Isolation and Reactions of a Tantalum−Imine Complex $TaCl_3(dme)(PhCH{=}NCH_2Ph)$

Kazuhiko Takai,* Takaya Ishiyama, Hidekuni Yasue, Takahumi Nobunaka, and Makoto Itoh

Department of Applied Chemistry, Faculty of Engineering, Okayama University, Tsushima, Okayama 700-8530, Japan

Toshiyuki Oshiki, Kazushi Mashima,* and Kazuhide Tani*

Department of Chemistry, Graduate School of Engineering Science, Osaka University, Toyonaka, Osaka 560-8531, Japan

Received May 14, 1998

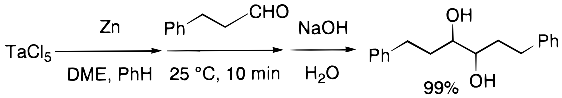
A new tantalum−imine complex $TaCl_3(dme)(PhCH{=}NCH_2Ph)$ (**1**) (DME = 1,2-dimethoxyethane) was synthesized by treatment of *N*-(benzylidene)benzylamine with low-valent tantalum derived from $TaCl_5$ and zinc in DME and toluene. The molecular structure of complex **1** was determined by X-ray crystallographic analysis, and its reactivity toward unsaturated compounds examined.

Introduction

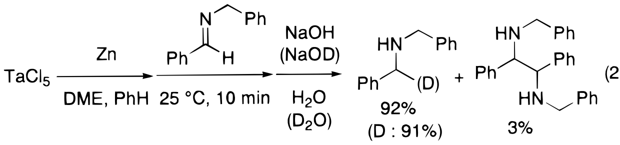
Although tantalum−imine complexes have been postulated as intermediates in several organometallic transformations,[1] only a few have been isolated and crystallographically characterized.[2] In these cases, the complexes were prepared by either transfer of a hydrogen from a tantalum−amine complex[2a] or reduction of a η^2-iminoacyl complex derived by insertion of isocyanide into a tantalum complex.[2b] However, the direct synthesis of a complex from an imine and a low-valent tantalum has not been reported.[3,4] Here we describe the direct synthesis of a tantalum−imine complex by treatment of a benzaldimine with a low-valent tantalum derived from $TaCl_5$ and zinc. The reactions of the complex with carbonyl compounds and an isocyanate are also examined.

Results and Discussion

Synthesis and Characterization of a Tantalum−Imine Complex. We have reported that the addition of an aldehyde to a low-valent tantalum derived from $TaCl_5$ and zinc in DME and benzene at 25 °C smoothly produces a McMurry-type 1,2-diol in an almost quantitative yield after alkaline workup (eq 1).[5] In contrast, treatment of an imine under the same reaction conditions gave a 1,2-diamine in only 3% yield, and almost all of the imine was reduced to the corresponding amine (eq 2). Quenching the reaction mixture with alkaline D_2O afforded a deuterated amine of 91% D content, which suggested the formation of a tantalum−imine complex as an intermediate in the reaction of eq 2. Thus, we attempted to isolate the tantalum−imine complex.



(1)



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A solution of *N*-(benzylidene)benzylamine in DME and toluene (1:1) was added at 25 °C to a low-valent

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10.1021/om980380l CCC: $15.00

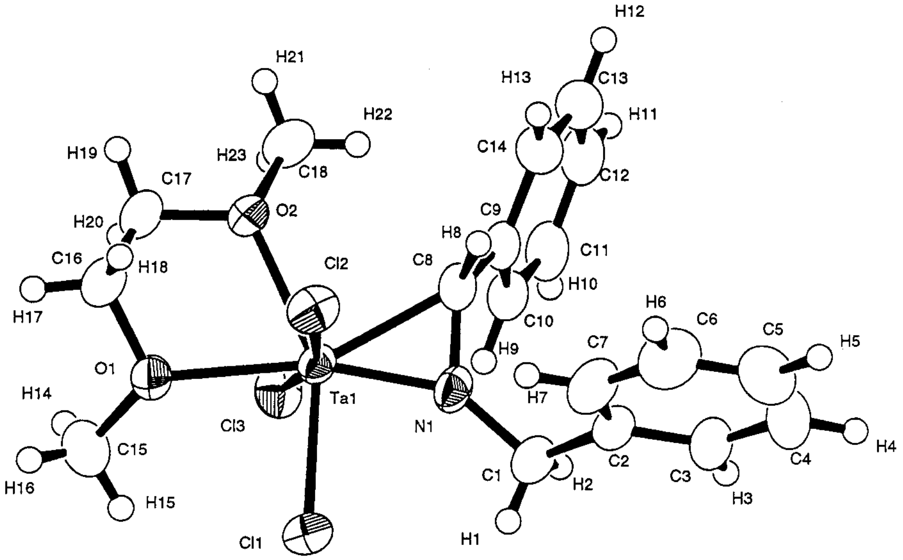


Figure 1. ORTEP drawing of $TaCl_3(dme)(PhCH{=}NCH_2Ph)$ (**1**) showing the atom-numbering scheme.

Table 1. Crystal and Refinement Data for Complex 1

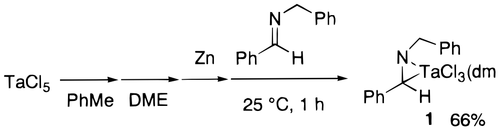
complex	$TaCl_3(dme)(PhCH{=}NCH_2Ph)$ (**1**)
formula	$C_{18}H_{23}Cl_3NO_2Ta$
fw	572.69
cryst syst	monoclinic
space group	$P2_1/n$ (No. 14)
a, Å	10.080(2)
b, Å	12.153(2)
c, Å	16.565(2)
β, deg	91.40(1)
V, Å^3	2028.6(5)
Z	4
D_{calcd}, g cm^{-3}	1.875
radiation	Mo Kα ($\lambda = 0.710\ 69$ Å)
cryst size, mm	0.4 × 0.4 × 0.5
abs coeff, cm^{-1}	58.16
scan mode	$\omega - 2\theta$
temp, °C	20
$2\theta_{max}$, deg	55.0
no. of data collected	5158
no. of unique data	4890 ($R_{int} = 0.057$)
no. of observations ($I > 3\sigma(I)$)	4035
no. of variables	226
R^a	0.030
R_wb	0.041
GOF	1.86
Δ, e Å^{-3}	1.41 (max.) −1.18 (min.)

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$. $w = 1/\sigma^2(F_o)$, Function minimized: $\sum w(|F_o| - |F_c|)^2$.

Table 2. Selected Bond Distances and Angles for 1

Bond Distances (Å)			
Ta(1)−C(8)	2.211(5)	Ta(1)−Cl(1)	2.389(1)
Ta(1)−N(1)	1.914(4)	Ta(1)−Cl(2)	2.383(1)
Ta(1)−O(1)	2.283(4)	Ta(1)−Cl(3)	2.368(1)
Ta(1)−O(2)	2.269(3)	C(8)−N(1)	1.401(6)
Bond Angles (deg)			
Cl(1)−Ta(1)−O(1)	80.74(10)	Cl(1)−Ta(1)−Cl(3)	94.04(5)
Cl(1)−Ta(1)−N(1)	83.4(1)	Cl(2)−Ta(1)−Cl(3)	163.36(5)
O(1)−Ta(1)−O(2)	71.6(1)	Cl(3)−Ta(1)−N(1)	98.8(1)
O(2)−Ta(1)−C(8)	85.7(2)	Cl(3)−Ta(1)−C(8)	98.9(1)
N(1)−Ta(1)−C(8)	38.9(2)	C(1)−N(1)−C(8)	128.9(4)
Cl(1)−Ta(1)−O(2)	152.26(9)		

tantalum derived from $TaCl_5$ (1.2 equiv) and zinc (1.8 equiv) in DME and toluene (1:1) over a period of 1 h. The color of the reaction mixture gradually turned from dark green to red. After the mixture was filtered from solid materials, heptane was slowly added to the filtrate. The solution was stored at −20 °C for 2 days to give the complex $TaCl_3(dme)(PhCH{=}NCH_2Ph)$ (**1**) as dark red crystals in 66% yield (eq 3).



(3)

1 66%

An X-ray diffraction study was carried out on a single crystal of **1**. Details of the structural refinement of the new complex **1** are given in Table 1. The molecular structure of **1** and the selected bond distances and angles are shown in Figure 1 and Table 2, respectively. The complex **1** adopts a seven-coordinate, distorted pentagonal bipyramidal geometry where two chloride ligands (Cl(2) and Cl(3)) occupy two apical positions. Thus, three chloride ligands are arranged in a meridional fashion. The bond angle Cl(2)−Ta(1)−Cl(3) (163.36(5)°) is deviated from 180° as a probable result of steric interactions among the ligands. The sum of the angles around the tantalum center in the pentagonal plane (360.3°) defined by Cl(1), O(1), O(2), C(8), and N(1) is in good accordance with the idealized value 360°. The dihedral angles between the plane of O(1)−Ta(1)−O(2) and that of N(1)−Ta(1)−C(8) is 7.9°. In the imine fragment, the bond angles around C(8) indicate an sp^3 character of C(8); the angles C(9)−C(8)−H(8) (111.8°) and N(1)−C(8)−H(8) (114.8°) are considerably smaller than 120° of the free ligand, respectively, although the N(1)−C(8)−C(9) angle is 120.0(4)°. The Ta(1)−C(8)−N(1) angle (59.1(3)°) is essentially identical with the value reported in $[Yb(\eta^2\text{-}Ph_2CNPh)(hmpa)_3]$ (Yb−C−N = 59.9(9)°)[3g] and $WN(t\text{-}Bu)CMe_2(Me)(Nt\text{-}Bu)[N(t\text{-}Bu)\text{-}CMe{=}CMe_2]$ (W−C−N = 58.9(7)°).[3h] The C(8)−N(1) bond length (1.401(6) Å) of the coordinated imine ligand is a little shorter than that of a typical C−N single bond (1.45 Å)[6] and is comparable with that (1.421(7) Å) of $TaCl_2(O(2,6\text{-}(i\text{-}Pr)_2(C_6H_3))_2(Me_3PCH{=}N(2,6\text{-}(i\text{-}Pr)_2\text{-}$

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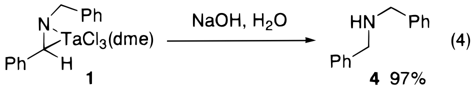
[6] The distance is very similar to those of the zirconium complex, $Cp_2Zr(thf)(PhCHNSiMe_3)$ (1.41(1) Å)[3a] and the titanium complex, $Ti(O(2,6\text{-}(i\text{-}Pr)_2(C_6H_3))_2(4\text{-}Ph(C_5H_4N))((PhCH_2)_2C{=}NBu\text{-}t)$ (1.421(7) Å).[3d]

(C_6H_3))) (**2**),[2b] but is longer than that (1.360(13) Å) of (Tp′TaCl(*t*-BuCH_2)(CH_2=NPh)) (**3**).[2a] The bond distance of Ta(1)–N(1) in **1** is 1.914(4) Å, which is a little shorter than those of **2** (1.932(6) Å) and **3** (1.956(4) Å). The torsion angle of Ta(1)–C(8)–N(1)–C(1) is 179.9(6)°; thus, the benzyl carbon attached to the nitrogen lies in a plane consisting of Ta and the imine C=N.

The ^1H and ^{13}C NMR of **1** were fully consistent with its structure determined by X-ray crystallographic study. In the ^1H NMR spectra, the methyl and methylene protons of the coordinated dme appear as two singlets at δ 4.16 and 4.27 ppm and a multiplet at δ 4.25–4.54 ppm, respectively. The ^{13}C spectrum of **1** indicates η^2-CN binding of the imine fragment. The imine carbon is found to resonate at δ 64.6 ppm, which is reasonable for a carbon atom within an azametallacycle ring.[3,4]

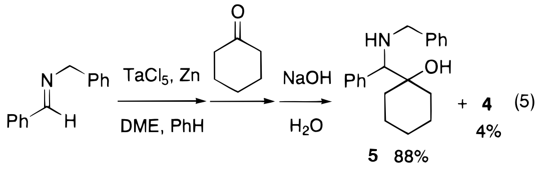
Furthermore, the azametallacycle structure of **1** was also confirmed by IR spectra. The complex **1** in Nujol exhibits characteristic ν(C–N) absorption at 1594 cm^{-1}, which indicates that the imine ligand is bound to the tantalum center in a η^2-fasion. This value is comparable to that observed in the η^2-imine group of the zirconocene complex (1588 cm^{-1})[3a] and shifts to lower frequency than that of the free *N*-(benzylidene)benzylamine ligand (1645 cm^{-1}).

Reactions of Tantalum–Imine Complex 1 with Unsaturated Compounds. Hydrolysis of the imine complex **1** with an alkaline solution produced dibenzylamine (**4**) in an almost quantitative yield (eq 4).[4c]



(4)

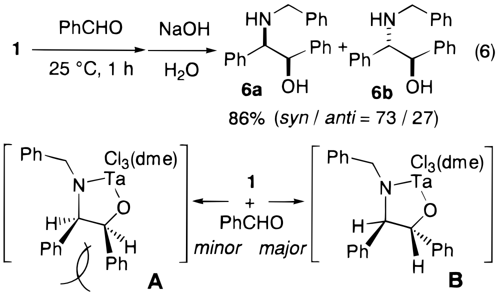
Treatment of the complex **1** with cyclohexanone in DME and toluene (1:1, 0.2 M) at 25 °C for 1 h gave the β-amino alcohol **5** in 94% yield. The coupling of *N*-(benzylidene)benzylamine and cyclohexanone could also be accomplished without isolation of the complex **1**; **5** was produced in 88% yield along with 4% yield of **4** (eq 5).[4a,c] The addition of neither THF nor pyridine, which are required in a similar insertion of carbonyl groups into tantalum–alkyne complexes,[5] was necessary to promote the reaction between the complex **1** and carbonyl compounds.



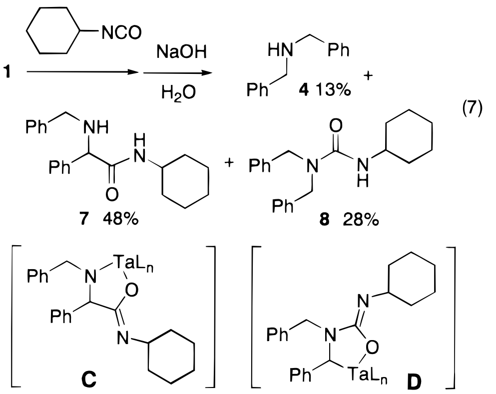
(5)

The complex **1** reacted with benzaldehyde to afford a diastereomeric mixture of β-amino alcohols **6a** and **6b** in 86% combined yields (syn/anti = 73/27) (eq 6). The syn selectivity can be explained by the aldehyde approaching the complex **1** to form a less hindered tantalacycle **B**.

The reaction of **1** with an isocyanate at 25 °C for 12 h gave α-amino amide **7** and urea **8** in 48% and 28% yields, respectively, along with dibenzylamine (**4**) in 13% yield (eq 7). The amide **7** was formed by insertion of the isocyanate into the Ta–C bond (**C**) and the urea **8** into the Ta–N bond of the complex **1** (**D**).[3b] The result is in sharp contrast to the reaction of Ta(NEt_2)$_3$(MeCH=NEt) with isocyanate, where the insertion occurs into the Ta–N bond exclusively.[1a]



(6)



(7)

Experimental Section

General Considerations, Measurements, and Materials. All reactions were carried out under an atmosphere of argon using standard Schlenk techniques. 1,2-Dimethoxyethane, benzene, and toluene were purified by distillation from sodium/benzophenone ketyl; *n*-heptane was purified by distillation from calcium hydride. Infrared spectra were obtained with a Nicolet Protégé 460 spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded on a Varian Gemini-200 or VXR-500 spectrometer. Chemical shifts are reported in δ units downfield from tetramethylsilane in the solvent as the reference signal. Melting points were measured with use of analytically pure samples. Elemental analyses were carried out by using a Perkin-Elmer 2400 CHNS/O analyzer.

Synthesis of TaCl$_3$(dme)(PhCH=NCH$_2$Ph) (1). In a 50 mL reaction flask was placed $TaCl_5$ (0.43 g, 1.2 mmol) under an argon atmosphere. To the salt was added benzene (5 mL) and DME (5 mL) successively at 25 °C. Zinc (0.12 g, 1.8 mmol) was added at 25 °C to a pale yellow solution of $TaCl_5$, and the mixture was stirred at 25 °C for 1 h. The color of the mixture turned to greenish dark blue in a slightly exothermic process. To the mixture was added at 25 °C a solution of *N*-(benzylidene)benzylamine (0.20 g, 1.0 mmol) in DME and benzene (1:1, 2 mL), and the whole mixture was stirred at 25 °C for 1 h. The color of the mixture gradually changed to red. After the mixture was filtered from solid materials, heptane (6 mL) was slowly added to the filtrate. The solution was stored at −20 °C for 2 days to give the complex $TaCl_3$(dme)(PhCH=NCH$_2$Ph) (**1**) as dark red crystals in 66% yield, mp 151–153 °C (DME–toluene–heptane). IR (Nujol/KBr): 1594 cm^{-1}. ^1H NMR (CD_2Cl_2): δ 4.16 (s, 3H), 4.27 (s, 3H), 4.25–4.54 (m, 4H), 5.94 (d, J = 12.8 Hz, 1H), 6.63 (d, J = 12.8 Hz, 1H), 6.85 (t, J = 7.3 Hz, 1H), 6.90–6.98 (m, 2H), 7.18–7.33 (m, 8H). ^{13}C NMR (CD_2Cl_2): δ 60.6, 64.6, 70.3, 72.3, 73.0, 75.7, 125.0, 126.3, 126.7, 127.5, 127.7, 129.3, 137.5, 143.2. Anal. Calcd for $C_{18}H_{23}Cl_3NO_2Ta$: C, 37.75; H, 4.05; N, 2.44. Found: C, 37.41; H, 4.17; N, 2.42.

Crystallographic Data Collection and Structure Determination of 1. (a) Data Collection. A suitable crystal of **1** was mounted in a glass capillary under argon atmosphere and then onto a goniometer head. Data for complex **1** were collected by a Rigaku AFC-7R diffractometer with a graphite-monochromated Mo Kα radiation and a 12 kW rotating anode generator. The incident beam collimator was 1.0 mm, and the crystal-to-detector distance was 235 mm. Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections, corresponded to the cells with dimensions listed in Table 2, where details of the data collection are summarized. The weak reflections ($I < 10\sigma(I)$) were rescanned (maximum of three rescans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. Three standard reflections were chosen and monitored every 150 reflections.

(b) Data Reduction. An empirical absorption correction based on azimuthal scans of several reflections was applied. The data were corrected for Lorentz and polarization effects. Each decay of intensities of three representative reflections was −0.56%, and thus linear correction factors were applied to observed data.

(c) Structure Determination and Refinement. The systematic absence (h 0 l) with $h + l =$ odd and (0 k 0) with k = odd indicated the space group $P2_1/n$ (no. 14). The structure of the complex was solved by Peterson methods (DIRDIF92 PATTY)[7] and refined by the full-matrix least-squares method. Measured nonequivalent reflections with $I > 3.0\sigma(I)$ were used for the structure determination. In the subsequent refinement the function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $|F_o|$ and $|F_c|$ are the observed and calculated structure factor amplitudes, respectively. The agreement indices are defined as $R = \sum||F_o| - |F_c||/\sum|F_o|$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$, where $w^{-1} = \sigma^2(F_o) = \sigma^2(F_o{}^2)/(4F_o{}^2)$. The positions of all non-hydrogen atoms and the hydrogen atom, H(8), were found from a difference Fourier electron density map. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms, except for H(8), were placed in calculated positions (C−H = 0.95 Å) and kept fixed. All calculations were performed using the TEXSAN crystallographic software package, and an illustration was drawn with ORTEP.

***N*,*N*-Dibenzylamine (4).** In a 50 mL reaction flask was placed $TaCl_3(dme)(PhCH{=}NCH_2Ph)$ (**1**, 0.52 g, 0.90 mmol), and toluene (3.7 mL) and DME (3.7 mL) were added at 25 °C. The resulting mixture was stirred at 25 °C for 1 h to give a red suspension. Aqueous solution of NaOH (15%, 0.90 mL) was added to the mixture, and the resulting mixture was stirred at 25 °C for an additional 1 h. The deposited white solid was removed by filtration with a Hyflo-Super Cel and washed well with ethyl acetate (3 × 5 mL). Organic extracts were concentrated in vacuo, diluted with ethyl acetate (10 mL), dried over $MgSO_4$, and concentrated again in vacuo. Purification of the crude product by column chromatography on silica gel (ethyl acetate−hexane, 1:10) gave *N*,*N*-dibenzylamine in 97% yield (0.17 g, 0.87 mmol) as a colorless oil.

2-(*N*-Benzylamino)-1-cyclohexyl-2-phenylethanol (5). In a 50 mL reaction flask was placed $TaCl_5$ (0.43 g, 1.2 mmol) under an argon atmosphere. To the salt was added benzene (5 mL) and DME (5 mL) successively at 25 °C. Zinc (0.12 g, 1.8 mmol) was added at 25 °C to a pale yellow solution of $TaCl_5$, and the mixture was stirred at 25 °C for 1 h. The color of the mixture turned to greenish dark blue in a slightly exothermic process. To the mixture was added at 25 °C a solution of *N*-(benzylidene)benzylamine (0.20 g, 1.0 mmol) in DME and benzene (1:1, 2 mL), and the whole mixture was stirred at 25 °C for 2 h. The color of the mixture gradually changed to red. A solution of cyclohexanone (0.12 g, 1.2 mmol) in DME and benzene (1:1, 2 mL) was added to the mixture at 25 °C, and the resulting mixture was stirred at 25 °C for 1 h. Aqueous NaOH solution (15%, 1.2 mL) was added, and the mixture was stirred at 25 °C for an additional 1 h. The deposited white solid was removed by filtration with a Hyflo-Super Cel and washed well with ethyl acetate (3 × 5 mL). Organic extracts were concentrated in vacuo, diluted with ethyl acetate (10 mL), dried over $MgSO_4$, and concentrated again in vacuo. Purification of the crude product by column chromatography on silica gel (ethyl acetate−hexane, 1:3) gave 2-(*N*-benzylamino)-1-cyclohexyl-2-phenylethanol in 88% yield (0.27 g, 0.88 mmol) as a colorless oil, bp 155 °C (bath temp, 0.9 Torr); IR (neat/NaCl): 3442, 3061, 3026, 2933, 2855, 1603, 1494, 1453, 1250, 1153, 1071, 1029, 969, 860, 739, 700, 668 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.90−1.81 (m, 10H), 2.01−3.20 (br, 2H), 3.49 (t, J = 6.5 Hz, 2H), 3.69 (d, J = 12.9 Hz, 1H), 7.22−7.41 (m, 5H). ^{13}C NMR ($CDCl_3$): δ 21.5, 21.8, 25.8, 32.3, 35.5, 51.7, 71.3, 72.8, 127.0, 127.3, 128.1, 128.3, 128.6, 139.7, 140.2. Anal. Calcd for $C_{20}H_{25}NO$: C, 81.31; H, 8.53; N, 4.74. Found: C, 80.96; H, 8.66; N, 4.62.

2-(*N*-Benzylamino)-1,2-diphenylethanol (6).[8] In a 50 mL reaction flask was placed **1** (0.50 g, 0.88 mmol) under an argon atmosphere, and toluene (3.6 mL) and DME (3.6 mL) were added at 25 °C. The resulting mixture was stirred at 25 °C for 1 h to give a red suspension. A solution of benzaldehyde (0.11 g, 1.1 mmol) in DME and benzene (1:1, 2.4 mL) was added to the reaction mixture at 25 °C. After being stirred at 25 °C for 1 h, an aqueous NaOH solution (15%, 0.88 mL) was added to the mixture, and the resulting mixture was stirred at 25 °C for an additional 1 h. The deposited white solid was removed by filtration with a Hyflo-Super Cel and washed well with ethyl acetate (3 × 5 mL). Organic extracts were concentrated in vacuo, diluted with ethyl acetate (10 mL), dried over $MgSO_4$, and concentrated again in vacuo. Purification of the crude product by column chromatography on silica gel (ethyl acetate−hexane, 1:1) gave 2-(*N*-benzylamino)-1,2-diphenylethanol in 86% yield (0.23 g, 0.76 mmol, syn/anti = 73/27) as a colorless oil. **(*R**,*R**)-Isomer (6a):** mp 125−126 °C; IR (neat/NaCl) 3061, 3028, 1601, 1495, 1453, 1377, 1236, 1196, 1110, 1038, 1028. cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.21−3.20 (bs, 2H), 3.50−4.02 (m, 3H), 4.60 (d, J = 8.5 Hz, 1H), 7.03−7.42 (m, 15H); ^{13}C NMR ($CDCl_3$) δ 51.2, 69.5, 126.8, 127.1, 127.4, 127.5, 127.8, 127.8, 128.2, 128.3, 128.4, 139.6, 141.0. **(*R**,*S**)-Isomer (6b):** mp 147−148 °C; IR (neat/NaCl) 3061, 3028, 1601, 1495, 1453, 1377, 1236, 1196, 1110, 1038, 1028. cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.21−3.20 (bs, 2H), 3.50−4.02 (m, 3H), 4.82 (d, J = 6.0 Hz, 1H), 7.03−7.42 (m, 15H); ^{13}C NMR ($CDCl_3$) δ 50.9, 67.7, 126.8, 126.9, 127.5, 127.5, 128.0, 128.0, 128.1, 128.1, 128.3, 139.1, 140.4.

Reaction of Complex 1 with Cyclohexyl Isocyanate. In a 50 mL reaction flask was placed complex **1** (0.55 g, 0.96 mmol) under an argon atmosphere, and toluene (4 mL) and DME (4 mL) were added at 25 °C. The resulting mixture was stirred at 25 °C for 1 h to give a red suspension. A solution of cyclohexyl isocyanate (0.14 g, 1.2 mmol) in DME and benzene (1:1, 2.4 mL) was added to the reaction mixture at 25 °C. After being stirred at 25 °C for 12 h, an aqueous NaOH solution (15%, 0.96 mL) was added to the mixture, and the resulting mixture was stirred at 25 °C for an additional 1 h. The deposited white solid was removed by filtration with a Hyflo-Super Cel and washed well with ethyl acetate (3 × 5 mL). Organic extracts were concentrated in vacuo, diluted with ethyl acetate (10 mL), dried over $MgSO_4$, and concentrated

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again in vacuo. Purification of the crude product by column chromatography on silica gel (ethyl acetate−hexane, 1:1) gave 2-benzylamino-*N*-cyclohexyl-2-phenylethanamide in 48% yield (0.15 g, 0.46 mmol, white solid), *N*,*N*-dibenzyl-*N*′-cyclohexylurea in 28% yield (87 mg, 0.27 mmol, white solid), and **4** in 13% yield (14 mg, 0.07 mmol), respectively. **2-Benzylamino-*N*-cyclohexyl-2-phenylethanamide (7):** mp 116−117 °C; IR (Nujol/NaCl) 3327, 2937, 2853, 2360, 1628, 1542, 1454, 1349, 1257, 1099, 1029, 892, 743, 696 cm^{-1}; ^{1}H NMR (CDCl$_3$): δ 1.20−2.00 (m, 11H), 3.78 (s, 3H), 4.20 (s, 1H), 7.11−7.34 (m, 10H); ^{13}C NMR (CDCl$_3$) δ 24.5, 25.3, 32.8, 47.4, 52.4, 66.8, 127.1, 127.8, 128.0, 128.4, 128.5, 139.2, 139.4, 170.6. Anal. Calcd for $C_{21}H_{26}N_2O$: C, 78.22; H, 8.13; N, 8.69. Found: C, 78.46; H, 8.40; N, 8.59. ***N*,*N*-Dibenzyl-*N*′-cyclohexylurea (8):** mp 137−138 °C; IR (Nujol/NaCl) 3320, 2934, 2854, 2361, 1608, 1548, 1496, 1455, 1413, 1353, 1252, 1236, 1085, 744, 699 cm^{-1}; ^{1}H NMR (CDCl$_3$) δ 0.85−1.99 (m, 11H), 4.48 (s, 4H), 7.23−7.34 (m, 10H); ^{13}C NMR (CDCl$_3$) δ 24.7, 25.5, 33.5, 49.3, 50.3, 127.2, 127.4, 128.7, 137.8, 157.8. Anal. Calcd for $C_{21}H_{26}N_2O$: C, 78.22; H, 8.13; N, 8.69. Found: C, 78.05; H, 8.29; N, 8.67.

Acknowledgment. This work was financially supported by a Grant-in-Aid for Science Research from the Ministry of Education, Science, Sports, and Culture, Japan.

Supporting Information Available: Crystallographic data of the complex **1** (18 pages). Ordering information is given on any current masthead page.

OM980380L