Ontario, for the high field spectra, Dr. J. W. Kolis for useful discussions, and the Natural Sciences and Engineering Research Council of Canada for financial support. G.J. thanks the France-Canada Scientific Exchange Program for a visiting professorship; M.M. is the recipient of an NSERC postgraduate Scholarship.

Registry No. **1**, 36834-86-3; **2**, 88008-84-8; **3**, 97950-29-3; **4**, 97919-68-1; **5**, 97950-30-6; **6**, 97950-31-7; **7**, 97919-69-2; **8**, 97950-32-8; **9**, 97950-33-9; **10**, 97919-70-5; C_5H_6, 542-92-7; $[CpNi(CO)]_2$, 12170-92-2; $[CpMo(CO)_3]_2$, 12091-64-4; Cp_2Ni, 1271-28-9; $Fe_2(CO)_9$, 15321-51-4.

Supplementary Material Available: Lists of calculated and observed structure factors, isotropic and anisotropic thermal parameters, and complete bond angles (19 pages). Ordering information is given on any current masthead page.

Communications

New Nickel *o*-Xylyl Complexes. Crystal and Molecular Structure of $Ni_3(CH_2C_6H_4Me\text{-}o)_4(PMe_3)_2(\mu_3\text{-}OH)_2$

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Summary: The formation of the new nickel *o*-xylyl derivatives $NiCl(\eta^1\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$, **1**, $NiCl(\eta^3\text{-}CH_2C_6H_4CH_3)(PMe_3)$, **2**, and $Ni_3(\eta^1\text{-}CH_2C_6H_4CH_3)_4(PMe_3)_2(\mu_3\text{-}OH)_2$, **3**, is reported. A crystal structure determination on **3** shows the complex crystallizes in the space group $P\bar{1}$ with a = 10.428 (2) Å, b = 12.646 (3) Å, c = 16.551 (3) Å, α = 70.68 (2)°, β = 79.54 (1)°, γ = 69.20 (2)°, V = 1920.4 Å^3, and Z = 2. The molecule possesses a distorted trigonal-bipyramidal $Ni_3(OH)_2$ core with the Ni(II) ions in a distorted square-planar environment. Apart from the hydroxo ions, the coordination sphere of the nickel(II) ions is completed by two *o*-xylyl groups for one of the Ni(II) and by one alkyl group and one PMe_3 ligand for the others.

The chemistry of organonickel compounds continues to attract much attention, both from a synthetic and from a structural point of view.[1] Following previous studies on the formation of nickel alkyl and acyl complexes,[2] we have investigated reactions of the *o*-methylbenzyl ligand, *o*-$CH_3C_6H_4CH_2$, with several nickel complexes. Here we report preliminary results based on reactions with $NiCl_2(PMe_3)_2$ and $Ni(cod)_2$ that lead to the formation[3] of a σ-bonded derivative, $NiCl(\eta^1\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$, **1**, a π-allyl derivative, $NiCl(\eta^3\text{-}CH_2C_6H_4CH_3)(PMe_3)$, **2**, and an unusual trimetallic compound, $Ni_3(\eta^1\text{-}CH_2C_6H_4CH_3)_4(PMe_3)_2(\mu_3\text{-}OH)_2$, **3**, containing two hydroxo groups triply bridging one NiR_2 fragment and two $Ni(R)(PMe_3)$ fragments.

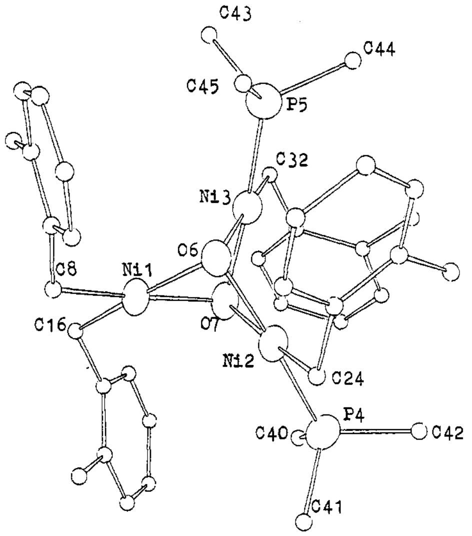

Figure 1. Molecular structure of **3** and atom labeling scheme. The C_6H_4Me labels have been ommitted for clarity. Important bond distances (Å) and angles (deg) include the following: Ni1–O = 2.000 (8) (av); Ni2–O7 and Ni3–O6 = 1.97 (1) (av); Ni2–O6 and Ni3–O7 = 1.92 (1) (av); Ni–C = 1.95 (1) (av); Ni–P = 2.110 (4) (av); O6–Ni–O7 = 72 (1) (av); Ni–O–Ni = 89 (1) (av); C–Ni–L (L = C or P) = 90.1 (5) (av).

Treatment of $NiCl_2(PMe_3)_2$ with $Mg(o\text{-}CH_3C_6H_4CH_2)Cl$ results in the formation of **1** or of a mixture of **1** and **2** depending upon the reaction conditions. From the resulting mother liquor, small amounts of very dark crystals of **3** can be collected after standing at −20 °C for 2–3 days. Although it is not easy to rationalize the formation of **3**, it is evident that adventitious water, possibly introduced at some stage during workup of the reaction, must play an important role. In fact, formation of **3** is not observed when the reaction is carried out with recently prepared Grignard solutions and under very strict anhydrous conditions.[4a] Complex **3** is very air sensitive, and although

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(2) Carmona, E.; González, F.; Poveda, M. L.; Atwood, J. L.; Rogers, R. D. *J. Chem. Soc., Dalton Trans.* **1980**, 2108–2116.

(3) The new compounds described in this paper have been fully characterized by analytical data and IR and multinuclear NMR spectroscopies.

(4) (a) The best yield of **3** is obtained as follows: 0.28 g of $NiCl_2(PMe_3)_2$ (1 mmol) in 30 mL of Et_2O was reacted at −40 °C, for ca. 30 min with 3 equiv of a 0.5 N solution of $Mg(o\text{-}CH_3C_6H_4CH_2)Cl$ in Et_2O. The resulting mixture was stirred at room temperature for 4 h, the solvent removed in vacuo, and the residue extracted with 40 mL of petroleum ether. Complex **3** was obtained in ca. 30%, together with small amounts of **1**, after concentration to ca. 5 mL, addition of a drop of water, and cooling at −30 °C for 48 h. (b) Crystal data: $C_{38}H_{56}Ni_3O_2P_2$; M = 782.5; triclinic; space group $P\bar{1}$; a = 10.428 (2) Å, b = 12.646 (3) Å, c = 16.551 (3) Å, α = 70.68 (2)°, β = 79.54 (1)°, γ = 69.20 (2)°, V = 1920.4 Å^3; ρ_{calcd} = 1.35 g cm^{-3}, Z = 2, $F(000)$ = 828; μ(Mo Kα) = 11.25 cm^{-1}. Intensity data collected on an Enraf-Nonius CAD4 diffractometer using the ω–2θ scan technique; 6817 independent reflections measured in a hemisphere, with $2 < \theta < 25°$; 1983 reflections considered observed, $I \geqslant 4\sigma(I)$ after data reduction. Structure solved by Patterson and Fourier techniques and refined by least-squares methods. The hydrogens attached to O(6) and O(7) were found by means of a Fourier difference map and included in the refinement with fixed contributions. The final R value was 0.044. All computations were made with the Oxford CRYSTALS package.[5] No absorption and no extinction correction were made.

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stable as a solid when kept under nitrogen, it decomposes quickly in solution even under inert atmosphere. This and its very low solubility in common organic solvents have made difficult its spectroscopic characterization.

Complex **3** has been investigated by X-ray crystallography[4b] and has been found to consist of discrete molecules with the structure shown in Figure 1. This structure may be described as a triangular array of metal atoms, with the two hydroxo groups bridging unsymmetrically the edges of the nearly equilateral Ni_3 triangle.[6] The $Ni_3(OH)_2$ core forms a distorted trigonal bipyramid, with the three nickel(II) ions in the equatorial plane at the relatively long Ni···Ni average separation of 2.75 (4) Å. While this distance is significantly shorter than the Ni···Ni distance in triangular $Ni_3(OH)$ cores[7] (3.04–3.14 Å), it is considerably longer than that observed in other trinuclear organometallic compounds of nickel,[8] indicating no or very weak Ni···Ni bonding interaction. As in the related complex $Ni_3(\eta^5\text{-}C_5H_5)_3S_2$ the relatively small intramolecular O···O distance at 2.30 (1) Å and the appreciably acute O–Ni–O angle (72 (1)° (av)) may be due[9] to a compression of the Ni_3O_2 skeleton due to the long Ni···Ni distances motivated by the absence of Ni···Ni bonding interaction. This is also manifested in the relatively short Ni···O separations (see below) exhibited by **3**, as compared with the average values of 2.05 (1) and 2.01 (1) Å found in other trimetallic complexes containing $Ni_3(OH)$ cores.[7]

Apart from the two hydroxo ions, the coordination sphere of the nickel(II) ions is completed by a trimethylphosphine and a xylyl ligand for Ni(2) and Ni(3) and by two xylyl groups for Ni(1). Each nickel(II) ion is therefore 4-coordinate and resides in a distorted square-planar environment, with the coordination plane approximately perpendicular to the Ni_3 triangle and bisecting the corresponding Ni–Ni–Ni angle. As expected, Ni(1) exhibits the longest Ni···O separations at 1.995 (8) and 2.004 (8) Å (for O(6) and O(7), respectively), while for Ni(2) and Ni(3), the Ni···O(6) and Ni···O(7) distances are 1.906 (9) and 1.956 (8) Å for Ni(2) and 1.975 (8) and 1.940 (8) Å for Ni(3), respectively. The Ni–C and Ni–P bond lengths have normal values and compare well with similar distances reported in the literature for other nickel alkyl complexes of trimethylphosphine.[2]

Compounds **1** and **2** are best prepared[10] by reaction of $Ni(cod)_2$ with $o\text{-}CH_3C_6H_4CH_2Cl$ in the presence of 2 and 1 equiv of PMe_3, respectively, as shown in eq 1 and 2.

$$\mathrm{Ni(cod)_2} + o\text{-}\mathrm{CH_3C_6H_4CH_2Cl} + 2\mathrm{PMe_3} \rightarrow \\ trans\text{-}\mathrm{NiCl}(\eta^1\text{-}\mathrm{CH_2C_6H_4CH_3})(\mathrm{PMe_3})_2 + 2\mathrm{cod} \quad (1)$$

$$\mathrm{Ni(cod)_2} + o\text{-}\mathrm{CH_3C_6H_4CH_2Cl} + \mathrm{PMe_3} \rightarrow \\ \mathrm{NiCl}(\eta^3\text{-}\mathrm{CH_2C_6H_4CH_3})(\mathrm{PMe_3}) + 2\mathrm{cod} \quad (2)$$

An excess of trimethylphosphine should be avoided in the former reaction, since **1** readily disproportionates under these conditions to give $NiCl_2(PMe_3)_2$, $Ni(PMe_3)_4$, and $C_{16}H_{18}$(1,2-bis(2-methylphenyl)ethane) as the final products. Both **1** and **2** are crystalline solids, very air sensitive in solution and in the solid state. Interconversion of both compounds takes place easily by addition of PMe_3 to solutions of **2** or by phosphine abstraction from **1** with $Ni(cod)_2$, as shown in eq 3.

$$\mathrm{NiCl}(\eta^1\text{-}\mathrm{CH_2C_6H_4CH_3})(\mathrm{PMe_3})_2 \underset{\mathrm{PMe_3}}{\overset{\mathrm{Ni(cod)_2}}{\rightleftharpoons}} \\ \mathrm{NiCl}(\eta^3\text{-}\mathrm{CH_2C_6H_4CH_3})(\mathrm{PMe_3}) \quad (3)$$

In addition, a room-temperature mixture of **1** and **2** shows only one set of proton resonances with chemical shifts average of those of **1** and **2**, clearly indicating that a rapid interconversion process is taking place.

Spectroscopic[11] and chemical studies for **1** and **2** are in accord with their formulations as η^1- and η^3-*o*-methylbenzyl complexes.[12] Interaction of **1** with CO affords the acyl complex *trans*-$NiCl(COCH_2C_6H_4CH_3)(PMe_3)_2$, while chloride metathesis with other anionic ligands produces the corresponding $NiX(\eta^1\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$ derivatives (X = Br, NCS, C_4H_4N, 2,5-$Me_2C_4H_2N$). The reactions of **2** with three- or five-electron donor ligands take place with concomitant change in the coordination mode of the *o*-methylbenzyl ligand from the η^3- to the η^1-bonding mode. For instance, NaCp produces the expected $(\eta^5\text{-}C_5H_5)Ni(\eta^1\text{-}CH_2C_6H_4CH_3)(PMe_3)$, and NaS_2CNR_2 (R = Me, *i*-Pr) gives $Ni(\eta^1\text{-}CH_2C_6H_4CH_3)(S_2CNR_2)(PMe_3)$. Interestingly, treatment of **2** with 4–5 equiv of *t*-BuNC yields red crystals of composition $NiCl(CH_2C_6H_4CH_3)(CNBu\text{-}t)_4$, for which spectroscopic studies suggest structure similar to that proposed for other similar derivatives.[13]

Acknowledgment. Generous support by the Comision Asesora de Investigación Científica y Técnica, CAICYT (to E.C.) is very gratefully acknowledged. P.P. and M.P. thank the CAICYT and Ministerio de Educación y Ciencia for support of research grants. We are also indebted to Professors Rafael Márquez and Amparo López, Facultad de Física de Sevilla, for the use of the diffractometer.

Registry No. **1**, 98170-36-6; **2**, 98170-37-7; **3**, 98170-38-8; $NiCl_2(PMe_3)_2$, 19232-05-4; $Ni(PMe_3)_4$, 28069-69-4; *trans*–$NiCl(COCH_2C_6H_4CH_3)(PMe_3)_2$, 98170-39-9; $NiBr(\eta'\text{–}CH_2C_6H_4CH_3)(PMe_3)_2$, 98170-40-2; $Ni(NCS)(\eta'\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$, 98170-41-3; $Ni(C_4H_4N)(\eta'\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$, 98170-42-4; $Ni(2,5\text{-}Me_2C_4H_2N)(\eta'\text{-}CH_2C_6H_4CH_3)(PMe_3)_2$, 98170-43-5; $(\eta^5\text{-}C_5H_5)Ni(\eta'\text{-}CH_2C_6H_4CH_3)(PMe_3)$, 98170-44-6; $Ni(\eta'\text{-}CH_2C_6H_4CH_3)(S_2CNMe_2)(PMe_3)$, 98170-45-7; $Ni(\eta'\text{-}CH_2C_6H_4CH_3)(S_2CN(i\text{-}Pr)_2)(PMe_3)$, 98170-46-8; NiCl-

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(6) An analogous doubly μ_3-hydroxo-bridged trinuclear Cu(II) complex has been reported recently. See: Comermond, J.; Dietrich, B.; Lehn, J. M.; Louis, R. *J. Chem. Soc., Chem. Commun.* **1985**, 74–76.

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(8) Raithby, P. R. "Transition Metal Clusters", Johnson, B. F. G., Ed.; Wiley: New York, 1980; Chapter 2.

(9) Vahrenkamp, H.; Uchtman, V. A.; Dahl, L. F. *J. Am. Chem. Soc.* **1968**, *90*, 3272–3273.

(10) To a cold (−60 °C) suspension of $Ni(cod)_2$ (1.92 g, ca. 7 mmol) in Et_2O (80 mL) were added PMe_3 (1.4 mL, ca. 14 mmol) and *o*-$ClCH_2C_6H_4Me$ (0.92 mL, ca. 7 mmol) via syringe, and the resulting mixture was stirred at room temperature for 7 h. After removal of the volatiles in vacuo, extraction with Et_2O (100 mL), and cooling at −30 °C, compound **1** was obtained as brown-orange prisms in ca. 90% yield. A similar procedure, using half of the amount of PMe_3, afforded **2** as red plates in ca. 70% yield.

(11) Analytical and spectroscopic data for **1** are as follows: ^1H NMR (200 MHz, C_6D_6) δ 0.94 (b s, 18 H, PMe_3), 1.57 (s, 2 H, $CH_2C_6H_4Me$), 2.50 (s, 3 H, $CH_2C_6H_4Me$), 7.07 and 7.62 (m and d, 3 H and 1 H, respectively, $CH_2C_6H_4Me$); $^{31}P\{^1H\}$ NMR (C_6D_6) δ −15.5 s; $^{13}C\{^1H\}$ NMR (C_6D_6) δ 5.06 (s, $CH_2C_6H_4Me$), 12.57 (b s, PMe_3), 21.28 (s, $CH_2C_6H_4Me$), 124.73, 125.33, 129.06, 131.87, 135.77, 146.86 (s, aromatics). Anal. Calcd for $C_{14}H_{27}P_2ClNi$: C, 47.8; H, 7.7. Found: C, 47.6; H, 7.8. Data for **2**: ^1H NMR (200 MHz, C_6D_6) δ 0.90 (b d, 9 H, $^2J_{HP}$ = 8 Hz, PMe_3), 1.09 (b s, 2 H, $CH_2C_6H_4Me$), 1.97 (s, 3 H, $CH_2C_6H_4Me$), 6.98–7.29 (m, 4 H, aromatics); $^{31}P\{^1H\}$ NMR (C_6D_6) δ −6.65 s; $^{13}C\{^1H\}$ NMR (C_6D_6) δ 15.21 (d, $^1J_{CP}$ = 29 Hz, PMe_3), 19.88 (s, $CH_2C_6H_4Me$), 20.6 (b s, $CH_2C_6H_4Me$), 107.9 and 114.7 (b s, aromatics bound to Ni), 127.98, 132.39, 132.67, 134.56 (s, aromatics). Molecular weight (cryoscopically in C_6H_6, N_2) calcd for $C_{11}H_{18}PClNi$: 275. Found: 274. Anal. Calcd for $C_{11}H_{18}PClNi$: C, 47.9; H, 6.5. Found: C, 47.7; H, 6.5. Preliminary X-ray studies on **2** are in accord with the η^3-allylic structure.

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$(CH_2C_6H_4CH_3)(CNBu\text{-}t)_4$, 98170-47-9; $Mg(o\text{-}CH_3C_6H_4CH_2)Cl$, 29875-05-6; $Ni(cod)_2$, 1295-35-8; $o\text{-}ClCH_2C_6H_4Me$, 552-45-4; $NaS_2CN(Me)_2$, 128-04-1; $NaS_2CN(i\text{-}Pr)_2$, 4092-82-4; t-BuNC, 7188-38-7; 1,2-bis(2-methylphenyl)ethane, 952-80-7.

Supplementary Material Available: Tables of fractional coordinates of atoms, thermal parameters, and observed and calculated structure factors for 3 (25 pages). Ordering information is given in any current masthead page.

A Novel Transition-Metal-Promoted Rearrangement of a Cyclopropenyl Cation. Synthesis and Crystal and Molecular Structure of a 1-3-η-Butadienyl Complex of Platinum

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Summary: Reaction of the methyldiphenylcyclopropenyl cation (**2**) with bis(triphenylphosphine)(ethylene)platinum(0) (**3**) affords the (1-3-η-butadienyl)platinum complex **6**, via an unprecedented ring-opening reaction coupled with a hydrogen shift. The crystal and molecular structure of **6** has been determined.

Reactions of cyclopropenyl cations with transition-metal complexes have afforded a diverse array of organometallic compounds. In addition to simple 3-η^1- and η^3-cyclopropenyl complexes, a number of other interesting and useful ligands have been derived from ring-opening or ring expansion reactions of these versatile three-membered organic rings.[3] An unusual mode of coordination for the intact triphenylcyclopropenyl ligand is found in its cationic complexes 1 containing the ML_2 fragment (M = Ni, Pd, Pt; L = PPh_3).[4,5] In these compounds the cyclopropenyl ring adopts a coordination mode perhaps best described as η^2, as evidenced by crystallographic determinations on compounds of all three metals of the nickel triad. These η^2-cyclopropenyl compounds are fluxional in solution, exhibiting a low-energy "ring-whizzing" of the ML_2 fragment. The pathway by which the metal perambulates around the periphery of the ring has been examined theoretically, and various points along this pathway are defined by the different conformational relationships between the ML_2 fragment and the cyclopropenyl ring which are observed in the solid-state structures of 1.[4]

We now report that this mode of coordination does not always result in isolable cyclopropenyl complexes and that when a methyl group is present on the three-membered ring, a novel skeletal rearrangement coupled with a hydrogen shift can occur to give the 1-3-η-butadienyl ligand, further extending the range of ligand types available from cyclopropenyl cation precursors.

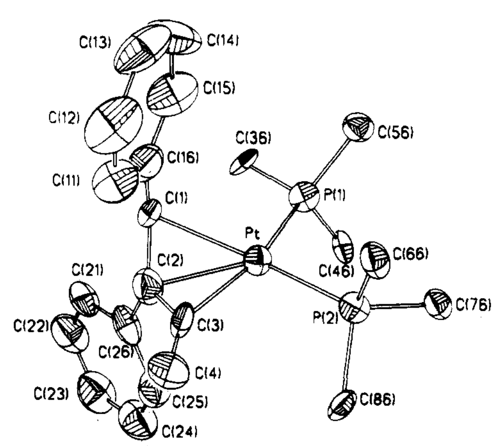
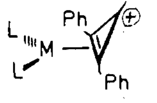


Figure 1. The molecular geometry and labeling scheme for **6**, drawn with 40% probability ellipsoids. Only the ipso carbon atoms of the phenyl groups of the triphenylphosphine ligands are shown. Selected bond lengths (Å): Pt–P(1), 2.339 (4); Pt–P(2), 2.289 (4); Pt–C(1), 2.291 (14); Pt–C(2), 2.201 (14); Pt–C(3), 2.075 (13); C(1)–C(2), 1.40 (2); C(2)–C(3), 1.46 (2); C(3)–C(4), 1.31 (2); Selected bond angles (deg): P(1)–Pt–C(1), 97.3 (4); P(1)–Pt–C(2), 120.3 (4); P(1)–Pt–C(3), 159.9 (4); P(2)–Pt–C(1), 155.0 (4); P-(2)–Pt–C(2), 134.3 (4); P(2)–Pt–C(3), 95.7 (4); C(1)–Pt–C(2), 36.2 (5); C(1)–Pt–C(3), 66.9 (5); C(2)–Pt–C(3), 39.7 (6); C(1)–C(2)–C(3), 115 (1); C(2)–C(3)–C(4), 140 (1); P(1)–Pt–P(2), 103.0 (1).

By analogy with the previously documented reaction of the triphenylcyclopropenyl cation,[5] it was anticipated that reaction of 2 with the platinum(0) complex 3 should afford the η^2-cyclopropenyl complex 4. It was envisaged that subsequent deprotonation of 4 would lead to the η^2-methylenecyclopropene compound 5, thus providing the first example of a transition-metal complex of this smallest cross-conjugated fulvene skeleton.[6] Contrary to these expectations, addition of an equimolar amount of tetrafluoroborate salt of 2 to a CH_2Cl_2 solution of 3 resulted in a sudden color change from pale yellow to bright orange, with subsequent rapid fading to a golden yellow color. Workup afforded high yields of a bright yellow crystalline solid, 6.[7] While microanalysis results indicated that this



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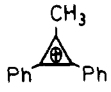

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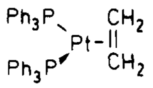

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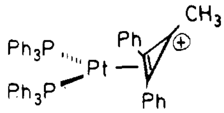

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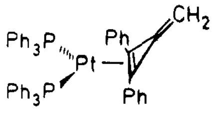

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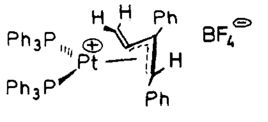

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(1) (a) Dartmouth College. (b) University of Delaware.

(2) Alfred P. Sloan Research Fellow, 1980–1984.

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