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SYNTHESIS AND MOLECULAR STRUCTURE OF LANTHANUM TRIS[CYCLOPENTADIENYLMOLYBDENOTRICARBONYLATE] TETRAHYDROFURANATE, $(THF)_5La[CpMo(CO)_3]_3 \cdot THF$

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Summary

The interaction of activated La powder with $[CpMo(CO)_3]_2$ or $Hg[CpMo(CO)_3]_2$ in THF resulted in the formation of crystalline $(THF)_5La[CpMo(CO)_3]_3 \cdot THF$ (I), which oxidized readily on exposure to air with cleavage of $[CpMo(CO)_3]_2$. Complex I was characterized by X-ray structural analysis. The La atom is bound to three equivalent $CpMo(CO)_3$ fragments via almost linear carbonyl bridges, Mo–C–O–La (MoCO 177.9(7)°). The coordination of La with the terminal CO group ($La–O_{av}$ 2.444(5) Å) leads to elongation of the C–O bond from an average value of 1.16(1) to 1.21(1) Å, and to shortening of the Mo–C bond from an average value of 1.943(9) to 1.874(9) Å, compared with the unbridged CO. The coordination of La is complemented to $CN = 8$ at the expense of binding of five THF molecules ($La–O(THF)_{av}$ = 2.580(6) Å; the sixth THF molecule is crystallizable).

Introduction

Of late, heterometal transition element complexes, being essentially highly-effective catalysts, have attracted much attention [1]. Of especial interest is the combination, in one molecule, of the carbonylate anions of the transition metals of Groups VI–VIII and the highly valent metal ions of Groups III–V. It has been noted [2] that the character of the bonding of Cp_2Ti^+ and $CpMo(CO)_3^-$ depends, for instance, on the solvating ability of the solvent: the $Cp_2Ti–Mo(CO)_3Cp$ complex described

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earlier [3] appears in benzene, whereas the paramagnetic $Cp_2(THF)Ti{-}OCMo(CO)_2Cp$ complex structurally characterized by X-ray crystallography [2], with coordination of Ti^{III} with respect to the oxygen atom of the carbonyl group at the Mo atom, is formed in THF. Such a type of carbonyl bridge is exceptionally interesting from the point of view of facilitating the rupture of the C–O bond in Fischer–Tropsch reactions [4], and is often realized when binding metal carbonyls to the elements of Group III [5]. The *f*-elements also enter the latter; for example, the X-ray structurally-investigated complexes $(Me_5C_5)_2Yb{-}OC{-}Co(CO)_3$ [6] and $[(Me_5C_5)_2Yb^+]_2[Fe_3(CO)_{11}]^{2-}$ [7]. Earlier we reported that the lanthanide atoms in such compounds are coordinated to the cyclopentadienyl ligands (for instance, in $Cp_2Ln(THF)_n{-}OC{-}Co(CO)_3$, where Ln = Sm, Ho, Yb; $n = 1$–4 [8]) or to Cl atoms in $(THF)_nCl_2Ln{-}Co(CO)_4$, where Ln = Sm, Dy, Ho, Yb, La; $n = 1$–4 [9].

The formation of fully-substituted lanthanide derivatives of transition metal carbonylates is also possible, e.g. $(THF)_nLn[Co(CO)_4]_3$ (Ln = Yb, Er) [10]. Such complexes are most promising from the viewpoint of their catalytic properties and preparation of polymetallic powders and coatings. Therefore, the method discovered by us is very important for synthesizing them by means of the interaction of binuclear metal carbonyls with activated metal lanthanides:

$$Co_2(CO)_8 + Ln/Hg \xrightarrow{THF} (THF)_nLn[Co(CO)_4]_3$$

where Ln = Tm [11,12], Sm, Yb [12]; $n = 3$–4.

$$M_2(CO)_{10} + Ln/Hg \xrightarrow{THF} (THF)_nLn[M(CO)_5]_3$$

where Ln = Sm, Tm, Yb; M = Mn, Re, $n = 1$–2 [12].

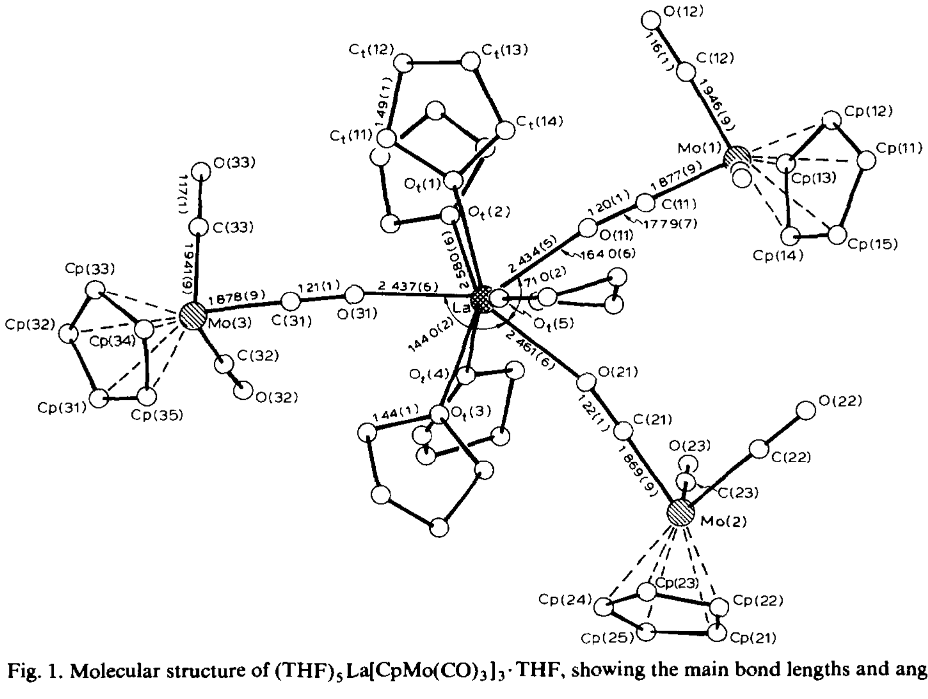

Fig. 1. Molecular structure of $(THF)_5La[CpMo(CO)_3]_3 \cdot THF$, showing the main bond lengths and angles.

In this paper we report the possibility of introducing, into the reaction, the $[CpMo(CO)_3]_2$ complex (with a strong Mo–Mo bond) and the relatively insufficiently active lanthane present at the boundary between the *d*- and *f*-elements (see ref. 13 for a preliminary report).

Results

Synthesis

Powder-like metal lanthane, activated by treatment with a small amount of $HgCl_2$ in THF, interacts completely with a solution of $[CpMo(CO)_3]_2$ in THF in the course of 4 h at room temperature to form complex I:

$$[CpMo(CO)_3]_2 + 2La/Hg \xrightarrow{\text{THF},20°\text{C}, 4\text{ h}} (THF)_5La[OCMo(CO)_2Cp]_3 \cdot THF$$

The same product was prepared by substituting $Hg[CpMo(CO)_3]_2$ for $[CpMo(CO)_3]_2$.

Complex I was isolated from THF/hexane by crystallization to form yellow crystals which dissolved moderately in THF, but did not dissolve in benzene or pentane. Complex I oxidized quite readily when exposed to air with cleavage of $[CpMo(CO)_3]_2$, which hindered the obtention of accurate analytical data. The IR spectrum of complex I (in KBr) revealed absorptions at 860, 1010, 1430 and 3080 cm^{-1} and stretching modes of the bridged (1660vs, 1740s, 1800s) and the terminal groups of CO (1900vs, 2000s). However, it is probably impossible to exclude hydrolysis which leads, for instance, to the formation of $CpMo(CO)_3H$; therefore, in order to obtain an unambiguous identification of complex I, it was necessary to carry out a comprehensive X-ray structural investigation.

Structure

Crystals of complex I may be referred to as possessing triclinic symmetry (spatial group $P\bar{1}$), having the following unit cell parameters: $a = 11.316(3)$; $b = 11.388(3)$; $c = 20.757(7)$ Å; $\alpha = 98.42(2)$, $\beta = 101.65(2)$, $\gamma = 92.53(2)°$, $V = 2584.1$ Å^3, $Z = 2$. In molecule I (Fig. 1) the La atom has a *CN* of 8, and is coordinated by five THF molecules (La–O 2.491(6)–2.580(6) Å) and three $CpMo(CO)_3$ groups, the latter being bound at the expense of the bridged fragments Mo–C–O → La (La–O 2.434(5)–2.461(1) Å) whose geometry is close to linear (MoCO 177.5(17), 178.3(7)°, COLa 164.0(6), 166.1(6)°). The C–O bonds in the bridged fragments are elongated to 1.20(1), 1.22(1) Å, whereas the Mo–CO bond is substantially reduced to 1.819(9), 1.876(9) Å compared with the corresponding values for two CO groups with end coordination at each Mo atom (Mo–C 1.941(9), 2.005(9) Å; C–O 1.13(1), 1.17(1) Å) (Tables 2 and 3). The coordination of the Mo atoms in the molecule is accomplished by binding to the planar cyclopentadienyl ligands (Mo–C_{aver} 2.40(3) Å). Finally, the unit cell contains one independent tetrahydrofuran solvate molecule.

Discussion

The interaction of $[CpMo(CO)_3]_2$ with metal lanthane activated by amalgamation is similar to the reduction of Mo^I to Mo^0 in a typical $[CpMo(CO)_3]_2$ reaction with sodium amalgam [14]. In the synthesis of I via a mercury derivative of

TABLE 1

ATOMIC COORDINATES (×10^4; for La and Mo, ×10^5) OF $(H_8C_4O)_5La[CpMo(CO)_3]_3 \cdot OC_4H_8$

Atom	*x*	*y*	*z*
La	31905(5)	2850(4)	−25036(3)
Mo(1)	18003(8)	37934(6)	−39713(4)
Mo(2)	4717(8)	30492(6)	−10671(4)
Mo(3)	62863(8)	−30163(6)	−16760(4)
O(11)	2881(6)	1909(5)	−3148(3)
O(12)	3117(7)	2494(6)	−5014(3)
O(13)	−435(7)	2023(5)	−4629(4)
O(21)	1847(6)	1652(5)	−2032(3)
O(22)	−563(7)	4320(7)	2261(4)
O(23)	2670(6)	4932(5)	−735(4)
O(31)	4448(6)	−1359(5)	−2294(3)
O(32)	7138(7)	−855(6)	−527(6)
O(33)	8080(7)	−1913(6)	−2417(4)
$O_t(1)$	3434(5)	−654(5)	−3659(3)
$O_t(2)$	5306(6)	1063(4)	−2614(3)
$O_t(3)$	2208(6)	−991(4)	−2614(3)
$O_t(4)$	4344(6)	1261(4)	−1376(3)
$O_t(5)$	1118(6)	−354(4)	−3174(3)
O_{tx}	6436(9)	−6163(9)	−4261(8)
C(11)	2436(8)	2637(7)	−3469(4)
C(12)	2607(9)	3004(4)	−4640(5)
C(13)	393(8)	2675(7)	−4389(5)
C(21)	1285(9)	2206(7)	−1664(4)
C(22)	−178(9)	3843(8)	−1814(5)
C(23)	1864(8)	4230(7)	−854(4)
C(31)	5164(8)	−2019(7)	−2062(4)
C(32)	6862(10)	−1649(9)	−935(5)
C(33)	7410(8)	−2338(8)	−2141(5)
$C_t(11)$	4415(9)	−1405(7)	−3791(5)
$C_t(13)$	3358(10)	−893(10)	−4809(5)
$C_t(14)$	2786(11)	−363(11)	−4281(5)
$C_t(21)$	6421(9)	784(8)	−2220(6)
$C_t(22)$	7424(12)	1152(16)	−2529(7)
$C_t(23)$	6889(12)	1637(13)	3140(5)
$C_t(31)$	1140(11)	−668(9)	−1557(7)
$C_t(32)$	887(14)	−1610(11)	−1149(7)
$C_t(33)$	1375(14)	−2562(10)	−1420(7)
$C_t(34)$	2489(11)	−2135(8)	−1627(5)
$C_t(41)$	4648(9)	2550(7)	−1229(5)
$C_t(42)$	4943(11)	2829(8)	−477(5)
$C_t(43)$	5324(11)	1696(8)	−256(5)
$C_t(44)$	4504(10)	766(8)	−771(5)
$C_t(51)$	103(9)	357(7)	−3375(5)
$C_t(52)$	−754(11)	−404(9)	−3946(6)
$C_t(53)$	−563(9)	−1653(8)	−3901(5)
$C_t(54)$	675(11)	−1601(7)	−3406(6)
$C_{tx}(1)$	7654(8)	−5717(11)	−3954(7)
$C_{tx}(2)$	7521(10)	−4479(9)	−3744(7)
$C_{tx}(3)$	6154(9)	−4289(8)	−3851(5)
$C_{tx}(4)$	5589(11)	−5506(10)	−4141(7)
Cp(11)	820(10)	5602(7)	−4115(5)
Cp(12)	2046(10)	5802(7)	−4130(5)

TABLE 1 (continued)

Atom	*x*	*y*	*z*
Cp(13)	2816(9)	5700(7)	−3484(5)
Cp(14)	1990(11)	5401(7)	−3074(5)
Cp(15)	822(10)	5354(7)	−3444(5)
Cp(21)	−626(10)	3409(7)	−201(5)
Cp(22)	−1420(8)	2647(7)	−743(5)
Cp(23)	−893(9)	1555(7)	−801(5)
Cp(24)	271(9)	1685(7)	−319(5)
Cp(25)	432(10)	2842(8)	60(5)
Cp(31)	6401(11)	−4507(7)	−975(5)
Cp(32)	7159(10)	−4811(7)	−1403(5)
Cp(33)	6454(12)	−5066(7)	−2045(5)
Cp(34)	5199(11)	−4925(8)	−2023(6)
Cp(35)	5205(10)	−4576(8)	−1338(7)

$[CpMo(CO)_3]_2Hg$, Hg^{II} reduces to Hg^0 with precipitation of metallic mercury. Such a method of substitution of an Hg^{II} ion by a Ln^{II} ion, or a Ln^{III} ion, was successfully used by us earlier in the synthesis of $LnCl_3(THF)_n$ from the following lanthanides in THF: Ln = Sm, Eu, Yb, $n = 3$; Ln = La, Eu, Sm, Yb, Tm [15,16]. Using THF dramatically accelerates the reduction of the metal lanthanides at the expense of solvation of the occurring Ln^{II} and Ln^{III} cations.

TABLE 2

BOND LENGTHS IN $(H_8C_4O)_5La[CpMo(CO)_3]_3\cdot OC_4H_8$

Bond	*d* (Å)	Bond	*d* (Å)
La–O(11)	2.434(5)	Mo(3)–C(31)	1.878(9)
La–O(21)	2.461(6)	Mo(3)–C(32)	2.005(9)
La–O(31)	2.437(6)	Mo(3)–C(33)	1.941(9)
La–O_t(1)	2.551(6)	Mo(3)–Cp(31)	2.383(9)
La–O_t(2)	2.580(6)	Mo(3)–Cp(32)	2.400(9)
La–O_t(3)	2.556(6)	Mo(3)–Cp(33)	2.375(9)
La–O_t(4)	2.508(5)	Mo(3)–Cp(34)	2.388(10)
La–O_t(5)	2.491(6)	Mo(3)–Cp(35)	2.380(10)
Mo(1)–C(11)	1.877(9)	O(11)–C(11)	1.200(10)
Mo(1)–C(12)	1.946(9)	O(12)–C(12)	1.162(12)
Mo(1)–C(13)	1.963(9)	O(13)–C(13)	1.148(12)
Mo(1)–Cp(11)	2.410(9)	O(21)–C(21)	1.220(10)
Mo(1)–Cp(12)	2.370(8)	O(22)–C(22)	1.163(13)
Mo(1)–Cp(13)	2.392(9)	O(23)–C(23)	1.149(12)
Mo(1)–Cp(14)	2.382(9)	O(31)–C(31)	1.210(10)
Mo(1)–Cp(15)	2.383(9)	O(32)–C(32)	1.131(11)
Mo(2)–C(21)	1.869(9)	O(33)–C(33)	1.168(12)
Mo(2)–C(22)	1.941(9)	O_t–C_t(THF)$_{av}$	1.455(11)
Mo(2)–C(23)	1.960(9)	C_t–C_t(THF)$_{av}$	1.473(21)
Mo(2)–Cp(21)	2.382(10)	Cp–Cp(Mo(1))$_{av}$	1.432(16)
Mo(2)–Cp(22)	2.414(10)	Cp–Cp(Mo(2))$_{av}$	1.428(13)
Mo(2)–Cp(23)	2.406(9)	Cp–Cp(Mo(3))$_{av}$	1.403(16)
Mo(2)–Cp(24)	2.387(9)	O_{tx}–C_{tx}(THF)$_{av}$	1.358(16)
Mo(2)–Cp(25)	2.394(9)	C_{tx}–C_{tx}(THF)$_{av}$	1.491(16)

The coordination of La^{III} to the oxygen atom of the terminal carbonyl group observed in structure I is very typical (see Introduction), and in accordance with the concept of Basolo and Pearson [17], it reflects the advantageous binding of Ln^{III}, as a hard Lewis acid, with a very "hard" basicity centre in the CpMo(CO)$_3^-$ anion. In this case, of interest is the extraordinary similarity of the structure of the above-indicated anion and the character of its binding with La(THF)$_5$ in I and with the CpTi^{III}(THF) fragment in the above-mentioned paramagnetic complex CpTi(THF)-OCMo(CO)$_2$Cp (II) [2] (bond lengths: Mo–CO$_{(bridge)}$ and C–O$_{(bridge)}$ are 1.874(7) and 1.201(0) Å, respectively; angle of MoCO$_{(bridge)}$ = 177.5(7)°, ν(CO) (cm^{-1}, THF) 1920s, 1890s, 1650s).

Essentially, the formation paths of I and II are also very similar to each other. Merola et al. [2] have described the formation of II from Cp$_2$Ti(CO)$_2$ and [CpMo(CO)$_2$]$_2$ as the initial transfer of two CO groups from Ti to two Mo atoms, followed by the reduction of [CpMo(CO)$_3$]$_2$ under the action of Cp$_2$Ti. The withdrawal of electron density from the oxygen atom of the CO group in CpMo(CO)$_3^-$ during coordination with a "hard" metal-containing Lewis acid of La^{III} or Ti^{III} results in a gradual decrease in the contribution of the additional

TABLE 3

BOND ANGLES (deg) IN $(H_8C_4O)_5La[CpMo(CO)_3]\cdot OC_4H_8$

Angle		Angle	
O(11)LaO(21)	71.0(2)	C(11)Mo(1)C(13)	89.0(4)
O(11)LaO(31)	143.3(2)	C(12)Mo(1)C(13)	86.7(4)
O(11)LaO$_t$(1)	76.6(2)	C(21)Mo(2)C(22)	84.3(4)
O(11)LaO$_t$(2)	73.5(2)	C(21)Mo(2)C(23)	86.2(4)
O(11)LaO$_t$(3)	143.9(2)	C(22)Mo(2)C(23)	87.6(5)
O(11)LaO$_t$(4)	104.4(2)	C(31)Mo(1)C(32)	87.1(4)
O(11)LaO$_t$(5)	80.3(2)	C(31)Mo(1)C(33)	86.5(4)
O(21)LaO(31)	144.0(2)	C(32)Mo(1)C(33)	86.8(4)
O(21)LaO$_t$(1)	137.3(2)	LaO(11)C(11)	164.0(6)
O(21)LaO$_t$(2)	120.3(2)	LaO(21)C(21)	164.4(6)
O(21)LaO$_t$(3)	77.5(2)	LaO(31)C(31)	166.1(6)
O(21)LaO$_t$(4)	73.1(2)	C$_t$(11)O$_t$(1)C$_t$(14)	108.0(1)
O(21)LaO$_t$(5)	74.5(2)	C$_t$(21)O$_t$(1)C$_t$(24)	110.0(1)
O(31)LaO$_t$(1)	75.9(2)	C$_t$(31)O$_t$(3)C$_t$(34)	107.0(1)
O(31)LaO$_t$(2)	76.0(2)	C$_t$(41)O$_t$(4)C$_t$(44)	110.0(1)
O(31)LaO$_t$(3)	71.9(2)	C$_t$(51)O$_t$(5)C$_t$(54)	107.0(1)
O(31)LaO$_t$(4)	84.2(2)	Mo(1)C(11)O(11)	177.9(7)
O(31)LaO$_t$(5)	113.9(2)	Mo(1)C(12)O(12)	176.2(7)
O$_t$(1)LaO$_t$(2)	74.1(2)	Mo(1)C(13)O(13)	179.5(8)
O$_t$(1)LaO$_t$(3)	118.8(2)	Mo(2)C(21)O(21)	177.5(7)
O$_t$(1)LaO$_t$(4)	143.3(2)	Mo(2)C(22)O(22)	179.8(7)
O$_t$(1)LaO$_t$(5)	73.2(2)	Mo(2)C(23)O(23)	179.1(8)
O$_t$(2)LaO$_t$(3)	140.1(2)	Mo(3)C(31)O(31)	178.3(9)
O$_t$(2)LaO$_t$(4)	71.4(2)	Mo(3)C(32)O(31)	178.3(8)
Ot(2)LaO$_t$(5)	141.8(2)	Mo(3)C(33)O(33)	180.0(8)
O$_t$(3)LaO$_t$(4)	82.4(2)	C$_t$C$_t$C$_t$ (av)	106.2(9)
O$_t$(3)LaO$_t$(5)	74.5(2)	C$_{tx}$C$_{tx}$C$_{tx}$(av)	105.4(9)
O$_t$(4)LaO$_t$(5)	143.5(2)	CpCpCp (av)	108.0(9)
C(11)Mo(1)C(12)	84.4(4)		

interaction of $\overrightarrow{O{=}C}$ and, accordingly, in an enhancement of the contribution of the dative interaction of $\overrightarrow{Mo{-}C}$. This manifests itself in the bond lengths of $Mo{-}C_{(bridge)}$ and $C{-}O_{(bridge)}$ in comparison with the Mo–CO(end) fragments (see above), and results in a sharp decrease in the ν(CO) frequency in the Mo–CO → La fragment (up to 1600 cm^{-1} in I and 1650 cm^{-1} in II compared with the low-frequency bond (1760 cm^{-1}) for the isolated $CpMo(CO)_3^-$ anions in the X-ray structurally-characterized ion complex with the $[Cp_3Mo_3(CO)_6]^+$ cation) [18]. It is possible that in complex II a weak interaction of the unpaired electron at Ti^{III} takes place with the carbon atom of the bridged CO which leads to a decrease in the Ti–O–C angle to 135.35(6)°, as compared with the average value 164.8(6)°, for the La–O–C angle in I.

Clearly, the La^{III} atom that does not have valent electrons is capable of being bonded only according to the type La–O–C–Mo observed in complex I. It is also obvious that such a possibility will favour the disruption of the C–O bond, i.e. it will facilitate, for instance, the hydrogenation of CO according to the Fischer–Tropsch process.

Experimental

All the operations were carried out in an atmosphere of pure argon using solvents which had been dehydrated by distillation over dispersed sodium (for hydrocarbons) or by benzophenone–sodium (for THF).

The complex $[CpMo(CO)_3]_2$ was prepared according to the procedure described in ref. 19. Powder-like metal was activated by treatment with a small amount of $HgCl_2$ in THF [16].

The IR spectrum was taken with a Specord IR-75, in a KBr pellet pressed into an argon current. Because of the extreme sensitivity of complex I to oxygen and moist air, single crystals of complex I were sealed in capillaries under argon. X-Ray structural analysis data were obtained on an autodiffractometer Syntex $P2_1$ at -120°C (λ Mo-K_α, θ–2θ scanning $2° \geqslant \theta \geqslant 58°$). 7254 reflexes were obtained, of which 6904 participated in the full-matrix anisotropic refinement for all the non-hydrogen atoms. The structure was solved by the direct method according to the MULTAN program, and was refined to $R = 0.048$, $R_w = 0.045$.

Preparation of $La(THF)_5[(u\text{-}OC)Mo(CO)_2Cp]_3THF$ (I)

La/Hg (1.4 g; 10.0 m*M*) was added to a dark-red solution of 2.4 g of 5.0 m*M* $[CpMo(CO)_3]_2$ in 6 ml of THF, and stirred for 4 h at room temperature; the colour of the solution changed to dark-orange. The solution was allowed to stand overnight and was then filtered off. Half of the THF volume was removed from the filtrate, to which 5 ml of heptane was added; the mixture was then left for 24 h at -5°C. The precipitated crystals were removed from the mother solution by decantation. The THF and pentane were washed and dried in an argon current. Yield 1.60 g (55%). Decomposition temperature 120°C (in a sealed capillary under argon).

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