Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$: Synthesis, X-ray structure and evaluation of its catalytic activity for the homogeneous hydrogenation of olefins and their mixtures

Pablo J. Baricelli [a],*, Luis G. Melean [a], Santiago Ricardes [a], Victor Guanipa [a], Mariandry Rodriguez [a], Carlos Romero [b], Alvaro J. Pardey [c], Sergio Moya [d], Merlin Rosales [e]

[a] Centro de Investigaciones Químicas, Facultad de Ingeniería, Universidad de Carabobo, Valencia, Carabobo 2005, Venezuela
[b] Departamento de Química Tecnológica, Facultad de Ingeniería, Universidad de Carabobo, Valencia, Venezuela
[c] Centro de Equilibrios en solución, Escuela de Química, Facultad de Ciencias, Universidad Central de Venezuela, Caracas, Venezuela
[d] Departamento de Química Aplicada, Facultad de Química y Biología, Universidad de Santiago de Chile, Santiago, Chile
[e] Laboratorio de Química Inorgánica, Departamento de Química, Facultad Experimental de Ciencias, Universidad del Zulia, Apdo. 526, Maracaibo, Venezuela

A R T I C L E   I N F O

A B S T R A C T

The complex Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$, was synthesized by the reaction of Mo(NCMe)$_3$(CO)$_3$ with two equivalents of PPh$_3$ and characterized by UV–Vis, IR, NMR and X-ray diffraction. This complex was used as a catalyst precursor for the hydrogenation of 1-hexene, styrene, cyclohexene and 2,3-dimethyl-1-butene and their mixtures under moderate conditions in homogeneous media. Under mild reaction conditions ($T = 373$ K, $P = 60$ atm), the substrates showed the following reactivity order: styrene > 1-hexene > cyclohexene > 2,3-dimethyl-1-butene. A quaternary equimolar mixture showed a different hydrogenation order: 1-hexene > cyclohexene > styrene > 2,3-dimethyl-1-butene; the presence of dibenzothiophene or mercury does not interfere with the activity of the catalyst.

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1. Introduction

Hydrogenation of olefins is one of the most important reactions in organometallic catalysis; indeed, it was the first reaction whose mechanism was well studied and understood and became the frame of reference for activity and selectivity of new catalysts [1]. Although the amount of work published on hydrogenation, both in homogeneous and heterogeneous media, by catalysts of rhodium, ruthenium and iridium is enormous, much less attention has been given to molybdenum complexes; even though Mo has been successfully used as an inexpensive catalyst for the ionic hydrogenation of the carbonyl double bond of ketones by Martins and co-workers [2a], and Bullock and co-workers [2b–e] among others. Most of the work published about molybdenum deals with heterogeneous or clustered systems, and has focused mainly on epoxidation of olefins [3] and hydrogenation of carbon monoxide [4].

Antecedent reports on homogenous hydrogenation of olefins by molybdenum catalysts are scarce. One of the first works was published by Tatsumi et al. [5] in which they studied the homogeneous hydrogen transfer reaction from secondary alcohols to ketones and α-olefins by *trans*-Mo(N$_2$)$_2$(dppe)$_2$ (dppe = Ph$_2$PCH$_2$CH$_2$PPh$_2$) and found it to be an efficient catalyst, although some internal bond migration occurred simultaneously. Fusi et al. [6] examined the homogenous hydrogenation of carbon–carbon multiple bonds by clusters of platinum, one of which, Pt(C$_6$H$_{11}$NC)$_2$[M(CO)$_3$(η^5-C$_5$-H$_5$)]$_2$, contained molybdenum; however, this catalyst showed poor performance. More recently, Fuchikami et al. [7] reported the activity of the binuclear complex Et$_4$N[(μ-H)Mo$_2$(CO)$_{10}$] in the hydrogenation of benzaldehyde and the hydrosilylation of isoprene and several conjugated olefins and benzaldehyde; unfortunately, this Mo-containing catalyst was unreactive towards the hydrogenation of alkenes.

Our group has recently reported the synthesis and catalytic activity of hydrogenation catalysts in homogeneous and biphasic systems based on Ru, Rh and W [8]. The current work presents the synthesis, characterization (including an X-ray study) and catalytic activity of the complex Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$ in the basically unexplored field of the homogenous olefin hydrogenation by a molybdenum species.

2. Materials and methods

2.1. General procedures

All manipulations were carried out under a nitrogen atmosphere using standard Schlenck techniques. Organic solvents were dried and purified by distillation over standard reagents under nitrogen prior to use. All the gases were of U.A.P. grade and

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* Corresponding author. Tel.: +58 241 8671655.
   E-mail address: pbaricel@uc.edu.ve (P.J. Baricelli).

purchased from AGA-Gases, Venezuela. The FTIR spectra were recorded in a Perkin Elmer Spectrophotometer 1000 FTIR, using samples as KBr disks. NMR analyses were performed in a Bruker AM 300 spectrometer. GC analyses were carried out on a Hewlett Packard 5890 Series II chromatograph with a flame ionization detector and an ultra 2.5% phenyl methyl silicone, 25 m, 320 μm column. Quantification was achieved using the internal standard method (naphthalene) and the peaks were identified by comparison with pure samples analyzed by GC/MS HP 5890/5971 coupled system using a Quadex PONA 5% phenyl methyl silicone, 25 m, 320 μm column. Atomic absorption analyses were performed with inductive coupled plasma–optical emission spectrometry (ICP-OES), Perkin Elmer-Optima 3100 XL.

2.2. Synthesis of the complex $Mo(CO)_3(NCMe)(PPh_3)_2$

The $Mo(CO)_3(NCMe)_3$ complex was prepared and characterized according the method reported by Tate et al. [9]. To a sample of $Mo(NCMe)_3(CO)_3$ (0.24 g, 0.80 mmol) dissolved in acetonitrile (10 mL) previously heated at 80 °C was added triphenylphosphine (0.48 g, 1.83 mmol). The resulting solution was refluxed at 60 °C with continuing stirring. After 12 h, a yellow-cream solid was filtered off and washed with acetonitrile and dried under vacuum. Yield 92%. Elemental Anal. Calc. for $Mo(CO)_3(NCMe)(PPh_3)_2$: C, 66.05; H, 4.46; N, 1.88; Mo, 12.87. Found: C, 65.66; H, 4.46; N, 1.62; Mo, 12.86%.

2.3. X-ray crystallography

A single crystal of $Mo(CO)_3(NCMe)(PPh_3)_2$ (0.35 mm × 0.38 mm × 0.40 mm), obtained through crystallization from cold acetonitrile, was selected under polarizing optical microscope and glued on a glass fiber. The X-ray data was collected at room temperature on a Rigaku AFC7S diffractometer with a Mercury CCD area detector, using Mo Kα monochromated radiation ($\lambda = 0.71073$ Å); empirical absorption correction was applied. The structure was solved by the direct method and refined by the full-matrix least-squares method on F^2, with anisotropic displacement parameters for non-hydrogen atoms, using the SHELXTL crystallographic software package. All hydrogen atoms on carbons were placed in their ideal positions and refined as riding atoms with a uniform value of U_{iso}. The final refinement was converged to give $R_1 = 0.0915$ and $wR_2 = 0.1974$ for all 7654 unique reflections.

2.4. Catalytic homogeneous hydrogenation

In a typical experiments, the catalyst precursor $Mo(CO)_3(NCMe)(PPh_3)_2$ (0.015 g, 0.019 mmol), 1-hexene (1.3 mL, 10.5 mmol), heptane (20 mL) and naphthalene (0.10 g) as internal standard were introduced into a glass lined stainless steel autoclave (Parr, 50 mL) fitted with internal mechanical stirring, a temperature control unit and a sample valve. The solution was purged three times with H_2, then charged with the required pressure and heated to the desired temperature. Samples of the reaction mixture were periodically extracted and the total pressure was adjusted via a high pressure reservoir; once the samples were withdrawn from the autoclave, they were cooled and analyzed by GC and GC–MS techniques.

3. Results and discussion

3.1. Synthesis of the complex $Mo(CO)_3(NCMe)(PPh_3)_2$

The complex was obtained as yellow microcrystals by reacting $Mo(CO)_3(NCMe)_3$ with two equivalents of triphenylphosphine in acetonitrile at 60 °C. The UV–Vis spectrum showed one adsorption band at 332 nm assigned to LMCT characteristics of a Mo(0) center coordinated with phosphorus ligand [10]. This band remains after 72 h at room temperature even at different dilutions of the complex in acetone, which is indicative of the good stability of the complex, demonstrating that it can be used as catalytic precursor for reactions in homogeneous phase.

The FTIR (Table 1) showed the characteristic bands of the PPh₃ ligand at 1600–1700, 730 and 670 cm⁻¹ assigned to the aromatic ring symmetric stretching and symmetric and asymmetric hydrogen overtones. Also, there were three prominent bands observed at 1897, 1840 and 1820 cm⁻¹ corresponding to the νCO vibrations, and a medium sharp band at 2020 cm⁻¹ assigned to νCN of acetonitrile coordinated to a metal center.

The ¹H NMR spectrum in deuterated chloroform showed a singlet at 1.96 ppm which is assigned to the acetonitrile protons and a multiplet centered at 7.30 ppm corresponding to the aromatic protons of the PPh₃ ligand in the corresponding 1:10 ratio. The ³¹P NMR (CDCl₃) spectrum showed a singlet at 39.30 ppm assigned to two equivalent P atoms coordinated to the Mo center. With all the information obtained from the elemental analysis, UV–Vis, FT-IR, ¹H NMR, and ³¹P NMR, we propose the formulation $Mo(CO)_3(NCMe)(PPh_3)_2$, which may present two possible structures: one in which the two phosphine are in *trans* position and other in which the two PPh₃ ligands are in a mutually *cis* position and each in *trans* position to a carbonyl ligand.

3.2. X ray structure

The structure of complex $Mo(CO)_3(NCMe)(PPh_3)_2$ was confirmed by single X-ray analysis. The ORTEP drawing is depicted in Fig. 1 and the crystal data are summarized in Table 2. Crystals of this complex were monoclinic and the structure was refined in the space group $P2(1)/c$ with four formula units per unit cell. The crystal structure is constituted by isolated molecules with no unusual intermolecular contacts; the coordination geometry around the metal atom consists of a distorted octahedron with two triphenylphosphine, three carbonyl and one acetonitrile ligand.

The two triphenylphosphine ligands are situated in the octahedral structure in a *cis* arrangement and each phosphorus atom, P(1) and P(2), in *trans* positions with respect to two carbonyl ligands, C(1)O and C(3)O, respectively; the bond distances of Mo–P(1) 2.6273 Å and Mo–P(2) 2.7295 Å are longer than the distances of the Mo–P bond *trans* to a carbonyl ligand reported for other similar molybdenum complexes, such as *cis*-$Mo(CO)_4(PR_3)_2$, $PR_3 = PMe_2Ph$ (2.525 and 2.533 Å), $PMePh_2$ (2.545 and 2.565 Å) and PPh_3 (2.576 and 2.577 Å) [11], $Mo(CO)_5$(5-methyldibenzophosphole) (2.4899 Å) [12], $Mo(CO)_5[PPh_2(o-C_6H_4OCH_2OCH_3)]$ (2.570 Å) [13] and *mer*-$Mo(CO)_3[P(OPh)_3]_3$ (2.3796, 2.4319 and 2.4390 Å) [14]. The main distortion from the ideal octahedral geometry is the bending of the bulky phosphine ligands towards the C(1)–O and C(3)–O ligands, in order to minimized steric repulsion [P(1)–Mo–

Table 1
Characterization of complex $Mo(CO)_3(NCMe)(PPh)_2$.

Spectroscopy analysis	Assignment
FT-IR (cm⁻¹)	1600–1700, 730, 670 ν(aromatic)
	1897, 1840, 1820 ν(CO)
	2020 ν(CN)
UV–Vis (nm)	332 (LMCT)
¹H NMR (ppm)	7.35 (m) (aromatic)
	2.04 (s) (CH₃)
³¹P NMR (ppm)	37.73 (s) (coordinated phosphine)
	38.51 (s) (coordinated phosphine)

(s) = singlet (m) = multiplet.
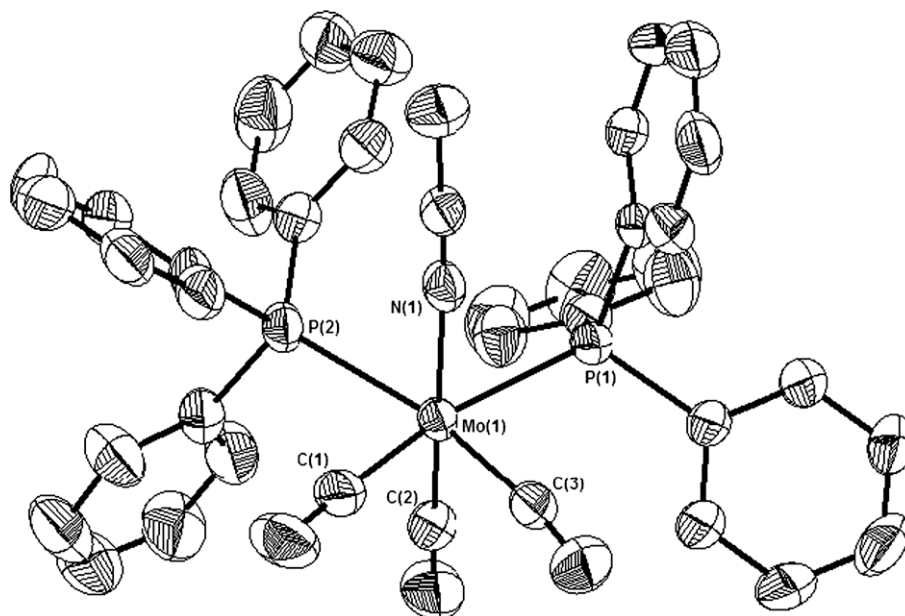
Fig. 1. Perspective drawing of Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$ with thermal ellipsoids at 50% probability displacement. Hydrogen atoms are omitted for clarity. Bond distances: Mo(1)–C(1) = 1.9985(3) Å, Mo(1)–C(2) = 2.0095(6) Å, Mo(1)–C(3) = 2.1110(5) Å, Mo(1)–P(1) = 2.6273(4) Å, Mo(1)–P(2) = 2.7295(5) Å, Mo(1)–N(1) = 2.2606(6) Å.

Table 2
Crystal data and structure refinement of compound Mo(CO)$_3$(NCMe)(PPh)$_2$.

Empirical formula	C$_{41}$H$_{33}$MoO$_3$P$_2$N
Formula weight	745.60
Crystal size (mm)	0.35 × 0.38 × 0.40
Crystalline system	Monoclinic
Space group	P 1 2(1)/c 1 (No. 14)
T (K)	293(2)
λ (Å)	Mo Kα 0.71073
a (Å)	19.191(4)
b (Å)	12.119(2)
c (Å)	18.464(4)
β (°)	90.35(3)
V (Å^{-3})	4.293, 9(2)
Z	4
ρ_{calc} (g cm^{-3})	1.226
μ (mm^{-1})	0.52
F(000)	1910
Reflections collected	7654
Index ranges (°)	$-20 \leqslant h \leqslant 20$
	$-14 \leqslant k \leqslant 14$
	$-17 \leqslant l \leqslant 21$
Data/Restraints/Parameters	7654/0/447
Residual index R_1[a]	0.0576
Weighted residual wR_2[b]	0.1974
Goodness-of-fit (GOF)[c]	1.249
Refinement method	Full-matrix least-squares

[a] Residual index $R = \sum |(|F_o| - |F_c|)| / \sum |F_o|$.
[b] Weighted residual $Rw = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{0.5}$.
[c] Goodness-of-fit (GOF) $= S = [\sum w(F_o^2 - F_c^2)^2 / (n - p)]^{0.5}$.

P(2) angle is 100.798(7)° whereas P(1)–Mo–C(3), P(2)–Mo–C(1) and C(1)–Mo–C(3) angles are 91.147, 89.924 and 78.117°, respectively. Similar structures have been reported by Song et al. [15] for the complex *fac*-Mo(CO)$_3$(dppb)(NCMe) in which X-ray diffraction techniques show an octahedral arrangement around the molybdenum atom and shorter distances between Mo–P and Mo–C due to the coordination of the two P atoms to the carbons of the benzene ring, and by Yih et al. [16] who reported the crystalline structure for the complex Mo(CO)$_3$(η^2-dppm)(η^1-SC$_5$H$_4$NH) with an octahedral arrangement between the molybdenum atom, dppm and the SC$_5$H$_4$NH ligand. The bond distances Mo–C in

Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$ are rather similar to those found in those other Mo–carbonyl complexes.

3.3. Olefin hydrogenation experiments

The activity of the complex Mo(CO)$_3$(NCMe)(PPh$_3$)$_2$ as a catalyst precursor for the homogeneous hydrogenation of olefins was investigated. Preliminary test were conducted using 1-hexene as a model molecule to determine the optimal operational conditions by varying the temperature from 333 to 393 K (60–120 °C), hydrogen pressure was varied from 41.1 to 82.2 atm (600–1200 psi), the S/C ratio was varied from 294/1 to 1000/1, keeping the stirring rate at 600 rpm. From this series of experiments, it was determined that the best operational conditions were T = 343 K, P = 60 atm, S/C ratio 531/1. Employing these operational parameters, the homogeneous hydrogenation of 1-hexene, cyclohexene, styrene and 2,3-dimethyl-1-butene were conducted. The results are summarized in Table 3.

The data in Table 3 show that styrene was hydrogenated to ethylbenzene with a 70% conversion in 8 h. The inductive effect of the aromatic ring and the impossibility to form structural isomers seem be operating in this substrate. Also, 1-hexene was converted to *n*-hexane in a 40% yield, cyclohexene was transformed to cyclohexane with a 29% conversion and finally the highly substituted olefin 2,3-dimethyl-1-butene was slowly converted to 2,3-dimethyl-1-butane with only 9% yield in the same period of time (8 h). The catalytic active specie generated from the catalytic precursor Mo(CO)$_3$(PPh$_3$)$_2$(CH$_3$CN) has a varying degree of activity on the olefin hydrogenation reaction depending on their steric hin-

Table 3
Homogeneous hydrogenation of olefins catalyzed by Mo(CO)$_3$(NCMe)(PPh)$_2$.

Olefin	Product	Conversion (%)	TOF (h^{-1})
1-Hexene	Hexane	40	27.6
Cyclohexene	Cyclohexane	29	20.0
Styrene	Ethylbenzene	70	48.4
2,3-Dimethyl-1-butene	2,3-Dimethylbutane	9	6.2

Conditions: P H$_2$ = 60 atm, T = 373 K, stirring = 600 rpm, S/C = 531/1, t = 8 h.

drance. The author theorized that the catalytic active species is either the hydride or the molecular complex $Mo(H_2)(CO)_3(PPh_3)_2$ analogous to those reported by Kubas et al. [17]. NMR studies aimed at the isolation and characterization of the active species are currently underway and will be reported elsewhere.

In order to confirm that the hydrogenation reaction proceeds via a molecular complex, the well known mercury test [18] was carried out in several independent experiments in the hydrogenation of 1-hexene as well as in the olefin mixtures. The results showed no interference at all of the experiments with the evolution of the reaction, confirming the absence of metallic particles or colloids during the catalysis.

3.4. Hydrogenation of olefin mixtures

Under the same reaction conditions used for the hydrogenation of the individual substrates mentioned above, we tested the performance of the precursor $Mo(CO)_3(NCMe)(PPh_3)_2$ in the hydrogenation reaction of a quaternary olefin mixture equimolar in 1-hexene (linear olefin), cyclohexene (cyclic olefin), styrene (aromatic olefin) and 2,3-dimethyl-1-butene (sterically hindered olefin) in order to simulate a C6 naphtha cut from a real refinery; in these experiments the S/C ratio refers to the total olefin content in the mixture. As shown in Fig. 2, for a period of 12 h, 1-hexene is hydrogenated faster than cyclohexene, styrene and 2,3-dimethyl-1-butene due to steric impediments and the competition for the active site of the catalyst. However, after the first 12 h, the hydrogenation of styrene increases to reach about 70% conversion to ethylbenzene after 24 h. From these observations we can infer that the strong inductive effect of the phenyl group in the styrene permits its hydrogenation when the steric effects are not predominant. Also, the reaction profiles of the hydrogenation of all olefins show an S-shaped curve probably due to the induction period required to form the catalytically active species in equilibrium from the original precursor. The total conversion was 70% for styrene, 42% for 1-hexene, 29% for cyclohexene and 9% for 2,3-dimethyl-1-butene. The same behavior was observed in the binary mixtures of 1-hexene with each olefin of the equimolar mixture described above.

3.5. Homogeneous hydrogenation of binary olefin mixtures in the presence of dibenzothiophene

In order to investigate if our catalytic molybdenum precursor $Mo(CO)_3(NCMe)(PPh_3)_2$ is resistant to the sulfur compounds pres-
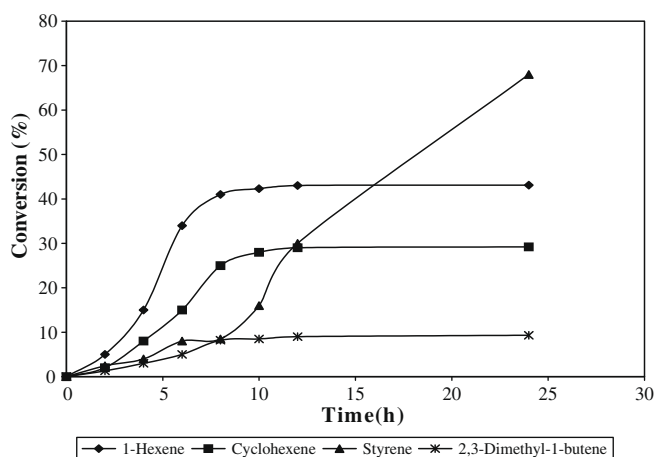

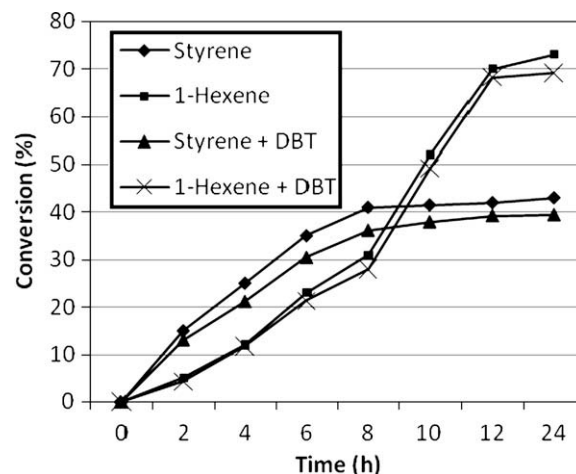**Fig. 3.** Homogeneous hydrogenation of a mixture of 1-hexene and styrene catalyzed by $Mo(CO)_3(NCMe)(PPh_3)_2$ in the presence and absence of dibenzothiophene. Conditions: P H$_2$ = 60 atm, T = 373 K, stirring = 600 rpm, S/C = 531/1, DBT = 0.02 mol.

ent in naphtha cuts, we carried out some preliminary studies in the homogeneous hydrogenation reaction of the binary mixture of 1-hexene and styrene with dibenzothiophene (30 ppm, 0.02 mol). The results are summarized in Fig. 3, the reaction profiles remain constant for both olefins in the presence of sulfur. Thus, it was demonstrated that under our best operational condition, our precursor is sulfur tolerant.

4. Conclusions

The complex $Mo(CO)_3(NCMe)(PPh_3)_2$, which was synthesized by reaction of $Mo(NCMe)_3(CO)_3$ with two equivalents of PPh$_3$ in acetonitrile at 60 °C and characterized by UV–Vis, IR, NMR and X-ray diffraction, proved to be an efficient catalyst precursor for the hydrogenation of 1-hexene, cyclohexene, styrene and 2,3-dimethyl-1-butene and styrene with the following hydrogenation order: styrene > 1-hexene > cyclohexene > 2,3-dimethyl-1-butene. The presence of 30 ppm of dibenzothiophene in the olefin binary mixtures does not affect the activity of the catalytic precursor, which is indicative of the sulfur tolerance of this catalyst. Also, the mercury test proved that the biphasic hydrogenation reaction proceed through a molecular complex.

Appendix A. Supplementary material

CCDC 736040 contains the supplementary crystallographic data for compound $Mo(CO)_3(NCMe)(PPh_3)_2$. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.06.042.

Fig. 2. Homogeneous hydrogenation of a quaternary olefin mixture using the complex $Mo(CO)_3(NCMe)(PPh_3)_2$. Conditions: P H$_2$ = 60 atm, T = 373 K, stirring = 600 rpm, S/C = 531/1).

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