (4) S(2)	-0.0068^{a} (5) S(6)
0.0052 ^a (4) S(3)	-0.0068^{a} (5) S(7)
-0.0050^{a} (4) S(4)	0.0068^{a} (5) S(8)
0.0001 (5) P(d1)	0.0172 (5) Pd(2)
-0.2459 (41) N(1)	-0.3239 (47) N(5)
-0.1399 (42) N(2)	-0.2994 (48) N(6)
0.0391 (45) N(3)	-0.3628 (49) N(7)
-0.2124 (40) N(4)	-0.1949 (44) N(8)
-0.1562 (39) C(1)	-0.2177 (45) C(21)
-0.0583 (34) C(2)	-0.1006 (38) C(22)
-0.0519 (32) C(3)	-0.0947 (35) C(23)
-0.1098 (40) C(4)	-0.2053 (44) C(24)
-0.0121 (41) C(5)	-0.2329 (44) C(25)
-0.0240 (33) C(6)	-0.1049 (35) C(26)
-0.0517 (36) C(7)	-0.0683 (34) C(27)
-0.1445 (42) C(8)	-0.1402 (41) C(28)

Equation of the plane for the entry of Pd(1): 11.6673(81)x - 0.1797(36)y - 14.0667(57)z = 0.8432(19). Rms deviation of fitted atoms = 0.0051. Equation of the plane for the entry of Pd(2): 11.8896(82)x + 0.2909(37)y - 14.0318(57)z = 0.4558(56). Rms deviation of fitted atoms = 0.0068. ^a Indicates atom used to define plane.

cations are generally in close agreement with values reported for the complex [FBzPy] [Ni(mnt)₂] [12b]. Two cations show Λ -shaped molecular configuration, the dihedral angles between benzene, pyridine rings and the reference plane, C(15)–C(14)–N(5) are 112.5 and 77.0° for the entry of F(1)-contained, respectively, (corresponding dihedral angles of 119.4 and 112.2° for the entry of F(2)-contained).

The anionic and cationic columns with separated-well stack along the direction of crystallographic c-axis as displayed in Fig. 1b. The adjacent $[Pd(mnt)_2]^-$ anions exhibit two types of overlapping patterns within an anionic column, the pattern with eclipsed plane over plane is labeled as pattern A (Fig. 2a) and two superimposed monomers rotated to each other as pattern B (Fig. 2b). Two $[Pd(mnt)_2]^-$ anions eclipse to lead the inter-plane (defined by four coordinated sulfur atoms) distance (3.39 Å) is almost identical to the separation of Pd···Pd, and the adjacent S...S overlapping pairs exist very shorter separations $(3.40 \text{ Å of } S(1)...S(5)^i, 3.38 \text{ Å of } S(2)...S(6)^i, 3.45 \text{ Å}$ of $S(3) \cdots S(7)^i$ and 3.43 Å of $S(4) \cdots S(8)^i$, symmetric code i=1-x, 0.5+y, 0.5-z in pattern A. The corresponding plane-to-plane distance is 3.64 Å, the separation of Pd···Pd is 4.06 Å, and the nearest S…S, S…Pd contacts are of 3.60 and 3.73 Å in Pattern B. In a cationic column, the adjacent cations stack in a boat-type conformation-like, which is shown in Fig. 3. The dihedral angles between the overlapping benzene rings are same as 8.6°, the corresponding center-to-center distances between the face-to-face stacking of aromatic rings are 4.01 Å and 3.97 Å, respectively, and too long to be of energetic significance [16].



Fig. 2. Patterns of overlapping of $[Pd(mnt)_2]^-$ anions in **2** (a) side view (b) top view.

3.2. Magnetic property of 2

The temperature dependence of magnetic susceptibility for **2** is shown in Fig. 4, which indicates very weak paramagnetic property in temperature range of 1.8-330 K. The magnetic susceptibilities in the temperature range of 30-330 K are negative, and exhibit weak paramagnetic Curie tail below 30 K, so weak paramagnetic property maybe arises from the magnetic impurities of the uncoupled spins, that is, this compound actually is almost diamagnetic.



Fig. 3. Stacking pattern with boat-conformation-like for the cations in 2.

The magnetic exchange scheme of a spin dimer with $s = \frac{1}{2}$ can be used to model the magnetic susceptibility for **2** based on the structural analysis, and the function of the magnetic susceptibility as temperature is described as the Eq. (1).

$$\chi_m = Ng^2 \mu_{\rm B}^2 / [k_{\rm B}T(3 + \exp(\Delta/k_{\rm B}T))] + C/T + \chi_0$$
(1)

where the first term contributes from the susceptibility of a spin dimer with $S = \frac{1}{2}$, Δ is the energy difference between triplet and singlet, *C* is Curie constant for the uncoupled paramagnetic impurity, and χ_0 is the sum of both core diamagnetic and temperature independent paramagnetic contributions, and other symbols have normal meanings. The fitting successfully give rise to the results of g=2.1, $\Delta/k_{\rm B}=1310$ K, $C=6.5\times10^{-3}$ emu K mol⁻¹ (the fraction



Fig. 4. The variety of magnetic susceptibility versus temperature (1.8–330 K) (circle is experimental data, and solid line is reproduced by calculation with the parameters of g=2.1, $\Delta/k_B=1310$ K, $C=6.5\times10^{-3}$ emu K mol⁻¹, $\chi_0=-2.0\times10^{-4}$ emu mol⁻¹).

of the paramagnetic impurity deduced from C value is ~ 1.7%), $\chi_0 = -2.0 \times 10^{-4}$ emu mol⁻¹, and the agreement factor is 3.2×10^{-6} .

4. Conclusion and remarks

In summary, an ion-pair compound, 1-(4'-F-benzyl)pyridinium bis(maleonitriledithiolato)palladium(III), has been synthesized. The crystal structural determination at room temperature revealed that the anions and cations in this compound stack into separated-well columns, which is similar to the compounds of both $[Ni(mnt)_2]^-$ and $[Pt(mnt)_2]^-$ series. Two types of anionic overlapping patterns within an [Pd(mnt)₂]⁻ column were found, one is two neighboring anions eclipses plane over plane (Pattern A) and another is two adjacent superimposed monomers rotates to each other (Pattern B), so the anionic column is dimerized. By comparison, the stacking columns of $[Ni(mnt)_2]^-$ anions in these series of ion-pair compounds are usually uniform, while the stacking columns of both $[Pd(mnt)_2]^-$ and $[Pt(mnt)_2]^-$ anions favor to be dimerized. These findings indicate the factors effecting on the stack structure in the types of [RBzPy]⁺[M(mnt)₂]⁻ compounds are not only from the cationic molecular configurations, but also from the anionic structural properties (such as the ionic radii of M, etc). The DC magnetic susceptibility measured in the temperature range of 1.8–330 K shown the compound 2 is almost diamagnetic. The magnetic susceptibility was modeled by using a spin dimer with $s = \frac{1}{2}$ to give the large energy gap of ca. 1310 K between singlet ground state and triplet excited state, which lead to the compound is nearly diamagnetic. Presently, We have been studying the control of the $[M(mnt)_2]^-$ (M=Ni, Pd or Pt) arrangements by introduced the type of RBzPy⁺ counter-cation with tunable molecular geometry. Although the trend that the influence of the substituent R- of the RBzPy⁺ cation on the anionic stacking structure and magnetic properties of the $[RBzPy][M(mnt)_2]$ compounds for both $[Pd(mnt)_2]^-$ and [Pt(mnt)₂]⁻ series are not extremely clear due to the lack of some crystal structural and magnetic data, some finding in [RBzPy][Ni(mnt)₂] compounds are significant, for example, the separations between adjacent Ni. Ni within an anionic column in those compounds decrease progressively as the order of the substituent R-=F-, Cl-, Br-, I- and NO₂- $(\approx$ CN–), each compound of them undergo a phase transition from the paramagnetic to the diamagnetic phase, and the corresponding phase transition temperature increase by degrees in the sequence of the substituent R = F, Cl-, Br-, I- and NO₂- (\approx CN-) [12,17]. In order to gain more information about the relationship of substituent effect, anionic stacking structure and magnetic property of these $[RBzPy][M(mnt)_2]$ compounds, the preparation of the new compounds, the determination of crystal structures of them, the investigation of their magnetic properties and the relevant theoretic analyses are in progress.

5. Supplemental materials

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publications, and the CCDC numbers is CCDC-208361. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44 1223 336 033; Email: deposit@ ccdc.cam.ac.uk).

Acknowledgements

The authors thank Ms E. Brücher for the magnetic property determinations. This project was financially supported from the National Natural Science Foundation of China (No. 29831010, 20171001 and 20371002), and one of authors (X. M. Ren) acknowledges Max-Planck Society for financial support.

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