Portability of the RNNMo(3+) core — Application to the synthesis of dinitrogen-derived trialkoxymolybdenum organodiazenido complexes¹

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Abstract: An alcoholysis strategy has been employed in the synthesis of new oligo- or polymeric trialkoxymolybdenum diazenido complexes $[R^1NNMo(O-1-Ad)_3]_x$ ($R^1 = Me \text{ or } C(O)OMe$) as well as their mononuclear THF solvates $R^{1}NNMo(O-1-Ad)_{3}(THF)$. Dinitrogen-derived diazenido precursor complexes $R^{1}NNMo(N[R]Ar)_{3}$ (R = i-Pr, Ar = 3,5- $C_6H_3Me_2$) react smoothly with 3 equiv. 1-adamantanol to liberate 3 equiv. HN(R)Ar without disruption of the diazenidomolybdenum(3+) core. X-ray structural investigations were carried out for diazenido complex MeNNMo(O-1-Ad)₃(THF) and for the corresponding cationic dimethyldiazenido complex [Me₂NNMo(O-1-Ad)₃(THF)]⁺, obtained as its triflate salt. The latter cation was found upon cobaltocene reduction to undergo N-N bond cleavage, producing nitride NMo(O-1-Ad)₃ (synthesized independently) and dimethylamine.

Key words: molybdenum, dinitrogen, bond cleavage, hydrazido, diazenido.

Résumé : On a utilisé une stratégie d'alcoolyse dans la synthèse de nouveaux complexes oligo- et polymériques du diazénido trialkoxymolybdène [R^1 NNMo(O-1-Ad)₃]_x (R^1 = Me ou C(O)OMe) et celle de leurs complexes monomoléculaires monosolvatés par le THF R¹NNMo(O-1-Ad)₃(THF). Les complexes précurseurs diazénido dérivés du diazote $R^{1}NNMo(N[R]Ar)_{3}$ (R = *i*-Pr, Ar = 3,5-C₆H₃Me₂) réagissent sans problème avec trois équivalents d'adamant-1-ol pour libérer trois équivalents de HN(R)Ar sans disruption du noyau diazénidomolybdène(3+). On a examiné les structures du complexe diazénido MeNNMo(O-1-Ad)₃(THF) et du complexe diméthyldiazénido cationique correspondant [Me₂NNMo(O-1-Ad)₃(THF)]⁺ isolé sous la forme de triflate. On a trouvé que la réduction de ce dernier cation par le cobaltocène conduit à un clivage de la liaison N-N qui conduit à la formation du nitrure NMo(O-1-Ad)₃ (qui a été synthétisé par une voie indépendante) et de la diméthylamine.

Mots clés : molybdène, diazote, clivage d'une liaison, hydrazido, diazénido.

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Introduction

Amide (NR₂¹⁻) ligands (1) and tripodal variants incorporating them have become established supporting ligands for the dinitrogen chemistry of metals from groups 4, 5, and 6 (2–16). In contrast, while early metal alkoxides and (or) aryloxides are abundant and their chemistry is wellestablished (17), the combination of alkoxide supporting ligands and dinitrogen activation-functionalization remains rare. A prime example of dinitrogen binding, activation, and cleavage to nitride by an early metal aryloxy system is provided by the niobium-calix[4]arene system reported by Floriani and co-workers (18). Furthermore, N₂ splitting in combination with intermetal N-atom transfer has been re-

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ported in a system that produces ${}^{15}NMo(O-t-Bu)_3$ from ${}^{15}N_2$ (19).

Since a common mode of reactivity for coordinated dinitrogen is that involving treatment with simple electrophiles (e.g., H⁺, Me⁺ synthons) and conversion to diazenido (NNH¹⁻ or NNMe¹⁻) units (20-24), and since such conversions are essentially absent from the known chemistry of early-metal alkoxides, we sought an alternative synthetic entry to trialkoxymolybdenum(IV) diazenido systems. Dinitrogenderived diazenido, hydrazido, and related ligands have interested theoreticians for at least a quarter century (25).

We have shown recently that certain Mo-ligand multiply bonded functional groups are "portable" in the sense that they can be retained while exchanging all the supporting ligands. Accordingly, the monomeric terminal phosphide (P^{3-}) complex PMo(O-1-MeCy)₃ was synthesized by treatment of $PMo(N[i-Pr]Ar)_3$ with 3 equiv. of 1-methyl cyclohexanol (26). In addition, a variety of Schrock-type alkyne metathesis catalysts R¹CMo(OR²)₃ could be synthesized by an analogous alcoholysis protocol, involving treatment of precursor $R^{1}CMo(N[i-Pr]Ar)_{3}$ with 3 equiv. of the desired alcohol or phenol, R^2OH (27, 28). The latter two examples reveal the "portability" of the MoP and MoC triple bond functional groups, attributable to their compatibility with the required protic reaction conditions in concert with the protolytic sen-

Scheme 1.



sitivity of the molybdenum-amide bonds. In the present work, we show that the same approach lends itself to the synthesis of a diazenido trialkoxymolybdenum(IV) system.

Results and discussion

Prepared by treatment of the corresponding anionic dinitrogen complex (29) (sodium salt) with, respectively, methyl tosylate (MeOTs) and methyl chloroformate are the diazenido complexes $R^1NNMo(N[i-Pr]Ar)_3$ **1a** ($R^1 = CH_3$, synthesis and structure reported previously) (29) and **1b** ($R^1 = C(O)OMe$). Gram quantities of **1a** could be obtained as red blocks in 58% isolated yield, as crystallized from ether (23). In contrast, **1b** was recovered in 94% yield as an analytically pure dark oil that dissolves in benzene to give a yellow solution.

The choice of MeOTs as the CH_3^+ source in the synthesis of **1a** was not random (29). A mild and non-oxidizing methylating agent is required to avoid both over-methylation and 1e oxidation of the anionic dinitrogen complex to its labile neutral counterpart (7). Furthermore, we had shown previously that sodium amalgam reduction of three-coordinate $Mo(N[t-Bu]Ar)_3$ in the presence of MeOTs under N₂ constituted a one-pot synthesis of MeNNMo(N[t-Bu]Ar)₃ (7). Structural precedent for molecules containing the [N=N C(O)OMe](1-) ligand, as found in **1b**, is extant (30).

Adamantanolysis of **1a** (Scheme 1) was accomplished by treating with 3 equiv. of 1-AdOH in THF at -25 °C, followed by warming to 25 °C and stirring for 4 h (Scheme 1). Solvent removal in vacuo, followed by suspension in pentane of the resulting orange solid, and subsequent collection by filtration, gave MeNNMo(OAd)₃(THF) (**2a**·THF) in 81% isolated yield. The filtrate was shown to contain, essentially exclusively, pure HN(*i*-Pr)Ar. A band at 1544 cm⁻¹ in the IR spectrum of **2a**·THF is assigned to v_{NN}, and this dia-

magnetic derivative exhibits ¹H and ¹³C NMR spectra consistent with a single 1-adamantyl environment.

The specific choice of adamantanol in this work derives from a combined consideration of steric bulk and solubility properties. The steric protection afforded by adamantoxide in complexes such as **2a**·THF is often sufficient to favor a mononuclear formulation, but in addition, such complexes have sufficiently low solubility in saturated hydrocarbon media for efficient separation from the nonvolatile HN(*i*-Pr)Ar aniline liberated in the alcoholysis reaction. In the latter respect, adamantoxy is superior to, for example, *tert*-butoxy.

Adamantanolysis of **1a** was also possible in the absence of THF, under more forcing conditions (toluene, 92 °C, 39 h). In this case, the orange product precipitated from the brown reaction mixture while still hot. The product obtained in this manner (2a, 30% yield) was only sparingly soluble in benzene, toluene, or chloroform, but dissolved quantitatively in THF to provide solvate 2a. THF. The properties of solvent-free 2a are reminiscent of those reported for nitrosyl $ONW(O-t-Bu)_3$ (31), and both compounds are presumably polymeric in the solid state, though the nature of the bridging interactions is not known in either case. For 2a, it may be surmised that the nucleophilic β -N atom of the diazenido ligand serves to provide the bridging contact, leading to infinite polymer formation. If so, then by analogy, the nitrosyl oxygen in ONW(O-t-Bu)₃ may behave similarly. Such structural features (i.e., topologically linear bridging N=NR or NO) are of interest given the paucity of data concerning such interactions (32), and their potential relevance to oxygen-atom or nitrene-group transfer reactions (33).

The molecular structure of **2a**·THF (Fig. 1) was determined via single-crystal X-ray diffraction. The overall molecular conformation is quite reminiscent of that observed for nitrosyl ONW(O-*t*-Bu)₃(py) (31), with local threefold axial symmetry in the trialkoxymetal core, an apical metal– ligand multiple bond, and a solvent adduct trans thereto. The **Fig. 1.** Thermal ellipsoid representation at 50% probability of **2a**·THF. Selected interatomic distances (Å) and angles (°): Mo-N1, 1.773(3); N1-N2, 1.250(4); Mo-O2, 1.902(2); Mo-O1s, 2.300(2); Mo-N1-N2, 171.8(3); N1-N2-C1, 118.7(3).



key structural parameters for the diazenidomolybdenum core in **2a**·THF (see caption to Fig. 1) are essentially unperturbed relative to those reported previously for **1a** (26), indicating that the exchange of alkoxide for anilide ligation, along with the inclusion of a trans solvent molecule, alters little the apical metal–ligand multiple bonding. The bond angles in the diazenido core reveal *sp* and *sp*² hybridization at, respectively, N1 and N2, as is typical of this functional group. The Mo-N1 distance is elongated by only ca. 0.1 Å relative to values typical of terminal MoN triple bonds (6), such that the resonance structure used to represent **2a**·THF in Scheme 1 is not by itself a complete description of the Mo—N bonding.

While **1b** was obtained only as a dark oil, its conversion to a solid derivative was accomplished via room-temperature adamantanolysis. Product **2b** was obtained accordingly as a yellow powder in 62% yield. The low solubility of **2b** in toluene suggests that like **2a**, the complex is polymeric in the solid phase. While in **2a** the β -N atom is likely to provide a bridging contact, in the case of **2b**, the carbonyl oxygen atom in the carboxylmethyl residue may alternatively serve this function. Carboethoxydiazenido molybdenum 3+ systems have been obtained from O₂Mo(dtc)₂ (dtc = *N*,*N*-dimethyldithiocarbamate) by treatment with the corresponding hydrazine, or by ethylchloroformate derivitization of dinitrogen complexes (N₂)₂Mo(dppe)₂ (dppe = 1,2-diphenylphosphinoethane) (34).

Nucleophilicity of the β -N in **2a**·THF was demonstrated by reaction of the complex with methyl triflate. The triflate salt [**3**]OTf was thereby produced as an orange powder in 82% isolated yield. Dimethylhydrazido complexes related to [**3**]OTf include the triaryloxy system Me₂NNV(dipp)₃ (dipp = 2,6-diisopropylphenoxy) studied with reference to vanadium-containing nitrogenase (35). The latter system displayed a short metal–nitrogen interatomic distance: V-N = **Fig. 2.** Thermal ellipsoid representation at 50% probability of [**3a**·THF][OTf] (disordered triflate anion omitted for clarity). Selected interatomic distances (Å) and angles (°): Mo-N1, 1.745(3); N1-N2, 1.296(5); Mo-O2, 1.860(3); Mo-O4, 2.297(3); Mo-N1-N2, 177.2(3); N1-N2-C2, 119.5(4); N1-N2-C2, 120.0(4); C2-N2-C1, 118.2(4).



1.653(3) Å, an elongated N—N bond: N-N = 1.311(4) Å, and near linearity at the α -N atom: V-N-N = 175.8(3)° (35). While the structure of unsolvated [3]OTf is unknown, solvate [3.THF]OTf was obtained straightforwardly and its structure determined crystallographically (Fig. 2). Structural parameters for the dimethylhydrazido ligand in [3-THF]OTf are very similar to those seen for the aforementioned triaryloxy vanadium system. Accordingly, the methylation event leading to [3]OTf may be viewed as a redox event, the metal undergoing a formal oxidation state change from 4+ to 6+ concomitantly. The bonding between metal and α -N is then accurately described as for a metal imido complex (25). On the other hand, donation of the dimethylamino lone pair of electrons into the metal- α -N π^* system leads to its observed planarity and suggests the alternative formulation, as drawn in Scheme 1 for the line drawings of [3]OTf and [3.THF]OTf. A related, structurally characterized molybdenum dimethylhydrazido complex cation was obtained by methyl triflate treatment of a trimethylsilyldiazenido complex (36).³

There has long been interest in the conversion of dinitrogen-derived hydrazido systems to amines, and it has been shown that treatment of cationic hydrazido complexes with LiAlH₄ can effect the conversion (37). Although not a clean transformation, it has been shown that [$\{ArN_3N\}MoNNMe_2$]OTf upon reduction with Na–Hg in THF undergoes N—N bond cleavage producing nitride [ArN_3N]MoN together with dimethylamine and other products (38), implying that neutral dimethylhydrazido complex [ArN_3N]MoNNMe₂ lacks sufficient stability for isolation. Presumably, fragmentation of the latter neutral system leads to nitride along with the [NMe₂] radical, the latter being

³Supplementary data may be purchased from the Directory of Unpublished Data, Document Delivery, CISTI, National Research Council Canada, Ottawa, ON K1A 0S2, Canada (http://cisti-icist.nrc-cnrc.gc.ca/irm/unpub_e.shtml for information on ordering electronically). CCDC 262546 and 262547 contain the crystallographic data for this manuscript. These data can be obtained, free of charge, via ww.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax +44 1223 336033; or deposit@ccdc.cam.ac.uk).

capable of hydrogen atom abstraction from THF solvent to produce dimethylamine. Sodium amalgam reduction of dimethylhydrazido [Me₂NNMo(N[t-Bu]Ar)₃][OTf] likewise gave rise to dimethylamine and the corresponding nitrido complex, $NMo(N[t-Bu]Ar)_3$ (7). Similarly, in the present work we find (Scheme 1) that cobaltocene reduction of [3]OTf produced cobaltocenium triflate, nitride NMo(O-1-Ad)₃, and dimethylamine, as determined spectroscopically. Nitride NMo(O-1-Ad)₃ was prepared independently using the method described by Chisholm and co-workers (39) for the synthesis of NMo(O-t-Bu)₃, for determination of the NMR spectroscopic characteristics of the former. The existence of neutral molybdenum(V) dimethylhydrazido complexes has been probed for electrochemically (38), and such systems remain interesting as putative reactive intermediates in this manifold of amine-producing N-N bond cleavage reactions.

Concluding remarks

Diazenido complexes synthesized from N_2 in the context of a simple supporting platform, namely the Mo(N[R]Ar)₃ (R = *i*-Pr, Ar = 3,5-C₆H₃Me₂) fragment, have been shown to be amenable to replacement of the full complement of ancillary ligands. Accordingly, trialkoxymolybdenum-supported diazenido systems have become available for study, expanding the scope of systems combining the chemistry of N₂ or N₂-derived ligands with an O-donor supporting framework. This approach, harnessing the portability of the diazenidomolybdenum(3+) core, provides an opportunity to study this functional group's behavior in a variety of contexts.

Experimental

General considerations

All operations were performed in a drybox under an atmosphere of purified nitrogen. Anhydrous ether, toluene, benzene, pentane, and hexane were purified according to the procedure of Grubbs and co-workers (40). Deuterated solvents were degassed and dried over molecular sieves (4 Å) and transferred under vacuum into a storage vessel. Molecular sieves (4 Å) and Celite® were activated in vacuo overnight at a temperature above 180 °C. Other chemicals were purified and dried by standard procedures or were used as received. IR spectra were recorded on a Bio-Rad 135 Series FT IR spectrometer. ¹H and ¹³C NMR spectra were recorded on a Varian VXR-500, Varian Mercury-300, Varian XL-300, or Varian Unity-300 spectrometer. ¹H and ¹³C NMR chemical shifts are reported with reference to solvent resonances. Dinitrogen complex $[Na(THF)][N_2Mo(N[i-Pr]Ar)_3]$ and compound 1a were prepared as previously reported (29, 41).

Synthesis of (Ar[i-Pr]N)₃MoN₂C(O)OMe (1b) (41)

To a solution of $[Na(THF)][N_2Mo(N[i-Pr]Ar)_3]$ (151 mg, 0.214 mmol) in ether (8 mL) was added a solution of methyl chloroformate (18 µL, 0.235 mmol) at -25 °C. The mixture was allowed to warm to room temperature and was stirred for a total of 3 h. The resultant mixture was filtered through a pad of Celite[®] on a sintered glass frit. The solvent was removed from the filtrate in vacuo to give **1b** as a dark brown oil (135 mg, 94% yield). FT IR (C₆D₆, KBr, cm⁻¹): v_{NN}

1654, v_{CO} 1212. ¹H NMR (C₆D₆, 25 °C) & 6.46 (3H, para), 6.32 (6H, ortho), 4.81 (3H, methine), 3.62 (3H, N₂C(O)OCH₃), 1.93 (18H, Ar-CH₃). ¹³C NMR (C₆D₆, 25 °C) & 165.1 (N₂C(O)OCH₃), 148.0 (ipso), 138.8 (meta), 127.5 (ortho), 125.2 (para), 64.4 (CH(CH₃)₂), 52.6 (N₂C(O)OCH₃), 25.4 (CH(CH₃)₂), 21.7 (aryl-CH₃). Anal. calcd. for C₃₅H₅₁N₅O₂Mo: C 62.77, H 7.68, N 10.46; found: C 62.55, H 7.79, N 10.41.

Synthesis of (AdO)₃Mo(N₂Me)(THF) (2a·THF) (41)

A solution of 1-adamantanol (667 mg, 4.38 mmol) in THF (4 mL) was added to a solution of 1a (884 mg, 1.41 mmol) in THF (8 mL) at -25 °C. The mixture was then stirred for 4.5 h at room temperature after which the solvent was removed in vacuo. The resultant orange powder was suspended in pentane and stirred for 20 min. This gave a suspension that was chilled to -25 °C, and from which orange powder was collected by filtration and washed with cold pentane. The filtrate was subjected twice to the same concentration-suspension-cooling-washing procedure to maximize the yield. The combined orange precipitate was dried in vacuo to yield 2a (759 mg, 81% yield). FT IR (THF, KBr, cm⁻¹): v_{NN} 1544. ¹H NMR (THF- d_8 , 25 °C) δ : 3.75 (3H, N₂CH₃), 3.60 (4H, THF), 2.13 (9H, CH), 1.92 (18H, CH₂), 1.76 (4H, THF), 1.66 (18H, CH₂). ¹³C NMR (THF-d₈, 25 °C) δ: 78.8 (OC(CH₂-)₃), 68.4 (THF), 47.6 (CH-CH₂), 43.8 (N₂CH₃), 37.5 (C(CH₂-)₃), 32.7 (CH-CH₂), 26.5 (THF). Anal. calcd. for C₃₅H₅₆N₂O₄Mo: C 63.24, H 8.49, N 4.22; found: C 63.16, H 8.4, N 4.16.

Synthesis of (AdO)₃Mo(N₂Me) (2a) (41)

(Ar[*i*-Pr]N)₃MoN₂Me (**1a**, 328 mg, 0.524 mmol), 1adamantanol (239 mg, 1.57 mmol), and toluene (12 mL) were installed in a flask equipped with a Teflon stopcock. The reaction mixture was stirred for 39 h at 92 °C. Then, the mixture was cooled to -25 °C, and the resulting orange precipitate was filtered off and washed with cooled toluene to give **2a** as an orange powder (92 mg, 30% yield). ¹H NMR (CDCl₃, 25 °C) δ : 3.77 (3H, NNCH₃), 2.10 (9H, CH), 1.88 (18H, CH₂), 1.56 (18H, CH₂). Anal. calcd. for C₃₁H₄₈N₂O₃Mo: C 62.82, H 8.16, N 4.73; found: C 62.58, H 8.25, N 4.67.

Synthesis of (AdO)₃MoNNC(O)OMe (2b) (41)

A toluene (4 mL) solution of $(Ar[i-Pr]N)_3MoN_2C(O)OMe$ (**1b**, 190 mg, 0.284 mmol) was added to 1-adamantanol (134 mg, 0.879 mmol). The mixture was stirred for 12 h, producing a yellow solution with a yellow precipitate. The yellow precipitate was filtered off, washed with cold toluene, and dried in vacuo to give **2b** as a yellow powder (113 mg, 62% yield). FT IR (C₆D₆, KBr, cm⁻¹) v: 2915, 2852, 1113, 1086, 1061. ¹H NMR (CDCl₃, 25 °C) δ : 3.70 (3H, N₂C(O)OCH₃), 2.32 (9H, CH), 2.10 (18H, CH₂), 1.65/1.62 (18H, CH₂). ¹³C NMR (CDCl₃, 25 °C) δ : 163.9 (N₂C(O)OCH₃), 86.8 (C(CH₂-)₃), 52.9 (CH-CH₂), 45.4 (N₂C(O)OCH₃), 36.2 (C(CH₂-)₃), 31.9 (CH-CH₂). Anal. calcd. for C₃₂H₄₈N₂O₅Mo: C 60.37, H 7.60, N 4.40; found: C 60.84, H 7.71, N 4.48.

Synthesis of [(AdO)₃Mo(NNMe₂)][OTf] ([3][Otf]) (41)

Methyl triflate (258 μ L, 2.28 mmol) was added to a toluene (8 mL) suspension of (AdO)₃MoN₂Me(THF) (**2a**·THF, 759 mg, 1.14 mmol) at -25 °C. The mixture was stirred for 3 h at room temperature. The resultant purple-red mixture was filtered through Celite[®] and the solvent was removed from the filtrate in vacuo. The crude red solid thereby obtained was suspended in ether–pentane 1:1 and cooled to –25 °C. An orange precipitate of [**3**][OTf] was collected by filtration and dried in vacuo (709 mg, 82%). FT IR (C₆D₆, KBr, cm⁻¹) v: 2915, 2852, 1113, 1086, 1061. ¹H NMR (C₆D₆, 25 °C) &: 3.34 (6H, NN(CH₃)₂), 2.11 (9H, CH), 2.06 (18H, CH₂), 1.52/1.49 (18H, CH₂). ¹³C NMR (C₆D₆, 25 °C) &: 86.6 (C(CH₂-)₃), 45.8 (CH-CH₂), 45.3 (NN(CH₃)₂), 36.6 (C(CH₂-)₃), 32.0 (CH-CH₂). Anal. calcd. for C₃₃H₅₁N₂O₆F₃SMo: C 52.37, H 6.79, N 3.70; found: C 52.48, H 6.74, N 3.78.

Synthesis of [(AdO)₃Mo(NNMe₂)(THF)][OTf] ([3·THF][OTf]) (41)

The complex [(AdO)₃Mo(NNMe₂)(THF)][OTf] ([**3**·THF][OTf]) was obtained simply by dissolving [**3**]OTf in THF. Unlike **2a**·THF, the THF ligand in [**3**·THF][OTf] can be removed by simply dissolving the complex in toluene and removing the solvent. A single crystal suitable for X-ray structure analysis was obtained by recrystallization from toluene–THF. ¹H NMR (C₆D₆, 25 °C) & 3.76 (4H, THF), 3.46 (6H, NN(CH₃)₂), 2.12 (9H, CH), 2.00 (18H, CH₂), 1.57/1.54 (18H, CH₂), 1.48 (4H, THF).

Independent synthesis of (AdO)₃MoN (41)

Sodium azide (801 mg, 12.3 mmol) was added to a THF (50 mL) suspension of MoCl₄(THF)₂ (3.92 g, 10.3 mmol) at room temperature, and the mixture was stirred for 1 h. Then, Li(OAd) (4.74 g, 30.0 mmol) was added, and the mixture was stirred for 4 h at room temperature. The resulting mixture was filtered through Celite[®], and the solvent was removed from the filtrate to give a dark, red-brown solid. The solid was stirred in boiling toluene (125 mL) and filtered through a pad of Celite® on a sintered glass frit, and the solid collected on the frit was washed with toluene. The filtrate was condensed and allowed to stand at room temperature for 36 h. The white precipitate was collected by filtration, washed with pentane, and dried in vacuo. The resulting powder was further suspended in cold toluene (25 mL), collected by filtration, and dried in vacuo to give (AdO)₃MoN as a white powder (0.98 g, 17% yield). FT IR (pyridine- d_5 , KBr, cm⁻¹) v: 2938, 2893, 2851, 1453, 1104, 1077, 940, 745. ¹H NMR (pyridine-d₅, 25 °C) δ: 2.24 (18H, CH₂), 2.17 (9H, CH), 1.63/1.61 (18H, CH₂). ¹³C NMR (pyridine-d₅, 25 °C) δ: 78.1 (C(CH₂-)₃), 46.1 (CH-CH₂), 36.9 (C(CH₂-)₃), 32.0 (CH-CH₂). Anal. calcd. for C₃₀H₄₅NO₃Mo: C 63.93, H 8.05, N 2.49; found: C 64.06, H 8.12, N 2.58.

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