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Chelates formed by a constrained bis(diphenylphosphino)xylene; the crystal structures of $[FeCl_2(anphos)]$ and [RhCl(CO)(anphos-monoxide)]

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Abstract

The indan derived diphosphine, *cis*-1,3-(diphenylphosphino)indan (anphos) is synthesised by the addition of $Ph_2P(BH_3)Li$ to *cis*-1,3-dibromoindan followed by deprotection with diethylamine. Anphos readily forms the bicyclic chelates [RhCl(CO)(anphos)], $[PtCl_2(anphos)]$, [PtCl(Me)(anphos)] and $[FeCl_2(anphos)]$. The crystal structures of $[FeCl_2(anphos)]$ and the monoxide complex, [RhCl(CO)(anphosO)] have been determined. Reaction of the diphosphine with $[Rh(acac)(CO)_2]$ under moderate hydroformylation conditions catalysed the formation of 1-heptanal and branched aldehydes from 1-hexene in a ratio of 1.5:1.

Keywords: Diphosphine; Hydroformylation; Synthesis and characterisation

1. Introduction

The bite angle and flexibility of diphosphines are important parameters when considering their potential for application in homogeneous catalysis [1]. *Ortho*-xylenyl diphosphines have a flexible C_4 backbone and these diphosphines have found several applications in catalysis: α,α-bis(diphenylphosphino)xylene (dppx) [2] with a calculated ligand-preferred bite angle of 90° has been reported as a ligand for Pt–Sn catalysed hydroformylation [3] and rhodium-catalysed CO_2 hydrogenation [4]. 1,2-Bis(di-*tert*-butylphosphino)xylene (dbpx) has been shown to be an excellent ligand for Pd-catalysed propene methoxycarbonylation, a reaction of potential commercial value [5].

Here we report the indan-derived diphosphine, anphos (**1**), which has a rigidified xylenyl backbone, and show that it readily forms chelates with square planar or tetrahedral metal centres.

2. Results and discussion

The anphos ligand was synthesised according to Scheme 1. The dibromo precursor **2** was made by the reaction of *N*-bromosuccinimide with indan [6]. Treatment of **2** with $LiPPh_2$ did not give anphos and the only detected product was Ph_2PH. However, Ph_2PBH_3Li reacted with **2** to give the borane-protected diphosphine $\mathbf{2}\cdot(BH_3)_2$ which upon treatment with diethylamine yielded anphos (**1**). Our attempts to make *cis*-1,3-(di-*tert*-butylphosphino)indan under similar conditions by reaction of **2** with Bu^t_2PLi or $Bu^t_2PLi\cdot BH_3$ were unsuccessful.

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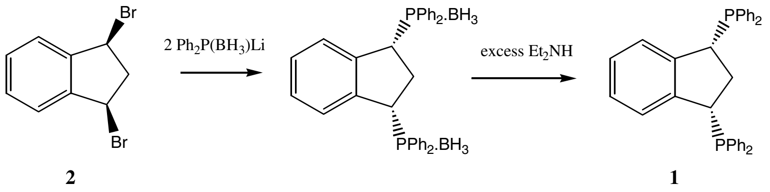
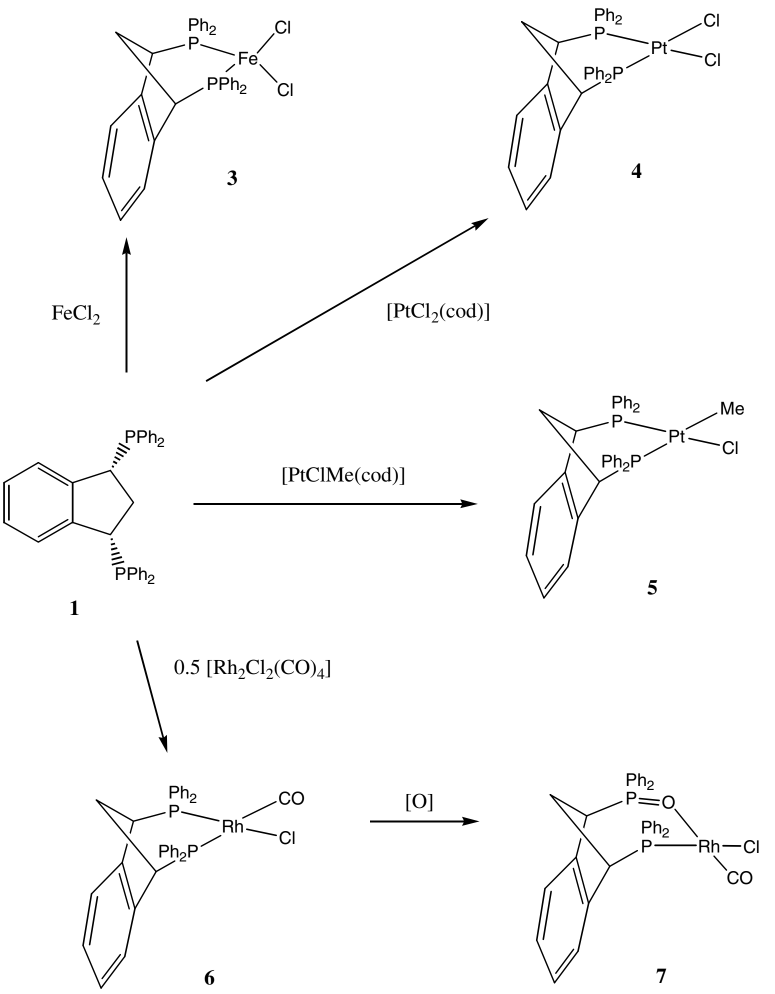


Scheme 1. Synthesis of anphos.



Scheme 2. Anphos coordination chemistry.

A summary of the coordination chemistry investigated with anphos is shown in Scheme 2.

Treatment of anhydrous $FeCl_2$ with anphos in CH_2Cl_2 gave **3** which has been fully characterised (see Section 4). Crystals of the same product **3** were obtained when anphos reacted with $[Fe(CO)_3(bda)]$ in $CDCl_3$ over several days. The crystal structure of **3** was determined and is shown in Fig. 1 (selected bond lengths and angles are given in Table 1).

It can be seen from the data that $[FeCl_2(anphos)]$ (**3**) adopts a strongly distorted tetrahedral structure, with bond angles for Cl(1)–Fe–Cl(2) and P(1)–Fe–P(2) of 118.22(3)° and 93.18(3)°, respectively. The bond angles in complexes of the type $[FeCl_2P_2]$ (where P_2 is a diphosphine or two monophosphines) span a range of values: $[FeCl_2(^iPr_2PCH_2CH_2P^iPr_2)]$ [7] for example, has a Cl–Fe–Cl bond angle of 118.0(4)° and a P–Fe–P value of 83.7(3)°, whereas for $[FeCl_2(^tBu_2PMe)_2]$ the corresponding angles are 110.97(3)° and 106.375(26)° [8]. Complexes of the type $[FeCl_2(PR_3)_2]$ and $[FeCl_2(diphos)]$ have been shown to insert diazoalkanes into the Fe–P bonds [9] and catalyse the reaction of propargyl sulphides with trimethylsilyldiazomethane to give homoallenylsilanes [10].

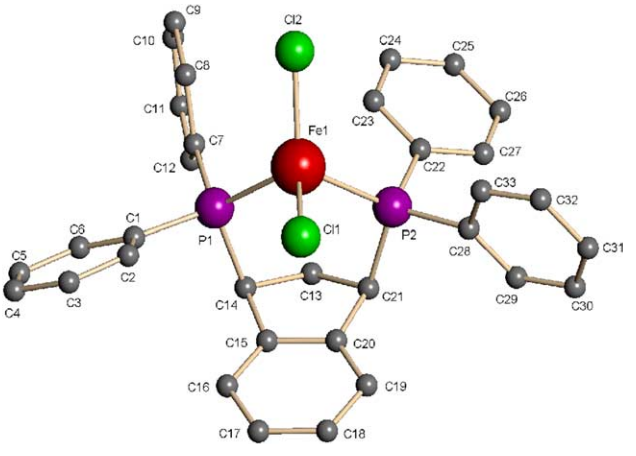


Fig. 1. The crystal structure of **3**.

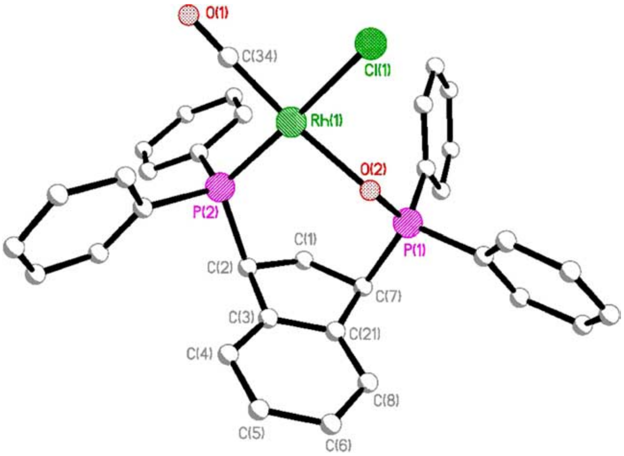


Fig. 2. The crystal structure of **7**.

Table 1
Selected bond lengths and angles for [FeCl$_2$(anphos)] (**3**)

Bond lengths (Å)		Angles (°)	
Fe(1)–Cl(2)	2.2292(7)	Cl(2)–Fe(1)–Cl(1)	118.22(3)
Fe(1)–Cl(1)	2.2297(7)	Cl(2)–Fe(1)–P(2)	114.80(3)
Fe(1)–P(2)	2.4390(8)	Cl(1)–Fe(1)–P(2)	103.77(3)
Fe(1)–P(1)	2.4627(8)	Cl(2)–Fe(1)–P(1)	113.15(3)
C(13)–C(21)	1.546(4)	Cl(1)–Fe(1)–P(1)	110.64(3)
C(13)–C(14)	1.548(4)	P(2)–Fe(1)–P(1)	93.18(3)
C(14)–C(15)	1.512(3)	C(21)–C(13)–C(14)	105.7(2)
C(15)–C(16)	1.391(3)	C(15)–C(14)–C(13)	102.3(2)
C(15)–C(20)	1.392(4)	C(16)–C(15)–C(20)	120.6(2)
C(16)–C(17)	1.390(4)	C(16)–C(15)–C(14)	128.6(2)
C(17)–C(18)	1.373(4)	C(20)–C(15)–C(14)	110.9(2)
C(18)–C(19)	1.383(4)	C(19)–C(20)–C(15)	120.4(2)
C(19)–C(20)	1.385(3)	C(19)–C(20)–C(21)	128.9(3)
C(20)–C(21)	1.518(3)	C(15)–C(20)–C(21)	110.7(2)
		C(20)–C(21)–C(13)	102.4(2)

Addition of 1 equivalent of anphos to [PtCl$_2$(cod)] gave **4** as a white solid. The ^{31}P{^{1}H} NMR spectrum of the product is a singlet at δ −3.5 ppm with $^1J_{PtP} = 3484$ Hz, consistent with the *cis*-PtCl$_2$ geometry. Similarly, addition of 1 equivalent of anphos to [PtClMe(cod)] gave **5**. The ^{31}P{^{1}H} NMR spectrum showed two doublets at δ 4.6 and 12.7 ppm ($^1J_{PP}$ 20 Hz) with $^1J_{PtP}$ 1704 and 4344 Hz, respectively and therefore the signal at 4.6 is assigned to the P *trans* to methyl.

The product of the reaction of anphos with [RhCl(CO)$_2$]$_2$ was assigned the chelate structure **6** on the basis of ^{31}P NMR, IR and mass spectrometric data (see Section 4). The ^{31}P{^{1}H} NMR spectrum showed two doublets of doublets at δ 7.7 ($J_{Rh-P} = 120.0$ Hz) and 32.8 ppm ($^1J_{Rh-P} = 168.4$ Hz) with $^2J_{P-P}$ of 47.0 Hz. The ^{31}P NMR and IR (ν_{CO} 2003 cm^{-1}) data for **6** are similar to those for the five-membered chelate [RhCl(CO)(dppe)] [11] and differ from the bridged binuclear complexes *trans*-[Rh$_2$Cl$_2$(CO)$_2$(μ-dppp)$_2$] and *trans*-[Rh$_2$Cl$_2$(CO)$_2$(μ-dppb)$_2$] formed by diphosphines with a larger bite angle. The constraints of the indan backbone decisively favour the formation of the chelate **6** rather than a binuclear [Rh$_2$Cl$_2$(CO)$_2$(μ-diphos)$_2$] species.

It was found that CH$_2$Cl$_2$ solutions of **6** slowly deposited red-brown crystals suitable for X-ray crystallography. The crystal structure revealed that the coordinated anphos had been mono-oxidised to give a P,O-chelate **7** (Fig. 2).

From Table 2 the degree of distortion in the interior angles of the five-membered indan ring in **7** can be clearly seen, the angles ranging from 103.6(8)° to 111.3(9)° (102.3(2)° to 110.9(2)° for **3**). The remaining bond lengths and angles are broadly as expected. Coordination at rhodium is square planar with the phosphine oxide function of the oxidised anphos ligand *trans* to carbonyl and the remaining phosphine *trans* to the chloride ligand, in accord with the concept of anti-symbiosis. The ^{31}P NMR spectrum of **7** (CDCl$_3$) gives the following data; P(1) 54.4 ppm, $^2J_{Rh-P} = 6.0$ Hz, $^3J_{P-P} = 2.8$ Hz; P(2) 53.1 ppm, $^1J_{Rh-P} = 179.1$ Hz, $^3J_{P-P} = 2.8$ Hz.

The anphos ligand (**1**) was tested for the rhodium-catalysed hydroformylation of 1-hexene. Under the

Table 2
Selected bond lengths and angles for [RhCl(CO)(anphos-O)] (**7**)

Bond lengths (Å)		Angles (°)	
Rh(1)–C(34)	1.800(13)	O(1)–C(34)–Rh(1)	178.5(11)
Rh(1)–O(2)	2.076(6)	P(1)–O(2)–Rh(1)	136.1(4)
Rh(1)–P(2)	2.232(3)	O(2)–Rh(1)–P(2)	96.50(19)
Rh(1)–Cl(1)	2.387(3)	C(34)–Rh(1)–Cl(1)	89.8(4)
P(1)–O(2)	1.517(7)	C(1)–C(7)–P(1)	111.4 (7)
C(1)–C(7)	1.563(13)	C(3)–C(2)–C(1)	105.4(8)
C(2)–C(3)	1.518(13)	C(21)–C(3)–C(2)	109.4(9)
C(3)–C(4)	1.369(13)	C(2)–C(1)–C(7)	104.8(8)
C(3)–C(21)	1.412(14)	C(3)–C(21)–C(7)	111.3(9)
C(8)–C(21)	1.370(14)	C(1)–C(7)–C(21)	103.6(8)
C(34)–O(1)	1.140(12)		

reaction conditions employed (7.5 bar H_2/CO, 36 °C, 16 h) the conversion to aldehydes was 91% with a *n*:iso ratio of 1.5:1. These results are similar to those for dppx which we tested under the same conditions and observed a conversion of 73% and a *n*:iso ratio of 2.4:1. Anphos and dppx react with the precatalyst $[Rh(acac)(CO)_2]$ in C_6D_6 to give species which have similar ^{31}P NMR parameters ($^1J_{Rh-P} = 186$ and 189 Hz, respectively).

3. Conclusion

The constrained bite angle in anphos has the effect of making its coordination chemistry resemble dppe in its propensity to chelate. In terms of hydroformylation catalytic activity and selectivity however, anphos performs similarly to its unconstrained analogue dppx. These results further confirm the complexity of the factors at work in hydroformylation catalysis.

4. Experimental

4.1. General methods

All operations were carried out under a N_2 atmosphere using standard Schlenk line techniques. Dichloromethane, diethyl ether, hexane, and tetrahydrofuran were dried and purged with nitrogen using the Grubbs system [12]. Anhydrous *n*-pentane was purchased from Aldrich. Carbon tetrachloride and acetone were dried over 4 Å molecular sieves. Diethylamine was distilled from potassium hydroxide. Indan, *N*-bromosuccinimide and $[Rh_2Cl_2(CO)_2]$ were purchased from Aldrich. $[PtCl_2$(1,5-cyclooctadiene)] [13] and [PtCl(Me)(1,5-cyclooctadiene)] [14] were prepared according to the literature procedures. $[Fe(CO)_3$(benzylideneacetone)] was a gift from BP chemicals but it can be prepared by the method of Brookhart and Nelson [15]. Anhydrous $FeCl_2$ was supplied by Fluka.

EI and FAB mass spectra were recorded on a Micromass MD800 and an Autospectrometer. Infrared spectra were recorded on a Perkin–Elmer Spectrum 1 Spectrometer as KBr disks or as powder samples using a universal ATR sampling accessory. The NMR spectra were recorded on JEOL ecp300 and 400 spectrometers (δ relative to $SiMe_4$ for ^1H and ^{13}C spectra and 85% H_3PO_4 for ^{31}P spectra) at 23 °C in $CDCl_3$ unless otherwise stated. $CDCl_3$, CD_2Cl_2 and C_6D_6 were purged with nitrogen and dried over 4 Å molecular sieves.

4.2. Synthesis of *cis*-1,3-dibromoindan (**2**)

Indan (5.0 g, 42.3 mmol) was dissolved in CCl_4 (70 cm^3) and *N*-bromosuccinimide (18.8 g, 105.8 mmol) was added as a solid. The reaction mixture was heated at reflux for 2 h resulting in a suspension in a pale peach solution. The solution was filtered, and the solid washed with carbon tetrachloride. The filtrates were combined and the solvent was removed in vacuo to give a pale orange oil. Hexane (5 cm^3) was added to this and the mixture was cooled to −5 °C, producing a pale yellow crystalline solid. This solid was recrystallised from acetone to give pale yellow crystals. Yield = 1.7 g, 6.0 mmol, 14%. ^1H NMR data: 2.94 (dt, 1H, $J = 16.2$, 2.8 Hz), 3.27 (dt, 1H, $J = 16.2$, 6.8 Hz), 5.50 (dd, 2H, $J = 6.8$, 2.8 Hz), 7.3–7.5 (m, 4H) ppm.

4.3. Synthesis of 1,3-bis(diphenylphosphino)indan (anphos) (**1**)

Diphenylphosphine (1.25 cm^3, 7.20 mmol) in THF (15 cm^3) was cooled to 0 °C and then $BH_3 \cdot THF$ (7.20 cm^3, 7.20 mmol, 1M solution in THF) was added slowly (over 10 min) to the stirred solution. The resulting solution of $PPh_2H \cdot BH_3$ was cooled to −78 °C and then ^nBuLi (4.50 cm^3, 7.20 mmol, 1.6 M solution in hexanes) was added dropwise over 10 min. The solution was then allowed to warm to room temperature. This was then added by syringe to a stirred, cooled (0 °C) solution of **2** (1.00 g, 3.60 mmol) in THF (10 cm^3). The ice-bath was removed and the reaction stirred for 3 h. An excess of diethylamine (30 cm^3) was added and the reaction mixture was stirred for 48 h. The volatiles were removed to give a white powder, which was extracted into pentane (40 cm^3). Filtration from the salt by-products followed by removal of the pentane in vacuo gave a white powder that was washed with 2×40 cm^3 of methanol. The product was dried at 50 °C for 5 h. Yield = 0.40 g, 22.8%. NMR (CD_2Cl_2): δ_H 7.16–7.59 (m, 20H, ArH), 6.82 (m, 2H, ArH), 6.37 (m, 2H, ArH), 3.96 (m, 2H, CH), 2.38 (m, 1H, CH_2), 1.99 (m, 1H, CH_2) ppm; δ_C 34.6 (t, CH_2), 41.8 (m, CH), 125.0-134.3 (m, CH, aromatic C) 137.7 (m, quaternary C), 144.3 (m, quaternary C) ppm; δ_P −7.4 (s) ppm; IR (solid, cm^{-1}): ν(aryl-H) 3068 (w), 746 (s), 737 (s), ν(C=C) 1598 (m), ν(C–H) 1478 (m), ν(aryl-P) 1431 (m); EI mass spectrum: *m*/*z* 486 (M^+), 502 ($M^+ + O$), 518 ($M^+ + 2O$). Elemental composition found by accurate mass measurement was within 1.6 ppm.

4.4. Synthesis of $[FeCl_2$(anphos)] (**3**)

4.4.1. From anhydrous $FeCl_2$

Anphos (1.50 g, 3.10 mmol) and anhydrous $FeCl_2$ (0.39 g, 3.10 mmol) were combined in a flask. The addition of dichloromethane (20 cm^3) yielded a suspension, which was stirred for 1 week. No solution ^{31}P NMR signal was observed. The solution was filtered off and cooled to 0 °C. The resultant white precipitate was filtered, washed with 5 cm^3 of dichloromethane and dried in vacuo at 45 °C for 4 h. Yield = 1.54 g, 81.0%. IR (solid, cm^{-1}): ν(aryl-H) 3051 (w), 749 (s), 743 (s), ν(C=C) 1590 (w), ν(C–H) 1484 (m), ν(aryl-P) 1435 (s). FAB mass spectrum: *m/z* 612 (M^+). Elemental Anal. $C_{33}H_{28}P_2FeCl_2$ requires: C, 64.63; H, 4.60. Found C, 64.45; H, 4.80%. Recrystallisation from $CDCl_3$ yielded green crystals (0.68 g, 35.8%).

4.4.2. From [Fe(CO)$_3$(benzylideneacetone)]

Anphos (0.05 g, 0.10 mmol) was dissolved in $CDCl_3$ (0.7 cm^3), solid [Fe(bda)$(CO)_3$] (0.03 g, 0.10 mmol) was added and the mixture transferred to an NMR tube. No immediate colour change was apparent and only uncoordinated anphos was detected by ^{31}P NMR. The tube was wrapped in aluminium foil and after 16 h the solution had become yellow and no anphos signal was detected by ^{31}P NMR. Over a period of weeks green crystals deposited which were suitable for X-ray cystallography.

*4.5. Synthesis of [PtCl$_2$(anphos)] (**4**)*

To a solution of [$PtCl_2$(1,5-cyclooctadiene)] (0.10 g, 0.26 mmol) in dichloromethane (20 cm^3) was added anphos (0.14 g, 0.28 mmol). The resulting solution was stirred for 1 h at room temperature, followed by removal of the solvent in vacuo to afford a white solid. This was washed with 2×10 cm^3 portions of diethyl ether, filtered and dried in vacuo. Yield = 0.07 g, 36.3%. ^{31}P NMR: −3.5 (s, $J_{Pt–P} = 3484$ Hz) ppm. FAB mass spectrum: *m/z* 717 ($M^+ - Cl$), 680 ($M^+ - 2Cl$).

*4.6. Synthesis of [PtClMe(anphos)] (**5**)*

A dichloromethane (20 cm^3) solution of anphos (0.14 g, 0.28 mmol) was added to [PtClMe(1,5-cyclooctadiene)] (0.10 g, 0.28 mmol). The pale yellow solution was stirred for 1 h, during which time a white precipitate appeared. The solid was filtered, washed with 2×5 cm^3 portions of cold chloroform and filtered again. The white product was dried in vacuo. Yield = 0.15 g, 73.6%. ^{31}P NMR: 4.6 (d, $J_{P–P} = 20$ Hz, $J_{Pt–P} = 1704$ Hz), 12.7 (d, $J_{P–P} = 20$ Hz, $J_{Pt–P} = 4344$ Hz) ppm. FAB mass spectrum: *m/z* 717 ($M^+ - CH_3$), 696 ($M^+ - Cl$).

*4.7. Synthesis of cis-[RhCl(CO)(anphos)] (**6**)*

To a solution of anphos (0.14 g, 0.29 mmol) in dichloromethane (20 cm^3) was added $[RhCl(CO)_2]_2$(0.06 g, 0.14 mmol). Evolution of CO ensued and the resultant yellow/brown solution was stirred for 1 h at room temperature. Removal of the solvent afforded a deep orange solid that was washed with 2×10 cm^3 portions of diethyl ether and dried in vacuo. Yield = 0.12 g, 66.2%. ^{31}P NMR: 7.7 (dd, $J_{Rh–P} = 120.0$ Hz, $J_{P–P} = 47.0$ Hz), 32.8 (dd, $J_{Rh–P} = 168.4$ Hz, $J_{P–P} = 47.0$ Hz) ppm. IR (KBr, cm^{-1}) 2003 s ν(CO). FAB mass spectrum: *m/z* 624 ($M^+ - CO$), 617 ($M^+ - Cl$), 589 ($M^+ - CO$, Cl).

4.8. Catalytic Hydroformylation of 1-hexene

The diphosphine (0.019 mmol) was added to a solution of [Rh(acac)$(CO)_2$] (5.0 mg, 0.019 mmol) in anhydrous C_6D_6 (3 cm^3) and the emerald green solution

Table 3
Important crystallographic parameters for **3** and **7**

Parameters	[$FeCl_2$(anphos)] (**3**)	[RhCl(CO)anphosO] (**7**)
Empirical formula	$C_{33}H_{28}Cl_2FeP_2$	$C_{37}H_{31}ClO_2P_2Rh$
Temperature (K)	173(2)	173(2)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
Unit cell dimensions		
a (Å)	9.1548(12)	10.3901(17)
b (Å)	16.818(2)	14.305(2)
c (Å)	19.420(2)	22.889(4)
α (°)	90	90
β (°)	100.485(2)	94.335(3)
γ (°)	90	90
Z	4	4
Absorption coefficient (mm^{-1})	0.825	0.707
Crystal size (mm^3)	0.10×0.20×0.40	0.15×0.80×0.50
Reflections collected	18809	13657
Independent reflections	6721 [*R*(int) = 0.0467]	4137 [*R*(int) = 0.1478]
Absorption correction	Multiscan	Multiscan
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	0.944	1.028
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0397$, $wR_2 = 0.0849$	$R_1 = 0.0631$, $wR_2 = 0.1378$
R indices (all data)	$R_1 = 0.0815$, $wR_2 = 0.0970$	$R_1 = 0.1382$, $wR_2 = 0.1656$

became orange. The solution was transferred under nitrogen to the autoclave which was then evacuated and refilled three times with a 1:1 mixture of H_2 and CO. The reaction mixture was stirred for 1 h at 36 °C and 7.5 bar of syngas. 1-Hexene (1.2 cm^3, 9.6 mmol) was injected into the autoclave and the reaction monitored by syngas uptake. After 16 h the reaction was stopped and the autoclave purged with nitrogen. The ratio of linear:branched products was calculated by integration of the ^{1}H NMR signals (in C_6D_6) for the aldehyde protons at δ 9.22 (*n*) and 9.15 (*i*).

4.9. Crystal structure analysis of **3** *and* **7**

X ray diffraction experiments on **3** as a benzene solvate and **7** were carried out at −100 °C on a Bruker SMART diffractometer using Mo Kα X-radiation, $\alpha = 0.71073$ Å. Crystal and refinement data are given in Table 3: absorption corrections were based on equivalent reflections and structures refined against all F_o^2 data with hydrogen atoms riding in calculated positions. In the case of **3** intensity data were very weak. A benzene solvate molecule was found in the electron density difference map, disordered about a special position. Attempts to model this disorder produced unsatisfactory results and an improved model was obtained by applying a diffuse solvent correction to the data using the Squeeze routine in the Platon suite of software [16]. Furthermore the value of $(2\,c\cos\alpha)/a$ is almost exactly $-1/3\sim$ indicating that twofold rotational twinning about the *a* axis is possible for the lattice of this crystal. A refinement was attempted on the data with an appropriate twinning matrix (1 0 0 0 −1 0 −0.3333 0 −1). This did not improve the final residuals and gave a scale factor for the second twin component of almost zero. These results would appear to indicate that a single crystal is present with no contribution from a second twin component.

5. Supplementary material

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 241326 for **3** and 241327 for **7**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(1223)336033 or e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk/conts/retrieving.html.

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