A zwitterionic nickel–olefin initiator for the preparation of high molecular weight polyethylene

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Dedicated to Professor Gerhard Erker, for his lasting contributions to Organometallic Chemistry and for his persistent and significant efforts in promoting the chemical sciences.

Abstract

The reaction of the sodium 3-(2,6-diisopropylphenylimino)-but-1-en-2-olato with $Ni(PMe_3)_2(\eta^1\text{-}CH_2C_6H_5)Cl$ provides 3-(2,6-diisopropylphenylimino)-but-1-en-2-olato(η^1-benzyl)(trimethylphosphine) nickel (**1**), which was structurally characterized. The addition of 2 equiv. of $B(C_6F_5)_3$ to **1** results in the formation of 2-tris(pentafluorophenyl)borate-3-(2,6-diisopropylphenylimino)-but-1-ene(η^3-benzyl)nickel (**2**), in which the borane coordinates to the O site of the ligand and forces binding of the olefin unit to the nickel center. The solid-state structure of **2** shows a zwitterionic structure with substantial positive charge at the nickel center. Compound **2** can be used to initiate the homopolymerization of ethylene to yield high molecular weight polyethylene.
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1. Introduction

Nickel-based olefin oligomerization and polymerization initiators are of interest in industrial and academic laboratories [1]. Cationic versions are the most commonly encountered [2]. Neutral species, although less active, are under investigation because of their higher tolerance toward functionalities [3]. Zwitterionic counterparts are the least common and provide an intermediate range of reactivities [4].

During our efforts at developing tandem catalytic processes [5], we discovered that the reactivity of SHOP-type catalysts such as $[(C_6H_5)_2PC_6H_4C(O)O\text{-}\kappa^2P,O]Ni(\eta^3\text{-}CH_2\text{-}CMeCH_2)$ [6] increases upon addition of $B(C_6F_5)_3$ [7]. Carbonyl coordination to the borane gives zwitterionic $[(C_6H_5)_2PC_6H_4C(O\text{-}B(C_6F_5)_3)O\text{-}\kappa^2P,O]Ni(\eta^3\text{-}CH_2CMeCH_2)$ and removes electron density from the nickel center. Exam-

ination of ligand/reactivity relationships for cationic Ni(diimine) initiators [2,8] led to the design and synthesis of $\{(H_3C)C[=NAr]C[O\text{-}B(C_6F_5)_3][=NAr]\text{-}\kappa^2N,N'\}\text{-}Ni(\eta^3\text{-}CH_2C_6H_5)$, with which high molecular weight polyethylene (PE) can be produced for bulky aromatic (Ar) substituents [9]. Several other ligand types have been reported that are amenable to the concept of Lewis acid attachment on a ligand site to redistribute electron density, including 3-(1-arylimino-ethyl)-acetylacetonato [10], 2-diphenylphosphinylbenzamido [11], *N*-(2-benzoylphenyl)-benzamido [12], α-iminoenamido [13], 2-(alkyldeneamino)-benzoato [14] and iminoamido pyridine [15], However, none of these systems have been able to produce high molecular weight PE in the absence of bulky Ar substituents.

Herein we show a zwitterionic Ni complex containing the simple and relatively small 3-(arylimino)-but-1-en-2-olato ligand, in which the active species is stabilized by a chelated olefin adduct. These complexes, which have reduced bulk, possess the ability to produce high molecular weight PE.

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2. Results and discussion

2.1. Synthesis and characterization of a zwitterionic nickel–olefin initiator

As shown in Scheme 1, synthetic access begins with 3-(2,6-diisopropylphenylimino)-butan-2-one [16], which is obtained by condensation of 2,6-diisopropylaniline with 2,3-butanedione. Deprotonation with NaH in THF provides the sodium salt in 80% yield. Subsequent reaction of the salt with $Ni(PMe_3)_2(\eta^1\text{-}CH_2C_6H_5)Cl$ [17] provides a new organometallic species, which by ^{1}H NMR spectroscopy (C_6D_6) contains the ligand fragment (δ: 4.91 and 4.63 ppm, $=CH_2$), an η^1-benzyl group (δ: 7.56 ppm, $CH_2C_6H_5$) and PMe_3 (δ: 0.90 ppm). The vicinal protons on the methylene unit adjacent to oxygen appear as singlets [18].

Single crystals of the product were obtained from a solution of pentane and the results are shown in Fig. 1. The product is thus (3-(2,6-diisopropylphenylimino)-but-1-en-2-olato)-(η^1-benzyl)-(trimethylphosphine)-nickel (**1**), as shown in Scheme 1.

Structural characterization of **1** reveals a distorted square-planar geometry with a *cis* relationship between the benzyl ligand and the imine nitrogen. The 3-(2,6-diisopropylphenylimino)-but-1-en-2-olato ligand coordinates to the nickel via the nitrogen and oxygen atoms with Ni–O and Ni–N bond lengths of 1.9031(16) and 1.9555(18) Å, respectively. The C1–C2 (1.344(3) Å) and C3–N (1.294(3) Å) distances are consistent with double bond character, whereas the C2–C3 (1.486(3) Å), C3–C4 (1.497(3) Å) and C2–O (1.315(3) Å) distances reveal single bond character. These data indicate localized bonding in the chelating ring.

The addition of 2 equiv. $B(C_6F_5)_3$ to **1** in toluene results in the formation of $Me_3P\text{-}B(C_6F_5)_3$, which precipitates out of solution, and a new organometallic product. ^{1}H NMR spectra of the product show the formation of two isomers in a 9:1 ratio. The upfield shift of the aromatic signals due to $-CH_2C_6H_5$ from 6.96–7.56 to 5.97–6.86 ppm indicates η^3-coordination. The vinyl protons are observed as a pair of doublets ($J = 3.5$ Hz) at 4.35 and 3.25 ppm. The ^{11}B NMR spectrum shows a signal at -2.6 ppm, consistent with boron–oxygen coordination. [1]H NOE spectroscopy between the vinyl group and the benzyl group indicates that the sets of isomers arise from pseudorotamers of the
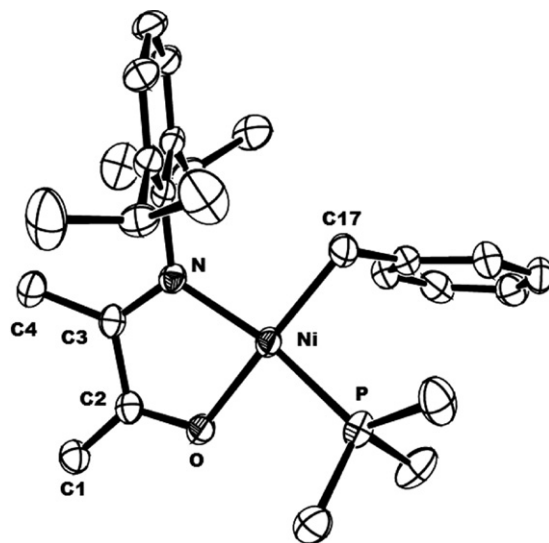

Fig. 1. ORTEP drawing of **1** at the 50% probability level. Hydrogen atoms are not shown for clarity.

benzyl ligand [15] and that the major isomer contains a *cis* relationship between the vinyl group and the benzyl group, as shown in Scheme 1.

Single crystals of compound **2** formed from a toluene solution and the structure is shown in Fig. 2. Isomer **2(A)** was obtained preferentially. The oxygen binds to the Lewis acidic boron and by doing so, forces coordination of the olefin to the nickel center. The distances from Ni to C1 and C2, are 2.109 (6) Å and 2.455(6) Å, respectively. The rotation of the C2–C3 bond and the C1–C2–C3–N torsional angle (41.6(8) °), are oriented to allow overlap between the π-orbital of the $-C=CH_2$ group and Ni. The C2–O distance (1.319(6) Å) is characteristic of a single bond, while the C1–C2 (1.371(7) Å) distance is more indicative of a double bond. These observations are consistent with the charge distribution as shown for structures **2(A)** and **2(B)** in Scheme 1. The C3–N distance (1.300(6) Å) is close to that observed in compound **1** (1.294(3) Å). The N–Ni bond length (1.900(4) Å) is 0.055 Å shorter than that of compound **1** (1.9555(18) Å), revealing a more electron deficient metal center in the zwitterionic compound.

9 (A) : 1 (B)

1　　　　　　　　　　　　　　**2**
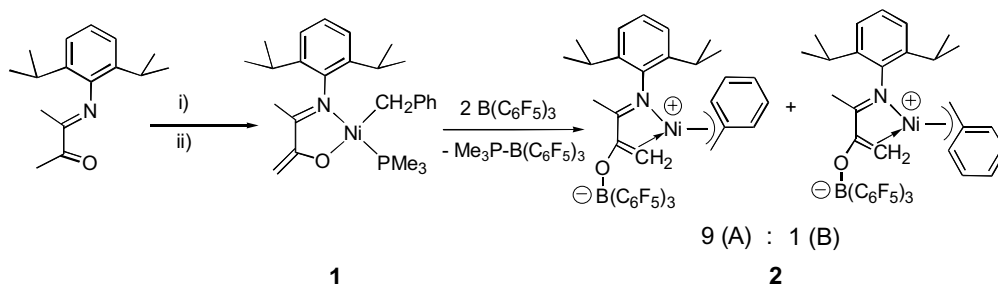
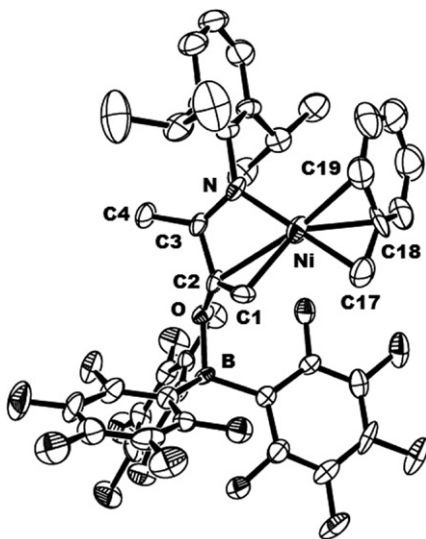Scheme 1. (i) NaH, (ii) $Ni(PMe_3)_2(\eta^1\text{-}CH_2C_6H_5)Cl$.

Fig. 2. ORTEP diagrams of the molecular structure of **2** at the 50% probability level. Hydrogen atoms are omitted for clarity.

2.2. Reactivity toward ethylene

Ethylene polymerization reactions with **1** or **2** were studied, and the results of these studies are listed in Table 1. The addition of ethylene at 100 psi to a solution of **2** in toluene at 30 °C results in the quick consumption of the monomer and polymer formation (entry 1). An additional 6 equiv. of $B(C_6F_5)_3$ increases the activity of the catalyst and decreases the PDI of the polymer (entry 2). Our current thinking is that $B(C_6F_5)_3$ acts as a scrubbing agent that removes impurities in the reaction medium that may interfere with the propagating sites. Thermal analysis indicates that the polymer has a high melting point, consistent with the highly linear polymer structure (7 branches/1000 carbons) revealed by ^{1}H NMR spectroscopy. The activity increases with temperature and at 75 °C (entries 3 and 4) it is comparable to those of cationic diimine nickel initiators [2]. The polymers formed at high temperature have broader PDIs, increased branching in the backbone (23 branches/1000 carbons) and lower melting points [1,19]. No reaction is observed when **1** is used alone (entry 5).

3. Conclusion

In summary, we have demonstrated that a zwitterionic nickel complex supported by the 3-(2,6-diisopropylphenylimino)-but-1-en-2-olato ligand can be used to prepare high molecular weight PE. The most noteworthy feature of the active site is that it is supported by an olefinic fragment, which lacks substantial steric hindrance. Such a structure departs from previous structure/reactivity relationships and theoretical predictions for cationic Ni(diimine) complexes, which require sterically demanding substituents to produce high molecular weight PE. Bonding from an olefin is considerably different from that of a nitrogen donor in that π-back bonding is a possibility. Whether this electronic feature is responsible for increasing the ratio of the rate of chain propagation, relative to chain transfer or termination rates is unknown at this stage and may require theoretical analysis for elucidation.

4. Experimental

All manipulations were performed under a nitrogen atmosphere using standard glove box and schlenk techniques. Toluene and THF were distilled from benzophenone ketyl. Pentane and hexane were dried over Na/K alloy. The toluene for polymerization use was purchased from Aldrich (anhydrous grade) and purified further over Na/K alloy. Tis(pentafluorophenyl)boron was provided by Boulder Scientific Company, Mead, Colorado and purified further by sublimation. Ethylene (99.99%) was purchased from Matheson Trigas and purified by passing through the Agilent moisture and oxygen/moisture traps. 2,3-Butanedione and 2,6-diisopropylaniline were purchased from Aldrich and used without further purification. 3-(2,6-Diisopropylphenylimino)-butan-2-one was prepared from condensation of 2,6-diisopropylaniline with 2,3-butanedione in benzene using a catalytic amount of p-toluenesulfonic acid. NMR spectra were recorded on Varian 400, 500 or Bruker 200 spectrometers. ^{1}H NMR spectra of the polyethylene were obtained in a mixed solvent (C_6D_6/1,2,4-trichlorobenzene 1:4 ratio in volume) at 115 °C. ^{1}H and ^{13}C NMR spectra were calibrated using signals from the solvent and are reported downfield from tetramethylsilane referenced to residual solvent ^{1}H and ^{13}C signals. ^{11}B NMR, ^{19}F NMR and ^{31}P NMR spectra were calibrated and reported downfield from external $BF_3 \cdot OEt_2$, $CFCCl_3$ and H_3PO_4, respectively. Elemental analyses were performed by Analytic Lab, Marine Science Institute, University of California, Santa Barbara. Gel permeation chromatograms (GPC) were measured on PL-GPC220 in trichlorobenzene at 135 °C, and the calibration curve was established against polystyrene standards. Thermal analysis was carried out on a TA instruments DSC 2920. T_m data were measured at a scan rate of 10 °C/min.

Table 1
Polymerization of ethylene[a] with **1** or **2**

Compd (μmol)	Additive (equiv.)	T [b]	Time (min)	A [c]	M_w [d]	PDI	T_m [b]
2 (6)	0	30	10	0.45	135.5	5.8	135
2 (3)	BCF(6)	30	8	2.2	134.8	2.5	134
2 (3)	BCF(6)	75	8	4.0	149.7	6.9	117
2 (2)	BCF(6)	75	5	6.9	132.3	6.8	118
1 (6)	0	30	10	0	–	–	–

[a] Polymerization conditions: 26 g toluene, 100 psi ethylene.
[b] °C.
[c] Activity in kg polymer/(mmol cat h).
[d] M_w ($\times 10^3$).

4.1. 3-(2,6-Diisopropylphenylimino)-but-1-en-2-olato (η^1-benzyl)(trimethylphosphine)nickel (1)

NaH (0.120 g, 5.0 mmol) was added to 3-(2,6-diisopropylphenylimino)-butan-2-one (1.23 g, 5.0 mmol) in 5 g of THF. After stirring overnight at room temperature, the solvent was removed under vacuum. The residue was dissolved in toluene and filtered through celite. Excess toluene was removed and pentane was added, the mixture was cooled to −35 °C to yield a white solid (1.08 g, 80 % yield). ^{1}H NMR (400 MHz, C_6D_6/THF-d_8 (5/1), 25 °C): $\delta = 7.14$–7.00 (m, 3H; imine-ph-H), 4.42 (s, 1H; CH_2), 4.23 (s, 1H; CH_2), 2.93 (septet, $J = 6.8$ Hz, 2H; iPr-CH), 1.88 (s, 3H; CH_3), 1.28 (d, $J = 6.8$ Hz, 6H; iPr-CH_3), 1.18 ppm (d, $J = 6.8$ Hz, 6H; iPr-CH_3). The sodium salt (227 mg, 0.85 mmol) and Ni(PMe$_3$)$_2$(η^1-benzyl)Cl (288 mg, 0.85 mmol) were mixed at −35 °C in toluene and allowed to warm to room temperature over 2 h. Excess toluene was then removed under vacuum and the residue was extracted with pentane. The solution was concentrated to ∼3 mL and cooled to −35 °C to yield an orange solid (295 mg, 74% yield). ^{1}H NMR (400 MHz, C_6D_6, 25 °C): $\delta = 7.56$ (d, $J = 7.2$ Hz, 2H, benzyl-ph-H^{2,6}), 7.03–6.96 (m, 6H; imine-ph-H and benzyl-ph-H^{3,4,5}), 4.91 (s, 1H; CH_2), 4.63 (s, 1H; CH_2), 3.60 (septet, $J = 6.8$ Hz, 2H; iPr-CH), 1.65 (s, 3H; CH_3), 1.32 (d, $J = 6.8$ Hz, 6H; iPr-CH_3), 1.04 (s, 2H; benzyl-CH_2), 1.01 (d, $J = 6.8$ Hz, 6H; iPr-CH_3), 0.74 ppm (d, $J = 10.0$ Hz, 9H; PCH$_3$); ^{13}C NMR (100 MHz, C_6D_6, 25 °C): $\delta = 181.88$ (imine), 169.24 (=C–O), 152.23, 142.86, 140.96, 130.35, 128.09, 127.27, 124.29, 123.45 (ph-C), 92.11 (CH_2), 28.85 (iPr-CH), 24.60 (iPr-CH_3), 24.37 (iPr-CH_3), 18.81 (CH_3), 12.87 (d, $J = 28$ Hz, PCH$_3$), 9.55 ppm (d, $J = 26$ Hz, benzyl-CH_2); ^{31}P NMR (162 MHz, C_6D_6, 25 °C): $\delta = -8.69$ ppm; Elemental analysis Calc. for $C_{26}H_{38}NOPNi$: C, 66.41; H, 8.14; N, 2.98. Found: C, 65.97; H, 8.24; N, 3.02%.

4.2. 2-Tris(pentafluorophenyl)borate-3-(2,6-diisopropylphenylimino)-but-1-ene(η^3-benzyl)nickel (2)

Compound 1 (94 mg, 0.20 mmol) and 2 equiv. of B(C$_6$F$_5$)$_3$ (205 mg, 0.40 mmol) were mixed in toluene (7.0 g). The color of solution changed from orange into purple immediately along with the formation of a white precipitate. After 90 min, hexanes were added and the reaction was then filtered after 15 min. The solvents were then removed under vacuum. The residue was washed with hexanes, and dried under vacuum to afford a purple solid (168 mg, 93% yield). ^{1}H NMR (500 MHz, C_6D_6, 25 °C. Two sets of peaks (9:1 ratio) were observed): major isomer, $\delta = 6.86$ (t, $J = 7.5$ Hz, 1H; imine-ph-H^4), 6.77 (d, $J = 7.5$ Hz, 1H; imine-ph-H$^{3\,or\,5}$), 6.67 (d, $J = 7.5$ Hz, 1H; imine-ph-H$^{3\,or\,5}$), 6.52 (t, $J = 7.5$ Hz, 1H; benzyl-ph-H^4), 6.31 (t, $J = 7.5$, 1H; benzyl-ph-H$^{3\,or\,5}$), 6.30 (t, $J = 7.5$, 1H; benzyl-ph-H$^{3\,or\,5}$), 6.25 (d, $J = 7.5$ Hz, 1H; benzyl-ph-H^6), 5.97 (d, $J = 7.5$ Hz, 1H; benzyl-ph-H^2), 4.35 (d, $J = 3.5$ Hz, 1H; CH_2), 3.25 (d, $J = 3.5$, 1H; CH_2), 2.38 (septet, $J = 7.0$ Hz, 1H; iPr-CH), 1.96

(septet, $J = 7.0$ Hz, 1H; iPr-CH), 1.63 (s, 3H; CH_3), 1.15 (d, $J = 2.5$ Hz, 1H; benzyl-CH_2), 0.95 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 0.88 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 0.81 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 0.58 (d, $J = 2.5$ Hz, 1H; benzyl-CH_2), 0.49 ppm (d, $J = 7.0$ Hz, 3H; iPr-CH_3); minor isomer, 6.61 (d, $J = 7.5$ Hz, 1H; benzyl-ph-H^4), 6.37 (d, $J = 7.5$ Hz, 1H; benzyl-ph-H^2), 5.02 (d, 1H; CH_2), 3.27 (d, 1H; CH_2), 1.50 (s, 3H; CH_3), 1.18 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 1.10 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 1.03 (d, $J = 7.0$ Hz, 3H; iPr-CH_3), 0.68 ppm (d, $J = 7.0$ Hz, 3H; iPr-CH_3), some signals of the minor isomer were not seen due to the overlap with those of the major isomer; ^{13}C NMR (100 MHz, CD_2Cl_2, 25 °C): $\delta = 183.55$ (imine), 164.40 (=C–O), 149.67, 147.27, 141.27, 139.99, 138.69, 138.20, 136.44, 135.30, 134.19, 130.04, 128.42, 124.75, 123.42, 115.74, and 113.87 (ph-C), 104.20 (CH_2), 29.06, 28.92 and 28.39 (iPr-CH and CH_3), 24.26, 24.22, 23.84 and 22.45 (iPr-CH_3), 19.33 ppm (benzyl-CH_2); ^{19}F NMR (376 MHz, C_6D_6, 25 °C): $\delta = -133.70$, −158.08, −164.28 ppm; ^{11}B NMR (161 MHz, CD_2Cl_2, 25 °C): $\delta = -2.6$ ppm; Elemental analysis Calc. for $C_{41}H_{29}NOBF_{15}Ni$: C, 54.34; H, 3.23; N, 1.55. Found: C, 54.06; H, 2.81; N, 1.50%.

4.3. Polymerization reactions

Typical polymerization of ethylene: B(C$_6$F$_5$)$_3$ (18 μmol in 1.0 g toluene) and compound 2 (3 μmol in 1.0 g toluene) were placed in a 100 mL steel autoclave reactor charged with 26.0 g of toluene. The reactor was then assembled and brought out of the glove box, ethylene was then added at 100 psi. Reaction length and temperature were varied as described in Table 1. The reaction was quenched by release

Table 2
Crystallographic data for compounds 1 and 2

Compound	1	2
Formula	$C_{26}H_{38}NOPNi$	$C_{41}H_{29}BF_{15}NONi$
Molecular weight	470.25	906.17
Crystal system	Monoclinic	Monoclinic
Space group	$P2(1)/c$	$P2(1)/c$
Unit cell parameters		
a (Å)	9.8143(6)	13.9249(16)
b (Å)	21.7333(13)	14.2751(16)
c (Å)	12.0168(7)	20.158(2)
α (°)	90	90
β (°)	94.571(2)	105.457(3)
γ (°)	90	90
V (Å^3)	2555.0(3)	3862.0(8)
Z	4	4
D_{calc} (Mg m^{-3})	1.223	1.558
λ (Mo Kα) (mm^{-1})	0.838	0.610
Crystal Size (mm)	$0.2 \times 0.1 \times 0.05$	$0.12 \times 0.1 \times 0.06$
Habit	Blades	Blades
Color	orange	purple
θ Range (°)	1.87–28.21	1.77–26.71
Parameters refined	423	561
$F(000)$	1008	1832
R_1, wR_2 [$I>2\sigma(I)$]	0.0418, 0.0913	0.0641, 0.01153
Maximum and minimum density (e Å^{-3})	0.831 and −0.233	0.601 and −1.160

of ethylene pressure and addition of acetone. The precipitated polymer was collected by filtration and dried under high vacuum overnight (see Table 2).

Acknowledgement

Appendix A. Supplementary material

CCDC 280467 and 280468 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2007.05.016.

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