Isomerism of Strain-Free Metallamacrocycle. Dichloropalladium(II) Complex of 1,2-Bis(dimethyl-3-pyridylsilyl)ethane

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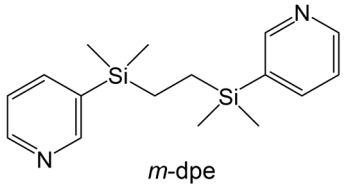
Modulation of the ring size of strain-free metallacyclic compounds *via* thermal energy has been established. The reactions of (COD)$PdCl_2$ with *m*-dpe ligand (COD = 1,5-cyclooctadiene; *m*-dpe = 1,2-bis(dimethyl-3-pyridylsilyl)ethane) in a mixture of acetone and ethanol at room temperature produce the metallacyclodimeric species, $[PdCl_2(m\text{-dpe})]_2$. The cyclodimer, $[PdCl_2(m\text{-dpe})]_2$, at the boiling temperature of the chloroform solution is completely converted to the cyclotrimer, $[PdCl_2(m\text{-dpe})]_3$. In the 0-55 °C range, the cyclotrimer completely returns to the cyclodimer in solution for 24 h. Both the cyclodimer and the cyclotrimer are locked in the solution below 0 °C. The equilibria in the 0-55 °C range are dependent on temperature and concentration.

Key Words : Palladium(II) complexes, Cyclodimer, Cyclotrimer, Interconversion, Isomerism

Introduction

Cyclic isomerism *via* chemical triggers is a hot issue in the construction of molecular machines and switches as well as an aesthetic appeal.[1-5] Especially, various metallamacrocyclic complexes have been studied for their important building blocks in the construction of functional supramolecular materials that can be utilized for molecular recognition, selective transformation, drug delivery systems, catalysts, storage, and biomimics.[6-15] One of facile synthetic methods is the ring-expansion[16-18] including cavity-control[19,20] by means of labile metal-ligand coordination and thermodynamic control. Some palladium(II) complexes of N-donor ligands have been utilized in the development of coordination materials such as catalysts,[21] rectangular building blocks,[22] submicrospheres,[23] desirable morphology,[24] and "magic ring" with the associative/dissociative dual character Pd-N bond.[25] Of the N-donor ligands, silicon-containing pyridyl ligands have been found to be useful for the synthesis of ring-skeletal structures since they are adjustable in bridging ability and length, possess flexible angles around silicon, and are conformationally nonrigid.[26-30] Equilibria of ionic palladium(II) metallacyclic compounds containing NO_3^-, PF_6^-, or OTf^- have been observed,[31,32] but ring-control in neutral palladium(II) complexes is very rare except our previous results. Recently, complete interconversion of $[PdCl_2(p\text{-psb})]_2$ (*p*-psb = 1,4-bis(dimethyl-4-pyridylsilyl)-benzene) into trimer *via* angle constraint was published,[33] but interconversion between macrocyclodimer and macrocyclotrimer containing non-rigid ligands is unprecedented.

In this context, new metallamacrocyclic complex was prepared for the reaction of dichloropalladium(II) with 1,2-bis(dimethyl-3-pyridylsilyl)ethane (*m*-dpe). Herein, we report a unique interconversion process of metallacyclodimers versus metallacyclotrimers *via* temperature effects on the strain-free metallacycles, *m*-dpe is a conformationally non-rigid spacer ligand that possesses a flexible alkyl chain between two pyridine rings, thereby imparting a conformational nonrigidity to the pyridyl groups.[34]



m-dpe

Experimental Section

Materials and Measurements. Palladium(II) chloride, 1,5-cyclooctadiene (COD), and 1,2-bis(chlorodimethylsilyl)ethane were purchased from Aldrich, and used without further purification. (COD)$PdCl_2$ was prepared according to the procedure described in the literature.[35] 1,2-Bis(dimethyl-3-pyridylsilyl)ethane (*m*-dpe) was prepared according to the previous papers.[36,37] ^{1}H NMR spectra were recorded on a Varian Mercury Plus 300 operating at 300.00 MHz, and the chemical shifts were relative to the internal Me_4Si. Infrared spectra were obtained on a Perkin Elmer 16F PC FTIR spectrophotometer with samples prepared as KBr pellets. Elemental microanalyses (C, H, N) were performed on solid samples by the Busan center, KBSI, using a Perkin Elmer 2400 CHNS analyzer. Mass spectrometric analysis *via* a fast atom bombardment technique was performed in chloroform using a KMS-700 Mstation Mass Spectrometer (Jeol, Japan) and an MS-MP9020D data system.

Preparation of $[PdCl_2(m\text{-dpe})]_2$. An acetone solution (7.0 mL) of *m*-dpe (4.36 mg, 0.01 mmol) was slowly diffused into an ethanol solution (7.0 mL) of (COD)$PdCl_2$ (2.85 mg, 0.01 mmol) in a Pyrex glass vial. Yellow crystals of $[PdCl_2(m\text{-dpe})]_2$ suitable for X-ray crystallographic characterization formed at the interface and were obtained in 65% yield after 3 days. Anal. Calcd for $C_{32}H_{48}Cl_4N_4Pd_2Si_4$: C, 40.21; H, 5.06; N, 5.86. Found: C, 40.19; H, 5.09; N, 5.85. ^{1}H NMR

Table 1. Crystal Data and Structure Refinement for $[PdCl_2(m\text{-dpe})]_2$

Empirical formula	$C_{16}H_{24}Cl_2N_2PdSi_2$
Formula weight	477.85
Space group	*C*2/*c*
a, Å	27.540(2)
b, Å	6.9146(5)
c, Å	23.048(2)
b, deg	106.701(2)
V, Å^3	4203.9(5)
Z	8
d_{cal}, gcm^{-3}	1.510
Crystal size (mm^3)	0.20 × 0.20 × 0.10
μ, mm^{-1}	1.251
Completeness to theta = 28.32°	92.7%
Data / restraints / parameters	4854 / 0 / 230
Goodness-of-fit on F^2	1.022
Final R indices [I>2σ(I)]	*R1* = 0.0500, *wR2* = 0.0945
R indices (all data)	*R1* = 0.1004, *wR2* = 0.1095

$R1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$. $wR2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma wF_o^2]^{1/2}$

($CDCl_3$, $SiMe_4$, ppm): 0.34 (s, 24H), 0.74 (s, 8H), 7.23 (t, *J* = 6.3 Hz, 4H), 7.72 (d, *J* = 7.2 Hz, 4H), 8.79 (d, *J* = 5.6 Hz, 4H), 8.86 (s, 4H). Mass: *m/e* = 921.3 $[M_D\text{-Cl}]^+$, calcd 921.0.

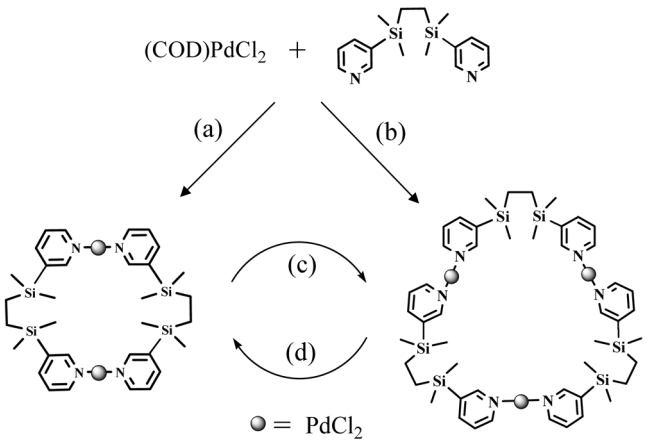
Preparation of $[PdCl_2(m\text{-dpe})]_3$. *Method 1*: a chloroform solution (5 mL) of $[PdCl_2(m\text{-dpe})]_2$ (5.2 mg, 0.005 mmol) was refluxed for 40 h at 63 °C. *Method 2*: direct reaction of $(COD)PdCl_2$ (8.55 mg, 0.03 mmol) in ethanol (15 mL) with *m*-dpe (13.08 mg, 0.03 mmol) in acetone (15 mL) was accomplished at reflux temperature for 24 h. Then, the solvent was evaporated to dryness to yield the thin yellow solid. Anal. Calcd for $C_{48}H_{72}Cl_6N_6Pd_3Si_6$: C, 40.21; H, 5.06; N, 5.86. Found: C, 40.26; H, 5.14; N, 5.89. ^{1}H NMR ($CDCl_3$, $SiMe_4$, ppm): 0.34 (s, 36H), 0.67 (s, 12H), 7.29 (t, *J* = 6.3 Hz, 6H), 7.80 (d, *J* = 7.2 Hz, 6H), 8.71 (d, *J* = 5.6 Hz, 6H), 8.80 (s, 6H). Mass: *m/e* = 1197.1 $[M_T\text{-L-HCl}]^+$, calcd 1198.0; 1063.6 $[M_T\text{-L-2HCl}]^+$, calcd 1064.0.

Crystal Structure Determination. X-ray data were collected on a Bruker SMART automatic diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) and a CCD detector at ambient temperature. Forty five frames of two dimensional diffraction images were collected and processed to obtain the cell parameters and orientation matrix. The data were corrected for Lorentz and polarization effects. The absorption effects were corrected using the empirical ψ-scan method. The structures were solved using the direct method (SHELXS 97) and refined by full-matrix least squares techniques (SHELXL 97).[38] The non-hydrogen atoms were refined anisotropically, and the hydrogen atoms were placed in calculated positions and refined only for the isotropic thermal factors. The chlorides were disordered. The crystal parameters and procedural information corresponding to the data collection and structure refinement are listed in Table 1.

Results and Discussion

Synthesis and Properties. The reaction of $(COD)PdCl_2$ with *m*-dpe ligand in a mixture of acetone and ethanol at room temperature yields discrete cyclodimers, $[PdCl_2(m\text{-dpe})]_2$, in high yield, as shown in Scheme 1. However, the reaction of $(COD)PdCl_2$ with *m*-dpe at reflux temperature gives only a cyclotrimeric species, $[PdCl_2(m\text{-dpe})]_3$. Furthermore, $[PdCl_2(m\text{-dpe})]_2$ at boiling temperature of chloroform solution is exclusively converted to the cyclotrimer, as will be explained in detail. The reactions were originally conducted in the 1:1 mole ratio of Pd(II) to dpe ligand, but the products were not significantly affected by either the mole ratio or the concentrations. The present products are remarkable in that there was no evidence for polymerization, not even under the high concentrations, and despite the lack of protective groups, in contrast to silver(I) complexes.[34] The results of an elemental analysis and the NMR spectra of the products were consistent with the desirable structures. The complex is stable solid which is soluble in common polar organic solvents.

Crystal Structure of $[PdCl_2(m\text{-dpe})]_2$. The crystal structure of $[PdCl_2(m\text{-dpe})]_2$ is depicted in Figure 1, and the



(a) reaction at room temperature
(b) reaction at reflux condition in $CDCl_3$
(c) reflux
(d) cooling

Scheme 1

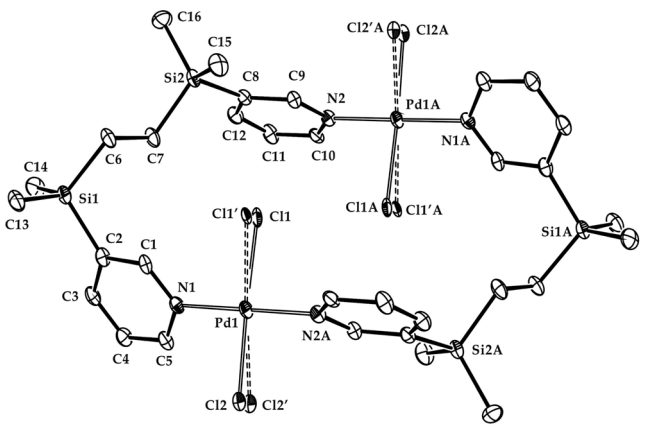


Figure 1. ORTEP view of $[PdCl_2(m\text{-dpe})]_2$. Chlorine atoms were disordered. All hydrogen atoms are omitted for clarity.

Table 2. Bond Lengths [Å] and Angles [°] for $[PdCl_2(m\text{-dpe})]_2$

Pd(1)-N(2)#1	2.009(4)	Pd(1)-N(1)	2.043(4)
Pd(1)-Cl(1)	2.28(2)	Pd(1)-Cl(2)	2.295(7)
Pd(1)-Cl(2')	2.301(8)	Pd(1)-Cl(1')	2.34(2)
N(1)-Pd(1)-Cl(1')	89.5(5)	Cl(2')-Pd(1)-Cl(1')	175.2(6)
N(2)#1-Pd(1)-Cl(1')	91.0(5)	C(14)-Si(1)-C(13)	112.1(3)
N(2)#1-Pd(1)-N(1)	175.8(2)	C(1)-N(1)-C(5)	117.7(4)
N(2)#1-Pd(1)-Cl(1)	86.4(6)	C(1)-N(1)-Pd(1)	122.3(3)
N(1)-Pd(1)-Cl(1)	93.6(6)	C(5)-N(1)-Pd(1)	119.6(4)
N(2)#1-Pd(1)-Cl(2)	88.0(2)	C(9)-N(2)-C(10)	118.7(4)
N(1)-Pd(1)-Cl(2)	91.8(2)	C(9)-N(2)-Pd(1)#1	119.2(3)
Cl(1)-Pd(1)-Cl(2)	174.0(5)	C(10)-N(2)-Pd(1)#1	122.2(3)
N(2)#1-Pd(1)-Cl(2')	87.8(2)	N(1)-C(1)-C(2)	124.8(5)
N(1)-Pd(1)-Cl(2')	92.0(2)	N(1)-C(5)-C(4)	122.3(5)
Cl(1)-Pd(1)-Cl(2')	173.8(5)	N(2)-C(9)-C(8)	124.8(4)
Cl(2)-Pd(1)-Cl(1')	175.3(6)	N(2)-C(10)-C(11)	121.0(5)

Symmetry transformations used to generate equivalent atoms: #1 −x, −y+1, −z.

relevant bond lengths and angles are listed in Table 2. The ORTEP shows that the single crystal consists of centrosymmetric cyclodimers, $[PdCl_2(m\text{-dpe})]_2$. The local geometry around the palladium(II) approximates to a typical square planar arrangement with two chlorides in *trans* position (Cl-Pd-Cl = 174.0(5)°, 173.8(5)°, 175.3(6)°, 175.2(6)°; N-Pd-N = 176(2)°; Pd-Cl = 2.28(2) Å, 2.295(7) Å, 2.301(8) Å, 2.34(2) Å; Pd–N = 2.009(4) Å, 2.043(4) Å). The neutral dpe ligand connects two palladium(II) ions to form a 22-membered macrocyclodimer for $[PdCl_2(m\text{-dpe})]_2$. The ligand acts as a horse-shoe tectonic, which is useful for the construction of molecular rectangles.[28] Cyclodimeric products are discrete molecules with no close intermolecular contacts. No other exceptional features, including those of either bond lengths or angles, were observed, indicating that the metallamacrocycle does not have any significant ring strain.

Isomerism between of $[PdCl_2(m\text{-dpe})]_2$ and of $[PdCl_2(m\text{-dpe})]_3$. The ^{1}H NMR spectra of $[PdCl_2(m\text{-dpe})]_2$ in chloroform at room temperature are consistent with the crystal structures. $[PdCl_2(m\text{-dpe})]_2$ is unusually non-rigid in solution under vigorous conditions. After the crystal was refluxed in chloroform, the signals of the *m*-pyridyl group exhibited two sets of ^{1}H resonances (7.23, 7.72, 8.79 and 8.86 ppm; 7.29, 7.80, 8.76 and 8.80 ppm), confirming the coexistence of two species in the solution. At initial state, the signals at 7.23, 7.72, 8.79 and 8.86 ppm were predominant, and finally the original signals were completely replaced by the signals at 7.29, 7.80, 8.76 and 8.80 ppm with similar splitting patterns as shown in Figure 2, indicating that the $[PdCl_2(m\text{-dpe})]_2$ in chloroform was transformed into a new species. The new, transformed product could be isolated and characterized by elemental analysis, IR, and mass data in addition to ^{1}H NMR. All of the data of the transformed compound are very similar to those of the original cyclodimer except for the mass data. The mass data of $[PdCl_2(m\text{-dpe})]_2$ and the new species were obtained to characterize the new species (Figure 3). As expected, the mass data of

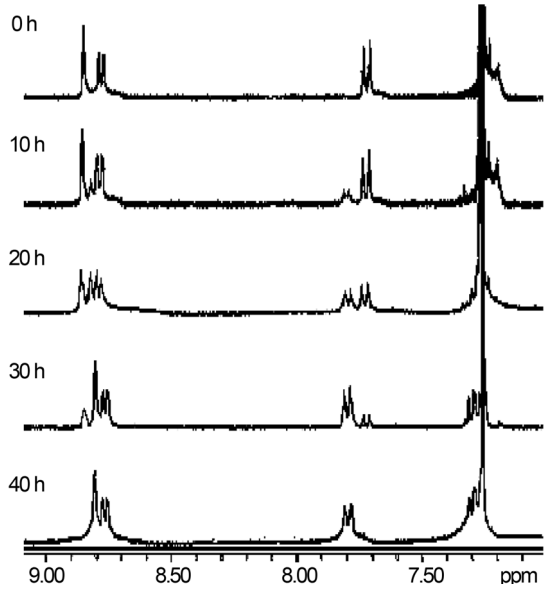


Figure 2. ^{1}H NMR spectral change on a conversion of $[PdCl_2(m\text{-dpe})]_2$ into $[PdCl_2(m\text{-dpe})]_3$ in $CDCl_3$ at 63 °C.

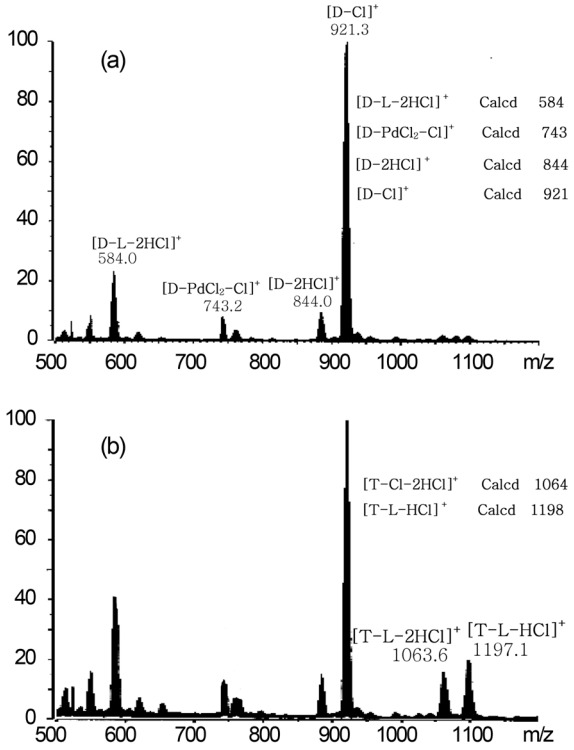


Figure 3. FAB-mass data of $[PdCl_2(m\text{-dpe})]_2$ (a) and $[PdCl_2(m\text{-dpe})]_3$ (b).

$[PdCl_2(m\text{-dpe})]_2$ (*m/e* = 921.3 $[M_D\text{-Cl}]^+$) indicated the dimeric products. In contrast, the main mass peaks of the new species (*m/e* = 1197.1 $[M_T\text{-L-HCl}]^+$; 1063.6 $[M_T\text{-L-2HCl}]^+$) clearly indicate that the transformed new species is a cyclotrimer of $[PdCl_2(m\text{-dpe})]_3$. Of course, the ^{1}H NMR spectrum indicates that the product prepared from the direct reaction at reflux temperature is the same cyclotrimer. Another interesting feature is that in the 0-55 °C range, the cyclotrimer completely returns to the cyclodimer in solution for 24 h.

Such a returning rate is relatively faster than our previous system.[39] In particular, this system completely returns to cyclodimer in contrast to the previous compound. Both the cyclodimer and the cyclotrimer are locked in the solution below 0 °C. The equilibria in the 0-55 °C range are dependent on temperature and concentration. High concentration favors the cyclotrimer. Cyclodimer/cyclotrimer interconversion *via* temperature can be explained by the entropy difference between the two ($3\mathbf{D} \rightleftarrows 2\mathbf{T}$; $\Delta G^o = \Delta H^o - T\Delta S^o$). If H and S favor opposite processes, spontaneity will depend on temperature.[40] The formation of cyclotrimers is predominant above the chloroform reflux temperatures, meaning that the entropy of two cyclotrimer molecules is remarkably higher than that of three cyclodimer molecules in solution at high temperatures (ΔH^o = positive; $T\Delta S^o$ = positive; $\Delta H^o < T\Delta S^o$; ΔG^o = negative). However, in the 0-55 °C temperature range, ΔG^o is positive because the ΔH^o term is larger than the $T\Delta S^o$ term. Thus, the reverse reaction is a spontaneous (ΔH^o = positive; $T\Delta S^o$ = positive; $\Delta H^o > T\Delta S^o$; ΔG^o = positive) one below the reflux temperatures. The cyclotrimer in chloroform below 0 °C is locked, indicating that the interconversion process requires somewhat higher activation energy. Thus, the conversion rate is slow. For the present 22-membered strain-free system, such an interconversion process is unprecedented. The driving force of the interconversion between cyclodimers and cyclotrimers seems to be the dual characters of Pd(II)-N bond and the entropy change rather than angle strain.

Conclusion

The present discrete 22-membered metallacyclic complex containing bidentate 1,2-bis(dimethyl-3-pyridylsilyl)ethane is an effective system for cyclic isomerism of ring strain-free metallacycle. This interconversion presents a synthetic strategy for obtaining successive ring control in metallacyclic compounds. Indeed, this unusual process can contribute to the desirable molecular cyclic materials applicable to sensor technology, transport, catalysis, and molecular switching.

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Supporting Information: IR spectra of $[PdCl_2(m\text{-dpe})]_2$. Crystallographic data for $[PdCl_2(m\text{-dpe})]_2$ is deposited at the Cambridge Crystallographic Data Centre under supplementary publication numbers CCDC-702597. Copies of available material may be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336 033, or e-mail: deposit@ccdc.cam.ac.uk.

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