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Copolymerization of olefins and CO catalyzed by new chiral arsine–oxazoline palladium complexes

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Abstract

Coordination of a chiral arsine–oxazoline ligand (**As**–**N**) toward (COD)$PdCl_2$ and (COD)PdMeCl leading to (**As**–**N**)$PdCl_2$ (**3**) and (**As**–**N**)PdMeCl (**4**) were reported. Both **3** and **4** were characterized by spectroscopic and X-ray single crystal diffraction methods. Treatment of **4** with $AgBF_4$ in the presence of acetonitrile resulted in the formation of [(**As**–**N**)PdMe(CH_3CN)]BF_4 (**6**). Complex **6** catalyzed the copolymerization of CO and olefin, but in low conversions. In fact, the palladium black and [(**As**–**N**)$_2$Pd]$(BF_4)_2$ (**7**) were formed from the copolymerization processes, which may explain the lower catalytic activity of **6**. Crystal structure of **7** was also determined.
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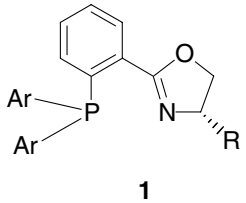
Keywords: Arsine; Heterobidentate; Palladium; Copolymerization; Chiral

1. Introduction

Alternating copolymerization of CO and olefin mediated by palladium complexes have attracted considerable attention in past years [1]. In particular, the stereo-regular copolymers from α-alkenes constitute an important application in this area [2]. These studies illustrate that both the electronic and steric environment of the ligand are crucial in controlling the selectivity and activity of the metal centers during the polymerization [2–14]. In contrast to symmetrical bidentate systems, ligands containing both hard and soft donor atoms provide better manipulation on the regularity of copolymers. Several chiral palladium **P**–**N** (phosphorus–nitrogen donors) complexes used as catalysts for copolymerization leading to highly stereoselective copolymers have been reported by Consiglio [2]. For comparison with the phosphine–oxazoline bidentate **1**, we report here the preparation, characterization and catalytic copolymerization of palladium complexes containing a chiral arsine–oxazoline ligand **2**.



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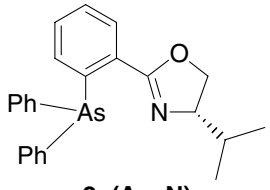

2 (As~N)

2. Results and discussion

2.1. Preparation of complexes and insertion study

Ligand **2** was prepared according to our previously reported procedures [15]. Reactions of equimolar amounts of **2** with Pd(COD)Cl_2 and Pd(COD)MeCl [COD = 1,5-cyclooctadiene] in dichloromethane solution afforded the corresponding complex [Pd(**2**)Cl_2] (**3**) and [Pd(**2**)MeCl]

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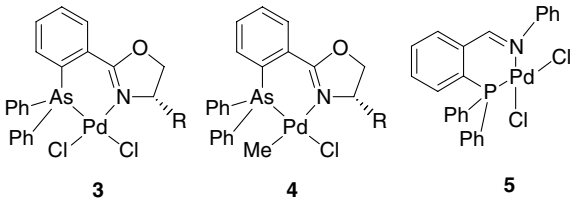
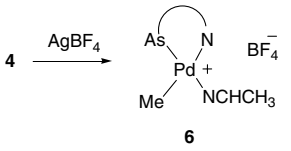
(**4**), respectively. Upon the treatment of silver salt in the presence of acetonitrile, the abstraction of chloride ligand of **4** gave the cationic complex **6** (Eq. 1).





(1)

Analytically pure products of **3** and **4**, which were characterized by spectroscopic methods and single-crystal analysis, were obtained in good yields. A slight shift of the proton at the chiral center for both palladium complexes indicates the coordination of metal ion toward ligand. Chemical shifts of ^{1}H NMR chemical shift (0.59 ppm) and ^{13}C NMR (−4.8 ppm) for Pd–CH_3 are in typical range for methyl group attached to the palladium center. However, the detail structural information comes from their crystal structural determination. ORTEP diagrams for **3** and **4** are shown in the Figs. 1 and 2, respectively.

As shown in Figs. 1 and 2, the palladium metal center was seated in a square planar arrangement with the chelation of arsine and oxazoline–nitrogen. Selected bond lengths and angles are shown in the Table 1. All bond distances and bond angles of **3** and **4** lie within the normal range. There is clearly a differentiation between Pd–Cl bond lengths in **3** due to the *trans* influence of donors between arsine and imine. The distance of Pd–Cl(1), *trans* to the arsine, appears to be longer than that *trans* to imine donor, which is similar to the related phosphine complex **5** [16]. This outcome suggests that the arsine donor resembling to phosphine, is a good σ-donor. The magnitude of the ligand bite angle N–Pd–As of 88.14(9)° is close to that found for a range of (P–N)$PdCl_2$ such as **5** [86.34(8)°]. The phenyl groups on As center are diastereotopic with one phenyl group at an equatorial position and the other at axial position. The axial phenyl group is arranged in *cis* fashion with respect to the isopropyl group of oxazoline ring.

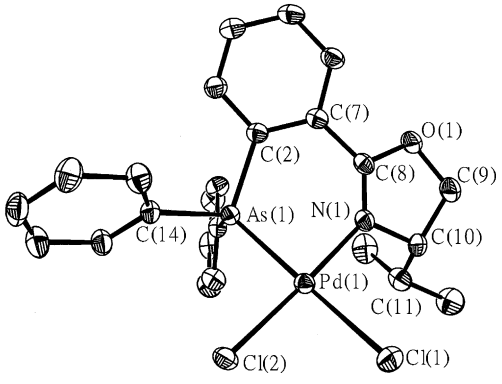


Fig. 1. Molecular structure of **3** (drawn with 30% probability ellipsoids).

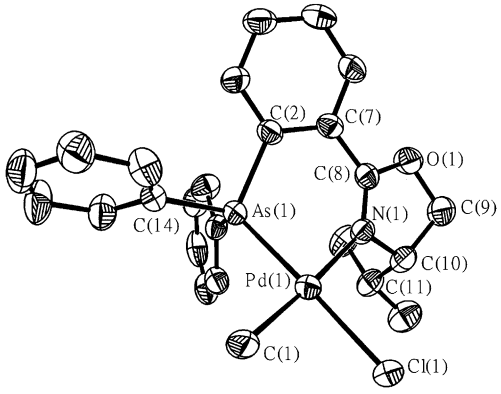


Fig. 2. ORTEP plot of **4** (30% probability ellipsoids).

Table 1
Selected bond distances (Å) and bond angles (°) for complexes **3** and **4**

Complex	**3** [X = Cl(2)]	**4** [X = C(1)]
Pd(1)–N(1)	2.046(3)	2.157(4)
Pd(1)–As(1)	2.3109(4)	2.3000(6)
Pd(1)–Cl(1)	2.368(1)	2.372(1)
Pd(1)–X	2.288(1)	2.057(5)
N(1)–Pd(1)–As(1)	88.14(9)	84.54(9)
N(1)–Pd(1)–Cl(1)	92.87(9)	94.0(1)
N(1)–Pd(1)–X	173.65(9)	176.3(2)
As(1)–Pd(1)–Cl(1)	174.79(4)	174.47(4)
As(1)–Pd(1)–X	87.98(3)	91.9(2)
Cl(1)–Pd(1)–X	91.44(4)	89.7(2)

For complex **4**, the methyl group is positioned *trans* to the nitrogen donor. Since the methyl group is *trans* to imine donor, the palladium–carbon distance [2.057(5) Å] is in the typical range of Pd–C bonds of the related species [6]. The angle of As(1)–Pd–N(1) [84.54(9)°] is much deviated from 90° even smaller than that in **3** presumably due to the steric relief of methyl group and arsine donor. The orientation of phenyl groups on arsine is quite similar to that in **3**. Thus, the two phenyl groups are diastereotopic with one phenyl group occupying an axial position and the other one at an equatorial position.

As for complex **6**, it appeared to be less stable and slowly decomposed in solution. However, it could be characterized by both NMR and mass spectroscopy. The presence of two weak bands around 2317 and 2288 cm^{-1} verify the coordinating acetonitrile ligand around the metal center, which is comparable to the recently reported palladium complexes with P–N ligand [6b]. All ^{1}H NMR signals of **6** are similar to those of **4**, except the shift at δ 2.39 for the coordinating acetonitrile. The chemical shift of Pd–Me in **6** appears to be slightly up-field than that of **4**.

2.2. Copolymerization

In order to understanding the reactivity, we investigated the catalytic property of these palladium complexes on

Table 2
Copolymerization of olefin and CO catalyzed by Pd(II) complexes

Entry	Catalyst	CO (psi)	Olefin	Solvent	Temp.	Yield (mg)	Productivity[a]
1	**3** (5.5 mg)	100	Styrene	CH_2Cl_2	r.t.	–	–
2	**6** (5.5 mg)	100	Styrene	CH_2Cl_2	r.t.	47	2.22
3	**6** (5.5 mg)	100	Styrene	CH_2Cl_2	100 °C	22	1.04
4	**6** (5.5 mg)	250	Styrene	CH_2Cl_2	r.t.	56	2.64
5	**6** (5.5 mg)	100	Styrene	CH_3CN	r.t.	10	–
6	**6** (2.8 mg)	100	Ethylene	CH_2Cl_2	r.t.	130	12.1
7	**6** (2.8 mg)	100	Norbornene	CH_2Cl_2	r.t.	11.3	–

[a] Productivity = g (polymer)/g (Pd) h.

copolymerization. While the complex **3** showed the poor activity, complex **6** could be used as a catalyst for copolymerization of CO and olefins (Table 2). In one of the typical reactions, **6** (5.5 mg) with CO (100 psi) and styrene (0.4 mL) in dichloromethane produces 47 mg of poly(CO–*alt*–styrene) [$M_N = 2900$; PDI = 1.4]. As shown in Table 2, complex **6** does catalyze the copolymerization of CO and olefins. The obtained styrene/CO copolymers were characterized by ^1H and ^{13}C NMR spectroscopy. The chirality of copolymers was analyzed by ^{13}C NMR spectral method and CD developed by Consiglio and coworkers [2a,17]. Fig. 3 shows the ^{13}C NMR spectrum in the region of the *ipso*-carbon atom for the copolymer of styrene/CO. The obtained copolymer shows essentially a single sharp band in that region (at δ 136.3 ppm), consistent with a highly ordered structure. In addition, the rotatory strength $\Delta\varepsilon = -1.02\ l\ cm^{-1}\ g^{-1}$ of the CD band in the region of the carbonyl and phenyl chromophors suggest the isotatic structure of poly(CO–*alt*–styrene) according to the literature data [2a,2e]. These results are essentially similar to those obtained from palladium–phosphine–oxazoline catalyst [2], suggesting that the chiral induction is irrelevant to the arsine donor, but to the configuration of substituents.

This cationic arsine–palladium complex does not show excellent catalytic activities for the copolymerization of CO and alkenes as compared to the phosphine analog [2d,2e,2f]. In fact, we isolated a bis ligand palladium complex $[(\mathbf{As}–\mathbf{N})_2Pd](BF_4)_2$ (**7**) and palladium black from the reaction mixture of the catalysis, which might explain one of the possible factors for lowering the catalytic activity. The formation of **7** appears to be the result of disproportionation of **6** during the catalysis, indicating the unstable nature of the active catalytic species.

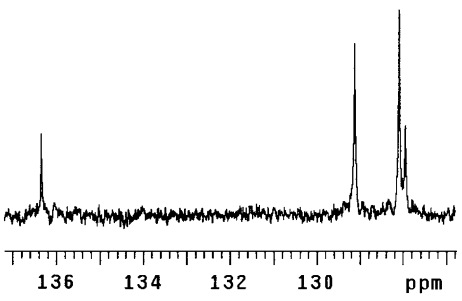


Fig. 3. ^{13}C NMR spectrum in the region of the *ipso*-carbon for the poly(CO–*alt*–styrene).

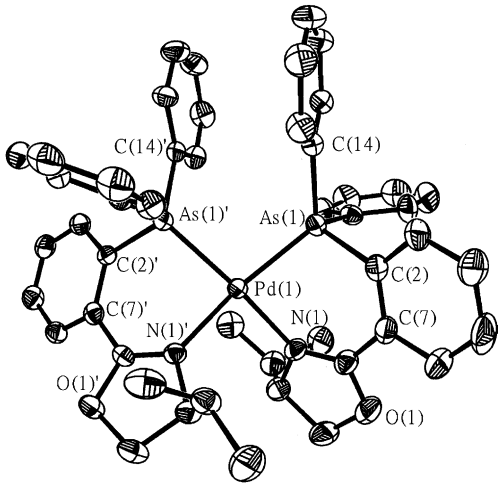


Fig. 4. ORTEP plot of **7**. Pd(1)–As(1), 2.3516(6) Å; Pd(1)–As(1)′, 2.3420(6) Å; Pd(1)–N(1), 2.077(4) Å; Pd–N(1)′, 2.088(4) Å; N(1)–Pd(1)–N(1)′, 91.2(2)°; N(1)–Pd(1)–As(1), 85.1(1)°; As(1)–Pd(1)–As(1)′, 99.49(2)°; N(1)′–Pd(1)–As(1)′, 86.2(1)°.

The detail coordination arrangement of complex **7** was established unambiguously by a single crystal X-ray diffraction study. In the crystal structure of **7** (Fig. 4), the palladium(II) center is *cis* coordinated by two **As**–**N** bidentate ligands arranged in a slightly distorted square planar geometry as illustrated by the angles around the metal. As shown in Fig. 4, the two six-membered metallacycles embody two **As**–**N** ligands with phenyl substituents on arsines positioned at both axial and equatorial directions. The Pd(1)–As(1), Pd(1)–As(1)′, Pd(1)–N(1), Pd–N(1)′ bond lengths are comparable to those in **3** and **4**. The crystal structure of **9** readily confirms the stereochemistry around the palladium center. This complex appears to be stable under aerobic conditions. No evidence for isomerization was observed even after allowing $CDCl_3$ solutions of **7** to stand for up to several days.

3. Conclusion

In this work, we have prepared and characterized the chiral palladium arsine–oxazoline complexes. The cationic palladium complexes chelated through an arsine–oxazoline

linkage exhibits moderate activities in copolymerization of olefins and carbon monoxide. However, the catalytic activity is poorer than that of the phosphine–oxazoline analog due to the decomposition of complex during the catalysis.

4. Experimental

4.1. General

General information. All reactions, manipulations and purifications steps were performed under a dry nitrogen atmosphere. Tetrahydrofuran was distilled under nitrogen from sodium benzophenone ketyl. Dichloromethane and acetonitrile were dried over CaH_2 and distilled under nitrogen. Other chemicals and solvents were of analytical grade and were used after degassed process. Ligand **As–N** was prepared accordingly to the method reported previously [15]. (*Caution*: arsine is highly toxic and should be handled in hood carefully!)

Nuclear magnetic resonance spectra were recorded in $CDCl_3$ or acetone-d_6 on either a Bruker AM-300 or AVANCE 400 spectrometer. Chemical shifts are given in parts per million relative to Me_4Si for ^1H and ^{13}C NMR, and relative 85% H_3PO_4 for ^{31}P NMR. Infrared spectra were measured on a Nicolet Magna-IR 550 spectrometer (Series-II) as KBr pallets, unless otherwise noted.

4.2. Synthesis and characterization

4.2.1. Complex **3** *[(Ar–N)PdCl$_2$]*

A mixture of **2** (10.3 mg, 24.7 μmol) and (COD)$PdCl_2$ (7.0 mg, 24.7 μmol) in dichloromethane was stirring for 0.5 h under N_2 atmosphere. The reaction mixture was filtrated through Celite followed by crystallization in dichloromethane and hexane to afford yellow crystalline solids (14 mg, 95%), which was suitable for X-ray analysis. ^1H NMR ($CDCl_3$, 400 MHz): δ 8.09 (d, 1H, $J = 7.2$ Hz, *ortho* to oxazolyl), 7.66 (dd, 1H, $J = 7.6$, 7.6 Hz, *para* to oxazolyl), 7.58–7.39 (m, 11H, As-*Ph*), 7.07 (d, 1H, $J = 7.2$ Hz, *ortho* to arsinyl), 5.51–5.49 (m, 1H, NC*H*), 4.44 (dd, 1H, $J = 10.0$, 9.2 Hz, *cis* to isopropyl), 4.33 (dd, 1H, $J = 5.2$, 9.2 Hz, *trans* to isopropyl), 2.80–2.70 (m, 1H, C*H* of isopropyl), 0.80 (d, 3H, $J = 6.8$ Hz, –CH_3), 0.11 (d, 3H, $J = 6.8$ Hz, –CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.9 (oxazolyl), 133.7, 133.6, 132.9, 132.7, 131.9, 131.5, 129.8, 129.2, 128.7, 128.6, 128.0, 126.7, 71.6 (N*C*H), 68.6 (O*C*H$_2$), 30.7 (*C*HMe$_2$), 18.5 (–*C*H$_3$), 13.2 (–*C*H$_3$). Anal. Calc. for $C_{24}H_{24}NOAsPdCl_2$: C, 48.47; H, 4.07; N, 2.36. Found: C, 48.40; H, 3.98; N, 2.15%.

4.2.2. Complex **4** *[(Ar–N)PdMeCl]*

Ligand **2** (31.8 mg, 76.2 μmol) and (COD)PdMeCl (20.2 mg, 76.2 μmol) were added together in CH_2Cl_2 (8 ml) with stirring under nitrogen. After 30 min, the solution was filtered through Celite under nitrogen and the filtrate was concentrated to afford the crude product, which was dissolved in dichloromethane. Upon addition of hexane, complex **4** was precipitated as a white solid (39.3 mg, 89.8%): ^1H NMR ($CDCl_3$, 400 MHz): δ 8.01 (dd, 1H, $J = 8.0$, 1.2 Hz, *ortho* to oxazolyl), 7.54–7.34 (m, 10H, As-*Ph*), 7.21–7.18 (m, 2H, Ar), 7.04 (dd, 1H, $J = 8.0$, 1.2 Hz, *ortho* to arsinyl), 5.16 (ddd, 1H, $J = 9.6$, 4.8, 3.2 Hz, NC*H*), 4.23 (dd, 1H, $J = 9.6$, 9.6 Hz, *cis* to isopropyl), 4.16 (dd, 1H, $J = 9.6$, 4.8 Hz, *trans* to isopropyl), 2.52 (dqq, $J = 7.2$, 7.2, 3.2 Hz, C*H*Me$_2$), 0.76 (d, 3H, $J = 7.2$ Hz, CH(CH_3)Me), 0.59 (s, 3H, Pd*Me*), 0.10 (d, 3H, $J = 7.2$ Hz, CH(CH_3)Me). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.4 (*C*(N)O), 133.7, 133.3, 133.1, 132.2, 132.1, 131.2, 130.9, 130.7, 130.6, 129.7, 129.3, 129.1, 129.0, 70.7 (N*C*H), 67.8 (O*C*H$_2$), 30.6 (*C*HMe$_2$), 18.9 (CH(*C*H$_3$)Me), 13.8(CH(*C*H$_3$)Me), −4.8 (Pd*Me*). Anal. Calc. for $C_{25}H_{27}NOAsPdCl$: C, 52.29; H, 4.74; N, 2.44. Found: C, 52.18; H, 4.64; N, 2.35%.

4.2.3. Complex **6** *[(Ar–N)PdMe(NCCH$_3$)$^+$BF$_4^-$]*

Complex **4** (5.9 mg, 10.3 μmol) and $AgBF_4$ (2.0 mg, 10.3 μmol) were mixed together in CH_2Cl_2 (1 mL) with stirring under nitrogen. Fresh-distilled acetonitrile (0.01 ml, 190 μmol) was added. The precipitate formed after supersonic vibration for 30 min. Reaction mixture was filtered through Ceilte under nitrogen, and then the excess acetonitrile and solvent were removed under vacuum. Light-yellow product (90%) was isolated. IR (CH_2Cl_2): 2317, 2288 cm^{-1} ($CH_3C{\equiv}N{-}Pd$); ^1H NMR ($CDCl_3$, 400 MHz): δ 8.11 (dd, 1H, $J = 8.0$, 1.2 Hz, *ortho* to oxazolyl), 7.61–7.35 (m, 10H, As *Ph*), 7.21–7.19 (m, 2H, Ar), 7.07 (d, 1H, $J = 8.0$ Hz, *ortho* to arsinyl), 4.71 (s, br, 1H, NC*H*), 4.48 (d, br, 1H, $J = 5.2$ Hz, *cis* to isopropyl), 4.22 (dd, 1H, $J = 9.2$, 5.2 Hz, *trans* to isopropyl), 2.39 (s, 3H, NCCH_3), 2.16 (s, br, 1H, C*H*Me$_2$), 0.72 (d, 3H, $J = 6.8$ Hz, CH(CH_3)Me), 0.44 (s, 3H, Pd*Me*), 0.29 (d, 3H, $J = 6.8$ Hz, CH(CH_3)Me). HRFAB for [$M^+ - CH_3CN$]: Calc.: 537.0359 ($C_{25}H_{27}NOAs^{105}Pd$), 538.0343 ($C_{25}H_{27}NOAs^{106}Pd$), 540.0348 ($C_{25}H_{27}NOAs^{108}Pd$); Found: 537.0356 ($C_{25}H_{27}NOAs^{105}Pd$), 538.0335 ($C_{25}H_{27}NOAs^{106}Pd$), 540.0331 ($C_{25}H_{27}NOAs^{108}Pd$).

4.2.4. Complex **7** *[(Ar–N)$_2$Pd(BF$_4$)$_2$]*

This complex was isolated from the residue of catalysis. ^1H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, 2H, $J = 7.2$ Hz, *ortho* to oxazolyl), 7.77–7.02 (m, 26H, Ar), 4.97–4.92 (m, 2H, NC*H*), 4.50–4.43 (m, 2H, *cis* to isopropyl), 4.31–4.28 (m, 2H, *trans* to isopropyl), 1.85–1.83 (m, 2H, C*H* of isopropyl), 0.81 (d, 6H, $J = 6.8$ Hz, CH(CH_3)Me), 0.29 (d, 6H, $J = 6.8$ Hz, CH(CH_3)Me). Anal. Calc. for $C_{48}H_{48}As_2B_2F_8N_2O_2Pd \cdot CH_2Cl_2$: C, 49.06; H, 4.20; N, 2.34. Found: C, 48.95; H, 3.99; N, 2.43%.

4.3. Copolymerization

To an autoclave (100 mL), the palladium complex in CH_2Cl_2(8 mL), CO (100 psi) and olefin were loaded. Reaction was kept stirring for 24 h at given temperature. The resulting reaction mixture was washed with 2 N HCl, and

Table 3
Crystallographic data of **3**, **4** and **7**

Complex	**3**	**4**	**7**
Formula	$C_{25}H_{26}AsCl_4NOPd$	$C_{25}H_{27}AsClNOPd$	$C_{49}H_{50}As_2B_2Cl_2F_8N_2O_2Pd$
F_w	679.59	574.25	1199.57
Crystal system	Monoclinic	Orthorhombic	Monoclinic
Space group	$P2_1$	$P2_12_12_1$	$P2_1$
a (Å)	10.3378(1)	10.7770(2)	12.1889(2)
b (Å)	12.8868(2)	13.1569(2)	14.8674(2)
c (Å)	10.8053(2)	17.5545(3)	14.0195(2)
α (°)	90	90	90
β (°)	112.0070(8)	90	91.130(1)
γ (°)	90	90	90
V (Å^3)	1334.61(3)	2489.09(7)	2540.08(6)
Z	2	4	2
D_{calc} (Mg/m^3)	1.691	1.532	1.569
$F(000)$	676	1152	1204
Crystal size (mm)	$0.20 \times 0.15 \times 0.10$	$0.25 \times 0.20 \times 0.15$	$0.20 \times 0.20 \times 0.15$
θ Range	2.12 – 27.48	1.93 – 27.48	1.45 – 27.46
Reflection collected	8931	14075	16860
Independent reflection	5426 ($R_{int} = 0.0295$)	5492($R_{int} = 0.0452$)	9686 ($R_{int} = 0.0297$)
Refined method	Full-matrix least squares on F^2		
$R[I > 2\sigma(I)]$	$R_1 = 0.0279$	$R_1 = 0.0347$	$R_1 = 0.0388$
	$wR_2 = 0.0707$	$wR_2 = 0.0786$	$wR_2 = 0.0943$
Goodness-of-fit on F^2	1.061	1.095	1.026

a white solid product was collected. Characterization of poly(CO–*alt*–olefin) were performed by NMR spectroscopy.

poly(CO–alt–styrene): IR (KBr, cm^{-1}) 3072 (w), 3039 (w), 2919 (w), 1706 (vs), 1613 (m), 1494 (m), 1461 (m), 1308 (m), 1056 (m), 765 (m), 698 (s); ^{13}C NMR (100 MHz, $CDCl_3$/HFIP = 1/1): δ 209.7 (*C*=O), 136.3 (C_{ipso}), 129.1 (C_{ortho}), 128.1 (C_{meta}), 127.9 (C_{para}), 52.9 (*C*Ph), 45.0 (CH_2C=O). *poly(CO–alt–ethylene):* IR (KBr, cm^{-1}) 2926 (m), 2860 (w), 1706 (vs), 1414 (s), 1341 (s), 1262 (m), 1056 (s), 811 (m), 599 (s), 546 (m). ^{13}C NMR (100 MHz, $CDCl_3$/HFIP = 1/1): δ 212.1 (*C*=O), 35.8 (CH_2).

4.4. Crystallography

Crystals suitable for X-ray determination were obtained for **3** · CH_2Cl_2, **4** and **7** · CH_2Cl_2 by slow diffusion of hexane into a dichloromethane solution at room temperature. Cell parameters were determined either by a Siemens SMART CCD diffractometer. Crystal data of these complexes are listed in Table 3. Other crystallographic data are deposited as supporting information.

Crystallographic data (excluding structure factors) for the structure reported in this work have been deposited with the Cambridge Crystallographic Data Center: CCDC-268713 for **7** · CH_2Cl_2, CCDC-268714 for **3** · CH_2Cl_2 and CCDC-268715 for **4**. Copies of this information can be obtained free of charge and by application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.ukwww: http://www.ccdc.cam.ac.uk.

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Appendix A. Supplementary data

The ^{13}C NMR spectrum of poly(alt–styrene–CO) is available from the authors. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2005.08.003.

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