

Dedicated to blessed memory of Prof. A.A. Pasynskii

Heterometallic Nickel Complexes with *N*-Heterocyclic Carbene: Synthesis, Structure, and Thermal Decomposition

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Abstract—The reaction of $(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})\text{Cl}$ with *n*-propylmercaptane and NEt_3 in CH_2Cl_2 affords the thiolate carbene nickel complex $(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})\text{S}^n\text{Pr}$ (**I**) (SIMes is 1,3-dimesitylimidazol-2-ylidene), which reacts with $\text{W}(\text{CO})_5(\text{THF})$ to form the heterometallic complex $(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})(\mu_2\text{-S}^n\text{Pr})\text{W}(\text{CO})_5$ (**II**). The reaction of complex **I** with $(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_2(\text{THF})$ affords compound $(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})(\mu_2\text{-S}^n\text{Pr})(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_2$ (**III**). The structures of compounds **I**, **II**, and **III** are determined by X-ray structure analysis (CIF file CCDC nos. 2024873 (**I**), 2024874 (**II**), and 2024875 (**III**)). According to the data of thermogravimetry and differential scanning calorimetry, the thermal decomposition of complexes **II** and **III** occurs stepwise in ranges of 101–500 and 119–550°C, and no ligand elimination is observed.

Keywords: nickel, NHC, SIMes, thermogravimetry, differential scanning calorimetry, heterometallic complexes

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INTRODUCTION

N-Heterocyclic carbenes (NHCs) based on imidazol-2-ylidene are the ligands with simultaneously strong σ -donor and weak π -acceptor properties, which makes it possible to use them as stabilizing ligands in the chemistry of organoelement compounds [1, 2]. On the one hand, these ligands can stabilize the nickel complexes in different oxidation states [3, 4]. On the other hand, owing to the variation of the substituents at the nitrogen atoms, they can form a sufficient shielding of the metal center thus providing a high catalytic activity of compounds of this type. We have previously shown [5, 6] the possibility of synthesizing the heterometallic nickel complexes with 1,3-dimethylimidazol-2-ylidene (MeIm). The thiolate complex of cyclopentadienylnickel with 1,3-dimesitylimidazol-2-ylidene (SIMes), $(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})\text{S}^n\text{Pr}$ (**I**), is used as the metal ligand for the synthesis of the heterometallic derivatives.

EXPERIMENTAL

All procedures associated with the synthesis and isolation of the compounds were carried out under argon and in dehydrated solvents. Compound

$(\eta^5\text{-C}_5\text{H}_5)\text{Ni}(\text{SIMes})\text{Cl}$ was synthesized using a known procedure [7]. Commercial nickelocene (99%, OOO Dalkhim), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (95+%, OOO Dalkhim), and $\text{W}(\text{CO})_6$ (97%, Sigma-Aldrich) were used as received. Triethylamine ($\geq 99.5\%$, Sigma-Aldrich) was dried over molecular sieves 3E Sigma-Aldrich. UV irradiation was carried out using a DRL 250V lamp without a glass flask covered with the luminophore. Chemical analyses were carried out on an EA3000 CHNS analyzer (Euro Vector). A Bruker Alpha FT-IR spectrometer with a Platinum ATR accessory for operating in the attenuated total reflectance mode was used for IR spectroscopy. ^1H (300.13 MHz) and ^{13}C (75.4 MHz) NMR spectra were recorded on a Bruker AV 300 spectrometer, and chemical shifts are presented relative to tetramethylsilane. Simultaneous (TG–DSC) thermal analysis (TG–DSC is thermogravimetry coupled with differential scanning calorimetry) of the synthesized complexes was carried out using an SDT Q-600 thermoanalyzer. Controlled heating was conducted in Al_2O_3 crucibles in a range of 20–600°C with a rate of 10°C/min in an argon flow (flow rate 250 mL/min), and the weight of the starting sample was varied in a range of 3.5–10.5 mg.

Synthesis of (η^5 -C₅H₅)Ni(SIMes)(S^{*n*}Pr) (I). *n*-Propylmercaptane (0.1 mL, 1.105 mmol) and triethylamine (0.12 mL, 0.865 mmol) were added to a pink solution of (η^5 -C₅H₅)Ni(SIMes)Cl (0.301 g, 0.649 mmol) in toluene (15 mL). The reaction mixture was stirred for 48 h. The resulting brown solution was filtered, and the solvents were removed under reduced pressure. The obtained brown oily residue was crystallized from hexane at -24°C. The yield of the green-brown crystals was 0.282 g (87%).

For C₂₉H₃₆N₂SNi (*FW* = 503)

Anal. calcd., %	C, 69.20	H, 7.21	N, 5.56
Found, %	C, 69.32	H, 7.32	N, 5.47

IR (ν , cm⁻¹): 3170 w, 3135 w, 3102 w, 3018 w, 2951 m.br, 2913 m.br, 2862 w, 2194 w, 1718 w.br, 1651 w, 1607 w, 1548 w, 1520 w, 1483 m.br, 1459 m.br, 1396 s, 1372 m, 1350 m, 1322 s, 1281 w, 1264 s, 1246 m, 1218 m, 1161 w, 1113 w, 1082 m, 1054 m, 1031 m, 1015 s, 985 m, 960 m, 923 s, 890 m, 848 s, 834 m, 824 m, 775 vs, 741 m, 725 vs, 692 vs, 645 m, 593 m, 575 m, 501 w, 452 w, 430 m. ¹H NMR (C₆D₆), δ , ppm: 1.01 (t, ³*J* = 7.11 Hz, 3H, Me, Pr), 1.53–1.77 (m, 4H, 2CH₂, Pr), 2.15 (s, 6H, 2Me, Mes), 2.22 (s, 4Me, Mes), 4.92 (s, 5H, Cp), 6.17 (s, 2H, 2CH, Im), 6.85 (s, 4H, 2CH, Mes). ¹³C{H} NMR (C₆D₆), δ , ppm: 15.0 (Me, Pr), 19.3 Me, Mes), 21.4 (Me, Mes), 28.8 (CH₂, Pr), 34.9, (CH₂, Pr), 91.7 (Cp), 123.8 (CH, Im), 129.6 (Mes), 136.6 (Mes), 138.0 (Mes), 139.0 (Mes), 174.8 (C, Im).

Synthesis of (η^5 -C₅H₅)Ni(SIMes)(μ_2 -S^{*n*}Pr)W(CO)₅ (II). *n*-Propylmercaptane (0.08 mL) and triethylamine (0.09 mL) were added to a pink solution of (η^5 -C₅H₅)Ni(SIMes)Cl (0.201 g, 0.433 mmol) in toluene (10 mL). The reaction mixture was stirred for 48 h. The resulting brown solution was filtered, and the solvents were removed under reduced pressure. A yellow solution obtained by the UV irradiation of W(CO)₆ (0.152 g) in THF (20 mL) for 30 min with water cooling was added to the brown oily residue. The solvent was removed under reduced pressure, and the residue was extracted with boiling hexane. Brown crystals were formed on storage at -24°C for 18 h and recrystallized from a toluene–hexane (1 : 1) mixture. The yield was 0.179 g (49%).

IR (ν , cm⁻¹): 2925 vw, 2057 vw, 1962 vw, 1904 vs, 1874 m, 1486 vw.br, 1400 vw, 1377 vw, 1319 vw, 1266 vw.br, 1034 vw.br, 925 vw, 853 vw, 785 vw, 734 vw, 698 vw, 678 vw, 599 vw, 584 vw.br. ¹H NMR (C₆D₆), δ , ppm: 0.70 (t, ³*J* = 7.07 Hz, 3H, Me, Pr), 1.17–1.37 (m, 2H, CH₂, Pr), 1.58–1.74 (m, 2H, CH₂, Pr), 2.03 (s, 12H, 4Me, Mes), 2.13 (s, 6H, 2Me, Mes), 4.89 (s, 5H, Cp), 5.99 (s, 2H, 2CH, Im), 6.78 (s, 4H, CH, Mes). ¹³C{H} NMR (C₆D₆), δ , ppm: 14.3 (Me, Pr), 19.1 Me, Mes), 21.4 (Me, Mes), 26.9 (CH₂, Pr),

44.4 (CH₂, Pr), 93.4 (Cp), 125.0 (CH, Im), 130.0 (Mes), 135.9 (Mes), 137.3 (Mes), 139.4 (Mes), 168.1 (C, Im), 201.1 (CO), 201.5 (CO).

For C₃₄H₃₆N₂O₅SNiW (*FW* = 827)

Anal. calcd., %	C, 49.36	H, 4.39	N, 3.38
Found, %	C, 49.78	H, 5.43	N, 3.38

Synthesis of (η^5 -C₅H₅)Ni(SIMes)(μ_2 -S^{*n*}Pr)(η^5 -C₅H₅)Mn(CO)₂ (III). A red solution obtained by the UV irradiation of C₅H₅Mn(CO)₃ (0.06 g, 0.24 mmol) in THF (15 mL) at -40°C for 50 min was added to Cp(Mes2Im)SNiPr (0.12 g, 0.24 mmol). The solvent was removed under reduced pressure. The resulting dark green oily residue was triturated with hexane (30 mL), dried in vacuo, and crystallized from a toluene–hexane (1 : 1) mixture at -24°C for 18 h. The yield of the dark green crystals was 0.085 g (52%).

IR (ν , cm⁻¹): 3129 vw.br, 2958 w.br, 2919 vw.br, 2866 vw, 1896 vs, 1827 vs.br, 1608 vw, 1567 vw, 1485 w.br, 1461 w.br, 1422 w, 1397 w, 1375 w, 1355 w, 1318 m, 1264 m, 1224 w, 1163 vw, 1112 vw, 1081 w, 1013 w.br, 974 vw, 960 vw, 924 w, 894 vw, 852 w, 836 w, 785 m, 735 m, 697 m, 660 w, 611 m, 586 m, 525 w, 477 w, 432 vw. ¹H NMR (C₆D₆), δ : 0.80 (t, ³*J* = 7.06 Hz, 3H, Me, Pr), 1.25 (m, 2H, CH₂, Pr), 1.56 (m, 2H, CH₂, Pr), 2.09 (s, 12H, Me, Mes), 2.17 (s, 6H, Me, Mes), 4.36 (s, 5H, Cp), 5.05 (s, 5H, Cp), 6.09 (s, 2H, 2CH, Mes₂Im), 6.85 (s, 4H, CH, Mes). ¹³C{H} NMR (C₆D₆), δ , ppm: 14.8 (Me, Pr), 19.3 Me, Mes), 21.5 (Me, Mes), 26.3 (CH₂, Pr), 45.4 (CH₂, Pr), 83.2 (Cp), 93.2 (Cp), 124.3 (CH, Im), 130.0 (Mes), 136.0 (Mes), 137.5 (Mes), 139.3 (Mes), 172.8 (C, Im), 238.1 (CO).

For C₃₆H₄₁MnN₂NiO₂S (*FW* = 679)

Anal. calcd., %	C, 63.64	H, 6.08	N, 4.12
Found, %	C, 63.58	H, 6.21	N, 3.98

X-ray structure analysis (XSA). Diffraction data for compounds I and II were obtained at 100 K and those for compound III were obtained at 240 K on a Bruker SMART APEX II diffractometer equipped with a Photon two-coordinate detector (graphite monochromator, $\lambda(\text{MoK}\alpha) = 0.71073 \text{ \AA}$, ω scan mode). An absorption correction was applied by the method of multiple measurements of equivalent reflections using the SADABS program [8]. The crystal structures were solved using the SHELXT program and refined by full-matrix least squares in the anisotropic (except for hydrogen atoms) approximation using the SHELXL [9] and OLEX2 [10] programs. The positions of the hydrogen atoms of the organic ligands were calculated geometrically and refined by the riding model. The crystallographic data and details of diffraction experiments are presented in Table 1.

Table 1. Crystallographic data and structure refinement details for compounds **I–III**

Parameter	Value		
	I	II	III
Empirical formula	C ₂₉ H ₃₆ N ₂ NiS	C ₃₄ H ₃₆ N ₂ NiO ₅ SW	C ₃₆ H ₄₁ MnN ₂ NiO ₂ S
<i>FW</i>	503.37	827.27	679.42
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
<i>a</i> , Å	8.6491(2)	11.0741(5)	10.8085(4)
<i>b</i> , Å	19.1568(4)	16.0171(8)	11.4279(4)
<i>c</i> , Å	16.0388(4)	19.7876(9)	15.6174(6)
α , deg	90	93.160(2)	75.4760(1)
β , deg	101.282(1)	93.460(2)	76.4640(1)
γ , deg	90	104.531(2)	66.5140(1)
<i>V</i> , Å ³	2606.10(10)	3382.4(3)	1693.04(11)
<i>Z</i>	4	4	2
ρ_{calc} , g/cm ³	1.283	1.624	1.333
μ , mm ⁻¹	0.844	4.058	1.024
<i>F</i> (000)	1072.0	1648.0	712.0
Crystal size, mm	0.21 × 0.17 × 0.15	0.13 × 0.13 × 0.1	0.23 × 0.17 × 0.12
Scan range over θ , deg	4.98–63.754	4.47–61.008	4.156–58.288
Range of indices <i>hkl</i>	–12 ≤ <i>h</i> ≤ 12, –27 ≤ <i>k</i> ≤ 25, –23 ≤ <i>l</i> ≤ 22	–15 ≤ <i>h</i> ≤ 13, –22 ≤ <i>k</i> ≤ 20, –28 ≤ <i>l</i> ≤ 27	–13 ≤ <i>h</i> ≤ 14, –15 ≤ <i>k</i> ≤ 15, –19 ≤ <i>l</i> ≤ 21
Measured reflections	51327	40541	18470
Independent reflections (<i>R</i> _{int})	8205 (0.0352)	18132 (0.0287)	8474 (0.0248)
Reflections with <i>I</i> > 2 σ (<i>I</i>)	7080	15289	11223
GOOF for <i>F</i> ²	1.060	1.021	1.058
<i>R</i> factors for <i>I</i> ≥ 2 σ (<i>I</i>)	<i>R</i> ₁ = 0.0433, <i>wR</i> ₂ = 0.0900	<i>R</i> ₁ = 0.0321, <i>wR</i> ₂ = 0.0585	<i>R</i> ₁ = 0.0512, <i>wR</i> ₂ = 0.1160
<i>R</i> factors for all reflections	<i>R</i> ₁ = 0.0547, <i>wR</i> ₂ = 0.0943	<i>R</i> ₁ = 0.0425, <i>wR</i> ₂ = 0.0618	<i>R</i> ₁ = 0.0731, <i>wR</i> ₂ = 0.1272
Residual electron density (max/min), e/Å ³	0.64/–0.45	1.19/–0.96	0.71/–0.47

The full tables of interatomic distances and bond angles, coordinates of atoms, and atomic shift parameters were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC nos. 2024873 (**I**), 2024874 (**II**), and 2024875 (**III**); <https://www.ccdc.cam.ac.uk/structures/>).

RESULTS AND DISCUSSION

Thiolate complexes based on cyclopentadienyl-nickel containing sterically loaded *N*-heterocyclic carbene ligands can be synthesized either by the treatment of the 17-electron complex ($\eta^5\text{-C}_5\text{H}_5$)Ni(NHC) with diaryl sulfide [11], or by the synthetically simpler

substitution of halogen by SR in ($\eta^5\text{-C}_5\text{H}_5$)Ni(NHC)Cl [12].

The use of the second approach made it possible to synthesize the ($\eta^5\text{-C}_5\text{H}_5$)Ni(SIMes)S^{*n*}Pr complex (**I**) by the reaction of ($\eta^5\text{-C}_5\text{H}_5$)Ni(SIMes)Cl with *n*-propylmercaptane and triethylamine. The single crystals of complex **I** were obtained by vapor diffusion. According to the XSA data (Fig. 1), the Ni–S and Ni–C bond lengths in compound **I** are 2.1873(5) and 1.874(2) Å, respectively, which are lower than the sum of covalent radii (SCR): $r_{\text{Ni}} + r_{\text{S}} = 2.29$ and $r_{\text{Ni}} + r_{\text{C}} = 1.97$ Å [13].

The reaction of complex **I** with the W(CO)₅(THF) adduct, which was synthesized by the UV irradiation

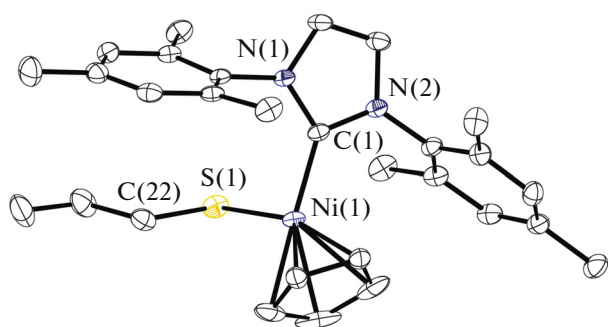


Fig. 1. Molecular structure of complex **I** (thermal ellipsoids of atoms of 50% probability). Hydrogen atoms are omitted for clarity. Selected bond lengths and bond angles: Ni(1)–S(1) 2.1873(5), Ni(1)–C(1) 1.874(2), N(2)–C(1) 1.368(2), and N(1)–C(1) 1.367(2) Å and C(22)S(1)Ni(1) 102.98(6)°.

of a solution of $W(CO)_6$ in THF, affords the $(\eta^5-C_5H_5)Ni(SiMe_3)(\mu_2-S^iPr)W(CO)_5$ complex (**II**) bearing the bridging thiolate ligand.

The IR spectrum of complex **II** exhibits bands at 2057, 1962, 1904, and 1874 cm^{-1} corresponding to stretching vibrations of the carbonyl groups of the $W(CO)_5$ fragment. According to the XSA data (Fig. 2), the Ni–S bond lengths in complex **II** (2.2139(8) and 2.2066(8) Å) are lower than the SCR ($SCR = r_{Ni} + r_S = 2.29$ Å [13]) but higher than a similar bond length in complex **I** and in the $(\eta^5-C_5H_5)Ni(MeIm)(\mu_2-S^iPr)W(CO)_5$ complex (**IIa**) with a similar structure (2.1933(7) Å) [5]. The Ni–C bonds with the carbene ligand are 1.903(3) and 1.900(3) Å, which are lower than the corresponding SCR ($r_{Ni} + r_C = 1.97$ Å) but higher than those in complex **IIa** (1.882(3) and 1.879(3) Å). This indicates a higher donor ability of the MeIm fragment compared to SiMe₃. The W–S bond lengths (2.5800(7) and 2.5931(7) Å) are lower than the corresponding SCR ($R_{Ni} + R_C = 1.97$ Å, $R_W + R_S = 2.67$ Å [13]) and comparable with those for complex **IIa**. In complex **II**, the NiSW angles are 116.46(3)° and 117.30(3)°. An increase in these values over those in complex **IIa** (109.66(3)° and 112.69(3)°) can be explained by the stronger steric repulsion of *N*-heterocyclic carbene and carbonyl groups of the $W(CO)_5$ fragment compared to complex **Ia**.

The reaction of complex **I** with the adduct $(\eta^5-C_5H_5)Mn(CO)_2(THF)$, which was synthesized by the UV irradiation of a solution of $(\eta^5-C_5H_5)Mn(CO)_3$ in THF, affords the heterometallic complex $(\eta^5-C_5H_5)Ni(SiMe_3)(\mu_2-S^iPr)(\eta^5-C_5H_5)Mn(CO)_2$ (**III**).

The IR spectrum of complex **III** exhibits bands at 1896 and 1827 cm^{-1} corresponding to stretching vibrations of the carbonyl groups of the $(\eta^5-C_5H_5)Mn(CO)_2$ fragment. The corresponding frequencies for the close in structure $(\eta^5-C_5H_5)Ni(MeIm)(\mu_2-$

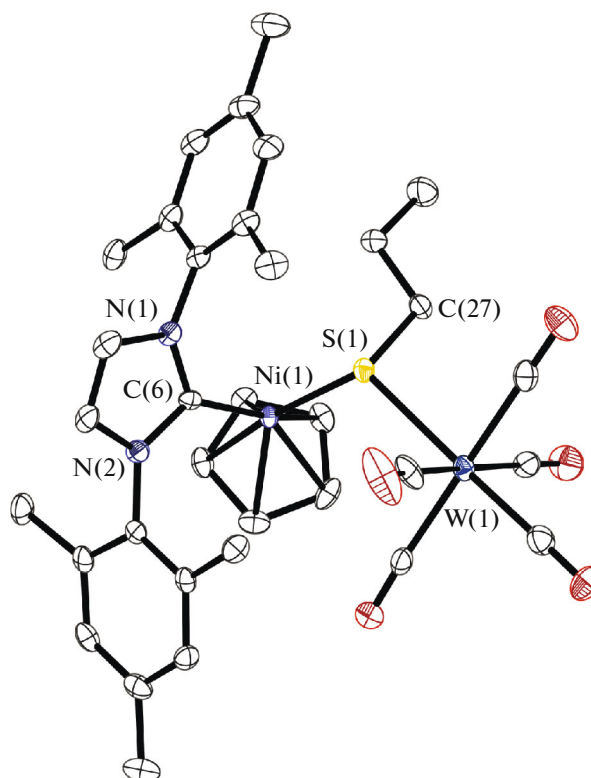


Fig. 2. Molecular structure of complex **II** (thermal ellipsoids of atoms of 50% probability). Hydrogen atoms are omitted for clarity. One of two independent molecules is shown. Selected bond lengths and bond angles: W(1)–S(1) 2.5931(7), W(1)–C(2) 2.045(3), W(1)–C(4) 2.036(3), W(1)–C(3) 2.048(4), W(1)–C(1) 1.975(3), W(1)–C(5) 2.035(4), Ni(1)–S(1) 2.2139(8), and Ni(1)–C(6) 1.903(3) Å and Ni(1)S(1)W(1) 116.46(3)°.

$S^iPr)Mn(CO)_2(\eta^5-C_5H_5)$ complex (**IIIa**) are 1891 and 1803 cm^{-1} [6]. According to the XSA data (Fig. 3), the Ni–S, Ni–C, and Mn–S bond lengths (2.2153(7), 1.898(3), and 2.3538(8) Å) are higher than the corresponding values in complex **IIa** (2.2087, 1.884, and 2.3308 Å) but lower than the SCR ($R_{Ni} + R_S = 2.29$ Å, $R_{Ni} + R_C = 1.97$ Å, $R_{Mn} + R_S = 2.44$ Å).

According to the TG–DSC data, the thermal decomposition of complex **II** is stepwise: one CO molecule is lost at 101–137°C, more two CO molecules are lost in a range of 137–156°C, the remained two CO molecules are eliminated in a range of 156–190°C, and other organic ligands are eliminated partially without pronounced boundaries to 500°C. The further heating to 550°C does not result in an additional mass loss. The residue (47.21%) can consist of nickel and tungsten sulfides (NiWS, 33.19%) contaminated with the pyrolysis products of the ligands (14.02%).

The thermal decomposition of complex **III** proceeds stepwise but without distinct boundaries: the organic ligands are partially eliminated at 119–550°C. The residue (47.21%) can consist of nickel and manga-

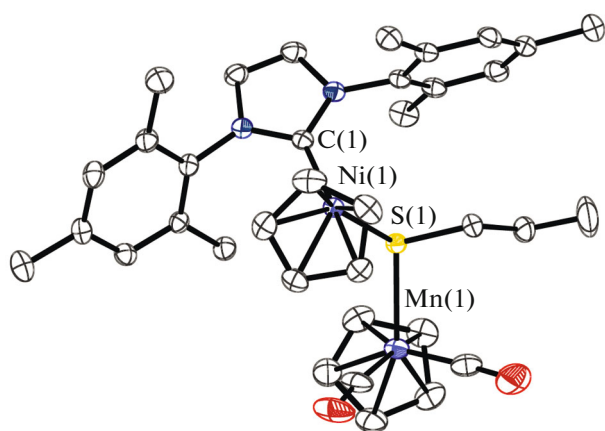


Fig. 3. Molecular structure of complex **III** (thermal ellipsoids of atoms of 30% probability). Hydrogen atoms are omitted for clarity. One of two independent molecules is shown. Selected bond lengths and bond angles: Ni(1)–S(1) 2.2153(7), Ni(1)–C(1) 1.898(3), Mn(1)–S(1) 2.3538(8), and S(1)–C(27) 1.828(3) Å and Ni(1)S(1)Mn(1) 122.11(3)°.

nese sulfides (NiMnS, 21.44%) contaminated with the pyrolysis products of the ligands (13.48%).

Complex **IIa** is characterized by the complete stepwise elimination of the ligands (at 92–182°C, five CO and C₃H₇; at 183–550°C, C₅H₅ and Me₂Im) to form a residue of NiWS (exp. 45.95%, theor. 44.35%).

Thus, 1,3-dimethylimidazol-2-ylidene is a more convenient ligand for the synthesis of precursors of inorganic materials than 1,3-dimesitylimidazol-2-ylidene. On the one hand, the present study describes the first examples for the thermal decomposition of the heterometallic complexes with 1,3-dimesitylimidazol-2-ylidene. On the other hand, this study continues the series of works on the isolobal analogy, an original example of which can be the publication of I.B. Sivaev dedicated to memory of A.A. Pasynskii [14].

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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