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Molybdenum compounds bearing pymS (pyrimidine-2-thiolato) and tertiary phosphine ligands

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In this paper we have reported mono- and dinuclear molybdenum compounds containing doubly bridging pymS (pyrimidine-2-thiolato) and $P(Fu)_3$ {(Fu = (2-furyl)phosphine)} or $P(OMe)_3$ (trimethyl phosphite). One-pot reaction between $[Mo(CO)_3(NCMe)_3]$ and pymSH (pyrimidine-2-thiol) in the presence of $P(Fu)_3$ in THF at 50 °C gives the previously reported eight coordinate compound $[Mo(\kappa^2\text{-pymS})_4]$ (**1**) in 17% yield and two new compounds $[Mo(CO)_4(P(Fu)_3)_2]$ (**2**) and $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pymS})_2(P(Fu)_3)_2]$ (**3**) in 51 and 15% yields, respectively. A similar reaction involving $P(OMe)_3$ furnishes two mononuclear compounds $[Mo(CO)_2(\kappa^2\text{-pyS})_2(P(OMe)_3)]$ (**4**) and $[Mo(CO)_4(P(OMe)_3)_2]$ (**5**) in 20 and 35% yields, respectively. Compounds **2** and **4** are characterized by single-crystal X-ray diffraction analysis in addition to IR, ^1H NMR and $^{31}P\{^1H\}$ NMR spectroscopic methods.

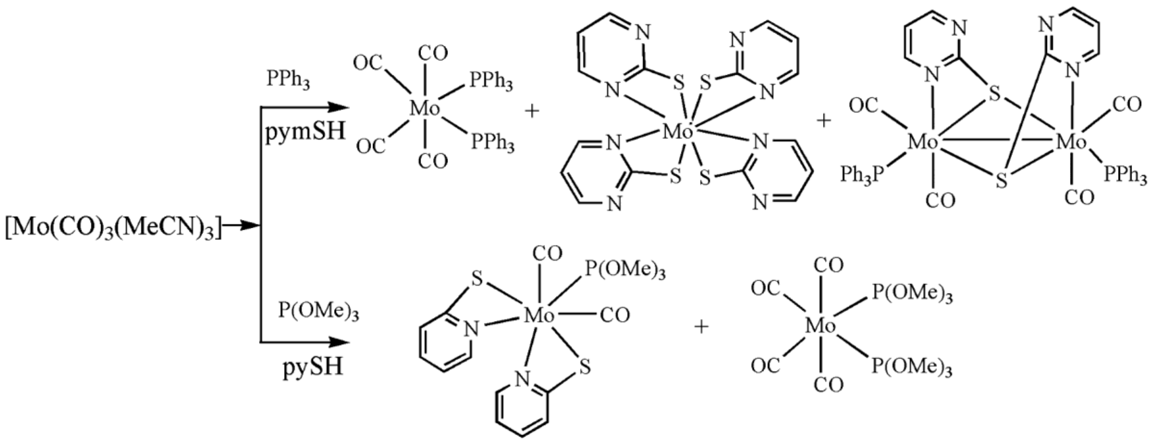
Keywords: Molybdenum carbonyls, pymSH, Tertiary phosphines, X-ray structures

Ambidentate donor ligands pySH (pyridine-2-thiol), pymSH and their deprotonated derivatives pyS (pyridine-2-thiolato) and pymS bear exocyclic sulfur and heterocyclic nitrogen atom(s) available for metal coordination. Several groups have demonstrated variety of coordination motifs of pyridine-2-thiolato ligands in several novel compounds in the past.[1-7] The chemistry of this class of ligands with low-valent metal carbonyls, especially Group 7 and 8 metals has been widely investigated.[8-13] Shi and co-workers found that the one-pot reaction between $[Mo(CO)_3(NCMe)_3]$, pySH and PPh_3 furnishes the dimolybdenum compound $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pyS})_2\text{-}(PPh_3)_2]$ and a mixed-valance trinuclear cluster $[Mo_3(CO)_6(\mu\text{-pyS})_2(\mu_3\text{-pyS})_2]$.[14] Recently, our group reported the binuclear compound $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pymS})_2(PPh_3)_2]$[15] along with the previously reported mononuclear compounds $[Mo(\kappa^2\text{-pymS})_4]$[16] and $[Mo(CO)_4(PPh_3)_2]$[17] from the one-pot reaction between $[Mo(CO)_3(NCMe)_3]$, pymSH and PPh_3. We also previously reported[18] the mononuclear compounds $[Mo(CO)_2\{P(OMe)_3\}(\kappa^2\text{-pyS})_2]$ and $[Mo(CO)_4\{P(OMe)_3\}_2]$ from the reaction of $[Mo(CO)_3(NCMe)_3]$ with pySH and $P(OMe)_3$ under conditions similar to those employed by Shi and co-workers (Scheme 1).

The organometallic chemistry of pymSH and pySH never cease to surprise us due to their similar functionalities and yet different chemistry. Therefore, we recently focused our attention towards reactivity of $[Mo(CO)_3(NCMe)_3]$ towards pymSH in the presence of a different subsidiary phosphine ligands such as $P(Fu)_3$ and $P(OMe)_3$ since they are potential alternatives of triphenylphosphine.

Materials and Methods

All reactions were performed under nitrogen atmosphere using standard Schlenk techniques unless otherwise stated. Reagent grade solvents were freshly distilled using appropriate drying agents prior to use. Infrared spectra were recorded on a Shimadzu IR Prestige-21 spectrophotometer. NMR spectra were recorded on a Bruker DPX 400 instrument. All chemical shifts are reported in δ unit and are referenced to the residual protons of the deuterated solvent (^1H) and external 85% H_3PO_4 (^{31}P) as appropriate. Elemental analyses were performed by Microanalytical Laboratories of Wazed Miah Science Research Centre at Jahangirnagar University. Products were separated in the air by TLC plates coated with 0.25 mm of silica gel (HF254-type 60, E. Merck, Germany). $Me_3NO\cdot 2H_2O$ was purchased from Lancaster and water was removed using a Dean-Stark apparatus by azeotropic distillation from benzene and the anhydrous Me_3NO was stored under nitrogen. $P(Fu)_3$, $P(OMe)_3$ and pySH were purchased



Scheme 1 —Schematic representation of the reaction of [Mo(CO)$_3$(NCMe)$_3$] with pySH and PymSH

from Sigma-Aldrich Chemical Company and used as received. [Mo(CO)$_3$(NCMe)$_3$] was prepared according to literature method.[14]

Reaction of [Mo(CO)$_3$(NCMe)$_3$] with pymSH and P(Fu)$_3$

To an acetonitrile solution (15 mL) of [Mo(CO)$_3$(NCMe)$_3$] (115 mg, 0.38 mmol) was added P(Fu)$_3$ (88 mg, 0.38 mmol) and pymSH (89 mg, 0.79 mmol) at 100 °C. The reaction mixture was allowed to cool to 50 °C and stirred for 2 h. The color of the solution changed from yellow to deep red. The solvent was removed under reduced pressure and the product was redissolved in a minimum volume of CH_2Cl_2 and chromatographed by TLC on silica gel. Elution with cyclohexane/acetone (7:3, v/v) developed several bands of which the first, second and third bands gave Mo(CO)$_6$, pymSH and P(Fu)$_3$, in order of elution. The fourth band gave the known compound [Mo(κ^2-pymS)$_4$][12] (**1**) (45 mg, 17%) as yellow crystals while the fifth and sixth bands afforded the new compounds [Mo(CO)$_4$(P(Fu)$_3$)$_2$] (**2**) (82 mg, 51%) and [Mo$_2$(CO)$_4$(P(Fu)$_3$)$_2$(μ-κ^2-pymS)$_2$] (**3**) (40 mg, 15%) as black crystals after crystallization from hexane/CH_2Cl_2 at 4 °C. The contents of the other bands were too small to complete characterization. Spectroscopic data for **2**: Anal. (%) Calc. for $C_{28}H_{18}MoO_{10}P$: C 52.44; H 2.83. Found: C 52.66; H 2.98. IR (cm^{-1}) (νCO, CH_2Cl_2): 2035 s, 1936 vs, 1921 vs. ^{1}H NMR (CDCl$_3$): δ 7.52 (s, 6H), 6.67 (d, 6H, J = 3.2 Hz), 6.37 (d, 6H, J = 1.2 Hz). ^{31}P{^{1}H} NMR (CDCl$_3$): δ –11.17 (s). Spectroscopic data for **3**: Anal. (%) Calc. for $C_{36}H_{24}MoO_{10}N_4P_2S_2$: C 50.02; H 2.69. Found: C 50.18; H 2.80. IR (cm^{-1}) (νCO, CH_2Cl_2):1920 vs, 1846 s. ^{1}H NMR (CDCl$_3$)**:** δ 9.10 (dd, 2H, J = 5.0, 2.5 Hz), 8.20 (dd, 2H, J = 5.0, 2.5 Hz), 7.78 (br s, 6H), 7.21 (br s, 6H), 6.87 (t, 2H, J = 5.0 Hz), 6.60 (m, 6H). ^{31}P{^{1}H} NMR (CDCl$_3$): δ –11.69 (s, 1P).

Reaction of [Mo(CO)$_3$(NCMe)$_3$] with pymSH and P(OMe)$_3$

To an acetonitrile solution (15 mL) of [Mo(CO)$_3$(NCMe)$_3$] (115 mg, 0.38 mmol) was added P(OMe)$_3$ (88 mg, 0.71 mmol) and pymSH (89 mg, 0.79 mmol) at 100 °C. The reaction mixture was allowed to cool to 50 °C and stirred under nitrogen atmosphere for 2 h. The color of the solution changed from yellow to deep red. The solvent was removed under reduced pressure and the residue was separated by TLC on silica gel. Elution with cyclohexane/acetone (7:3, v/v) developed several bands of which the first, second and third bands gave unconsumed starting material Mo(CO)$_6$, pymSH and P(OMe)$_3$, respectively. The fifth band afforded the new compound [Mo(CO)$_2$(κ^2-pymS)$_2$(P(OMe)$_3$)] (**4**) (82 mg, 51%) as red crystals after crystallization from hexane/CH_2Cl_2 at 4 °C while the fourth bands gave the known compound [Mo(CO)$_4$(P(OMe)$_3$)$_2$][14] (**5**) (40 mg, 15%). The contents of the other bands were too small to complete characterization. Spectroscopic data for **4**: Anal. (%) Calc. for $C_{13}H_{15}MoO_5N_4PS_2$: C 31.33; H 3.03. Found: C 31.50; H 3.20. IR (cm^{-1}) (νCO, CH_2Cl_2): 1963 s, 1886 vs. ^{1}H NMR (CDCl$_3$): δ 8.48 (dd, 2H, J = 4.8, 2.4 Hz), 8.37 (dd, 2H, J = 4.8, 2.4 Hz), 6.79 (t, 2H, J = 4.8 Hz), 3.64 (d, 9H, J = 12.0 Hz). ^{31}P{^{1}H} NMR (CDCl$_3$): δ 165.3 (s).

X-ray crystallography

Single crystals of **2** and **4** suitable for X-ray diffraction analysis were grown by slow diffusion of hexane into a CH_2Cl_2 solution. Suitable crystals were mounted on a Bruker Nonius Kappa CCD diffractometer using a Nylon loop with inert oil and the diffraction data were collected at 162.98 and 193.0 K using Mo-Kα radiation (λ = 0.71073). Unit cell determination, data reduction, and absorption corrections were carried out using Apex3 v 2016.1-0. The structures were solved by direct methods and refined by full-matrix least-squares on the basis of F2 using ShelXS[19] within the Olex2[20] graphical user interface. Non-hydrogen atoms were refined anisotropically, and hydrogen atoms were included using a riding model. Pertinent crystallographic parameters are given in Table-1.

Results and Discussion

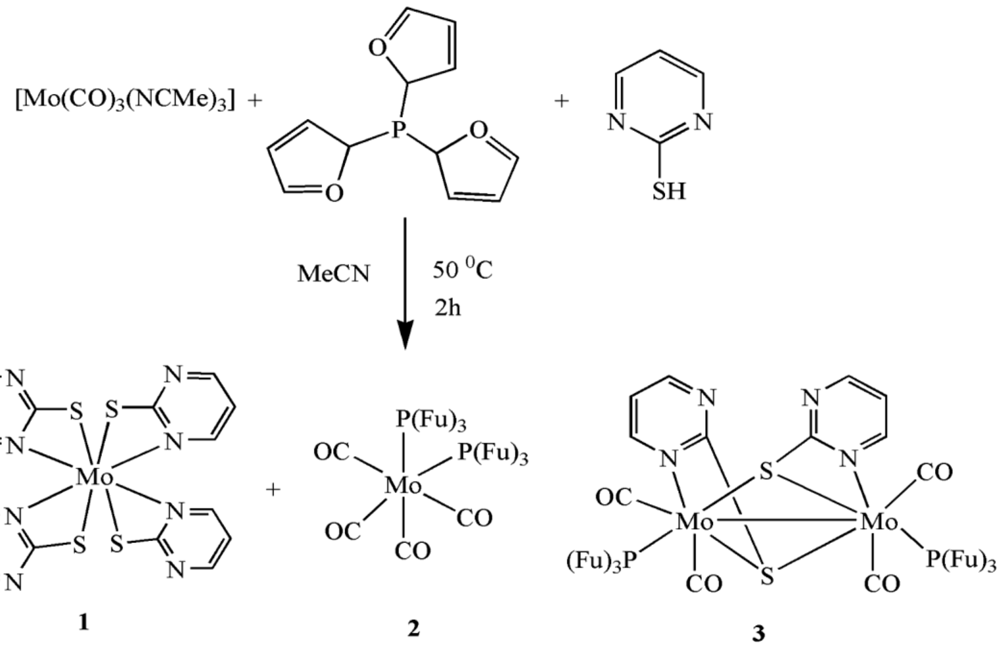
One-pot synthesis of mononuclear [Mo(CO)$_4$(P(Fu)$_3$)$_2$] (2) and binuclear [Mo$_2$(CO)$_4$((μ-κ^2-pymS)$_2$(P(Fu)$_3$)$_2$] (3) by the reaction between [Mo(CO)$_3$(NCMe)$_3$], pymSH and P(Fu)$_3$

One-pot reaction between [Mo(CO)$_3$(NCMe)$_3$], pymSH and P(Fu)$_3$ in acetonitrile at 60 °C afforded two new compounds; mononuclear [Mo(CO)$_4$-(P(Fu)$_3$)$_2$] (**2**) and the binuclear compound [Mo$_2$(CO)$_4$(μ-κ^2-pymS)$_2$(P(Fu)$_3$)$_2$] (**3**) in 51 and 15% yields, respectively (Scheme 2) and the previously reported eight coordinate mononuclear compound [Mo(κ^2-pymS)$_4$][16] (**1**) in 17% yield. The new compound **2** has been characterized by spectroscopic data, elemental analysis and single crystal X-ray diffraction studies and **3** have been characterized by spectroscopic data and elemental analysis.

The solid-state molecular structure of **2** is shown in Fig. 1 together with selected bond distances and bond angles in Table 2. The molecule **2** is an octahedral mononuclear Mo compound with two P(Fu)$_3$ ligands and four CO ligands satisfying the six coordination of Mo where two P(Fu)$_3$ ligands lie at the *cis* position. The Mo–P bond distances in **2** [Mo01–P1 = Mo01–P1^1 = 2.4901(13) Å] are a bit shorter to those observed in *cis*-[Mo(CO)$_4$(PPh$_3$)$_2$][13] (2.525(2) and 2.533(2) Å) and in compound [Mo(CO)$_4$(κ^2-dppm)][18] [2.535(3) and 2.501(2) Å]. The P1–Mo01–P1^1 bond angle of 97.89(6)° indicates that the two P(Fu)$_3$ ligands are coordinated to the molybdenum atom at the *cis* position which is smaller compared to the bond angle [P1–Mo–P2 = 104.62(7)°] in *cis*-[Mo(CO)$_4$(PPh$_3$)$_2$][17] and comparable to 97.5, 100.3 and 99.3° in *cis*-[Mo(CO)$_4$(PR$_3$)$_2$][17] where R = Me, Et, n-Bu. Among the four carbonyls, two lie *cis* and the remaining two lie *trans* to one another with Mo–C bond lengths [Mo01–C1 = Mo01–C1^1 2.039(5) and (Mo01–C2 = Mo01–C2^1 1.994(5) Å] with the molybdenum atom. The compound **2** was also characterized by IR, ^{1}H NMR and ^{31}P{^{1}H } NMR and found completely consistent with the solid-state structure. The IR spectrum showed three strong CO absorptions at 2035, 1936 and 1921 cm^{-1} indicating the presence of terminal CO bonded to Mo and is consistent with the crystal structure revealed by X-ray. The ^{1}H NMR reveals one singlet at δ 7.52 and

Table 1 — Crystallographic data and structure refinement for **2** and **4**

Compound	**2**	**4**
Identification code	CCDC 1895417	CCDC 1895409
Empirical formula	$C_{28}H_{18}O_{10}P_2Mo$	$C_{26}Mo_{1.98}O_{10}N_8S_4P_2H_{30}$
Formula weight	672.30	994.89
Temperature/K	162.98	193.0
Crystal system	monoclinic	Triclinic
Space group	P2/n	P-1
a/Å	9.323(4)	9.014(4)
b/Å	8.620(4)	9.094(4)
c/Å	17.580(7)	12.395(6)
α/°	90	81.729(16)
β/°	91.859(17)	70.086(12)
γ/°	90	74.350(16)
Volume/Å^3	1412.1(10)	918.4(7)
Z	2	1
ρ_{calc}g/cm^3	1.581	1.799
μ/mm^{-1}	0.634	1.056
F(000)	676.0	499.0
Crystal size/mm^3	0.358 × 0.208 × 0.121	0.221 × 0.19 × 0.071
Radiation	Mo Kα (λ = 0.71073)	Mo Kα (λ = 0.71073)
2θ range for data collection/°	6.438 to 54.342	5.076 to 54.294
Index ranges	$-11 \le h \le 11$, $-11 \le k \le 11$, $-22 \le l \le 22$	$-11 \le h \le 11$, $-11 \le k \le 11$, $-15 \le l \le 15$
Reflections collected	19356	24788
Independent reflections	3127 [R_{int} = 0.0214, R_{sigma} = 0.0141]	4060 [R_{int} =0.0235, R_{sigma} = 0.0146]
Data/restraints/ parameters	3127/9/163	4060/0/240
Goodness-of-fit on F^2	1.038	1.105
Final R indexes [I>=2σ (I)]	R_1 = 0.0608, wR_2 = 0.1555	R_1 = 0.0192, wR_2 = 0.0506
Final R indexes [all data]	R_1 = 0.0624, wR_2 = 0.1568	R_1 = 0.0206, wR_2 = 0.0517
Largest diff. peak/ hole / e Å^{-3}	3.30/-1.71	0.50/-0.53



Scheme 2 — Schematic representation for the synthesis of $[Mo(CO)_4(P(Fu)_3)_2]$ (**2**) and $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pymS})_2(P(Fu)_3)_2]$ (**3**)

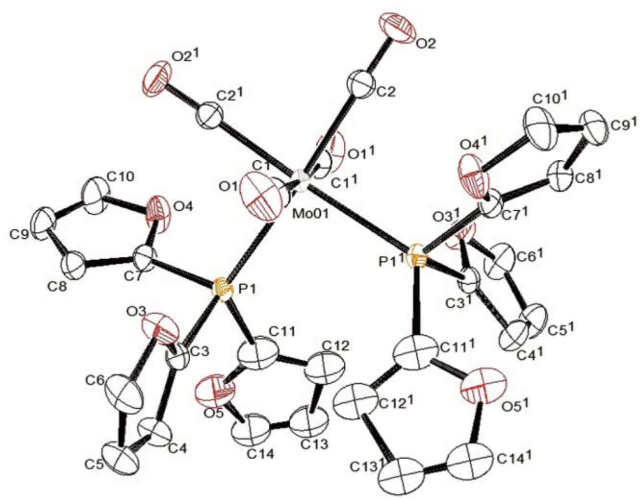


Fig. 1 — Molecular structure of $[Mo(CO)_4(P(Fu)_3)_2]$ (**2**) showing 50% probability atomic displacement ellipsoids. Hydrogen atoms are omitted for clarity.

two doublets at δ 6.67 and 6.37 corresponding to furyl protons. The $^{31}P\{^1H\}$ NMR gave only one singlet at δ –11.17 indicating that two phosphorus atoms are in equivalent chemical environment.

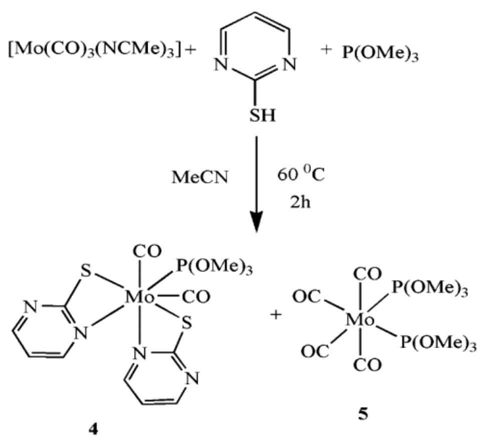
Unfortunately single crystals of compound $[Mo_2(CO)_4((\mu\text{-}\kappa^2\text{-pymS})_2(P(Fu)_3)_2]$ (**3**) could not be obtained after several attempts. Compound **3** (Scheme 2) was characterized on the basis of infrared, ^1H NMR, $^{31}P\{^1H\}$ NMR and elemental analysis. The infrared spectra of **3** exhibits two strong absorption bands in the carbonyl stretching region at 1920 and 1846 cm^{-1} which are very similar to that of the PPh_3 containing analogue $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pyS})_2(PPh_3)_2]$[14] and $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pymS})_2(PPh_3)_2]$[15] derived from the reactions of $[Mo(CO)_3(NCMe)_3]$ with pySH/pymSH and PPh_3/$P(Fu)_3$. The aromatic region of the ^1H NMR spectrum of compound **3** shows two doublet of doublets at δ 9.10 and 8.20, two broad singlets at δ 7.78 and 7.21 and one triplet at δ 6.87 and a multiplet at δ 6.60 assigned to the ring protons of the pyrimidine-2-thiolato and furyl ligands. The $^{31}P\{^1H\}$ NMR spectrum shows a singlet at δ –11.69 indicating that the two phosphorus nuclei of $P(Fu)_3$ are equivalent which supports the proposed formulation.

Synthesis of mononuclear $[Mo(CO)_2\{P(OMe)_3\}(\kappa^2\text{-pymS})_2]$ (4) by the reaction between $[Mo(CO)_3(NCMe)_3]$, pymSH and $P(OMe)_3$

The reaction of $[Mo(CO)_3(NCMe)_3]$ with pymSH and $P(OMe)_3$ under conditions similar to those employed by Shi and co-workers affords two mononuclear compounds $[Mo(CO)_2\{P(OMe)_3\}$-$(\kappa^2\text{-pymS})_2]$ (**4**) and $[Mo(CO)_4\{P(OMe)_3\}_2]$ (**5**) in 30 and 18% yields respectively, after chromatographic separation and recrystallization (Scheme 3). Compound **4** is new, while compound **5** has been previously reported and structurally characterized.[18] We did not characterize any di- or tri-molybdenum products in this reaction.

Table 2 — Selected bond distances (Å) and angles (°) for compounds **2** and **4**

Compound	Bond distances (Å)	Bond angles (°)	Symmetry code
2	Mo01–P1= Mo01–P1[1] 2.4901(13)	P1–Mo01–P1[1] 97.89(6)	(3/2–X, +Y, 3/2–Z)
	Mo01–C1 = Mo01–C1[1] 2.039(5)	C1–Mo01–C1[1] 177.0(3)	
	Mo01–C2 = Mo01–C2[1] 1.994(5)	C2–Mo01–C2[1] 90.2(3)	
4	Mo1–P1 2.4084(8)	N1–Mo1–S1 64.45(4)	(1–X, 2–Y, –Z)
	Mo1–N1 2.2270(15)	N3–Mo1–S2 64.89(4)	
	Mo1–N3 2.2230(16)	N3–Mo1–N3 81.85(6)	
	Mo1–S1 2.5571(11)	S2–Mo1–S1 141.46(3)	
	Mo1–S2 2.5003(11)	P1[1]–Mo1–C1 103.59(6)	
	Mo1–C1 1.9698(19)	P1[1]–Mo1–C2 73.92(6)	
	Mo1–C2 1.9705(19)	S1–Mo1–C1 83.0(6)	



Scheme 3 — Schematic representation for the synthesis of [Mo(CO)$_2$(κ^2-pymS)$_2$(P(OMe)$_3$)] (**4**).

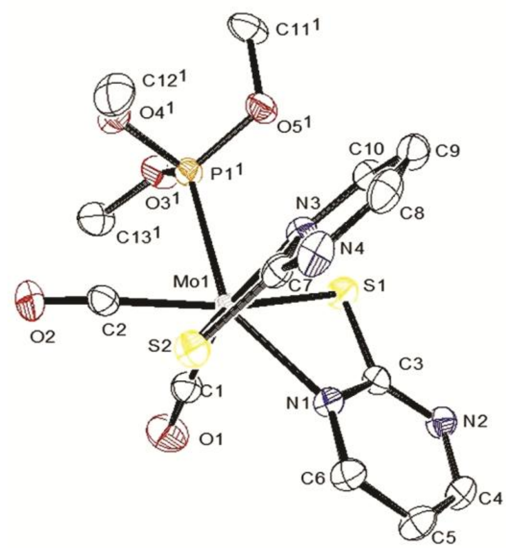


Fig. 2 — Molecular structure of one of the two crystallographically unique subunits of [Mo(CO)$_2$(κ^2-pyS)$_2$-(P(OMe)$_3$)] (**4**) showing 50% probability atomic displacement ellipsoids. Hydrogen atoms are omitted for clarity.

Two crystallographically distinct molecules were found in the asymmetric unit of **4**. Since the variations in the respective bond parameters in the two molecules are chemically insignificant, the molecular structure of one of these molecules is depicted in Fig. 2 and selected bond distances and bond angles in Table 2. The molecule **4** has a seven-coordinated molybdenum atom, comprising of two chelating pymS, two CO and a single P(OMe)$_3$ ligand. The carbonyls are situated in *cis* arrangement and the overall molecular structure is similar to the capped trigonal prismatic structures of [Mo(κ^2-pyS)$_2$(CO)$_2${P(OMe)$_3$}],[18] [M(κ^2-pyS)$_2$(CO)$_2$-(PMe$_2$Ph)],[6] [M(κ^2-pymS)$_2$(CO)$_3$][6] and [M(κ^2-pyS)$_2$-(CO)$_3$][6] (M = Mo or W). The P(OMe)$_3$ ligand occupies the capping position in the distorted monocapped trigonal prism of **4**. The average N-Mo-S chelate angle is 64.45(4)° which is similar to average N-Mo-S bond angle of 64.91(9) in [Mo(κ^2-pyS)$_2$(CO)$_2$-{P(OMe)$_3$}].[22] The Mo–P, Mo–N and Mo–S bond distances [Mo1–P1 = 2.4084(8), Mo1–N1 = 2.2270(15), Mo1–N3 = 2.2230(16), Mo1–S1 = 2.5571(11) and Mo1–S2 = 2.5003(11) Å] are consistent with their single bond designation and comparable to those bond distances reported for mononuclear molybdenum compounds [Mo(κ^2-pymS)$_4$],[16] [Mo(κ^2-pyS)$_2$(NO)$_2$][22] and [Mo(κ^2-pyS)$_2$(CO)(κ^2-dppm)].[23]

The compound **4** was also characterized by infrared, ^{1}H NMR and ^{31}P{^{1}H} NMR and found completely consistent with the solid-state structure. The IR spectrum showed two strong CO absorptions at 1963 and 1886 cm^{-1} indicating the presence of terminally bonded CO. The ^{1}H NMR spectrum shows two doublet of doublets at δ 8.48 and δ 8.37, one triplet at δ 6.79 assigned to the aromatic protons of the heterocyclic ring and a doublet at δ 3.64 due to the methyl protons of the P(OMe)$_3$ ligand. The ^{31}P{^{1}H} NMR gave a singlet at *δ* 165.3 implying a coordinated P(OMe)$_3$ ligand.

Conclusions

In summary, the one-pot reaction between $[Mo(CO)_3(NCMe)_3]$, pymSH and $P(Fu)_3$ in acetonitrile at 60 °C afforded two new compounds; a mononuclear $[Mo(CO)_4(P(Fu)_3)_2]$ (**2**) and a binuclear compound $[Mo_2(CO)_4(\mu\text{-}\kappa^2\text{-pymS})_2(P(Fu)_3)_2]$ (**3**). The reactions also yielded a previously reported eight coordinate mononuclear compound $[Mo(\kappa^2\text{-pymS})_4]$ (**1**). Compound **2** is a new example of a 6 coordinate molybdenum compound containing two $P(Fu)_3$ ligands in a *cis* position. On the other hand, an analogous reaction of $[Mo(CO)_3(NCMe)_3]$ and pyrimidine-2-thiol in the presence of a different auxiliary phosphine ligand $P(OMe)_3$ under similar reaction conditions yielded two compounds; a mononuclear bicarbonyl compound $[Mo(CO)_2(\kappa^2\text{-pymS})_2(P(OMe)_3)]$ (**4**) where the pymS ligand adopted κ^2 coordination fashion while $P(OMe)_3$ is terminally coordinated. The second compound is a known mononuclear six coordinate Mo compound $[Mo(CO)_4(P(OMe)_3)_2]$ (**5**) bearing four terminal carbonyl and two $P(OMe)_3$ ligands. The proposed binuclear molybdenum compound **3** where the pymS, we think is coordinated in κ-fashion was characterized by IR, ^1H NMR and $^{31}P\{^1H\}$ NMR spectroscopy.

Supplementary Data

Supplementary Data associated with this article are available in the electronic form at: http://nopr.niscair.res.in/jinfo/ijca/IJCA_59A(06)741-746_SupplData.pdf. CCDC 1895417 and CCDC 1895409 contain supplementary crystallographic data for **2** and **4,** respectively. These data may be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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