

Two Compounds with $trans\text{-}[\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2]^-$ Anion

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Abstract. By refluxing a mixture of $(\text{NH}_4)_2[\text{MoCl}_5(\text{H}_2\text{O})]$ and KSCN in γ -picoline, crystalline $trans\text{-}[(\gamma\text{-pic})_2\text{H}][\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2]$ (**1**) was prepared. After addition of $\text{P}(\text{C}_6\text{H}_5)_4\text{Br}$ to the solution of **1** in acetonitrile, $trans\text{-}[\text{P}(\text{C}_6\text{H}_5)_4][\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2] \cdot 2\text{CH}_3\text{CN}$ (**2**) was isolated. Both compounds were characterized by IR spectroscopy, and their crystal structures were determined. X-ray structural analysis revealed the presence of $trans\text{-}[\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2]^-$ anions, with N-bonding of the NCS groups in both compounds. The positions of the $\nu(\text{CN})$ and $\delta(\text{NCS})$ bands are in agreement with the crystal-structure results. The results of analyses were compared with the results of the related $trans\text{-}(\text{py}_2\text{H})[\text{Mo}(\text{NCY})_4\text{py}_2]$ ($\text{Y} = \text{S}, \text{O}$) complexes.

Keywords: molybdenum(III), thiocyanato-*N*, γ -picoline, crystal structures

INTRODUCTION

A series of compounds with trivalent d-transition elements, containing $trans\text{-}[\text{MX}_4\text{py}_2]^-$, and $mer\text{-}[\text{MX}_3\text{py}_3]$ ($\text{M} = \text{Ti}, \text{V}, \text{Ta}, \text{Cr}, \text{Mo}, \text{W}, \text{Tc}, \text{Re}, \text{Fe}, \text{Ru}, \text{Os}, \text{Rh}, \text{Ir}$; $\text{X} = \text{Br}, \text{Cl}$; $\text{py} = \text{pyridine}, \gamma\text{-picoline}$) coordination species were structurally characterized. There are also several $cis\text{-}[\text{MX}_4\text{py}_2]^-$ ($\text{M} = \text{Mo}, \text{Re}, \text{Os}$) complexes whose structures have been described.¹ Much rarer are the corresponding $trans\text{-}[\text{M}(\text{NCY})_4\text{L}_2]^-$ and $mer\text{-}[\text{M}(\text{NCY})_3\text{L}_3]$ ($\text{Y} = \text{S}, \text{O}$) compounds. Only the structures of two cyanato-*N* ($[\text{Mo}(\text{NCO})_3\text{py}_3]$,² $(\text{py}_2\text{H})[\text{Mo}(\text{NCO})_4\text{py}_2]$)² and seven thiocyanato-*N* ($[\text{Os}(\text{NCS})_3\text{py}_3]$,³ $[\text{Cr}(\text{NCS})_3(\gamma\text{-pic})_3] \cdot 4/3\gamma\text{-pic}$,⁴ $\text{K}[\text{Cr}(\text{NCS})_4\text{py}_2] \cdot 4\text{py}$,⁴ $\text{Hg}[\text{Co}(\text{NCS})_4\text{py}_2]$,⁵ $(\gamma\text{-picH})_2[\text{Mn}(\text{NCS})_4(\gamma\text{-pic})_2] \cdot 2\gamma\text{-pic}$,⁶ $(\gamma\text{-picH})_2[\text{Fe}(\text{NCS})_4(\gamma\text{-pic})_2] \cdot 2\gamma\text{-pic}$,⁶ $(\text{py}_2\text{H})[\text{Mo}(\text{NCS})_4\text{py}_2]$)⁷ complexes have been reported in the literature.

EXPERIMENTAL

All starting compounds and solvents, which were obtained from commercial sources, were of analytical grade quality and used without further purification. $(\text{NH}_4)_2[\text{MoCl}_5(\text{H}_2\text{O})]$ was prepared by the electrolysis of a solution prepared from MoO_3 (1 M) and NH_4Cl (2 M) in concentrated hydrochloric acid, and was checked by elemental (C, H, N) analysis, the powder diffraction technique^{8,9} and vibration spectroscopy.

Syntheses

$trans\text{-}[(\gamma\text{-pic})_2\text{H}][\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2]$ (**1**)

A mixture of KSCN (10.0 g, 0.103 mol) and $(\text{NH}_4)_2[\text{MoCl}_5(\text{H}_2\text{O})]$ (5.0 g, 15 mmol) in γ -picoline (25 mL) was refluxed in an argon atmosphere for 8 hours at *ca.* 115 °C. The solution was cooled down to room temperature and the resulting crystalline product was filtered off. To remove by-products and the remainder of the reactants, the product was washed with water, subsequently with methanol and dried in a vacuum (4.5 g, 6.4 mmol, 42 %).

Anal. Calcd. mass fractions of elements, w/%, for $\text{C}_{28}\text{H}_{29}\text{N}_8\text{MoS}_4$ ($M_r = 701.77$) are: C 47.92, H 4.17, N 15.97; Found: C 47.91, H 4.08, N 15.69; $\Lambda(\text{CH}_3\text{CN})$ ($\text{Scm}^2\text{mol}^{-1}$): 132; IR (nujol) $\tilde{\nu}_{\text{max}}/\text{cm}^{-1}$: 2046 $\nu(\text{NC})$, 1505, 805, 554, 496 ($\gamma\text{-pic}$), 482 $\delta(\text{NCS})$, 312 $\nu(\text{Mo-N}(\text{NCS}))$, 280 $\nu(\text{Mo-N}(\gamma\text{-pic}))$.

Crystallization of 1. 0.50 g (0.71 mmol) of **1** was dissolved in an argon atmosphere in 80 mL of acetonitrile and the suspension was stirred for 30 min, filtered and gradually cooled down to 8 °C. Yellow crystals suitable for X-ray structure determination were isolated (123 mg, 0.175 mmol, 24.6 %).

$trans\text{-}[\text{P}(\text{C}_6\text{H}_5)_4][\text{Mo}(\text{NCS})_4(\gamma\text{-pic})_2] \cdot 2\text{CH}_3\text{CN}$ (**2**)

1.5 g (2.1 mmol) of **1** was dissolved in an argon atmosphere in 45 mL of acetonitrile, 1.0 g (2.4 mmol) of $[\text{P}(\text{C}_6\text{H}_5)_4]\text{Br}$ was added, and the solution was

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filtered and slowly cooled down to -4 °C. Brown-green single crystals were collected after several days (1.26 g, 1.35 mmol, 63.1 %).

Anal. Calcd. mass fractions of elements, w/%, for C₄₄H₄₀N₈MoPS₄ ($M_r = 936.04$) are: C 56.46, H 4.31, N 11.97; Found: C 56.21, H 4.34, N 11.01; $A(\text{CH}_3\text{CN})$ (S cm² mol⁻¹): 134; IR(nujol) $\tilde{\nu}_{\text{max}}$ /cm⁻¹: 2040 $\nu(\text{NC})$, 1501 (γ -pic), 1586, 1187, 1163, 1108, 998 ([P(C₆H₅)₄]⁺), 813 (γ -pic), 756, 691, 616 ([P(C₆H₅)₄]⁺), 551 (γ -pic), 528 ([P(C₆H₅)₄]⁺), 496 (γ -pic), 456, 485 (sh) $\delta(\text{NCS})$, 309 $\nu(\text{Mo-N}(\text{NCS}))$, 277 $\nu(\text{Mo-N}(\gamma\text{-pic}))$.

Measurements

Elemental CHN analyses were performed by a Perkin-Elmer 2400 CHN elemental analyzer.

Conductivity measurements were performed on an Iskra MA 5964 conductometer (with an Iskra HEK 1213 electrode). Samples were prepared as 1.0×10^{-3} M solutions in acetonitrile and measured at room temperature.

Infrared spectra were recorded as a nujol suspension by a Perkin-Elmer 1720 X FT-IR instrument in the 4000–400 cm⁻¹ region (with KBr plates) and a Perkin-Elmer 2000 FT-IR instrument in the 700–30 cm⁻¹ region.

X-ray powder diffraction data of the prepared starting material, (NH₄)₂[MoCl₅(H₂O)], was obtained using an Enraf Nonius Guinier camera with Cu-K α radiation.

The X-ray single crystal data were collected using Mo-K α radiation on an Enraf-Nonius CAD4 and Nonius Kappa CCD diffractometer for **1** and **2**, respectively. The diffraction data were processed using XTAL3.6¹⁰ and DENZO¹¹ for **1** and **2**, respectively. Structures of both compounds were solved by direct methods using SIR97¹² program and were refined by full matrix least squares on F using XTAL3.6¹⁰. The resulting crystal data and details concerning data collection and refinement for both compounds are quoted in Table 1. A drawing of the asymmetric unit of **1** (Figure 1), and of the coordination anion of **2** (Figure 2) were produced using PLATON.¹³

Table 1. Crystal data, data collection and structure refinement parameters for compounds **1** and **2**

Compound	1	2
Chemical formula	C ₂₈ H ₂₉ MoN ₈ S ₄	C ₄₄ H ₄₀ N ₈ MoPS ₄
Formula weight	701.077	936.04
Crystal system	monoclinic	monoclinic
Space group	<i>C</i> 2/ <i>c</i> (No. 15)	<i>P</i> 2 ₁ / <i>n</i> (No. 14)
<i>a</i> / Å	31.543(8)	16.8069(2)
<i>b</i> / Å	12.247(4)	13.7730(2)
<i>c</i> / Å	19.495(7)	19.7742(3)
β / °	118.52(3)	99.9615(6)
<i>Z</i>	8	4
<i>V</i> / Å ³	6617(4)	4508.35(11)
<i>D</i> _{calc} / g cm ⁻³	1.409	1.379
$\mu(\text{Mo-K}\alpha)$ / cm ⁻¹	0.68	0.552
Crystal shape	plate	needle
Crystal color	yellow	brown-green
Crystal size / mm	0.76 × 0.50 × 0.032	0.68 × 0.08 × 0.08
$\lambda(\text{Mo-K}\alpha)$ / Å	0.71069	0.71073
Temperature / K	293(2)	115(1)
Scan type	ω	ω
Intensity change / %	-1.47	
θ (max) / °	28.0	27.5
No. of refl. measured	17669	64570
No. of indep. refl.	7947	10306
Observed reflections	3546 ($I > 2.5 \sigma(I)$)	7350 ($F^2 > 2.5 \sigma(F^2)$)
No. of contributing reflections	5902	9079
No. of parameters	370	553
Final <i>R</i> indices: <i>R</i> , <i>R</i> _w	0.074, 0.081	0.045, 0.030
Largest peak and hole ($\Delta\rho_{\text{min}}/\Delta\rho_{\text{max}}$) / e Å ⁻³	-1.994, 1.628	-1.13, 0.75
<i>R</i> _{int}	0.046	0.072

RESULTS AND DISCUSSION

Crystal Structures

X-ray diffraction analyses reveals that compounds **1** and **2** crystallize in monoclinic space groups, *C*2/*c* for **1** and *P*2₁/*n* for **2**, both containing *trans*-[Mo(NCS)₄(γ -pic)₂]⁻ anions (Figure 1, Figure 2). The coordination polyhedron of the MoN₆ moiety is an octahedron, slightly elongated via the (γ -pic5)N5–Mo–N6(γ -pic6) axes. The best planes through γ -picoline-ligand rings are almost parallel to each other, 4.4(4)° for **1**, and 9.15(8)° for **2**, and are almost perpendicular to the (MoNCS) plane, defined by the molybdenum and N atoms of the

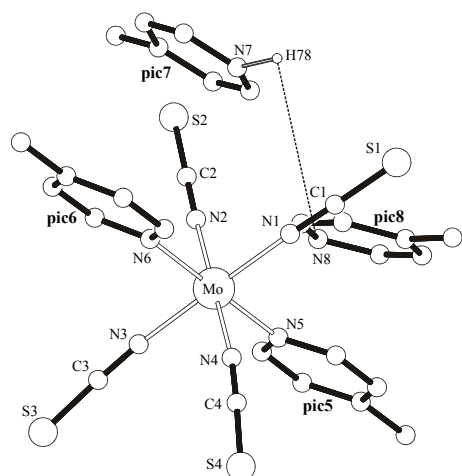


Figure 1. PLATON¹³ drawing of the *trans*-[(γ -pic)₂H][Mo(NCS)₄(γ -pic)₂]⁻ anion of **1**. Hydrogen atoms (with the exception of H78) are omitted for clarity.

thiocyanato-*N* groups (N1, N2, N3, N4). The deviation from a rectangular angle is larger in the case of compound **2** (87.42(6) and 85.65(8)°), than in that of **1** (89.8(3) and 89.4(3)°). In the related *trans*-(py₂H)-[Mo(NCS)₄py₂]⁷ and *trans*-(py₂H)[Mo(NCO)₄py₂]² the pyridine rings are in »staggered« (85.9(4)°) and »perfect staggered« conformation, and the (MoNCS) plane-pyridine angles are almost 90° and 45°, respectively.

The Mo–N(thiocyanato) bond distances are within the 2.080(9)–2.108(10) Å range for **1**, and 2.079(2)–2.093(2) Å range for **2**. Similar bond lengths, from 2.083(7) to 2.106(7) Å, were found in the complex *trans*-(py₂H)[Mo(NCS)₄py₂]³. The Mo–N(γ -pic) distances in **2** are *ca.* 0.01 Å longer than in **1** (Table 2), and comparable with the Mo–N(γ -pic) distances found in compounds with [MoX₄(γ -pic)₂]⁻ (X = Br, Cl) species, ranging from 2.18 to 2.25 Å.^{14–16}

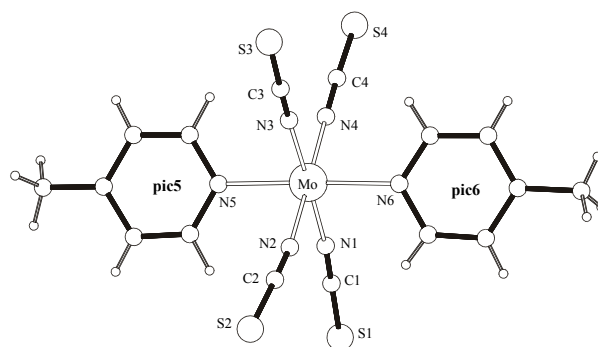


Figure 2. PLATON¹³ drawing of the *trans*-[Mo(NCS)₄(γ -pic)₂]⁻ anion of **2** with an atom-numbering scheme.

Table 2. Selected distances (expressed in Å) and angles (expressed in °) in *trans*-[Mo(NCS)₄(γ -pic)₂]⁻ for compounds **1** and **2**

	1	2		1	2
Mo–N1	2.080(9)	2.0822(19)	Mo–N2	2.108(10)	2.091(2)
Mo–N3	2.090(9)	2.093(2)	Mo–N4	2.094(10)	2.079(2)
Mo–N5	2.176(8)	2.184(2)	Mo–N6	2.173(8)	2.186(2)
N1–C1	1.154(14)	1.147(3)	N2–C2	1.146(17)	1.099(3)
N3–C3	1.159(14)	1.114(3)	N4–C4	1.153(17)	1.146(3)
C1–S1	1.613(12)	1.636(2)	C2–S2	1.613(14)	1.675(2)
C3–S3	1.622 (11)	1.665(2)	C4–S4	1.616(14)	1.635(3)
N1–Mo–N2	87.5(4)	89.37(8)	N1–Mo–N3	178.8(3)	178.37(8)
N1–Mo–N4	92.9(4)	92.09(8)	N1–Mo–N5	87.6(3)	88.00(7)
N1–Mo–N6	92.0(3)	89.33(7)	N2–Mo–N3	92.6(4)	89.34(8)
N2–Mo–N4	179.5(4)	178.52(8)	N2–Mo–N5	91.3(3)	89.62(8)
N2–Mo–N6	88.7(3)	90.47(8)	N3–Mo–N4	87.0(4)	89.19(8)
N3–Mo–N5	91.3(3)	90.98(7)	N3–Mo–N6	89.2(3)	91.69(8)
N4–Mo–N5	88.3(3)	90.22(7)	N4–Mo–N6	91.7(3)	89.77(8)
N5–Mo–N6	179.6(3)	177.33(7)	Mo–N2–C2	171.7(9)	173.5(2)
Mo–N1–C1	172.4(9)	174.01(19)	Mo–N3–C4	168.0(8)	175.8(2)
Mo–N3–C3	169.3(9)	176.3(2)	N2–C2–S2	179.0(11)	178.2(2)
N1–C1–S1	178.7(9)	179.8(2)	N4–C4–S4	177.9(9)	177.6(2)
N3–C3–S3	177.5(11)	178.6(2)			

The structure of **1** is completed by $[(\gamma\text{-pic})_2\text{H}]^+$ cations, the rings of which are held together by a strong hydrogen bond, with the N7...N8 contact distance being 2.68(2) Å and an angle of 152.7°. In other structures containing $[(\gamma\text{-pic})_2\text{H}]^+$ cations, the N...N contact distances range between 2.629 and 3.071 Å.^{17–23} The angle between the best planes through the γ -picoline-cation rings is 18.2(7)°. In *trans*-(py_2H)[Mo(NCY)₄py₂] (Y = S, O) this angle is 70.7(6)° for the thiocyanato and precisely 90° for the cyanato compound.^{2,7} In the compound **2**, $[\text{P}(\text{C}_6\text{H}_5)_4]^+$ cations were found, the P–C(phenyl) distances ranging from 1.793(2) to 1.802(2) Å. Both structures are stabilized by π - π stacking between γ -picoline-anion rings from the neighbouring asymmetric units (mean interplanar separation and centroid...centroid distances, 3.620(7) and 3.474, 3.432 Å for **1**, and 3.7055(13) and 3.540, 3.548 Å for **2**), and in the case of compound **1** also between the γ -picoline-anion and γ -picoline-cation rings of the same unit (mean interplanar separation, 3.894(9) and 3.779(10) Å, and centroid...centroid distances, 3.352, 3.546 and 3.476, 3.439 Å).

Spectroscopy

The broad band within the 2100–1900 cm^{-1} region, at 2046 and 2040 for compounds **1** and **2**, respectively, indicates N-bonding of the NCS ligands in the *trans*-[Mo(NCS)₄(γ -pic)₂][−] anion. In the spectrum of the related compound with pyridine this band is observed at 2052 cm^{-1} .⁷ Bands at *ca.* 1500, 810 550 and 495 cm^{-1} (1505, 805, 554, 496 cm^{-1} for **1**, and 1501, 813, 551, 496 cm^{-1} for **2**) can be attributed to the vibrations of the coordinated γ -picoline molecules (Figure 3).^{24,25} Within the interval of $\delta(\text{NCS})$ frequencies, 450–490 cm^{-1} for the thiocyanato-*N* compounds,^{24,26} a weakly split band at 482 cm^{-1} is observed in the spectrum of **1**, and in the spectrum of **2** at 456 cm^{-1} with a noticeable shoulder at 485 cm^{-1} . The $\delta(\text{NCS})$ band for the related *trans*-(py_2H)[Mo(NCS)₄py₂] appeared as a single band at 481 cm^{-1} .⁷ All Mo–N stretching bands (Mo–N(NCS), and Mo–N(γ -pic)) occurred only in the far-infrared region. The most noticeable is band of the stretching vibration of the Mo–N(NCS) bond, 312 cm^{-1} for **1**, and 309 cm^{-1} for **2**. In the spectrum of *trans*-(py_2H)[Mo(NCS)₄py₂] a band at 311 cm^{-1} is observed (Figure 4).⁷ The bands at 1586, 1187, 1163, 1108, 998, 756, 691, 616, 528 cm^{-1} probably belong to the vibrations of the tetraphenylphosphonium cation in **2** (Figure 3).

CONCLUSION

In the prepared compounds the octahedrally coordinated species *trans*-[Mo(NCS)₄(γ -pic)₂][−] were identified. The analyses (X-ray diffraction analysis, vibrational

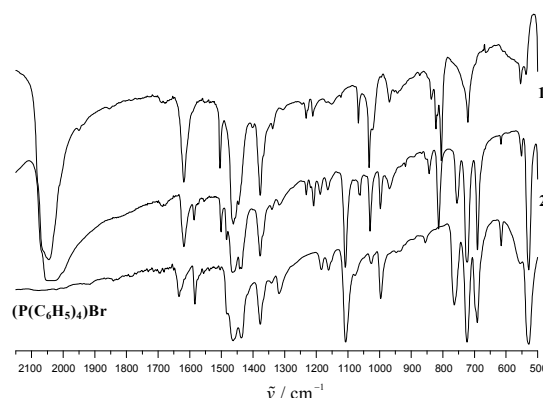


Figure 3. Vibrational spectra of the compounds **1** and **2**, and the spectrum of the commercial $[\text{P}(\text{C}_6\text{H}_5)_4]\text{Br}$, within the 2150–500 cm^{-1} region.

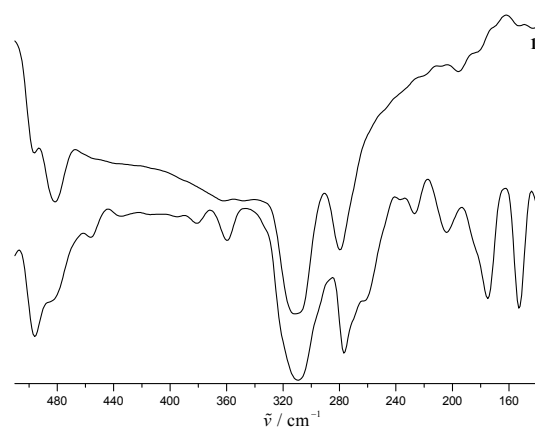


Figure 4. Vibrational spectra of the compounds **1** and **2**, within the 520–140 cm^{-1} region.

spectroscopy) reveal that the NCS groups are bonded to the Mo atom via nitrogen atoms, similarly as in the related *trans*-(py_2H)[Mo(NCY)₄py₂] (Y = S, O) complexes.^{2,7} In the anions of both presented compounds, the N-donor-aromatic rings are in »eclipsed«, while in the related pyridine complexes in »staggered« conformation.

Supplementary Materials. Crystallographic data for structures reported in this paper have been deposited with the *Cambridge Data Center*, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@cc-dc.ac.uk) and can be obtained on request, free of charge, by quoting the publication citation and the deposition numbers 667913 and 667914.

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SAŽETAK

Dva spoja s $\text{trans-[Mo(NCS)}_4(\gamma\text{-pic)}_2\text{]}^-$ anionom

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Refluksiranjem smjese $(\text{NH}_4)_2[\text{MoCl}_5(\text{H}_2\text{O})]$ i KSCN u γ -pikolinu pripravljen je kristalinični $\text{trans-}[(\gamma\text{-pic)}_2\text{H}][\text{Mo(NCS)}_4(\gamma\text{-pic)}_2]$ (**1**). Dodatkom $\text{P}(\text{C}_6\text{H}_5)_4\text{Br}$ u spoj **1** otopljen u acetonitrilu izoliran je $\text{trans-}[\text{P}(\text{C}_6\text{H}_5)_4][\text{Mo(NCS)}_4(\gamma\text{-pic)}_2] \cdot 2\text{CH}_3\text{CN}$ (**2**). Oba spoja analizirana su IR spektroskopijom, te im je određena kristalna struktura. Röntgenska strukturalna analiza otkrila je prisutnost $\text{trans-}[\text{Mo(NCS)}_4(\gamma\text{-pic)}_2]^-$ aniona s N-vezanim NCS grupama u oba spoja. Položaji $\nu(\text{CN})$ i $\delta(\text{NCS})$ vrpce u skladu su s rezultatima strukturalne analize. Rezultati analize uspoređeni su s rezultatima srodnih $\text{trans-}(\text{py}_2\text{H})[\text{Mo(NCY)}_4\text{py}_2]$ ($\text{Y} = \text{S}, \text{O}$) kompleksa.