Supporting Information

Synthesis and Characterization of a New Binucleating Tetraphosphine Ligand Based on 1,2-Phenylene Chelates and the Structures of Dinickel Tetrachloride Complexes of the Ligand.

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General Considerations: All synthetic work was done under inert atmosphere conditions using N_2, Schlenk line, or glove box manipulations. Organic solvents and chemicals were from Sigma-Aldrich; Et_2Zn was from Strem Chemicals. (H)(Ph)PCH_2P(Ph)(H) and Et_2PCl were prepared as described previously.[1] Solvents were obtained dry and under N_2 or degassed with N_2, no further solvent purification was done. NMR data was collected on a Bruker DPX 250 MHz spectrometer with a computer tunable quad-probe (^{1}H, ^{13}C, ^{19}F, ^{31}P) or an Avance III 400 MHz spectrometer equipped with a double resonance probe with two computer tunable coils (inner coil tunable from ^{107}Ag to ^{31}P; outer coil tunable from ^{19}F to ^{1}H). NMR data was analyzed and simulated using MestReNova (v8.1.1). IR data was collected on a Bruker Tensor 27 system using a Pike Diamond Miracle ATR cell and a room temperature DTGS detector and analyzed via OPUS v7.2. High resolution MS data was obtained from an Agilent 6210 LC electrospray system. GC-MS data was obtained from an Agilent 6890N GC and 5975B MS system using a J&W HP-5MS 30 m × 0.250 mm capillary column.

Et,ph-P4-Ph Synthesis, 2M & 2R

Synthesis of (Cl)(Ph)PCH_2P(Ph)(Cl), 4.

A Schlenk flask was charged with 6.0 g (25.8 mmoles) of (H)(Ph)PCH_2P(Ph)(H), **3**, 12.23 g (51.7 mmoles) of C_2Cl_6 and 50 mL of diethyl ether. The flask was attached to a reflux condenser and refluxed for 24 hours. Completion of the reaction was indicated by the solution becoming light pink in color and some white solid precipitating out of solution. The flask was then removed from the condenser and the mixture concentrated under vacuum. As it was concentrated some additional white solid precipitated. The white solid is unreacted C_2Cl_6. The concentrated solution was then filtered over a plug of Celite using a coarse frit funnel. The remaining solvent was removed under vacuum leaving behind a light pink, slightly viscous oil. Yields are typically between 60-67% and purity is 98% or higher based on ^{31}P NMR. The ^{31}P NMR can show only a single resonance at 81.7 ppm despite the presence of a 1:1 mixture of diastereomers. Whether a single or two resonances are observed depends on solvent and concentration. The two diastereomer resonances can be easily seen in C_6D_6 under more dilute conditions.

^{31}P{^{1}H} NMR (161.976 MHz, C_6D_6): 81.3 (s) and 81.0 (s)

^{1}H NMR (250.130 MHz, C_6D_6): δ 7.5 (m, 4H), 7.2 (m, 6H), and 2.9 (br s, 2H).

^{13}C{^{1}H} NMR (62.895 MHz, $CDCl_3$): δ 134.4 (m), 127.7, 125.5 (m), 73.9 (m), 42.2 (t, J = 42.3 Hz).

Synthesis of 1-(Et_2P)-2-iodobenzene, 6.

The following procedure was conducted in aluminum foil-wrapped glassware to exclude light. A Schlenk flask was charged with 25.00 g (75.8 mmoles) of 1,2-diiodobenzene and 80 mL of THF and cooled in an ice bath. A second Schlenk flask was charged with 26.1 mL (75.8 mmoles) of a 2.9 M THF solution of *i*PrMgBr. The room temperature Grignard solution was added dropwise to the 0°C flask with the 1,2-diiodobenzene via cannula. It is important that the Grignard flask is kept at room temperature to ensure that the Grignard does not precipitate out of

[1] Laneman, S. A.; Fronczek, F. R.; Stanley, G. G. *Inorg. Chem.* **1989**, *28*, 1872-1878.

solution. The cooled reaction flask was stirred at 0ºC for ~18 hours (overnight) using a large wide-mouth dewar filled with ice. The reaction flask is then cooled to –25°C and a solution of Et_2PCl (9.72 g, 78 mmoles) dissolved in 90 mL of THF was added dropwise via cannula. The yellow solution was then allowed to warm to room temperature and stirred overnight. The next day 80 mL of water was added and the organic layer was separated. The aqueous layer was extracted with three 50 mL portions of diethyl ether. The extracts and organic layer were combined and dried over Na_2SO_4. Solvents were removed under vacuum leaving a yellow oil. The product was isolated via short-path distillation to yield 16.0 g of an air- and *light-sensitive* colorless liquid (72% yield). Yields are usually between 70-75% and purity is usually greater than 99% via ^{31}P NMR.

$^{31}P\{^1H\}$ NMR (101.254 MHz, C_6D_6): δ 0.3 (s).

^1H NMR (250.130 MHz, C_6D_6): δ 7.7 (br m, 1H), 7.2 (sharp m, *J* = 7.3 Hz, 2H), 6.8 (sharp m, *J* = 7.3 Hz, 1H), 1.5 (m, 4H), and 0.9 (m, *J* = 7.3 Hz, *J*P,H = 7.7 Hz, 6H).

$^{13}C\{^1H\}$ NMR (62.895 MHz, C_6D_6): δ 142.3 (d, *J* = 15.3 Hz), 139.5 (s), 139.4 (d, *J* = 15.3 Hz), 108.5 (d, *J* = 40.3Hz), 77.4 (d, *J* = 30.7 Hz), 76.6 (s), 19.3, and 9.5 (d, *J* = 13.4 Hz).

Synthesis of *racemic,meso*-et,ph-P4-Ph, 2R and 2M.

A Schlenk flask was charged with 12.00 g of 1-(Et_2P)-2-iodobenzene, **6** (41.1 mmoles) and 50 mL of THF and wrapped with Al foil to exclude light. The solution was cooled to 0ºC and treated with 14.17 mL (41.1 mmoles) of a 2.9 M THF solution of *i*PrMgBr via cannula. The solution was allowed to stir for 8 hours at 0ºC, then cooled to –25ºC using a dry ice/acetone bath and 6.18 g (20.5 mmoles) of (Cl)(Ph)PCH_2P(Ph)(Cl), **4**, dissolved in 20 mL of THF was added dropwise via cannula. The solution was then allowed to warm to room temperature and stirred overnight. The next day 30 mL of water were added and the organic layer was separated from the aqueous layer. The aqueous layer was extracted with three 30 mL portions of diethyl ether. The organic layer and extracts were combined and dried over Na_2SO_4. The solution was concentrated under vacuum and then passed through a short alumina column to remove some of the impurities present. At this point *meso*-et,ph-P4-Ph, **2M**, is the primary diastereomer present (95-98%). Short-path vacuum distillation gave unreacted 1-(Et_2P)-2-iodobenzene, **6**, as the only fraction leaving a mixture of **2M** and **2R** in the distillation flask. Distillation to remove unreacted **6** epimerizes **2M** to produce a 1:1 mixture of **2M** and **2R**.

200 mL of 1-butanol was then added to the remaining yellow paste and warmed with a heat gun. As it was warmed a small amount of white solid precipitated out of solution. The solution was filtered to remove this inorganic impurity (soluble only in water) and the 1-butanol was removed under vacuum leaving behind *racemic,meso*-et,ph-P4-Ph in greater than 96% purity. An alternative to the alumina column cleanup is dissolving the slightly yellow paste in ethanol and placing it in a –20ºC freezer. The P4 ligand gradually precipitates out of solution and adheres to the sides of the flask. The remaining ethanol solution is poured out of the flask leaving behind *racemic,meso*-et,ph-P4-ph in greater than 95% purity. This step is done after removing the inorganic impurity.

The ^{31}P NMR values listed below were determined from an AA'BB' spin simulation using MestReNova (version 8.1.1) and manually optimized to fit the experimental spectrum of each diastereomer. A & A' represent the external phosphines (P_{ext}), while B & B' are the internal methylene bridged phosphorus centers (P_{int}).

$^{31}P\{^{1}H\}$ NMR **2M** (161.976 MHz, CD_2Cl_2): δ P_{ext} = –27.68, P_{int} = –31.42; $J_{P_{ext}\text{-}P_{int}}$ = 146 Hz, $J_{P_{int}\text{-}P_{int}}$ = 111 Hz, $J_{P_{ext}\text{-}P_{int2}}$ = 5 Hz, $J_{P_{ext}\text{-}P_{ext}}$ = 1 Hz.

^{1}H NMR **2M** (400.130 MHz, CD_2Cl_2): δ 7.67 (ddt, *J* = 6.3, 3.1, 1.8 Hz, 2H), 7.59 (ddq, *J* = 8.7, 5.3, 1.9 Hz, 4H), 7.33 – 7.25 (m, 2H), 7.20 – 6.98 (m, 10H), 3.08 (dt, *J* = 13.2, 3.6 Hz, 1H), 2.80 (dt, *J* = 13.2, 4.2 Hz, 1H), 1.61 – 1.51 (m, 2H), 1.50 – 1.33 (m, 2H), 0.95 (dt, *J* = 14.9, 7.6 Hz, 3H), 0.77 (dt, *J* = 14.9, 7.6 Hz, 3H).

High resolution mass spectrometry **2M**: 561.2159 amu (calc, $M+H^+$); 561.2168 amu (exp, $M+H^+$)

$^{31}P\{^{1}H\}$ NMR **2R** (161.976 MHz, CD_2Cl_2): δ P_{ext} = –27.35, P_{int} = –30.68; $J_{P_{ext}\text{-}P_{int}}$ = 142 Hz, $J_{P_{int}\text{-}P_{int}}$ = 122 Hz, $J_{P_{ext}\text{-}P_{int2}}$ = 2 Hz, $J_{P_{ext}\text{-}P_{ext}}$ = 0 Hz

^{1}H NMR **2R** (400 MHz, Benzene-d_6): δ 7.73 (ddt, *J* = 8.1, 4.7, 1.4 Hz, 4H), 7.46 (dh, *J* = 6.5, 1.5 Hz, 2H), 7.25 (ddt, *J* = 7.1, 2.9, 1.5 Hz, 2H), 7.19 – 6.94 (m, 10H), 2.99 (t, $J_{P\text{-}H}$ = 3.4 Hz, 2H), 1.61 – 1.32 (m, 4H), 0.94 (dt, *J* = 14.9, 7.6 Hz, 3H), 0.80 (dt, *J* = 14.8, 7.6 Hz, 3H).

High resolution mass spectrometry **2R**: 561.2159 amu (calc, $M+H^+$); 561.2157 amu (exp, $M+H^+$)

Column Chromatographic Purification and Separation of crude 2M and 2R

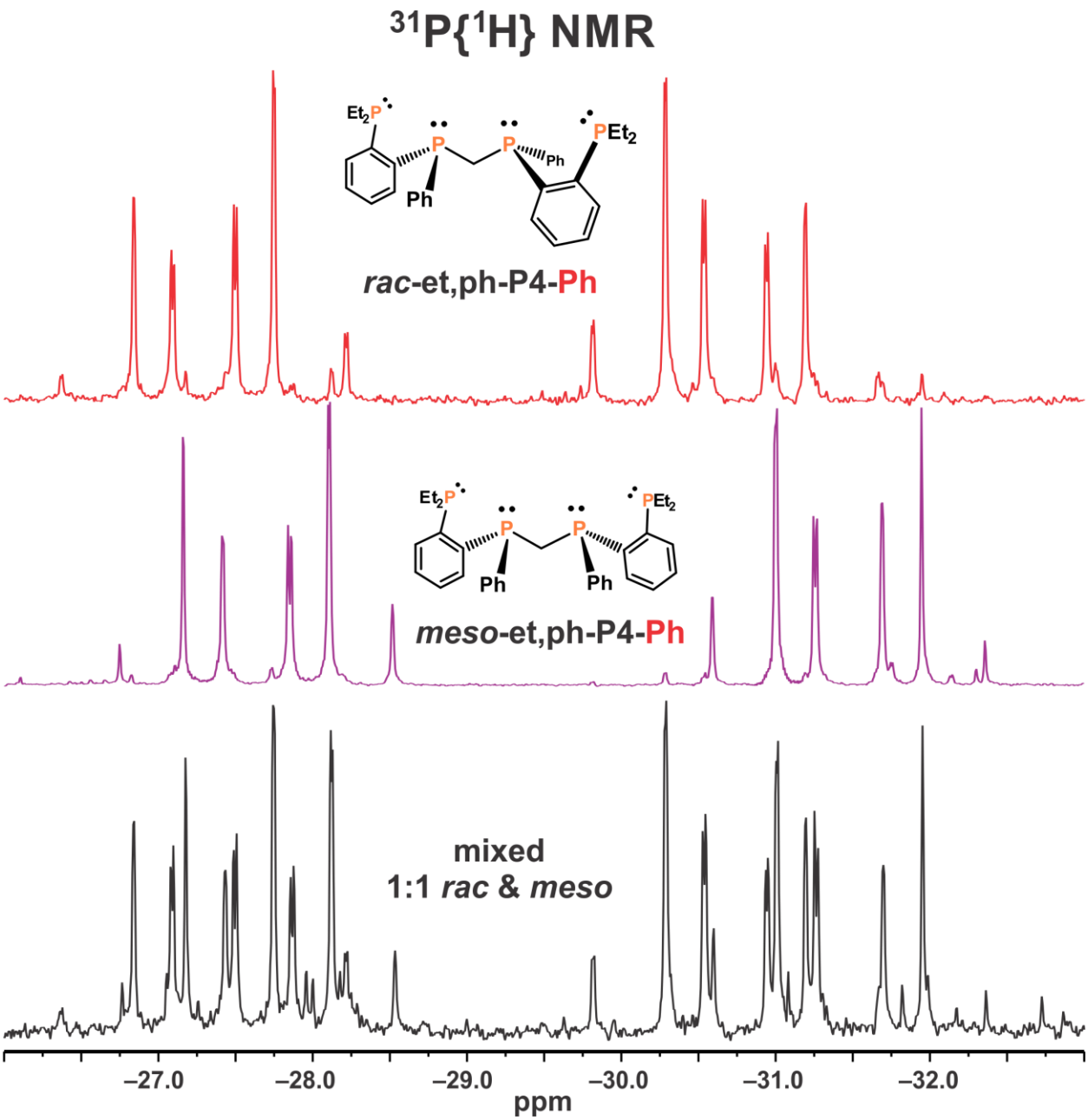
Initial Column Purification: Although the et,ph-P4-Ph ligand diastereomers react slowly with O_2, especially in the presence of moisture, all the chromatography described in this section can be done in air using dry (as received from Sigma-Aldrich) N_2-degassed solvents. The crude et,ph-P4-Ph product mixture (1:1 **2M**/**2R**, epimerized by heating) was initially purified by column chromatography on neutral alumina (4 × 12 cm column) with CH_2Cl_2 as the eluent. This step removes some of the unreacted small arm, 1-(Et_2P)-2-iodobenzene, **6**, and almost all of the other phosphorus impurities that likely contain the P3 intermediate species and any oxidized phosphines. Prior to this purification step, the crude product mixture contains about 76% et,ph-P4-Ph, **2M** & **2R**, 16% of 1-(Et_2P)-2-iodobenzene, **6**, and 8% of other phosphorus impurities based on ^{31}P NMR analysis. Up to 15 g of the crude product mixture can be partially purified at one time via this simple column procedure. The product mixture after this chromatographic purification and solvent removal consisted of a white pasty mass that contained a mixture of *rac* and *meso* diastereomers of et,ph-P4-Ph (**2M** & **2R**), 88%, and 1-(Et_2P)-2-iodobenzene, **8** (12% based on ^{31}P NMR).

Diastereomer Separation & Purification: To separate *rac* and *meso* diastereomers of et,ph-P4-Ph and the remaining 1-(Et_2P)-2-iodobenzene, **6**, 0.9 g. of the mixture from the previous chromatographic separation was subjected to a second chromatography on neutral alumina (Grade IV, 4 × 20 cm column) eluting with a 1:4 CH_2Cl_2/hexane solvent mixture. Optimum column flow rate was about one drop of solvent per second. A total of 38 × 10 ml fractions were collected and analyzed by TLC and ^{31}P NMR. Fractions 1-5 contained a total of 0.1 g of 1-(Et_2P)-2-iodobenzene, **6**, fractions 6-11 contained only solvent, fractions 12-21 contained **2M** (0.2 g) and a small amount of unidentified phosphine impurities, fractions 22-30 contained a mixture of **2M** and **2R** (0.3 g), and fractions 31-38 contained **2R** (0.3 g).

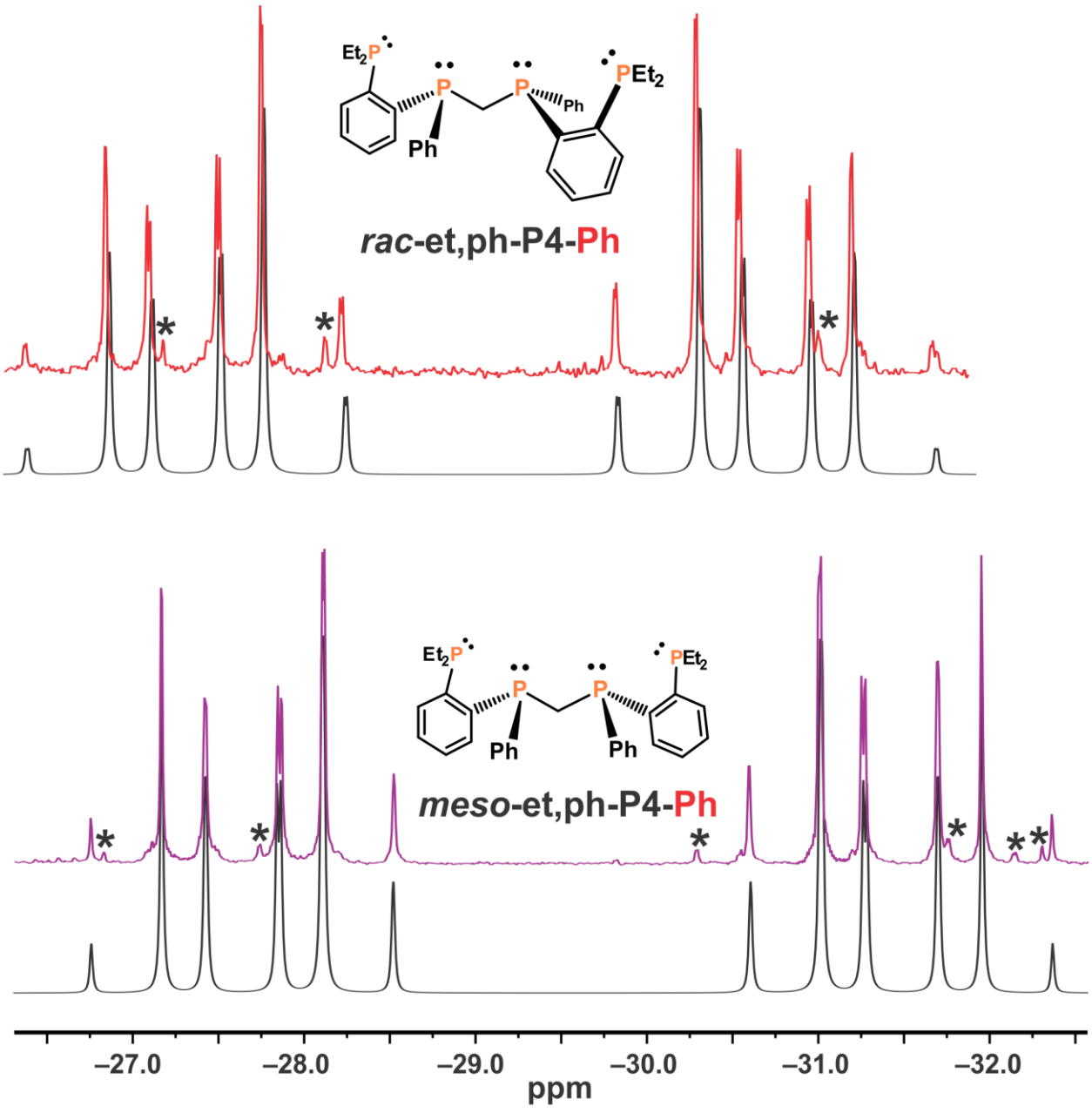
Important notes: **2M** and **2R** slowly react with CH_2Cl_2 to give unidentified products that we are still characterizing. So it is important to remove CH_2Cl_2 solvent from the et,ph-P4-Ph ligands as soon as possible. If one desires **2M**, do not heat and epimerize the P4 mixture from the initial

synthesis, which is 95-98% **2M** (aside from impurities). The second column chromatographic separation will then yield far more pure **2M**.

NMR (experimental and simulated) and IR spectra of 2M and 2R

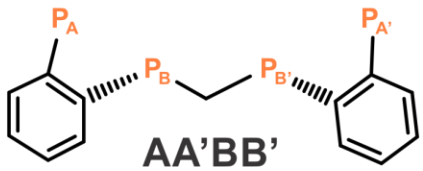


$^{31}P\{^{1}H\}$ NMR: Simulation & Experimental



* = impurities (mix of other P4 diastereomer & P3 intermediate species)

$^{31}P\{^{1}H\}$ NMR: Simulation & Experimental

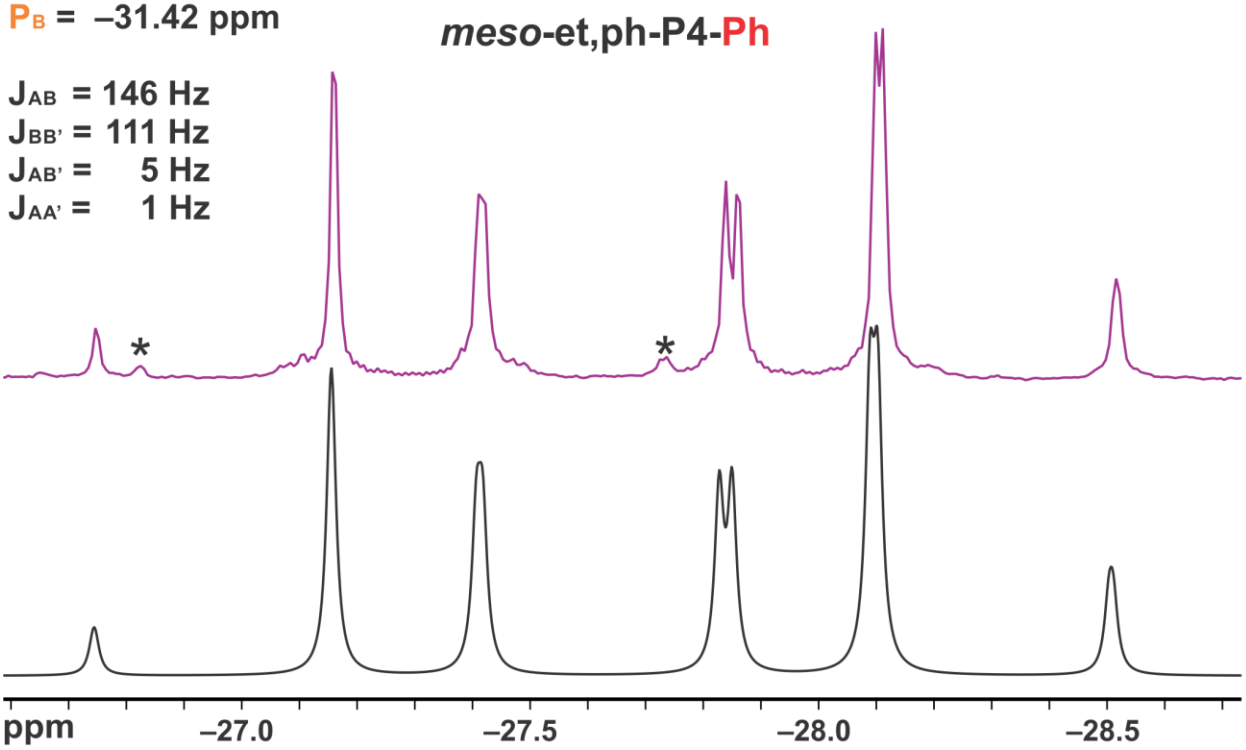


AA'BB'

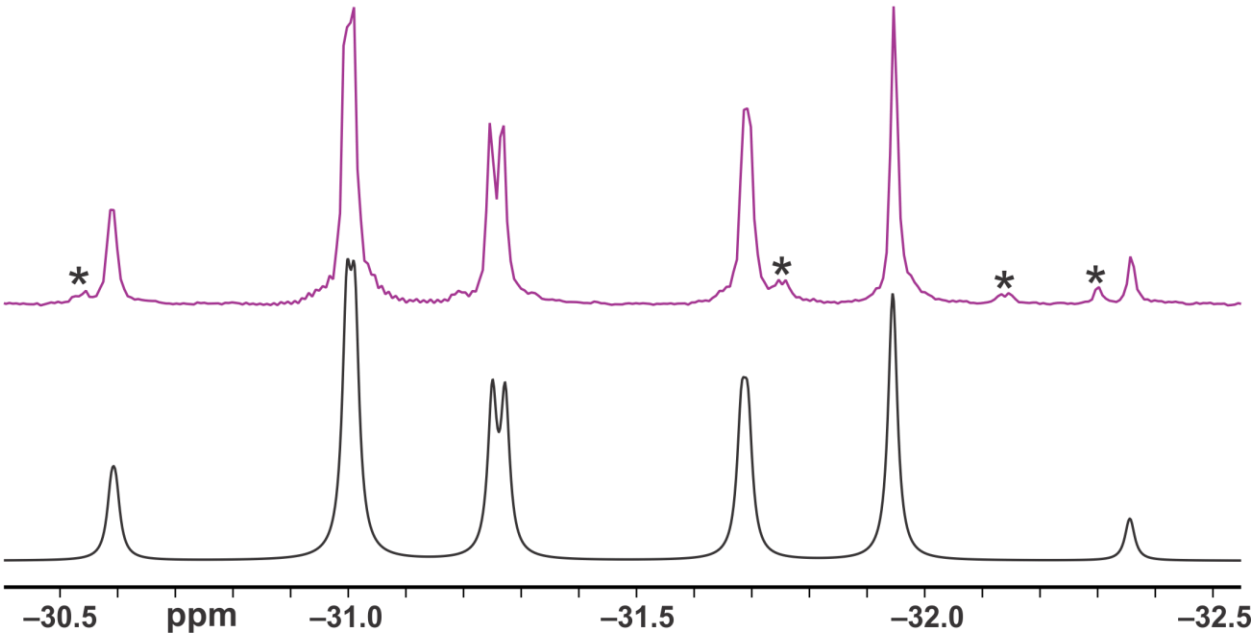
P_A = –27.68 ppm
P_B = –31.42 ppm

meso-et,ph-P4-Ph

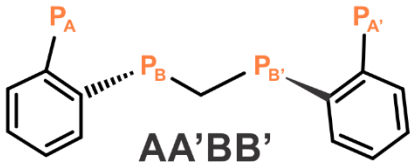
J_{AB} = 146 Hz
$J_{BB'}$ = 111 Hz
$J_{AB'}$ = 5 Hz
$J_{AA'}$ = 1 Hz



* = impurities (mix of other P4 diastereomer & P3 intermediate species)



$^{31}P\{^{1}H\}$ NMR: Simulation & Experimental

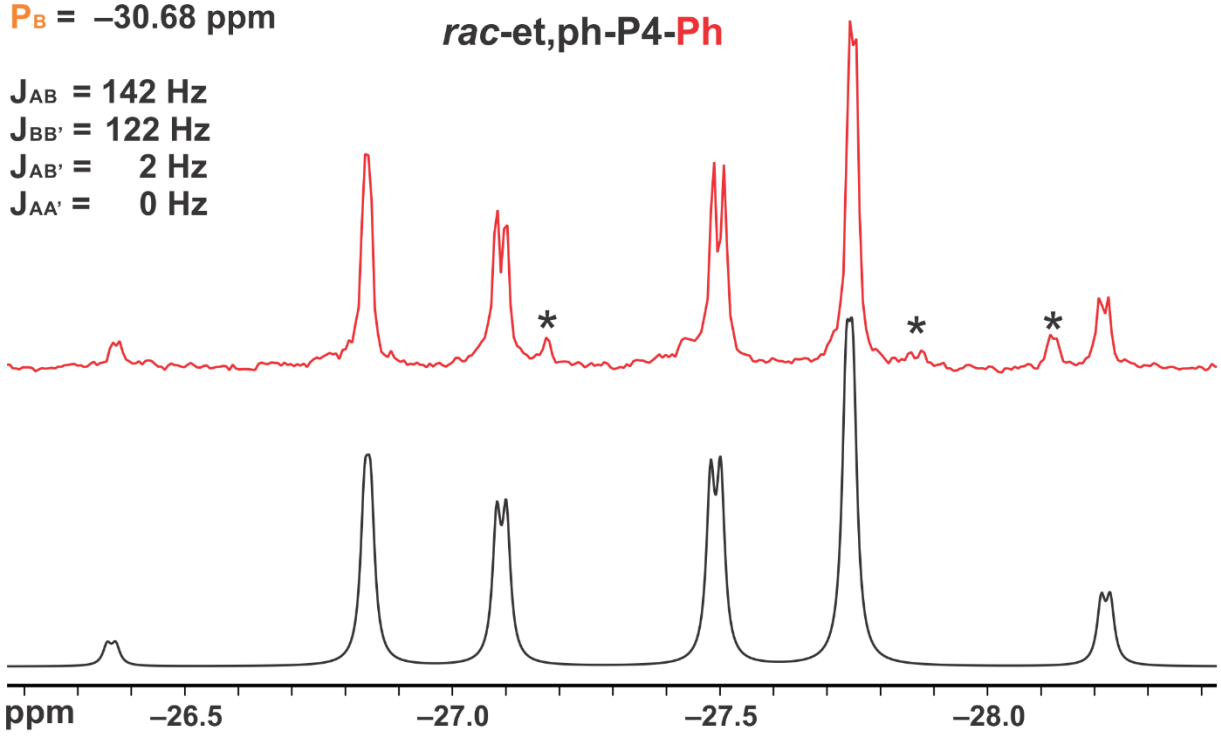


AA'BB'

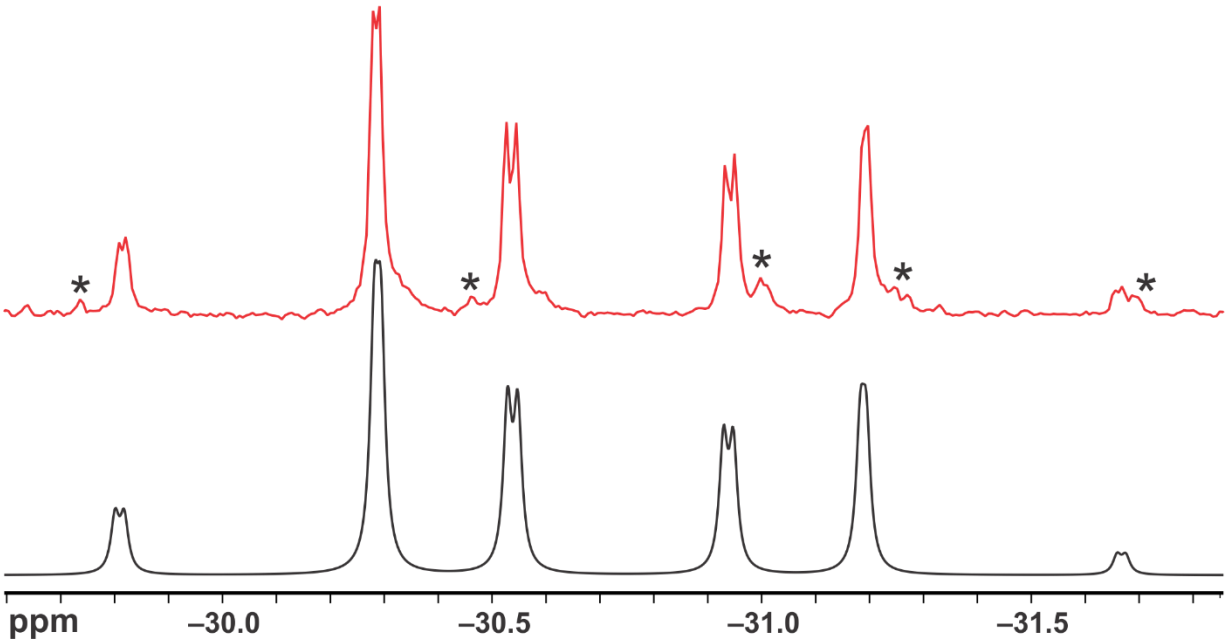
P_A = –27.35 ppm
P_B = –30.68 ppm

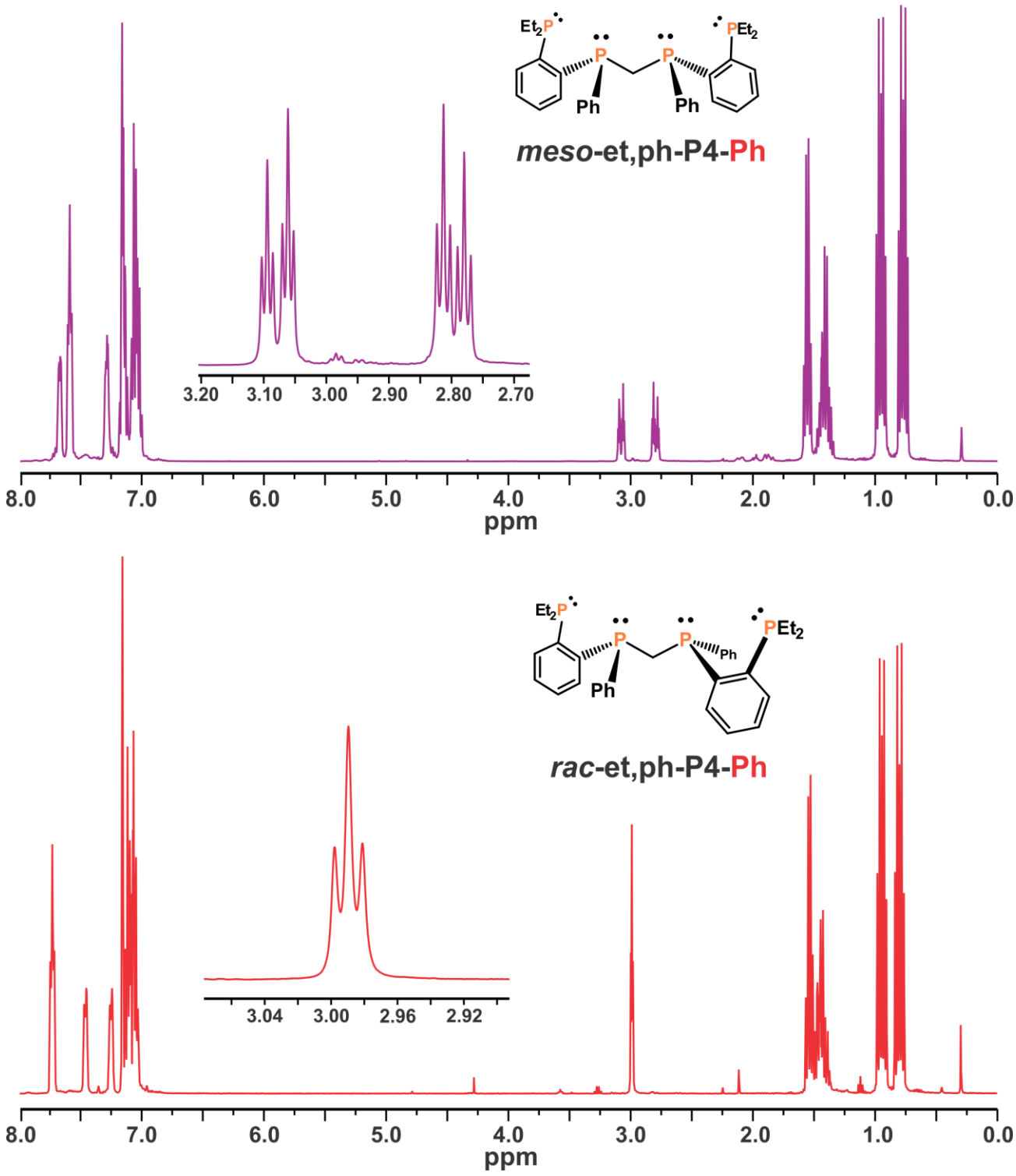
rac-et,ph-P4-Ph

J_{AB} = 142 Hz
$J_{BB'}$ = 122 Hz
$J_{AB'}$ = 2 Hz
$J_{AA'}$ = 0 Hz

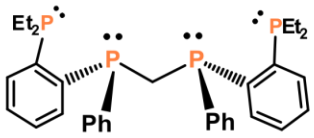


* = impurities (mix of other P4 diastereomer & P3 intermediate species)



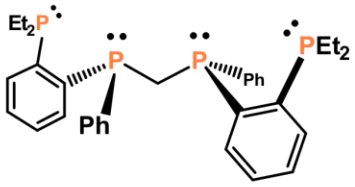

^{1}H NMR (400.130 MHz)
Et2P
PEt2
P
P
Ph
Ph
meso-et,ph-P4-Ph
3.20
3.10
3.00
2.90
2.80
2.70
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm
Et2P
PEt2
P
P
Ph
Ph
rac-et,ph-P4-Ph
3.04
3.00
2.96
2.92
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

^{1}H NMR (400.130 MHz)



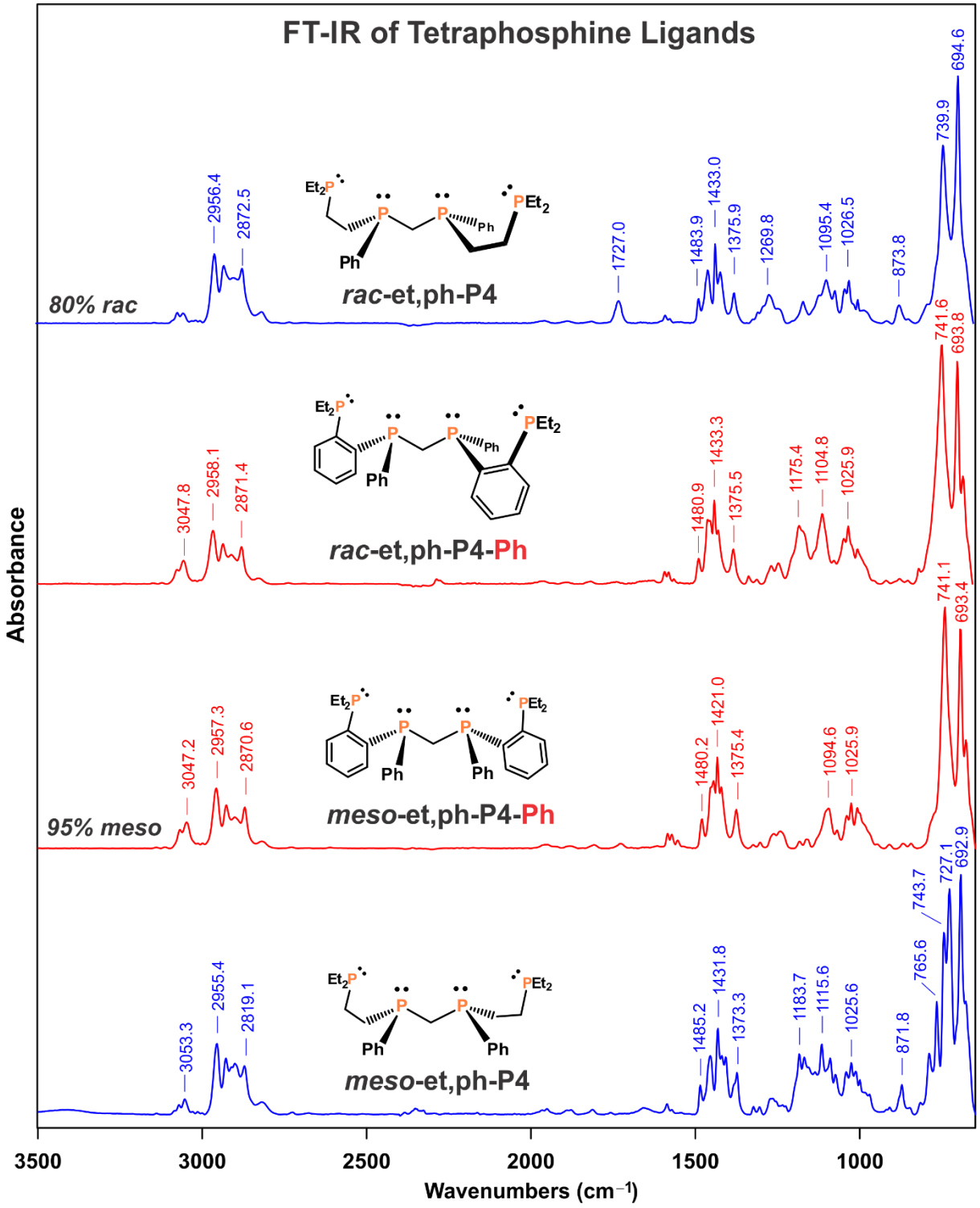
***meso*-et,ph-P4-Ph**

^{1}H NMR (400.130 MHz)



rac-et,ph-P4-**Ph**

IR Spectra of 1M, 2M, 2R, and 1R.



Synthesis of *rac*- and *meso*-Ni_2Cl_4(et,ph-P4-Ph), 7R and 7M

2.21 g (394 mmoles) of mixed *rac*,*meso*-et,ph-P4-Ph were dissolved in 100 mL of CH_2Cl_2 and placed into a Schlenk flask creating a clear, colorless solution. Another Schlenk flask was charged with 1.87 g (787 mmoles) $NiCl_2 \cdot 6H_2O$ and 100 mL of 1-butanol and heated with a heat gun to dissolve all of the Ni, creating a clear, green solution. The ligand solution was added dropwise via cannula to the rapidly stirring $NiCl_2$ solution. As the addition proceeded the solution became orange in color and slowly darkened. After the addition the solution was a very dark red color. The solution was allowed to stir overnight during which an orange powder precipitated out of solution. The powder was collected via filtration, washed with 1-butanol and dried in air. NMR analysis revealed this to be pure Ni_2Cl_4(*meso*-et,ph-P4), **7M**.

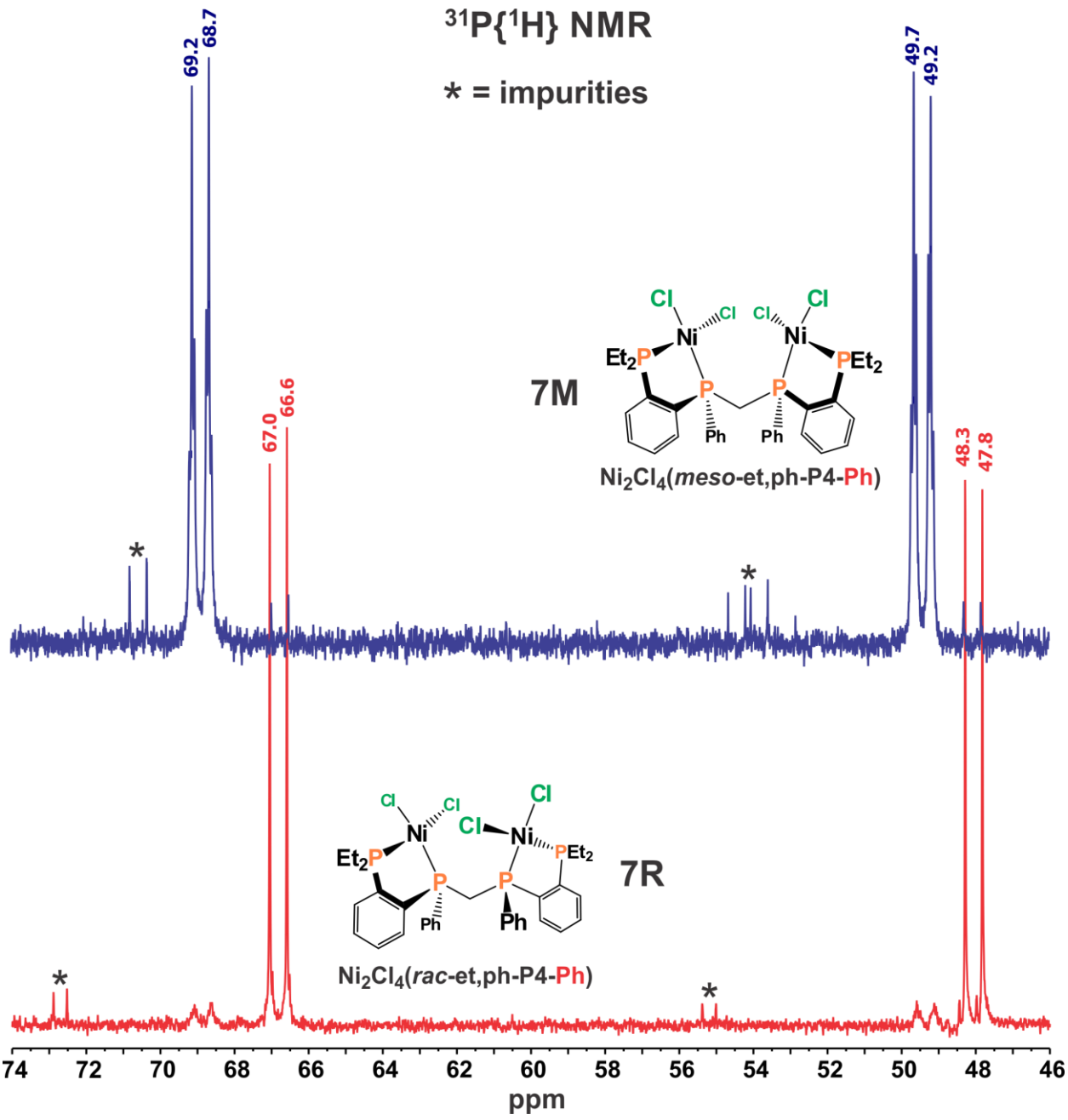
The remaining 1-butanol/dichloromethane solution was evaporated to dryness leaving behind a red/black residue. The residue was dissolved in CH_2Cl_2 and then filtered to remove any unreacted $NiCl_2$. The CH_2Cl_2 was concentrated under vacuum. NMR analysis of this solution revealed it to be composed mostly (~85% based on ^{31}P) of the *racemic* Ni complex, Ni_2Cl_4(*rac*-et,ph-P4), **7R**, with a small amount of *meso* (**7M**) and one other unidentified compound. A large excess of hexanes was added to the concentrated CH_2Cl_2 solution and an orange powder precipitated. The powder was collected via filtration, washed with hexanes and dried under vacuum. NMR analysis of this powder revealed it to be an 87/13 mixture of **7R/7M** bimetallic complexes. Dissolving this powder in hot acetonitrile and allowing it to sit in air evaporating for 1 to 2 days causes pure **7R** to selectively crystalize out. This step can be repeated two to three times with further concentration of the solution but eventually the remaining solution becomes enriched with the *meso* complex and **7R** will no longer crystallize.

Ni_2Cl_4(*meso*-et,ph-P4), 7M: $^{31}P\{^1H\}$ NMR (162 MHz, CD_2Cl_2) δ 71.2 (pseudo-dt, J_{P-P} = 75 Hz), 51.7 (pseudo-dt, J_{P-P} = 75 Hz),

^1H NMR (400 MHz, CD_2Cl_2) δ 8.61-8.50 (m, 2H), 7.80-7.71 (m, 4H), 7.65-7.60 (m, 6H), 7.44-7.38 (m, 2H), 7.29-7.22 (m, 4H), 4.44-4.31 (m, 1H), 4.25-4.14 (m, 1H), 2.51-2.37 (m, 2H), 2.21-1.99 (m, 4H), 1.91-1.76 (m, 2H), 1.18 (dt, 6H, J_{HP} = 18.6 Hz, J_{HH} = 7.6 Hz), 1.03 (dt, 6H, J_{HP} = 20.3 Hz, J_{HH} = 7.5 Hz)

Ni_2Cl_4(*rac*-et,ph-P4), 7R: $^{31}P\{^1H\}$ NMR (162 MHz, CD_2Cl_2) δ 69.1 (pseudo-d, J_{P-P} = 75 Hz), 50.3 (pseudo-d, J_{P-P} = 75 Hz),

^1H NMR (400 MHz, CD_2Cl_2) δ 9.59-9.53 (m, 2H), 8.14-8.06 (m, 2H), 8.02-7.95 (m, 4H), 7.83-7.76 (m, 2H), 7.55-7.40 (m, 8H), 4.57 (t, 2H), 2.39-2.26 (m, 2H), 1.88-1.72 (m, 2H), 1.57-1.42 (m, 4H), 1.02 (dt, 6H, J_{HP} = 20 Hz, J_{HH} = 7.5 Hz), 0.70 (dt, 6H, J_{HP} = 17.9 Hz, J_{HH} = 7.6 Hz)


31P{1H} NMR
* = impurities
69.2
68.7
49.7
49.2
67.0
66.6
48.3
47.8
7M
Ni2Cl4(meso-et,ph-P4-Ph)
7R
Ni2Cl4(rac-et,ph-P4-Ph)
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
ppm

Crystallography on 7M and 7R

X-ray quality crystals of both diastereomers were grown from CH_2Cl_2, acetonitrile, and acetone. **7R** grew without any solvate while **7M** grew as three different crystal morphs with each of the solvents present in the unit cell. The *meso* structures with acetone as the solvate had two molecules per asymmetric unit and the lowest R factor (3.7%), followed by the acetonitrile structure (6.5%). The CH_2Cl_2 solvated **7M** structure was poorly resolved with multiple disorder problems (R = 17%) and will not be reported.

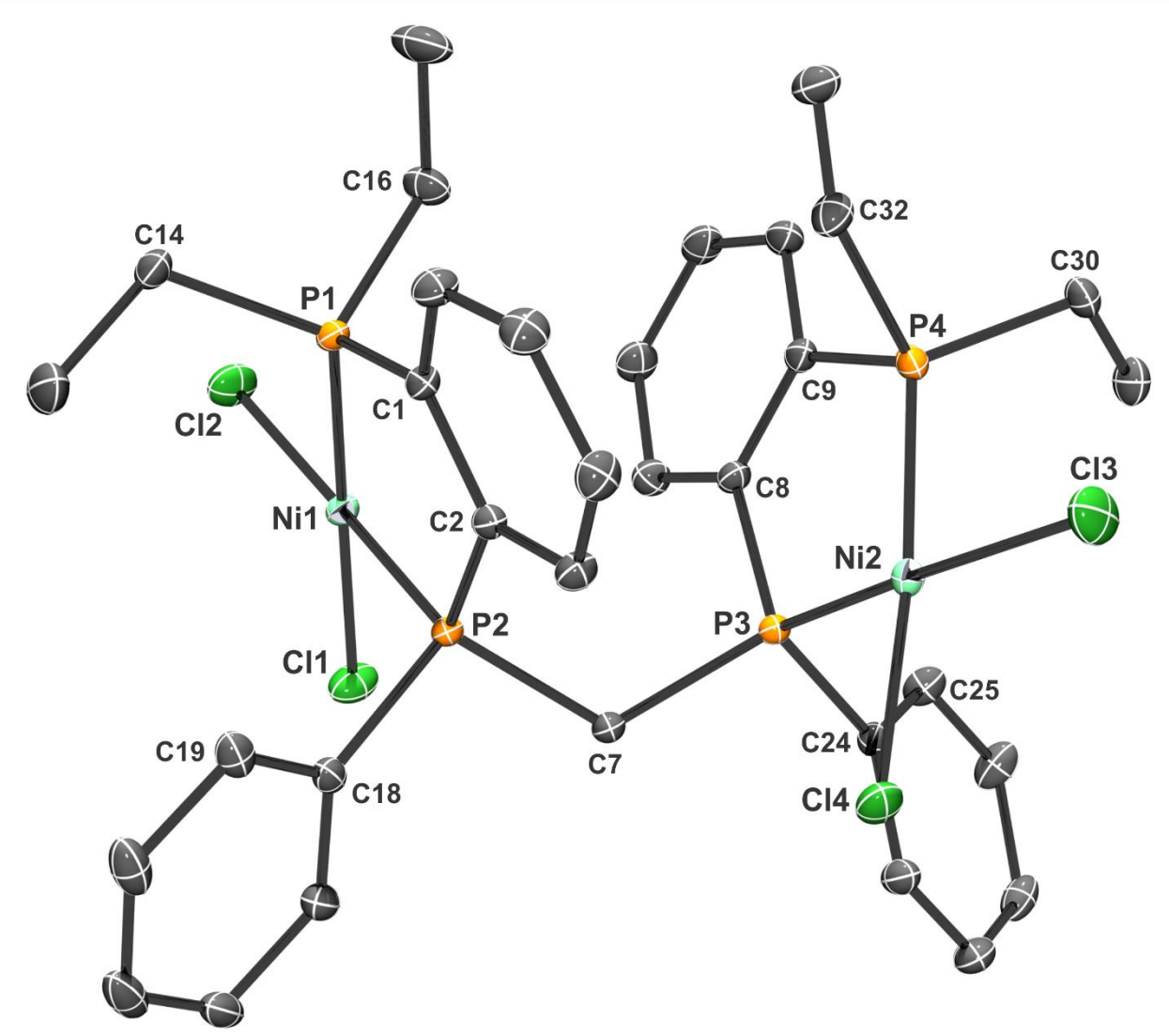
Table 1

Experimental details

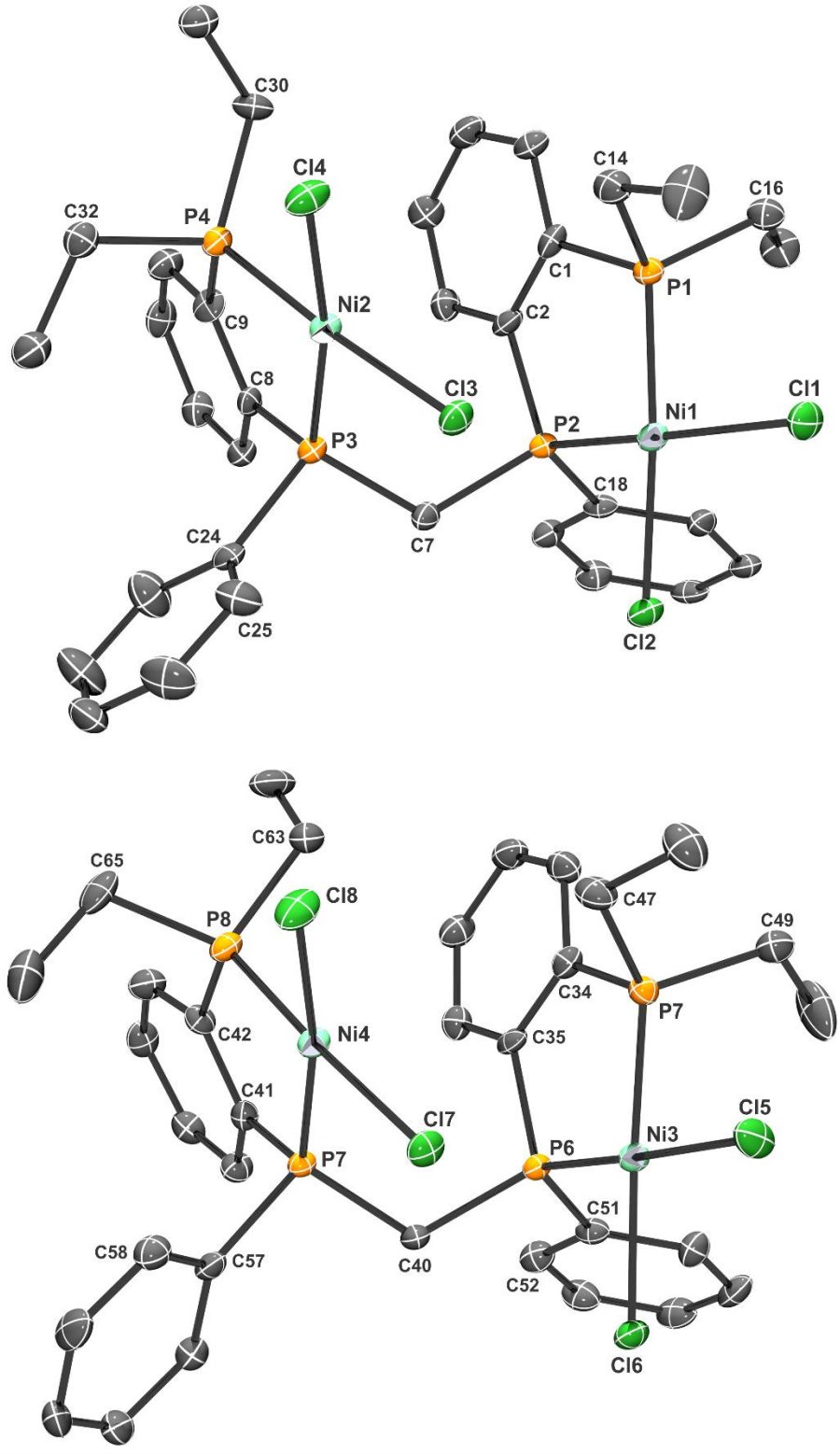
	(7R)	(7M_acetone)	(7M_MeCN)
Crystal data			
Chemical formula	$C_{33}H_{40}Cl_4Ni_2P_4$	$C_{33}H_{40}Cl_4Ni_2P_4 \cdot C_3H_6O$	$C_{33}H_{40}Cl_4Ni_2P_4 \cdot C_2H_3N$
M_r	819.75	877.83	860.80
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1$	Monoclinic, $P2_1$
Temperature (K)	90	90	90
a, b, c (Å)	11.4847 (3), 16.9323 (3), 17.8790 (4)	18.924 (4), 10.5062 (14), 20.027 (3)	9.2838 (4), 10.5305 (5), 19.7959 (8)
α, β, γ (°)	90, 92.277 (2), 90	90, 104.962 (10), 90	90, 101.489 (3), 90
V (Å^3)	3474.05 (13)	3846.8 (11)	1896.53 (14)
Z	4	4	2
Radiation type	Mo $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	1.60	5.57	5.63
Crystal size (mm)	0.16 × 0.13 × 0.06	0.17 × 0.08 × 0.01	0.11 × 0.08 × 0.04
Data collection			
Diffractometer	Bruker Kappa *APEX*-II DUO diffractometer	Bruker Kappa Apex-II CCD area detector diffractometer (with an Oxford Cryosystems cryostream cooler)	Bruker Kappa *APEX*-II DUO diffractometer
Absorption correction	Multi-scan *SADABS* (Sheldrick, 2002)	Multi-scan *SADABS* (Sheldrick, 2002)	Multi-scan *SADABS* (Sheldrick, 2002)
T_{min}, T_{max}	0.784, 0.910	0.442, 0.946	0.577, 0.806
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	89227, 14411, 11686	13645, 10448, 9294	12038, 5264, 4979
R_{int}	0.037	0.032	0.059
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.796	0.603	0.601
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.068, 1.03	0.037, 0.091, 1.00	0.065, 0.167, 1.11
No. of reflections	14411	10448	5264
No. of parameters	410	859	416
No. of restraints	0	1	1
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.82, −1.12	0.45, −0.43	1.01, −0.53
Absolute structure	–	Flack H D (1983), Acta Cryst. A39, 876-881	1718 Friedel pairs (Flack, 1983)
Absolute structure parameter	–	0.014 (13)	0.08 (4)

Computer programs: Bruker *APEX2*, Bruker (2006) Apex-II, Bruker *SAINT*, *SHELXS97* (Sheldrick, 2008), *SIR97* (Altomare *et al.*, 1999), *ORTEP-3 for Windows* (Farrugia, 1997), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL97* (Sheldrick, 2008) and *publCIF* (Westrip, 2010), Bruker *SHELXTL* (Sheldrick, 2008).

7R ORTEP (hydrogen atoms omitted for clarity, 50% probability ellipsoids)



7M Acetone solvate – two molecules per asymmetric unit **(hydrogen atoms omitted for clarity, 50% probability ellipsoids)**



7M Acetonitrile solvate

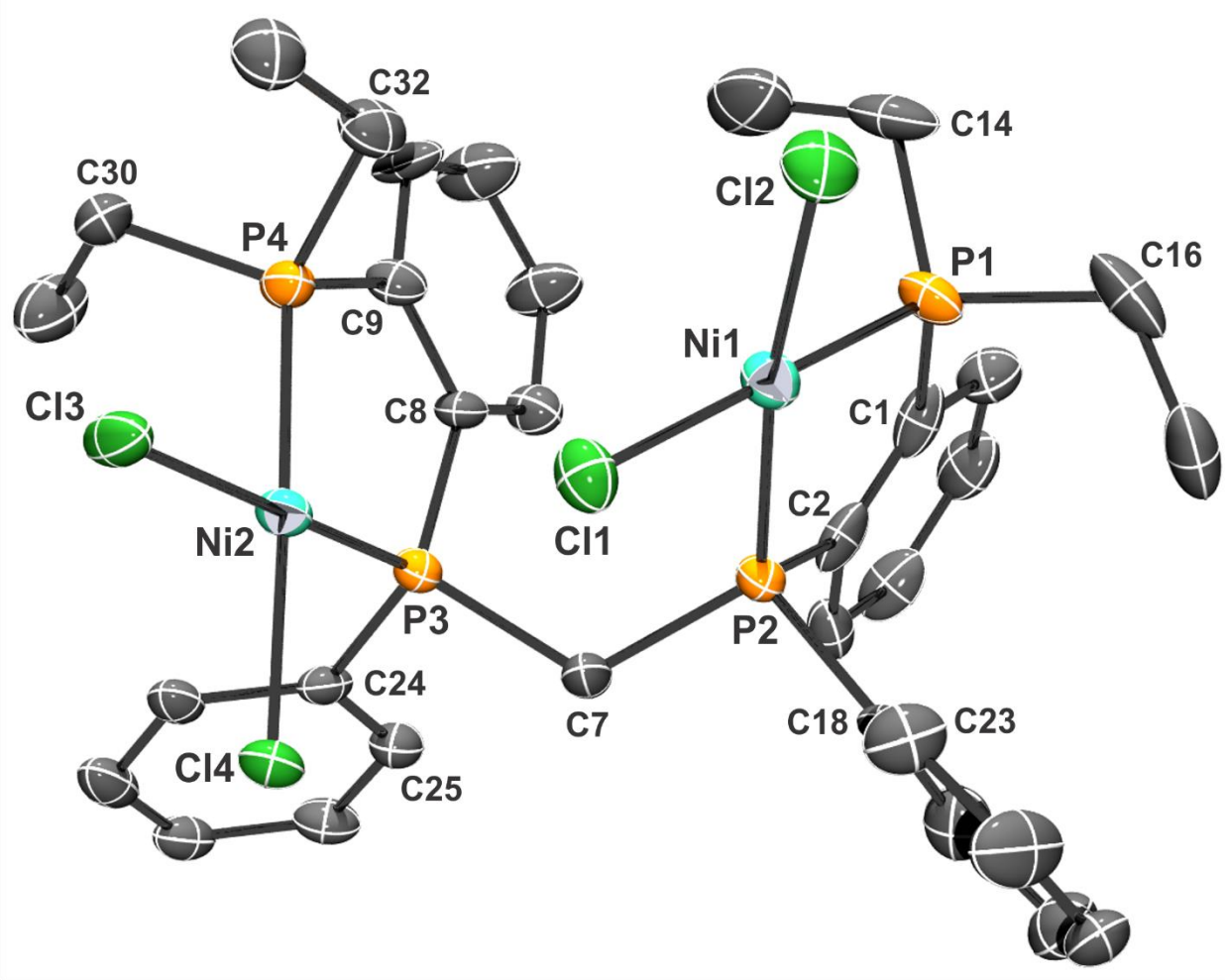


Table of Selected Bond Distances and Angles of **7R**, **7M-acetone (first molecule = 7M-A1, second molecule = 7M-A2)**, **7M-acetonitrile** (**7M-AN**) with Comparisons to Ni_2Cl_4(*rac*-et,ph-P4), **8R**, and Ni_2Cl_4(*meso*-et,ph-P4), **8M**.

	7R	**8R**[#]	**7M-A1**	**7M-A2***	**7M-AN**	**8M**[#]
Ni1-Cl1	2.1898 (3)	2.205 (2)	2.1921 (14)	2.2133 (14)	2.196 (3)	2.208 (2)
Ni1-Cl2	2.1871 (4)	2.194 (2)	2.2310 (12)	2.2169 (12)	2.200 (3)	2.195 (2)
Ni1-P1	2.1416 (3)	2.153 (2)	2.1401 (13)	2.1392 (13)	2.145 (3)	2.144 (2)
Ni1-P2	2.1298 (3)	2.134 (2)	2.1196 (14)	2.1363 (14)	2.131 (3)	2.139 (2)
Ni2-Cl3	2.1921 (4)	2.173 (2)	2.2047 (14)	2.1973 (13)	2.247 (3)	2.187 (2)
Ni2-Cl4	2.2057 (4)	2.214 (2)	2.1939 (13)	2.1824 (14)	2.226 (2)	2.209 (2)
Ni2-P3	2.1324 (3)	2.134 (2)	2.1298 (14)	2.1151 (13)	2.135 (3)	2.136 (2)
Ni2-P4	2.1379 (4)	2.141 (2)	2.1434 (14)	2.1318 (14)	2.143 (2)	2.160 (2)
Ni1•••Ni2	5.9031 (1)	5.417 (1)	4.434 (1)	4.272 (1)	4.404 (1)	6.272 (1)
Ni2•••Cl1	----	----	3.078 (1)	3.037 (1)	3.024 (3)	----
Cl1-Ni1-Cl2	94.352 (14)	94.90 (8)	95.52 (5)	95.68 (5)	96.21 (12)	95.17 (8)
P1-Ni1-P2	88.385 (13)	86.86 (8)	88.04 (5)	88.65 (5)	87.21 (11)	86.50 (8)
Cl1-Ni1-P1	177.654 (15)	175.63 (8)	175.68 (6)	174.92 (6)	176.04 (12)	176.75 (9)
Cl1-Ni1-P2	89.375 (13)	90.25 (7)	88.09 (5)	87.09 (5)	89.84 (10)	90.27 (8)
Cl2-Ni1-P1	87.855 (14)	87.86 (8)	88.72 (5)	88.12 (5)	87.01 (11)	88.06 (9)
Cl2-Ni1-P2	175.467 (15)	174.26 (8)	169.58 (6)	172.44 (6)	171.44 (12)	175.55 (9)
Cl3-Ni2-Cl4	94.581 (15)	95.89 (9)	95.17 (5)	96.73 (5)	94.92 (9)	94.13 (8)
P3-Ni2-P4	87.771 (13)	86.84 (8)	87.90 (5)	88.09 (5)	88.49 (11)	86.37 (7)
Cl3-Ni2-P3	167.563 (15)	173.48 (9)	169.58 (6)	170.59 (6)	175.76 (11)	175.07 (9)
Cl3-Ni2-P4	87.954 (15)	86.74 (9)	87.08 (5)	87.50 (5)	88.49 (11)	89.79 (8)
Cl4-Ni2-P3	90.859 (13)	90.46 (8)	90.75 (5)	88.53 (5)	87.11 (10)	89.58 (8)
Cl4-Ni2-P4	173.842 (14)	175.9 (1)	173.91 (6)	172.88 (6)	173.05 (12)	175.30 (9)
P2-C7-P3	118.60 (6)	119.3 (3)	119.5 (2)	117.5 (2)	120.2 (4)	121.7 (4)
Ni1-P2•••P3-Ni2	130.14 (1)	105.82 (5)	43.33 (1)	41.90 (1)	41.29 (1)	160.04 (5)

The atom labels for **8R** and **8M** have been set to match those used for the new structures to make comparisons easier.

* The atom labeling for the second molecule in the asymmetric unit has been adjusted to match that of the first molecule to make comparisons easier.