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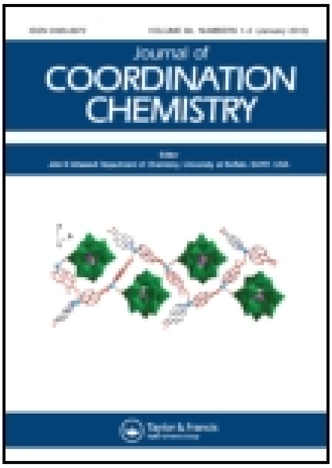

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Synthesis, X-ray studies, and catalytic activity of tridentate Schiff base dioxo-molybdenum(VI)

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Synthesis, X-ray studies, and catalytic activity of tridentate Schiff base dioxo-molybdenum(VI)

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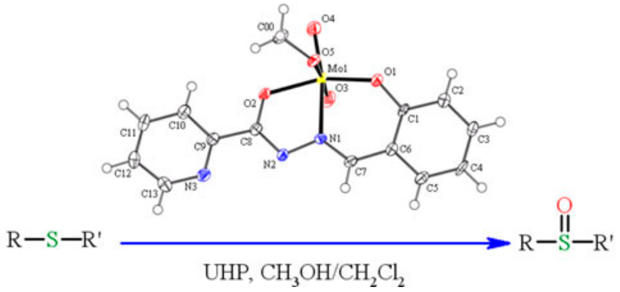

The reaction of a solution of $MoO_2(acac)_2$ in CH_3OH and salicylidene 2-picoloyl hydrazone as a tridentate ONO donor Schiff base (ONO) afford a six-coordinated Mo(VI) complex $[MoO_2(ONO)(CH_3OH)]$, with a distorted octahedral configuration. $[MoO_2(ONO)(CH_3OH)]$ was isolated as an air-stable crystalline solid and fully characterized by single-crystal X-ray structure analysis. $[MoO_2(ONO)(CH_3OH)]$ shows reactivity in the oxidation of sulfides to their corresponding sulfoxides using urea hydrogen peroxide as the oxidant at room temperature under air.

Keywords: Dioxo-Mo(VI) complex; Schiff base; Crystal structure; Sulfide oxidation

1. Introduction

Organic sulfoxides are important compounds to synthesize various chemically and biologically significant molecules [1–5]. Oxidation of sulfides is a common method for preparing sulfoxides [6–9]. There are several reagents for this conversion, but many have the problem of over oxidation of sulfoxides to sulfones. Different solvents, oxidants, and catalysts are used in improving the procedure [10–12]. The use of transition metal complexes as catalytic

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*Corresponding authors. Email: mamini@maragheh.ac.ir (M. Amini); dboghaei@sharif.edu (D.M. Boghaei) system is based on readily available, inexpensive, and environmentally benign oxidants like hydrogen peroxide is very attractive [13–15].

Metal complexes with Schiff bases as ligands play an important part in coordination chemistry as a whole, and there have been many reports on their applications in homogeneous and heterogeneous catalysis [16, 17]. Among various transition metal complexes, Schiff basedioxo-Mo(VI) complexes are one of the most efficient catalysts for oxidation reactions [18–21].

Recently, we used Schiff basedioxo-Mo(VI) complexes as efficient catalysts for oxidation of olefins and sulfides [22, 23]. Here, we describe the synthesis of a new dioxo-Mo(VI) complex [$MoO_2(ONO)(CH_3OH)$] ((ONO) = salicylidene 2-picoloyl hydrazone) and the use of this complex as a catalyst in oxidation of sulfides in the presence of urea hydrogen peroxide (UHP) as an oxidant at room temperature under air (scheme 1).

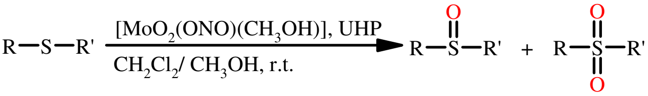
2. Experimental

2.1. *General procedure*

Chemicals and solvents were purchased from Fluka and Merck Chemical companies. Salicylidene 2-picoloyl hydrazone (ONO) and dioxo-molybdenum acetylacetonate, $MoO_2(acac)_2$, were synthesized according to published procedures [22, 24]. The elemental analysis (carbon, hydrogen, and nitrogen) of the compound was obtained from a Carlo ERBA Model EA 1108 analyzer. FT-IR spectrum was obtained by using a Unicam Matson 1000 FT-IR spectrophotometer using KBr disks at room temperature. ^{1}H spectra of the free ligand and complex in DMSO-d_6 were obtained using a Bruker FT-NMR 500 MHZ spectrometer. The products of oxidation reactions were determined and analyzed by an HP Agilent 6890 gas chromatograph equipped with a HP-5 capillary column and a flame-ionization detector. Crystal evaluation and data collection were performed on a Bruker APEX diffractometer (Mo Kα radiation, $\lambda = 0.71073$ Å).

2.2. *Synthesis of [$MoO_2(ONO)(CH_3OH)$]*

To a solution of $MoO_2(acac)_2$ (1.00 mM) in 10 mL of methanol was added a solution of salicylidene 2-picoloyl hydrazone (ONO) (1.00 mM) in 10 mL of methanol. The above solution gave a yellow solid when stirred. The solid was filtered off, washed with methanol, and dried. Crystals of the complex were obtained by diffusion of methanol into a solution of the complex in DMSO. Yield: 0.311 g, 78%. Anal. Calcd for $C_{14}H_{13}MoN_3O_5$: C, 42.12; H, 3.28; N, 10.52. Found: C, 41.89; H, 3.17; N, 10.60.



Scheme 1. The oxidation of sulfides by the [$MoO_2(ONO)(CH_3OH)$]/UHP catalytic system.

2.3. *General procedure for sulfide oxidation*

To a solution of sulfide (0.2 mM), chlorobenzene (0.2 mM) as internal standard, and [MoO_2(ONO)(CH_3OH)] (0.01 mM) in a 1 : 1 mixture of CH_3OH/CH_2Cl_2 (1 mL) was added 0.2 mM UHP as an oxidant. The mixture was stirred at room temperature and the reaction progress was monitored by GC. Assignments of products were made by comparison with authentic samples.

3. Results and discussion

3.1. *Complex characterization*

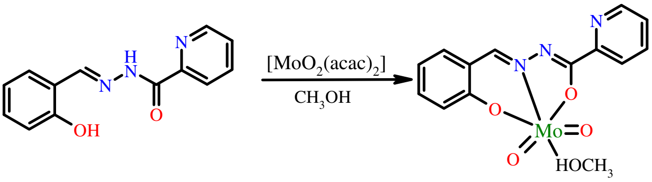
The complex was obtained by reaction of 1 equiv. of salicylidene 2-picoloyl hydrazone and $MoO_2(acac)_2$ (scheme 2). The fast color change from a pale yellow to a yellow-orange solution during the synthesis indicated the coordination of ligand. The new tridentate Schiff base dioxo-molybdenum(VI) complex is yellow, stable to air and light, and soluble in DMF and DMSO, but has low solubility in methanol, ethanol, and acetonitrile.

Elemental analysis and spectral data confirm the assigned composition of [MoO_2(ONO)(CH_3OH)]. IR spectra of the complex clearly showed the coordination of the ligands, as exemplified by the shift of the absorption band assigned to C=N bonds from 1621 (free ligand) to 1607 cm^{-1} (Mo complex) [25, 26]. Also, the $\nu_{(Mo=O)}$ bands of complex are at 916 and 981 cm^{-1} [22].

Molar conductivities of ligand and its complex in DMSO were 3.7 and 2.3 $\Omega^{-1}cm^2 M^{-1}$, respectively. These results show that the ligand coordinates to the metal ions to form neutral coordination compound. Their molar conductances are much smaller than those of 1 : 1 electrolytes [27], thus they are all considered to be nonelectrolytes.

^1H NMR spectrum of the complex has been recorded in d_6-DMSO solution. A sharp singlet appeared for OH proton of phenolic group of the ligand in the region $\delta = 11.49$ ppm is absent in the complex, and also a significant downfield shift ($\Delta\delta = 0.71$ ppm) of the signal for the azomethine (–CH=N–) proton in the complex relative to the corresponding ligand demonstrates the coordination of ligand through oxygen of the deprotonated phenolic group and azomethine nitrogen [28]. The complex shows multiplets at δ 6.88–8.73 ppm due to the aromatic protons. The coordinated methanol molecule displayed two signals at around $\delta = 3.15$ and 4.08 ppm in the ^1H NMR spectrum of complex [26].

To confirm the coordination mode of salicylidene 2-picoloyl hydrazone in this complex as well as the stereochemistry of the complex, the structure of the complex has been determined by X-ray crystallography, and an ORTEP view of the complex is shown in figure 1.



Scheme 2. Synthesis of [MoO_2(ONO)(CH_3OH)].

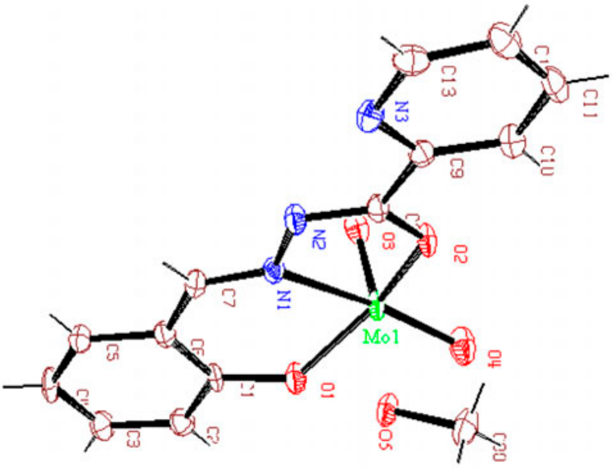


Figure 1. ORTEP diagram of [MoO_2(ONO)(CH_3OH)] with thermal ellipsoids drawn at a 50% probability limit.

The crystal lattice is stabilized by intermolecular hydrogen bonds O–H···N between hydrogen of coordinated methanol molecule and the aromatic N of Schiff base ligand belonging to the two neighboring complexes, to form an infinite 1-D polymeric array along the *a* axis (figure 2).

The summary of single-crystal X-ray structure refinement is shown in table 1. The complex crystallizes in the monoclinic crystal system and space group *P121/n1*. The structure of the complex is mononuclear, consisting of discrete monomeric units of [MoO_2(ONO)(CH_3OH)], with a distorted octahedral arrangement. The structure shows that the ligand is

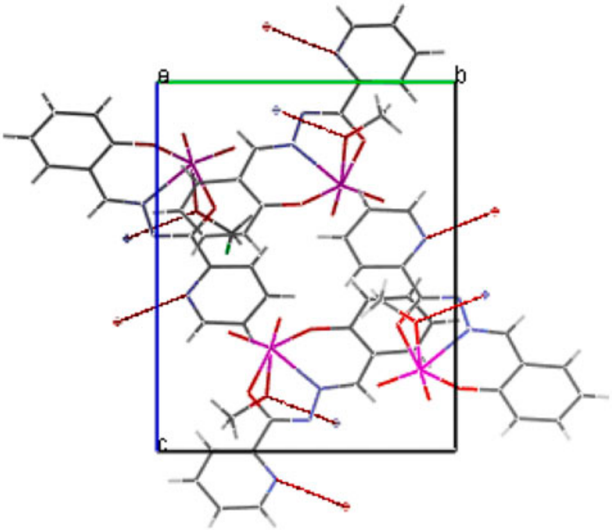


Figure 2. Depicting weak intermolecular O–H···N interactions of the 1-D polymeric array along the *a* axis for [MoO_2(ONO)(CH_3OH)].

Table 1. Crystal data and structure refinement for [MoO_2(ONO)(CH_3OH)].

	MoO_2(ONO)(CH_3OH)
Empirical formula	$C_{14}H_{12}MoN_3O_5$
Formula weight	398.21
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	*P1 21/n 1*
Unit cell dimensions	a = 11.477(3) Å
	α = 90°
	b = 10.124(3) Å
	β = 108.505(5)°
	c = 13.213(4) Å
	γ = 90°
Volume	1455.9(7) $Å^3$
Z	4
Density (calculated)	1.817 g/cm^3
Absorption coefficient	0.931 mm^{-1}
F (0 0 0)	796
Crystal size	0.080 × 0.140 × 0.200 mm
Theta range for data collection	2.05°–28.70°
Index ranges	$-15 \le h \le 15$, $-13 \le k \le 13$, $-17 \le l \le 17$
Reflections collected	19,814
Independent reflections	6374 [R(int) = 0.0383]
Completeness to theta = 28.70°	99.7%
Absorption correction	Multi-scan
Max. and min. transmission	0.8920 and 0.7850
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3761/0/209
Goodness-of-fit on F^2	1.083
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0573, wR_2 = 0.1431
R indices (all data)	R_1 = 0.0788, wR_2 = 0.1554
Largest diff. peak and hole	1.814 and −1.761 e $Å^{-3}$

Notes: $R_{int} = \Sigma|F_o^2 - F_o^2(\text{mean})|/\Sigma[F_o^2]$. $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. GOOF = $S = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$. $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $w = 1/[\sigma(F_o^2) + (aP)^2 + bP]$ where P is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$.

coordinated to molybdenum in the expected tridentate fashion through O, N, and O, in addition to two *cis*-oxido groups and one methanol. The ligand binds the metal center at O, N, and O forming five-membered and six-membered chelate rings with a bite angle of O(1)–Mo(1)–N(1) = 81.53(15)° and O(2)–Mo(1)–N(1) = 71.70(14)° and a bond length of Mo(1)–O(1) = 1.925(3) (Å), Mo(1)–O(2) = 2.005(3) (Å), Mo(1)–O(3) = 1.686(4) (Å), Mo (1)–O(4) = 1.699(4) (Å), Mo(1)–O(5) = 2.320(4) (Å) and C(8)–N(2) = 1.293(6) (Å).

3.2. *Catalytic activity*

In order to evaluate the catalytic activities of [MoO_2(ONO)(CH_3OH)] for the oxidation of sulfides, the reactions were optimized according to the oxidation of methylphenyl sulfide (MPS) through investigation of the influence of solvent, the amount of the catalyst, and the amount of the UHP.

To find the best solvent for sulfide oxidation, dichloromethane, chloroform, acetonitrile, acetone, methanol, and 1 : 1 mixture of CH_3OH/CH_2Cl_2 were employed as solvents. Among the solvents examined, 1 : 1 mixture of CH_3OH/CH_2Cl_2 was the best for this protocol (table 2). The effect of amount of catalyst on the conversion and the selectivity of the MPS oxidation at

Table 2. The effect of various conditions in the oxidation of MPS by $[MoO_2(ONO)(CH_3OH)]$/UHP.

Entry	Amount of catalyst (mM)	Amount of UHP (mM)	Solvent (1 mL)	Conversion (%)[a]	Selectivity to sulfoxide (%)[b]
1	0.01	0.4	CH_2Cl_2	33	100
2	0.01	0.4	$CHCl_3$	28	100
3	0.01	0.4	CH_3CN	66	99
4	0.01	0.4	CH_3OH	74	98
5	0.01	0.4	CH_3COCH_3	54	100
6	0.01	0.4	CH_2Cl_2 : CH_3OH	100	51
7	0	0.4	CH_2Cl_2 : CH_3OH	Trace	–
8	0.005	0.4	CH_2Cl_2 : CH_3OH	88	96
9	0. 015	0.4	CH_2Cl_2 : CH_3OH	100	46
10	0.01	0.1	CH_2Cl_2 : CH_3OH	65	99
11	0.01	0.2	CH_2Cl_2 : CH_3OH	97	94
12	0.01	0.3	CH_2Cl_2 : CH_3OH	100	68

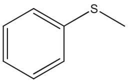
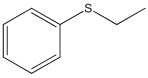
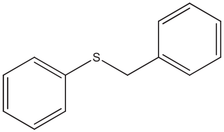
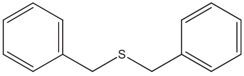
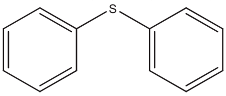
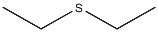
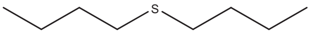
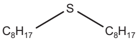
[a] TON = (mM of sulfoxide + mM of sulfone)/mM of catalyst.
[b] Selectivity to sulfoxide = (sulfoxide%/(sulfoxide% + sulfone%)) × 100.

room temperature for 30 min in CH_3OH/CH_2Cl_2 was also studied. The conversion of MPS monotonously increases with the addition of catalyst of 0 to 0.01 mM (table 2). When the amount of catalyst is increased to 0.015 mM, the selectivity of the MPS oxidation reduces from 94 to 83%, where the conversion of MPS was increased from 97 to 100% (table 2, entry 9). Reaction without catalyst proceeds very slightly. Hence, the amount of catalyst enhances the reaction rate for selective oxidation of sulfides. The amount of UHP could significantly affect the MPS conversion and methylphenylsulfoxide selectivity (table 2, entries 10–13). When the amount of UHP increased from 0 to 0.2 mM, the conversion of MPS increased from 0 to 97%. With a further increase of UHP to 0.3 mM, the selectivity to methylphenylsulfoxide decreases from 94 to 68%, while the conversion of MPS increased from 97 to 100%. In other words, selectivity to sulfoxide would appear to be better for reactions with 1 equiv. of the oxidant compared to 1.5 equiv. of UHP.

A series of structurally diverse sulfides were subjected to the oxidation using $[MoO_2(ONO)(CH_3OH)]$ as the catalyst and UHP as oxidant. Arylalkyl (table 3, entries 1 and 2), arylbenzyl (table 3, entry 3), dibenzyl (table 3, entry 4), diaryl (table 3, entry 5), and dialkyl (table 3, entries 6–8) sulfides underwent clean and selective oxidation to the corresponding sulfoxide under air, in impressive selectivity (91–100%). The very good conversions of substrates, depending on the nature of the sulfide of 71–97% (TON = 14.2–19.4), were obtained for all cases. In the case of dialkyl sulfides (table 3, entries 6–8), no over-oxidation to sulfone was observed. The highest and the lowest conversions were obtained for methylphenyl sulfide (97%) and diphenyl sulfide (71%), respectively (table 3, entries 1 and 5).

The mechanism for oxidation of various sulfides to sulfoxides using $[MoO_2(ONO)(CH_3OH)]$ has been proposed by comparison with the literature report (scheme 3) [4]. The changes of UV–vis absorbance spectra (figure 3) of the complex before and after adding UHP indicate that the reaction starts with nucleophilic attack of H_2O_2 to dioxo-Mo(VI)

Table 3. Oxidation of sulfides catalyzed by $[MoO_2(ONO)(CH_3OH)]$/UHP.[a]

Entry	Substrate	Conversion (%)[b] (TON)[c]	Selectivity (%)[d]
1		97 (19.4)	94
2		91 (18.2)	96
3		90 (18)	97
4		88 (17.6)	97
5		71 (14.2)	91
6		89 (17.8)	100
7		90 (18)	100
8		84 (16.8)	100

[a] The molar ratios for $[MoO_2(ONO)(CH_3OH)]$: substrate : oxidant are 1 : 20 : 20. The reactions were performed in (1 : 1) mixture of CH_2Cl_2/CH_3OH (1 mL) under air at room temperature.

[b] The GC yields (%) are measured relative to the starting sulfide.

[c] TON = (mM of sulfoxide + mM of sulfone)/mM of catalyst.

[d] Selectivity to sulfoxide = (sulfoxide%/(sulfoxide% + sulfone%)) × 100.

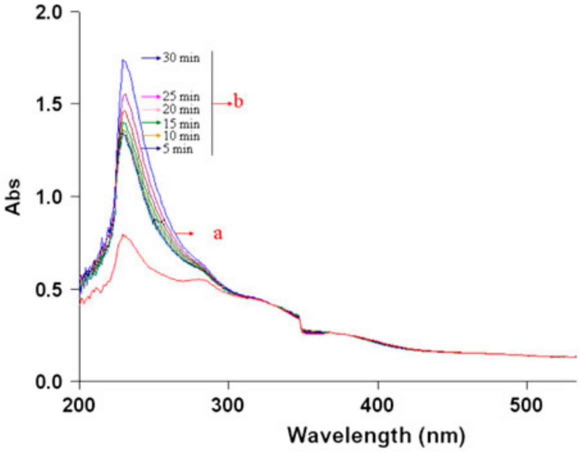
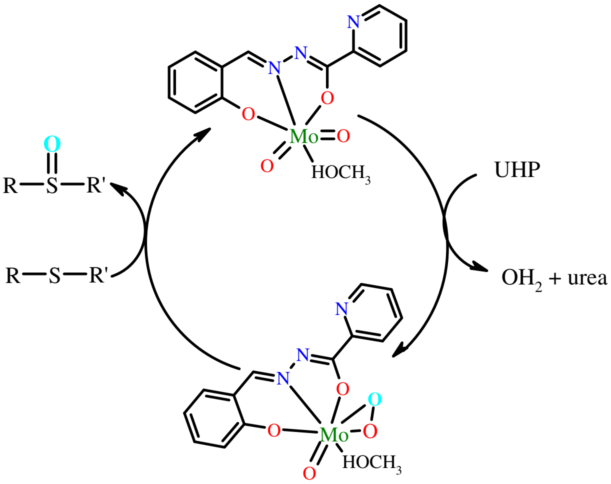


Figure 3. UV–vis absorption spectra: (a) absorbance of complex, (b) spectral changes for the reaction of complex and UHP.

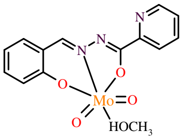
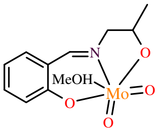
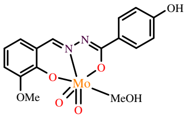
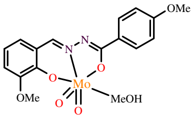
complex and the conversion of dioxo-Mo(VI) complex to oxo-peroxo Mo(VI) complex. The attack of the uncoordinated sulfide on one oxygen of the peroxido ligand, yielding sulfoxide and generating the molybdenum(VI) dioxido complex completes the catalytic cycle (scheme 3).



Scheme 3. Proposed mechanism for sulfide oxidation by the [$MoO_2(ONO)(CH_3OH)$]/UHP catalytic system.

In order to show the merit and efficiency of the present catalytic system in comparison with recently reported protocols, we compared the results of the methylphenyl sulfide oxidation in the presence of other catalysts. As shown in table 4, our catalytic system is superior to some of the previously reported catalysts in terms of reaction conditions and conversion. In contrast to similar, previously reported systems, the catalytic system presented in this paper does not suffer from the harsh reaction conditions, such as using large amount of catalyst (table 4, entry 2), long reaction time (table 4, entries 3 and 4), and using an additive (table 4, entries 3 and 4). As illustrated in table 4, all dioxo-Mo(VI) complexes are almost equally effective sulfide oxidation catalysts in different conditions (table 4, entries 1–4).

Table 4. Recently reported catalytic systems for oxidation of sulfides by dioxo-Mo(VI) catalytic systems.

Entry	Catalyst	Condition	Conversion (%)	Selectivity (%)	Ref.
1		1 M% of catalyst/UHP/rt/30 min	97	94	Present work
2		5 M% of catalyst/UHP/rt/30 min	92	100	[29]
3		1 M% of catalyst/H_2O_2/TMB/ 10 °C/2 h	100	100	[30]
4		1 M% of catalyst/H_2O_2/TMB/ 10 °C/2 h	100	100	[30]

4. Conclusion

We have synthesized and characterized a dioxo-Mo(VI) complex and have used it as catalyst in sulfide oxidation. The complex is catalytically active in the oxidation of sulfides to their respective sulfoxides under homogeneous conditions. This study affords new vistas of designing of new dioxo-Mo(VI) complexes for use as homogeneous oxidation catalysts.

Supplementary material

The CIF file of crystal structure of complex has been deposited with the CCDC, No. 986254. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223 336 033; or Email: deposit@ccdc.cam.ac.uk.

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