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Synthesis and X-ray crystal structures of two trinuclear molybdenum clusters coordinated by *p*-nitrobenzoate: $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(L)$

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Abstract

Two new trinuclear molybdenum clusters $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(L)$ (dtp = diethyldithiophosphate, L = Py and DMF) have been synthesized by the ligand substitution reaction with *p*-nitrobenzoic acid using the precursors $Mo_3S_4(dtp)_4(H_2O)$ and $Mo_3S_4(dtp)_3(CCl_3COO)(Py)$, respectively. Their structures have been determined by X-ray diffraction analysis and confirmed by ^{31}P NMR spectra. Both compounds contain $[Mo_3S_4]$ cores in which three Mo atoms form an isosceles triangle and each Mo atom is octahedrally coordinated. High asymmetry of coordination on the different Mo atoms is observed. The ligand replacement of –dtp by the aromatic acid makes the whole molecule more stable in a dipolar solvent. The coordination difference between Py and DMF also affects the core structure, resulting in the corresponding Mo–Mo distances being different in these two similar cluster compounds. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Trinuclear molybdenum cluster; Substitution reaction; X-ray crystal structures; ^{31}P NMR spectra

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1. Introduction

The significance of molybdenum in biosystems, especially in nitrogenase [1,2], leads to our interest to study molybdenum–sulfur compounds. Among the molybdenumclusters obtained so far, the incomplete cuboidal molybdenum sulfido cluster $Mo_3S_4(dtp)_3(\mu\text{-dtp})(H_2O)$, where dtp = diethyldithiophosphate, has been extensively studied due to the fascinating ligand properties of μ-dtp. Several derivatives containing aliphatate ligands were synthesized and characterized structurally [3]. Recently we have reported some aromatic acid-substituted derivatives, which exhibit interesting structures and properties [4,5]. Herein we report the synthesis and structures of two new compounds $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(Py)$ (**I**) and $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)$ (DMF) (**II**), obtained by different synthesis designs.

2. Experimental

All reactions were carried out in air. A. R. grade *p*-nitrobenzoic acid and the solvents *N*,*N*-dimethylformamide (DMF), pyridine (Py), anhydrous ethanol and dichloromethane were used without purification. $Mo_3S_4(dtp)_3(\mu\text{-dtp})(H_2O)$, $Mo_3S_4(dtp)_3(\mu\text{-}CCl_3COO)$(Py) were prepared according to the literature methods [6,7]. IR spectra were recorded on a Nicolet Magna 750 Fourier spectrophotometer in KBr. ^{31}P NMR spectra were measured on a Varian Unity-500 NMR spectrometer in $CHCl_3$ with reference to 85% H_3PO_4. Elemental analyses were determined on an EA 1110 elemental analyzer in Xiamen University.

2.1. Synthesis

2.1.1. Preparation of $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(Py)$ (I)

A solution of $Mo_3S_4(dtp)_3(\mu\text{-dtp})(H_2O)$ (0.10 g, 0.085 mmol) and *p*-nitrobenzoic acid (0.02 g, 0.129 mmol) in 40.0 cm^3 $EtOH/CH_2Cl_2$ (v/v = 1:1) was refluxed while

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stirring at 80 °C for 1 h. Then several drops of Py were added and the mixture was stirred further for 10 min. The resultant deep-brown mixture was then filtered. Black crystals of $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(Py)$ were obtained when the filtrate was kept at room temperature (r.t.) for a few days. Yield: 0.04 g (38.8%); *Anal.* Calc. for $C_{24}H_{39}Mo_3N_2O_{10}P_3S_{10}$: C, 23.69; H, 3.23; N, 2.30; S, 26.29. Found: C, 23.83; H, 3.45; N, 2.55; S, 26.20%. IR (KBr disc, cm^{-1}): ν 1603 (m), 1537 (s), 1392 (s), 1344 (s), 1009 (s), 964 (s), 648 (s), 536 (s).

2.1.2. Preparation of $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(DMF)$ (II)

A solution of $Mo_3S_4(dtp)_3(\mu\text{-}CCl_3COO)(Py)$ (0.10 g, 0.082 mmol) and $p\text{-}NO_2C_6H_4COOH$ (0.02 g, 0.129 mmol) in 3.0 cm^3 DMF was stirred at 80 °C for 1 h. After being filtrated, 20.0 cm^3 EtOH was added to the solution. When the solution was kept at room temperature for 3 days, black crystals of $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(DMF)$ were obtained. Yield: 0.065 g (65.4%); *Anal.* Calc. for $C_{22}H_{41}Mo_3N_2O_{11}P_3S_{10}$: C, 21.82; H, 3.41; N, 2.31; S, 26.43. Found: C, 22.37; H, 4.01; N, 2.46; S, 26.33%. IR (KBr disc, cm^{-1}): ν 1645 (s), 1537 (s), 1394 (m), 1344 (s), 1014 (s), 642 (m).

2.2. Crystal structure determination

Data collection of $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(Py)$ (**I**) and $Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(DMF)$ (**II**) were conducted on an Enraf–Nonius CAD4 and a Siemens SMART CCD diffractometer, respectively, with graphite-monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å) at 293(2) K. The data reductions and structural analyses were performed on a workstation using Enraf–Nonius MOLEN/VAX Software or SMART CCD Software and the structures were refined on F^2 through the use of the SHELXL-PC programs [8–10]. Crystal structure plots were drawn using ORTEP II [11]. The positions of the molybdenum atoms were determined by direct methods, and successive difference electron density maps located the remaining non-hydrogen atoms. All the non-hydrogen atoms were refined anisotropically by full-matrix least-squares technique. Hydrogen atoms were included but not refined. The crystallographic data are listed in Table 1.

3. Results and discussion

3.1. Synthesis

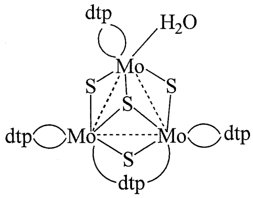
The precursor in this work $Mo_3S_4(dtp)_3(\mu\text{-}dtp)(H_2O)$

Table 1
Crystallographic data for compounds **I** and **II**

	I	**II**
Chemical formula	$C_{24}H_{39}Mo_3N_2O_{10}P_3S_{10}$	$C_{22}H_{41}Mo_3N_2O_{11}P_3S_{10}$
Formula weight	1216.90	1210.90
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Crystal dimension (mm)	0.75 × 0.25 × 0.08	0.40 × 0.35 × 0.30
Unit cell dimensions		
a (Å)	13.6576(1)	10.556(3)
b (Å)	13.8672(1)	13.840(3)
c (Å)	14.6284(1)	16.994(5)
α (°)	100.16(1)	85.01(2)
β (°)	114.69(1)	77.22(2)
γ (°)	97.27(1)	67.75(2)
V (Å^3)	2415.0(3)	2241.0(10)
Z	2	2
D_{calc} (g cm^{-3})	1.673	1.795
θ Range for data collection (°)	1.53–25.97	1.23–25.03
Absorption correction	empirical psi-scan	empirical
Reflections collected	9481	11578
Reflections observed ($>2\sigma$)	5622	5528
Data/restraints/parameters	9449/0/424	7786/1/460
R_1, wR_2 ($I>2\sigma(I)$) [a]	0.0827, 0.2041	0.0525, 0.1105
Transmission min, max	0.8044, 0.9996	0.6511, 1.0000
Goodness-of-fit, S	0.971	1.006
Largest shift	0.001	0.050
Largest different peak (e Å^{-3})	2.04	0.82

[a] $R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$. $wR = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2}$.

has three different substitution sites [3,7]: bridging μ-dtp, terminal –dtp, and the loosely-coordinated ligand H_2O, as shown below:



It has been confirmed that the loosely-coordinated ligand H_2O, the bridging –dtp, as well as the terminal –dtp are dynamically labile in polar solvents such as acetone and $CHCl_3$. These ligands sites can be replaced by other ligands to a different extent, forming a group of new cluster derivatives, which are more stable than the precursor in dipolar solvents.

The synthesis reactions of the title compounds can be described as follows:

$$Mo_3S_4(dtp)_4(H_2O) + p\text{-}NO_2C_6H_4COOH \xrightarrow[Py]{EtOH/CH_2Cl_2} Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(Py) \quad (1)$$

$$Mo_3S_4(dtp)_3(CCl_3COO)(Py) + p\text{-}NO_2C_6H_4COOH \xrightarrow{DMF} Mo_3S_4(dtp)_3(p\text{-}NO_2C_6H_4COO)(DMF) \quad (2)$$

Compound **I** was obtained via the ligand substitution of Py for H_2O, and of the bridging ligand p-$NO_2C_6H_4COO^-$ for μ-dtp. Similarly, the replacement of CCl_3COO^- by p-$NO_2C_6H_4COO^-$ and of Py by DMF gives compound **II**.

It is noteworthy that, for the first time, we have obtained the expected substitution products using $Mo_3S_4(dtp)_3(RCOO)(Py)$ instead of $Mo_3S_4(dtp)_4(H_2O)$ as the starting material (see reaction (2)). Attempts to prepare the same product from $Mo_3S_4(dtp)_4(H_2O)$ in DMF failed. Lu et al. [12] suggested that the coordination capability of substituents such as toluene-3,4-dithiol (TDT), 1-Pyrrolidinecarbodithioic acid (Hdtcpyr), diethyldithiocarbamic acid (Hdtc), RCOOH and Hdtp, well conforms to their respective acidity. In general, lower acidity corresponds to stronger coordination capability. The pK_a of p-$NO_2C_6H_4COOH$ is 3.42, while that of CCl_3COOH is 0.64, so reaction (2) can take place.

This can explain well reaction (1). Nevertheless, our further experiments indicate that the same products can also be crystallized out when $Mo_3S_4(dtp)_3(CH_3COO)(Py)$ or $Mo_3S_4(dtp)_3(CH_3CH_2COO)(Py)$ is used as the precursor in similar experiments. This is contradictory to the acidity interpretation because the pK_a value of either CH_3COOH (4.76) or CH_3CH_2COOH (4.87) is higher than that of p-$NO_2C_6H_4COOH$ (3.42). We claim that the stability of the clusters in the polar solvent will determine whether the reaction will happen. The bulky aromatic acid-substituted compounds are more dynamically stable and easier to deposit in the polar solvents used. In DMF, $Mo_3S_4(dtp)_4(H_2O)$ dissociates too much but $Mo_3S_4(dtp)_3(RCOO)(Py)$ is relatively stable. This enables the substitution to take place. More discussion on the synthesis will be presented in the following ^{31}P NMR spectra section.

By choosing ligands of different chelating properties for the precursor $Mo_3S_4(dtp)_3(\mu\text{-}L')(L)$, ($L' =$ dtp, $HCOO^-$, CH_3COO^-, CH_2COO^-, CCl_3COO^-; $L =$ H_2O, CH_3CN, Py, etc.), we can design many kinds of derivatives with desired structures and functions. For example, by the specific binding capability of the groups on the benzene ring, oligomerized and polymerized cluster compounds can be rationally synthesized [13]. Other important applications of this reaction are in the exploitation of new catalysts [14,15] and in the synthesis of new optical limiting materials [16].

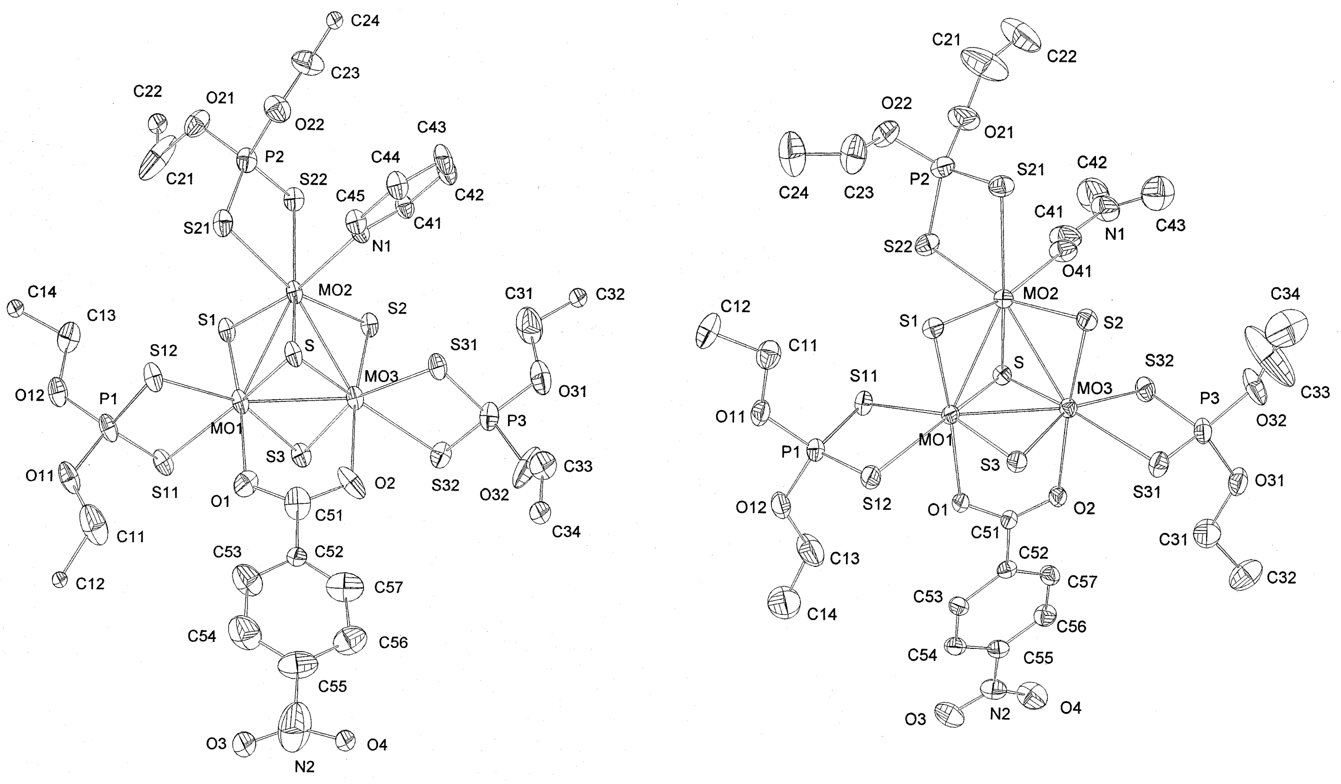
3.2. Crystal structure

The crystal structures of compounds **I** and **II** are shown in Fig. 1. Selected bond lengths and angles for the two compounds are presented in Tables 2 and 3, respectively. The structures of compounds **I** and **II** are similar to the typical incomplete cuboidal structures reported [17]. Each Mo atom is octahedrally coordinated with high distortion as in the case of other analogous clusters. The molecular core is $[Mo_3S_4]$, in which three Mo atoms construct an isosceles triangle. Direct bonding is found between each pair of Mo atoms. The ligand p-nitrobenzoate bridges Mo1 and Mo3 via its –COO chelating group. The solvated Py or DMF are coordinated to the remaining Mo atom without –COO coordination.

In compound **I**, the bond length of Mo2–N1 (Py) is 2.347(7) Å, far longer than that of normal Mo (+4)–N (2.0–2.18 Å), but similar to that of Mo–N (2.385–2.40 Å) in $Mo_3S_4(\mu\text{-}O_2CR)(dtp)_3(Py)$ ($R = H$, CH_3, CH_2CH_3) [18,19], showing that Py is also loosely coordinated to Mo2 in this molecule.

In compound **II**, Mo2–O41 is 2.219(6) Å, comparable to the Mo–O (DMF) distance (2.278 Å) of $Mo_3S_4(dtc)_4(DMF)$ [20], but shorter than that of Mo–O (H_2O) (2.361 Å) in $Mo_3S_4(dtp)_4(H_2O)$ [21], which means DMF is coordinated stronger than H_2O.

The only difference between compounds **I** and **II** is the loosely-coordinated ligands, Py and DMF. However, their effects on the $[Mo_3S_4]$ core and on the alignment of the bridging ligand p-nitrobenzoate are obvious. The average Mo–Mo distance of compound **I** is 2.732 Å, shorter than that of compound **II** (2.741 Å). The average Mo–μ-S and Mo–μ_3-S distances of compound **I** are 2.286 and 2.334 Å, respectively, shorter than those of compound **II** (Mo–μ-S 2.294 Å, Mo–μ_3-S 2.335 Å). Hence the $[Mo_3S_4]$ core in **I** is more tightly packed than that in **II**. We can conclude that DMF is



(I) **(II)**

Fig. 1. ORTEP drawings of compounds **I** and **II** with thermal ellipsoids at the 20% probability level.

a stronger ligand than Py and it draws more electrons of Mo into the Mo–O bond, weakening the central Mo–Mo bond. This is consistent with the fact that $[Mo_3S_4(H_2O)_9]^{4+}$ has the shortest average Mo–Mo distance [22], because H_2O is very loosely coordinated to Mo. *p*-Nitrobenzoates in these two molecules are coordinated in different orientations. The dihedral angles measured between the plane (Mo1, Mo2, Mo3) and the benzene plane (C52, C53, C54, C55, C56, C57) is 146.6° for **I** and 52.4° for **II**.

3.3. ^{31}P NMR spectra

The ^{31}P NMR spectra of the two compounds were measured in $CHCl_3$, showing two peaks at 109.7 (T1) and 108.8 ppm (T2) for compound **I** and 110.1 (T1) and 107.4 ppm (T2) for compound **II** (Fig. 2). The assignment of the ^{31}P resonances is consistent with the literature [7]. The relative positions and their line widths are determined by the loosely-coordinated ligands [4,7]. We have measured ^{31}P NMR spectra to study the kinetic behavior in organic solvents for all the substitution derivatives from $Mo_3S_4(dtp)_4(H_2O)$ and $[Mo_3S_4(H_2O)_9]^{4+}$ using aliphatic and aromatic acids as the substituent ligands. The result shows that, once the precursor is selected, the choice of solvent used in the synthesis is quite important to obtain the desired derivatives bridged by organic acids. In low polar solvents like ethanol, *o*-dichlorobenzene, the loosely-coordinated ligand is kinetically labile, but other ligands are quite stable [7]. However, in more polar solvents such as Py, DMF, both the loosely-coordinated ligand and the bridging ligand –dtp or RCO_2 are kinetically labile. Furthermore, for the derivatives substituted by aromatic acids, the influence of solvent is negligible. The spectra in this experiment show that all the moieties of compounds **I** and **II** are stable in highly polar solutions, so are the other aromatic acid-substituted derivatives. Whether we can get a desired product in the ligand substitution reaction is determined by the relative stability of both the precursor and product in solution. In practice, $Mo_3S_4(dtp)_4(H_2O)$ is a good precursor in solvents of low polarity like ethanol and *o*-dichlorobenzene, but not in polar solvents such as Py and DMF because it then becomes unstable. $Mo_3S_4(dtp)_3(RCO_2)(L)$ of aliphatic-bridged acids are good precursors in Py and DMF, but they cannot be the final products because of their lability in strong polar solvents.

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

Bond lengths			
Mo1–Mo2	2.744(1)	Mo2–S2	2.296(3)
Mo1–Mo3	2.703(1)	Mo2–N1	2.347(7)
Mo1–S	2.337(2)	Mo3–S	2.339(3)
Mo1–S1	2.273(3)	Mo3–S2	2.278(3)
Mo1–S3	2.292(3)	Mo3–S3	2.290(3)
Mo1–O1	2.277(9)	Mo3–O2	2.309(9)
Mo2–Mo3	2.7499(3)	O1–C51	1.311(16)
Mo2–S	2.327(3)	O2–C51	1.348(16)
Mo2–S1	2.289(2)		
Bond angles			
Mo1–Mo2–Mo3	58.95(3)	S2–Mo2–Mo1	97.58(8)
Mo1–Mo3–Mo2	60.42(3)	S2–Mo2–Mo3	52.74(7)
Mo3–Mo1–Mo2	60.63(3)	S2–Mo2–S	105.25(10)
S–Mo1–Mo2	53.78(7)	S2–Mo3–S	105.43(10)
S–Mo2–Mo1	54.14(6)	S2–Mo3–S3	94.00(10)
S–Mo2–Mo3	54.10(7)	S2–Mo3–O2	170.9(3)
S–Mo1–Mo3	54.72(7)	S3–Mo1–Mo3	53.80(7)
S–Mo3–O2	83.5(2)	S3–Mo1–Mo2	97.28(8)
S1–Mo2–Mo1	52.76(7)	S3–Mo3–S	107.84(10)
S1–Mo2–Mo3	97.36(8)	S3–Mo3–O2	84.7(2)
S1–Mo1–O1	172.4(2)	O1–Mo1–Mo3	86.2(2)
S1–Mo1–S	105.57(10)	O1–Mo1–Mo2	34.33(19)
S1–Mo2–S	105.40(9)	O1–Mo1–S3	84.9(2)
S1–Mo2–S2	96.69(10)	O1–C51–C52	113.5(13)
S1–Mo1–S3	93.74(10)	O2–C51–C52	112.7(13)
S1–Mo2–N1	168.3(2)		

Table 3
Selected bond lengths (Å) and bond angles (°) for compound **II**

Bond lengths			
Mo1–Mo2	2.760(1)	Mo2–S2	2.303(2)
Mo1–Mo3	2.697(1)	Mo2–O41	2.219(6)
Mo1–S	2.340(2)	Mo3–S	2.338(2)
Mo1–S1	2.294(2)	Mo3–S2	2.293(2)
Mo1–S3	2.297(2)	Mo3–S3	2.297(2)
Mo1–O1	2.230(5)	Mo3–O2	2.248(5)
Mo2–Mo3	2.766(1)	O1–C51	1.272(8)
Mo2–S	2.328(2)	O2–C51	1.267(8)
Mo2–S1	2.279(2)		
Bond angles			
Mo1–Mo2–Mo3	58.42(3)	S1–Mo2–Mo1	53.10(6)
Mo1–Mo3–Mo2	60.69(3)	S2–Mo2–Mo1	97.81(6)
Mo1–Mo3–O2	84.04(13)	S2–Mo3–Mo1	99.86(6)
Mo1–Mo2–O3	58.42(3)	S2–Mo2–S	104.81(7)
Mo2–Mo3–O2	132.41(13)	S2–Mo3–Mo2	53.15(6)
Mo3–Mo1–Mo2	60.89(3)	S3–Mo3–Mo2	97.40(6)
S–Mo1–Mo3	54.75(5)	S3–Mo1–Mo2	97.53(6)
S–Mo2–Mo1	53.95(5)	S3–Mo3–Mo1	54.05(5)
S–Mo3–Mo1	54.83(5)	S3–Mo1–Mo3	54.04(5)
S–Mo3–Mo2	53.47(5)	S3–Mo3–S	108.27(7)
S–Mo2–Mo3	53.80(5)	O1–Mo1–S1	173.42(14)
S–Mo1–Mo2	53.53(5)	O1–Mo1–Mo2	133.87(13)
S1–Mo2–S	105.34(7)	O1–Mo1–Mo3	84.77(13)
S1–Mo1–Mo2	52.61(6)	O1–Mo1–S	82.10(14)
S1–Mo1–S	104.46(7)	O1–C51–C52	116.4(6)
S1–Mo1–Mo3	99.37(6)	O2–C51–C52	117.7(6)
S1–Mo2–S2	98.22(8)		

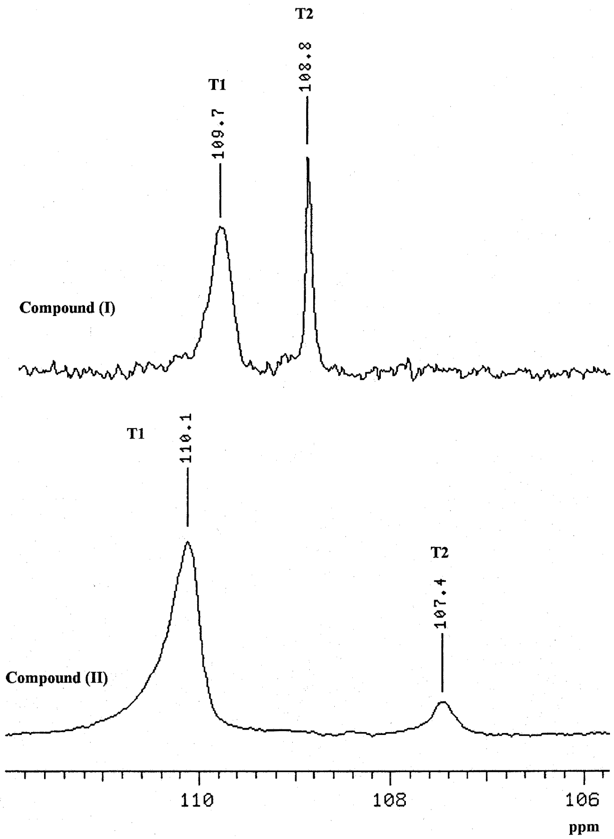


Fig. 2. ^{31}P NMR spectra of compounds **I** and **II**.

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 152332 and 152333. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1233-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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