catalysts and immiscible alloys.

A temperature-variable investigation of the solid-state magnetic properties of this high-nuclearity heterometallic cluster was undertaken because the even-electron decametal cluster $H_2Os_{10}C(CO)_{24}$ was found to exhibit Curie-like behavior below 70 K with a magnetic moment of 0.62 μ_B which was attributed to "quantum-size" effects.[22] Magnetic susceptibility measurements of the $[PPh_3Me]^+$ salts of **1** and the $[Ni_6(CO)_{12}]^{2-}$ dianion were carried out with a SHE SQUID magnetometer ($5\ K < T < 280\ K$) in magnetic fields from 1 to 40 kG. The magnetic data[23] (Figure 3) expectedly showed the $[Ni_6(CO)_{12}]^{2-}$ dianion to be diamagnetic but surprisingly revealed **1** to be paramagnetic with an effective magnetic moment of 2.08 μ_B indicative of one unpaired electron. This paramagnetism was substantiated by powder EPR measurements ($10\ K < T < 300\ K$; 9.243 GHz) with a spectrum (Figure 4, supplementary material) at 10 K exhibiting a line shape expected for an axially symmetric spin-$^1/_2$ system ($g_\parallel = 2.279$; $g_\perp = 2.027$).

Effective magnetic moments indicative of one unpaired electron have been found in several other high-nuclearity metal clusters.[24] We rationalize that the existence of an odd electron in **1** necessitates the presence of either one hydrido ($x = 1$) or three hydrido ($x = 3$) atoms. Our formulation of **1** as a monohydrido $[Rh_5Ni_6(CO)_{21}H_x]^{3-}$ trianion (with $x = 1$) is consistent with an analysis which indicates that the hydrogen atom could occupy *one* of the two tetrahedral Rh_4 cavities with reasonable, equivalent Rh(ax)–H and Rh(eq)–H distances of 1.85 Å; in fact, the calculated hydrogen position is in the near vicinity of a residual density peak found from a difference map. Attempts to isolate deprotonated species of **1** have been unsuccessful.

A detailed bonding description of **1** will be deferred until the presence of the hydrogen atom(s) is ascertained from a planned neutron diffraction investigation. Current research involves reactions of the $[Ni_6(CO)_{12}]^{2-}$ dianion with a number of ruthenium, osmium, rhodium, and iridium carbonyl clusters with the hope of nickel plating the metal polyhedral cores.

Acknowledgment. This research was financially supported by a Presidential Young Investigator Award to A.M.S. from the National Science Foundation (CHE 83-51881) and by a Grant to L.F.D. from the National Science Foundation (CHE 83-15312). We are indebted to Johnson Matthey Inc. (West Deptford, NJ) for supplying a sample (on loan) of hydrated rhodium trichloride which was used to prepare $Rh_2(CO)_4Cl_2$. We thank Dr. Norman M. Edelstein (Lawrence Berkeley Laboratory) for the use of his EPR spectrometer and Dr. Charles F. Campana (X-Ray Division, Nicolet Analytical Instruments, Madison, WI) for valuable assistance during the X-ray data collection on the Nicolet P3F diffractometer and in the use of SHELXTL.

Registry No. **1**-$3PPh_3Me\cdot{}^1/_2Me_2CO$, 101953-05-3; $[PPh_3Me]_2{}^+[Ni_6(CO)_{12}]^{2-}$, 101916-20-5; $Rh_2(CO)_4Cl_2$, 14523-22-9; Ni, 7440-02-0; Rh, 7440-16-6.

Supplementary Material Available: Figure 2 shows two views of the overall configuration of the $[Rh_5Ni_6(CO)_{21}H_x]^{3-}$ trianion (**1**), Figure 4 presents an EPR spectrum of a powder sample of $[PPh_3Me]_3{}^+[Rh_5Ni_6(CO)_{21}H_x]^{3-}\cdot{}^1/_2Me_2CO$ at 10 K and 9.243 GHz, and four tables list the atomic parameters and appropriate interatomic distances for $[PPh_3Me]_3{}^+[Rh_5Ni_6(CO)_{21}H_x]^{3-}\cdot{}^1/_2Me_2CO$ (7 pages). Ordering information is given on any current masthead page.

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[22] (a) Benfield, R. E.; Edwards, P. P.; Stacy, A. M. *J. Chem. Soc., Chem. Commun.* **1982**, 525–526. (b) Johnson, D. C.; Benfield, R. E.; Edwards, P. P.; Nelson, W. J. H.; Vargas, M. D. *Nature* (*London*) **1985**, *314*, 231–235.

[23] Samples were loaded into kel-f containers and sealed under dry argon. The χ_M values were corrected for the diamagnetism of the kel-f containers and for trace amounts of ferromagnetic impurities (<0.2%). For **1**, the χ_M values over $5\ K < T < 280\ K$ obey the equation $\chi_M = C/(T - \theta) + \chi_0$, where the first term represents the Curie-Weiss temperature-dependent behavior and the second term is a temperature-independent constant; parameters obtained from a data fitting are $C = 0.515\ K\ mol^{-1}$, $\theta = -11.0\ K$, and $\chi_0 = -8.0 \times 10^{-4}\ mol^{-1}$.

[24] (a) $[Pt_3Fe_3(CO)_{15}]^-$ ($\mu_{eff} = 1.6\ \mu_B$): Longoni, G.; Manassero, M.; Sansoni, M. *J. Am. Chem. Soc.* **1980**, *102*, 7973–7974. Longoni, G.; Morazzoni *J. Chem. Soc., Dalton Trans.* **1981**, 1735–1737. (b) $[Co_6(CO)_{14}C]^-$ ($\mu_{eff} = 1.37\ \mu_B$): Albano, V. G.; Chini, P.; Ciani, G.; Sansoni, M.; Strumolo, D.; Heaton, B. T.; Martinengo, S. *J. Am. Chem. Soc.* **1976**, *98*, 5027–5028. Albano, V. G.; Chini, P.; Ciani, G.; Sansoni, M.; Martinengo, S. *J. Chem. Soc., Dalton Trans.* **1980**, 163–166. Beringhelli, T.; Morazzoni, F.; Strumolo, D. *J. Organomet. Chem.* **1982**, *236*, 109–118. (c) $[Rh_{12}(CO)_{23}(C)_2]^{3-}$: Albano, V. G.; Braga, D.; Martinengo, S.; Strumolo, D. Atti del XIV Congresso della Societá Chimica Italiana, Catania, 1981; p 275. Beringhelli, T.; Morazzoni, F.; Strumolo, D. *J. Organomet. Chem.* **1982**, *236*, 109–118. (d) $[Co_{13}(CO)_{24}(C)_2]^{4-}$ ($\mu_{eff} = 1.45\ \mu_B$): Albano, V. G.; Braga, D.; Chini, P.; Ciani, G.; Martinengo, S. *J. Chem. Soc., Dalton Trans.* **1982**, 645–649. Beringhelli, T.; Morazzoni, F.; Strumolo, D. *J. Organomet. Chem.* **1982**, *236*, 109–118.

Epoxy Silyl Ether Rearrangements: A New, Stereoselective Approach to the Synthesis of β-Hydroxy Carbonyl Compounds

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Reported herein is a new method for the synthesis of β-hydroxy carbonyl compounds which is stereospecific, simple, and highly efficient. The new process depends on the site-specific 1,2-rearrangement as represented by Scheme I.[1,2]

The rearrangement of epoxy silyl ether **1** giving the aldol adduct **2** is effected by the use of titanium tetrachloride. Some examples are given in Table I. The typical experimental procedure is provided by the synthesis of 2-butyl-2-methyl-1-hydroxy-3-heptanone (**3**) from epoxy silyl ether **4** (entry 2). To a solution of **4** (272 mg, 1 mmol) in CH_2Cl_2 was added at −78 °C a 1 M CH_2Cl_2 solution of $TiCl_4$ (1.1 mL, 1.1 mmol). The mixture was stirred at −78 °C for 10 min, poured into aqueous HCl, and extracted with CH_2Cl_2. The organic layer was washed with brine, dried over Na_2SO_4, and concentrated. The residual oil was chromatographed on silica gel (ether/hexane, 1:1 as eluant) to give **3** (150 mg, 75% yield) as a colorless oil.

The characteristics of the new method follow. (1) The starting epoxy silyl ethers can be readily prepared stereo- and enantioselectively from a wide variety of simple precursors such as allylic alcohols or α,β-epoxy carbonyl compounds. (2) Of all the Lewis acids examined, $TiCl_4$ has proved to be the most efficacious. (3) The facile migration of the alkyl group was observed in the reaction of α,α,β-trisubstituted derivative **1** (entries 1–6 and 20–22). Here the aryl group migrates preferentially, leaving the alkyl group intact (entry 6). (1) In the case of β-nonsubstituted derivative **1**, only aryl and alkenyl groups participated in the selective 1,2-migration, reflecting the low migratory aptitude of the alkyl group (entries 7–15).[3] The rearranged α-alkenyl aldols were already

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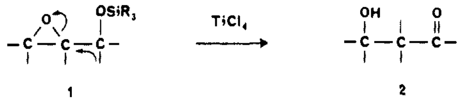
[1] This process represents the potential synthetic equivalent of stereoselective aldol methodologies. For recent reviews of the stereoselective aldol condensations, see: Evans, D. A.; Nelson, J. V.; Taber, T. R. *Top. Stereochem.* **1982**, 13. Mukaiyama, T. *Org. React.* **1982**, *28*, 203. Heathcock, C. H. In *Asymmetric Synthesis*; Morrison, J. D., Ed.; Academic Press: New York, 1984; Vol. 3B, p 111. Heathcock, C. H. In *Comprehensive Carbanion Chemistry*; Buncel, E., Durst, T., Eds.; Elsevier: New York, 1984; Vol. 5B, p 177. For related approaches to aldol adducts, see: Schreiber, S. L.; Hoveyda, A. H.; Wu, H.-J. *J. Am. Chem. Soc.* **1983**, *105*, 660. Curran, D. P. *Ibid.* **1983**, *105*, 5826. Fujita, M.; Hiyama, T. *Ibid.* **1985**, *107*, 8294.

[2] For another type of the epoxy alcohol rearrangement with Ti(O-*i*-Pr)$_4$, see: Morgans, D. J., Jr.; Sharpless, K. B.; Traynor, S. G. *J. Am. Chem. Soc.* **1981**, *103*, 462.

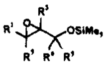
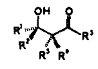
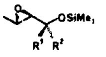
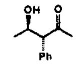
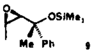
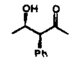
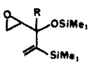
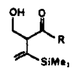
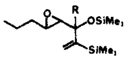
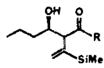
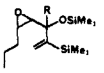
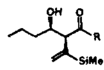
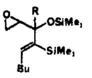
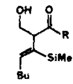
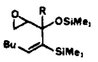
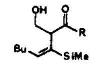
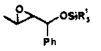
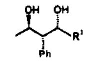
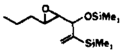
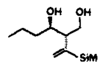
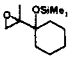
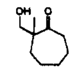
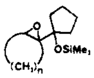
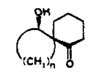
0002-7863/86/1508-3827$01.50/0

Scheme I



shown to be an important class of compounds as versatile building blocks for macrolide synthesis.[4] (5) The rearrangement is highly stereoselective with respect to the geometry of the epoxide moiety and not to the configuration of the alkoxy α-carbon. This observation is quite useful for synthetic applications. For example, both the erythro-trans isomer **5** and the threo-trans isomer **6** gave rise to the same threo aldol **7** as a sole isolable product, whereas the erythro aldol **8** was obtained exclusively from the threo-cis isomer **9** (entries 8–10, 12, and 13).[5] Thus, the observed stereoselectivity can be interpreted as the rigorous anti migration of the phenyl and alkenyl groups to the epoxide moiety in which electrostatic repulsion is minimized. (6) In certain cases the epoxy alcohols are also employable for the rearrangement (entries 2, 11–15, and 22). (7) The α-(trimethylsilyl)alkenyl groups migrate stereospecifically with complete retention of the olefin geometry, indicating the fully concerted nature of the rearrangement (entries 14 and 15). $BF_3{\cdot}OEt_2$ is also employable for the substrates possessing the readily migrating α-silylalkenyl moiety (entries 11–15).[6] (8) The α-monosubstituted derivative **1** on treatment with $TiCl_4$ afforded the unstable β-hydroxy aldehyde which can be reduced in situ with Et_3SiH to furnish 1,3-diols (entries 16 and 19). Notably, use of the bulky triphenylsilyl group was effective to improve the yield of the reaction (entry 17). Moreover, selective coupling of epoxy silyl ether with allyltrimethylsilane can be possible, thereby allowing the stereocontrolled chain extention with the rigorous establishment of the three contiguous chiral centers (entry 18).[7] (9) The one-carbon ring expansion is accomplished by the use of carbocyclic ethers (entries 20–22). (10) The reaction serves as a straightforward entry to the selective synthesis of spirocyclic cis aldols (entries 21 and 22).[8] The starting epoxy silyl ethers can be easily derived from the coupling of two kinds of cycloalkanone components by using the Shapiro reaction.[9]

Table I. Stereoselective Rearrangement of Epoxy Silyl Ethers[a]

entry	substrate	product	yield, %[b]
	 **4**	 **3**	
1	$R^1 = R^2 = H; R^3 = R^4 = R^5 = Me$		48
2	$R^1 = R^2 = H; R^3 = Me; R^4 = R^5 = Bu$		75 (58)[c]
3	$R^1 = H; R^2 = R^3 = R^4 = R^5 = Me$		48 (0)[c]
4	$R^1 = H; R^2 = R^3 = Me; R^4 = R^5 = Bu$		68
5	$R^1 = R^2 = R^3 = R^4 = R^5 = Me$		38
6	$R^1 = R^2 = H; R^3 = R^5 = Me; R^4 = Ph$		75 (18)[c]
7	$R^1 = R^2 = R^3 = H; R^4 = vinyl; R^5 = Pr$		58
		 7	
8	**5**: $R^1 = Ph, R^2 = Me$		95
9	**6**: $R^1 = Me, R^2 = Ph$		79
10	 9	 8	81
11			85 (81)[c,d]
12			98 (72)[c,d]
13			75 (55)[c,d]
14			95 (91)[c,d]
15		 (R = CH$_2$CH$_2$Ph)	93 (85)[c,d]
			
16	$R^1 = Me$	$R^2 = H$	58[e]
17	$R^1 = Ph$	$R^2 = H$	74[e,f]
18	$R^1 = Me$	$R^2 = CH_2CH{=}CH_2$	80,[g] 74[h]
19			71[e]
20			79 (0)[c]
			
21	$n = 3$		90
22	$n = 4$		89 (50)[c]

[a] Unless otherwise specified, reaction was carried out by using 1.1 equiv of $TiCl_4$ in CH_2Cl_2 at −78 °C for 10–30 min. [b] Isolated yield. [c] Use of epoxy alcohol as substrate. [d] Use of $BF_3{\cdot}OEt_2$ as Lewis acid. [e] Rearrangement was performed in the presence of Et_3SiH. [f] Isolated yield after treatment with Bu_4NF in THF at room temperature, since the reaction gave rise to a mixture of diol and its mono(triphenylsilyl)ether. [g] In the presence of $CH_2{=}CHCH_2SiMe_3$. [h] Initial treatment of the epoxy silyl ether with $TiCl_4$ followed by addition of $CH_2{=}CHCH_2SiMe_3$.

The new process disclosed above clearly demonstrates the versatility and practical utility in selective organic synthesis. Further, the general availability of chiral epoxy silyl ethers by combining use of the Sharpless asymmetric epoxidation[10] enabled the new, stereocontrolled approach to the asymmetric synthesis of various aldols and related 1,3-diols. Thus, chiral epoxy alcohols **10**[11] and **11**[12] were converted via the usual oxidation–alkylation sequence to epoxy silyl ethers **12**[13] and **13**[14] which without separation of two diastereomers were subjected to the rearrangement to furnish chiral aldol **14**[15,16] ($[\alpha]^{28}{}_D$ −125° (c 1.07, $CHCl_3$)) and

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(3) A similar tendency of the migratory aptitude was observed previously in the asymmetric pinacol-type rearrangement. See: Suzuki, K.; Katayama, E.; Tsuchihashi, G. *Tetrahedron Lett.* **1983**, *24*, 4997.

(4) Masamune, S.; Choy, W. *Aldrichimica Acta* **1982**, *15*, 47. Masamune, S.; Kaiho, T.; Garvey, D. S. *J. Am. Chem. Soc.* **1982**, *104*, 5521. Boschelli, D.; Ellingboe, J. W.; Masamune, S. *Tetrahedron Lett* **1984**, *25*, 3395.

(5) The isomeric epoxy silyl ethers **5** and **6** were prepared by the Sharpless epoxidation of the trans allylic alcohol followed by chromatographic separation of the two diastereomers. The stereochemical assignment of **5** and **6** were made according to the literature: Rossiter, B. E.; Verhoeven, T. R.; Sharpless, K. B. *Tetrahedron Lett.* **1979**, 4733.

(6) Suzuki, K.; Katayama, E.; Tsuchihashi, G. *Tetrahedron Lett.* **1984**, *25*, 1817.

(7) The stereochemistry of the diol (>95% pure) was determined by comparison with other diastereomeric diols which were prepared via an alternative route.

(8) The cis configuration of the spirocyclic aldols was established by TLC comparison with those of the authentic cis and trans isomers which were synthesized independently.

(9) Shapiro, R. H. *Org. React.* **1976**, *23*, 405. Chamberlin, A. R.; Stemke, J. E.; Bond F. T. *J. Org. Chem.* **1978**, *43*, 147.

(10) Katsuki, T.; Sharpless, K. B. *J. Am. Chem. Soc.* **1980**, *102*, 5974. Hill, J. G.; Sharpless, K. B.; Exon, C. M.; Regenye, R. *Org. Syn.* **1984**, *63*, 66.

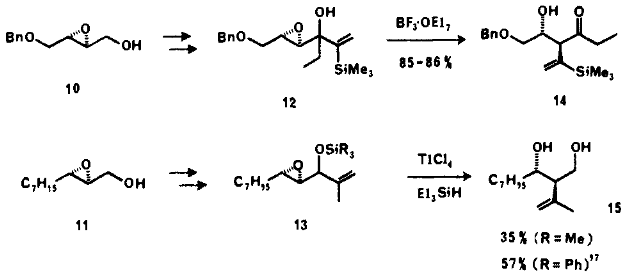
(11) Katsuki, T.; Lee, A. W. M.; Ma, P.; Martin, V. S.; Masamune, S.; Sharpless, K. B.; Tuddenham, D.; Walker, F. J. *J. Org. Chem.* **1982**, *47*, 1373. The optical purity of **10** (>95% ee) was substantiated by ^1H NMR analysis in the presence of the chiral shift reagent, $Eu(hfc)_3$, after converting to its acetate.

(12) $[\alpha]^{20}{}_D$ −30.0° (c 1.03, MeOH): 92% ee by capillary GLC after conversion to the (R)-(+)-MTPA ester.

(13) The epoxy alcohol **12** was prepared from **10** according to the following sequence: (i) Swern oxidation followed by alkylation with EtMgBr (77%), (ii) Swern oxidation (83%), and (iii) alkylation with $H_2C{=}C(SiMe_3)Li$ (87%).

(14) Derived from **11** by (i) Swern oxidation (81%), (ii) alkylation with isopropenyllithium (62%), and (iii) silylation with R_3SiCl (R = Me or Ph)/imidazole in DMF (90–95%).

1,3-diol **15**[16,17] ($[\alpha]^{20}_{D}$ −16.8° (*c* 1.02, MeOH)), respectively, with rigorous stereoselectivities.



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[15] Separate treatment of each diastereomer (erythro-trans and threo-trans isomers) of **12** with $BF_3 \cdot OEt_2$ gave rise to the same threo alcohol **14** in 85% and 86% yield, respectively.

[16] The diastereomeric isomer of **14** or **15** was not detected by TLC, ^1H NMR, capillary GLC, and/or HPLC analysis

[17] Rearrangement of the epoxy silyl ether **13** (R = Ph) yielded a mixture of **15** and its mono(triphenylsilyl) ether which was directly treated with Bu_4NF in THF at room temperature.

Chemistry of Platinum Hydrides: A Platinum(II) *cis*-Dihydride or a Platinum(0) η^2-Dihydrogen Complex?

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A complete understanding of the interconversion processes involving molecular hydrogen, η^2-dihydrogen species and hydride complexes is essential for the significant development of metal-catalyzed hydrogen chemistry. Recently, stable complexes containing η^2-dihydrogen ligands bonded to early transition metals such as chromium,[1-3] molybdenum,[4,5] tungsten,[4,5] and iron[6] have been reported although for the heavier transition metals comparable species of only ruthenium[6] and iridium[7] have been described. For the iron complex *trans*-$[Fe(\eta^2\text{-}H_2)(H)(dppe)_2]BF_4$, for which the X-ray crystal structure determination shows the coordination of the η^2-dihydrogen ligand to be similar to that in $[W(CO)_3(P\text{-}i\text{-}Pr_3)_2(\eta^2\text{-}H_2)]$, solution NMR spectroscopy suggests an exchange process between monohydride and η^2-dihydrogen ligands. We now report the preparation of a new series of complexes $[(Cy_2P(CH_2)_nPCy_2)PtH_2]$, n = 2, 3, or 4, (**1**, **2**, and **3**, respectively), in which the bulky chelating ligand forces the unusual *cis*-dihydride geometry and for which physical data (dynamic ^1H NMR) provides evidence for a correlated molecular rotation of the hydride ligands about the platinum center and chemical evidence displays easy displacement of dihydrogen. These species are therefore the first heavy-transition-metal dihydrides to provide evidence of dynamic processes involving a *cis*-dihydride ↔ η^2-dihydrogen exchange.

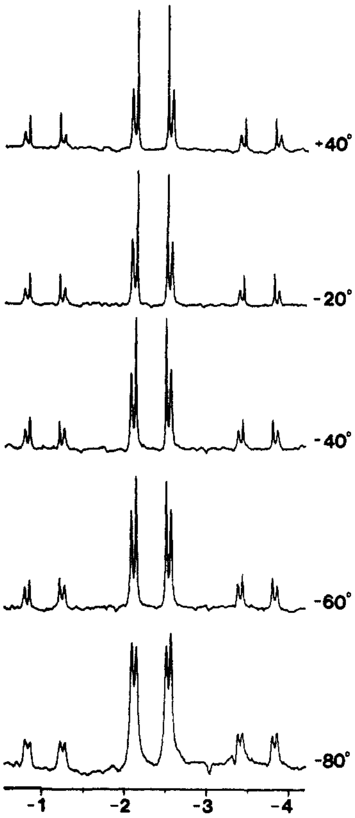

Figure 1. Variable-temperature (°C) ^1H NMR spectra of the hydride resonance of **3** in toluene-d_8 (spectral frequency = 400.13 MHz).

The title compounds may be prepared by subjecting either of the complexes $[(Cy_2P(CH_2)_nPCy_2)Pt^0]$[8a] (**4**) or $[(Cy_2P(CH_2)_nPCy_2)Pt(C_2H_4)]$ (**5**) to a pressure of hydrogen.[8b]

$$[(Cy_2P(CH_2)_nPCy_2)Pt^0] + H_2 \xrightarrow{\text{pressure}} [(Cy_2P(CH_2)_nPCy_2)PtH_2]$$

$$[(Cy_2P(CH_2)_nPCy_2)Pt(C_2H_4)] + H_2 \xrightarrow{\text{pressure}} [(Cy_2P(CH_2)_nPCy_2)PtH_2]$$

If the ethylene complexes **5** are treated with a *flow* of hydrogen gas, the *cis*-dihydride species are formed first with displacement of the coordinated ethylene but lose hydrogen and dimerize to form $[(Cy_2P(CH_2)_nPCy_2)Pt(\mu\text{-}H)]_2$ (**6**) (complete within 1 h for **1**). The same complexes (**6**) are also produced from solutions of the *cis*-dihydride species on standing. The analogous nickel(II) dimer $[(Cy_2P(CH_2)_3PCy_2)Ni(\mu\text{-}H)]_2$ has been prepared and structurally characterized.[10]

On dissolution of complexes **2** and **3** in benzene solution, a dynamic process is observed by NMR spectroscopy involving interchange of ligand position with retention of P–H spin correlation which is typified by the variable-temperature ^1H NMR spectra of the hydride region, Figure 1, for **3**. Such a process is strictly intramolecular. We have recently observed a similar line shape for the species *cis*-$[H(R_3Si)Pt(PCy_3)_2]$[11] (R = variously

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[8] (a) Hexane and benzene solutions of $[(Cy_2P(CH_2)_3PCy_2)Pt^0]$ are very pale orange/yellow and on cooling colorless crystals are obtained which react with, e.g., $HSiPh_3$, to form $[(Cy_2P(CH_2)_3PCy_2)Pt(SiPh_3)H]$. The monomeric nature of **4** is presumed by comparison with dimer $[(t\text{-}Bu_2P(CH_2)_3P\text{-}t\text{-}Bu_2)Pt^0]_2$,[9] which crystallizes as red hexagonal platelets, and from $^{31}P\{^1H\}$ NMR spectra in which no evidence for $^2J_{P-Pt}$ has been observed. (b) Complexes **4** and **5** are prepared by reduction of the corresponding platinum(II) dichlorides $[(Cy_2P(CH_2)_nPCy_2)PtCl_2]$ with 2 equiv of sodium naphthalide and addition of ethylene in the case of **5**.

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