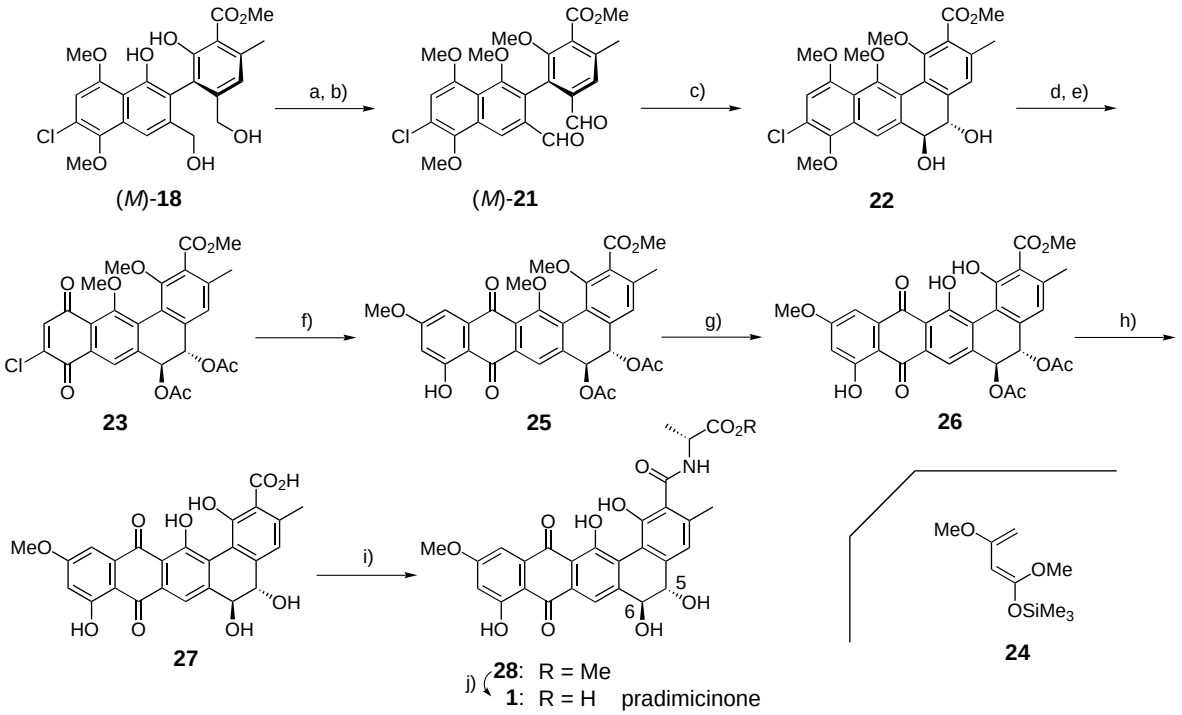

Scheme 5. Total synthesis of pradimicinone (**1**). a) MeI, K_2CO_3/acetone, 40 °C, 11 h (81 %); b) MnO_2/CH_2Cl_2, 24 h (79 %); c) SmI_2/THF, 0 °C, 5 min (quant.); d) Ac_2O, DMAP/pyridine, 0.5 h (quant.); e) $Ce(NH_4)_2(NO_3)_6/CH_3CN$, H_2O, 0 °C, 5 min (quant.); f) **24**/THF, 0 °C → room temperature, 2 h; SiO_2, 12 h, then K_2CO_3/CH_2Cl_2, THF, 4 h (90 %); g) BCl_3/CH_2Cl_2, −10 °C, 30 min (99 %); h) 2 M NaOH (aq), 70 °C, 2 h; H_3O^+; i) D-Ala-OMe · HCl, BOP, Et_3N/DMF, 1.5 h (2 steps, 80 %); j) 0.1 M NaOH, 15 min; H_3O^+ (quant.). BOP = benzotriazol-1-yloxytris(dimethylamino)phosphonium hexafluorophosphate.

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[14] That the diastereomer **20a** has the requisite chirality (*M*) was judged by the CD spectra of the two enantiomers of **18**, derived from **20a** and **20b**, respectively.

[15] Determined by HPLC analysis (DAICEL CHIRALCEL OD-H (25 cm, 0.46 cm diameter), hexane/*i*PrOH 9/1).

[16] Prepared by the degradation of benanomicin A,[1] kindly provided by Meiji Seika, Ltd. The ^{1}H NMR spectra of **28** (and **1**) are highly dependent on concentration, pH, temperature, and other factors, which makes their identification difficult. However, ^{1}H NMR measurement on a mixed sample of synthetic and authentic materials fully coincided.

[17] The same sequence of conversions was also applied to racemic **18**. In the samples of **28** (and **1**) thus obtained, additional peaks in the ^{1}H NMR spectra were observed arising from the 5,6-bis-epimer (relative to the D-alanine moiety).

Enantiomerically Pure Cyclic *trans*-1,2-Diols, Diamines, and Amino Alcohols by Intramolecular Pinacol Coupling of Planar Chiral Mono-Cr(CO)$_3$ Complexes of Biaryls**

Nobukazu Taniguchi, Takeshi Hata, and Motokazu Uemura*

Enantiomerically pure 1,2-diols, diamines, and amino alcohols have found widespread use as chiral ligands in asymmetric reactions.[1] Although a reductive coupling of carbonyl or imine compounds, pinacol coupling, is the most direct way to synthesize 1,2-diols or diamines, highly stereoselective formation of these compounds is problematic.[2] We

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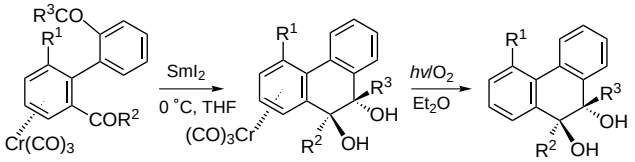
report the stereoselective synthesis of cyclic *trans*-1,2-diols, diamines, and amino alcohols in enantiomerically pure form by an intramolecular pinacol coupling of the planar chiral mono-$Cr(CO)_3$ complexes of biphenyls with dicarbonyl, diimino, or iminocarbonyl groups.

Optically pure mono-$Cr(CO)_3$-complexed biphenyls as starting materials were prepared by palladium-catalyzed cross-coupling of the planar chiral tricarbonyl(bromobenzene)chromium complexes with phenylboronic acids according to a known procedure.[3] This cross-coupling reaction gave the $Cr(CO)_3$-complexed biaryls in high selectivity depending on the *ortho* substituent of arylboronic acids. Thus, *o*-formylphenylboronic acid was coupled with the planar chiral tricarbonyl(2,6-disubstituted 1-bromobenzene)chromium complexes[4] to afford the thermodynamically more stable products, in which the formyl group of the chromium-uncomplexed phenyl ring is oriented *anti* to the $Cr(CO)_3$ moiety.

An intramolecular pinacol coupling[5] of enantiomerically pure tricarbonyl(2,2′-diformyl-1,1′-biphenyl)chromium (**1**, $R^1=R^2=R^3=H$) with samarium diiodide in THF at 0 °C afforded the chromium-complexed 9,10-*trans*-dihydroxy-9,10-dihydrophenanthrene (**2**, $R^1=R^2=R^3=H$) in 81% yield without any formation of stereoisomers (Scheme 1; Table 1, entry 1). The stereochemistry of the pinacol cyclization product **2** was determined by X-ray crystallography[6] of the corresponding racemic diacetate chromium complex. The proximal benzylic acetoxyl group to the chromium-complexed phenyl ring was found to be *anti* to the $Cr(CO)_3$ moiety, and the distal benzylic acetoxyl group was oriented *syn* (Figure 1). Photo-oxidative demetalation of **2** in diethyl ether gave (−)-(*S,S*)-9,10-dihydroxy-9,10-dihydrophenanthrene (**3**,[7] $R^1=R^2=R^3=H$) in a quantitative yield.



1 **2** **3**

Scheme 1. Synthesis of enantiomerically pure *trans*-1,2-diols **3**.

Similarly, the mono-$Cr(CO)_3$-complexed biphenyls with keto-aldehyde functionality at the 2- and 2′-positions produced stereoselectively the corresponding cyclic *trans*-diols under the same reaction conditions (Table 1, entries 3–5). This cyclic *trans*-1,2-diol formation is in sharp contrast to the intramolecular reductive cyclization of 1,5- or 1,6-dicarbonyl compounds giving the corresponding five- or six-membered *cis*-diols.[8] Suzuki et al. have found independently that axially chiral biaryldicarbaldehydes without the $Cr(CO)_3$ moiety also produced *trans*-1,2-diols by the intramolecular pinacol cyclization.[9] Although either chromium-complexed or chromium-free biphenyls having the carbonyl group at the 2- and 2′-positions produced the stereochemically identical cyclic *trans*-1,2-diols by the intramolecular pinacol cyclization, the reductive coupling using the planar chiral $Cr(CO)_3$-complexed biaryls is a significant procedure for the preparation of enantiomerically pure cyclic *trans*-1,2-diols if the biaryl substrates such as 2,2′-diformyl-1,1′-biphenyl have no axial chirality or undergo easily axial isomerization.

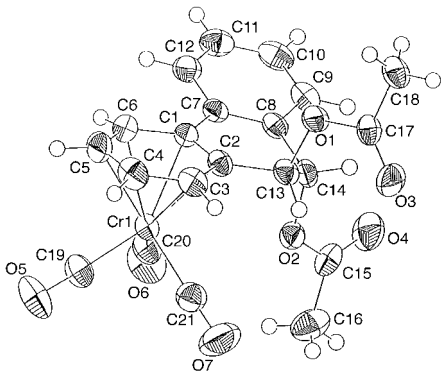
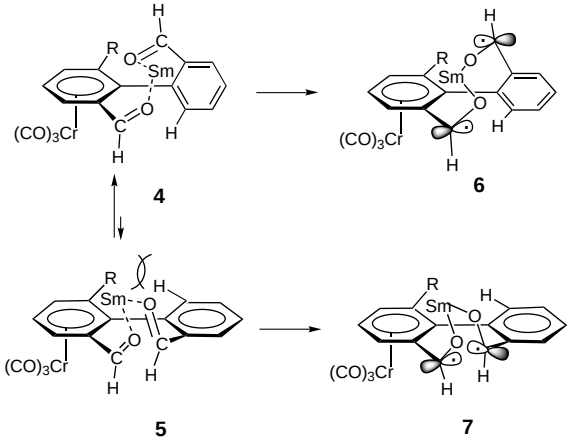


Figure 1. X-ray structure of the racemic diacetate of **2** ($R^1=R^2=R^3=H$).

A plausible reaction mechanism of the pinacol coupling would be as follows (Scheme 2). Samarium diiodide approaches from the *exo* side of the $Cr(CO)_3$ moiety, generating a bis-ketyl radical intermediate. Among two possible inter-



Scheme 2. Plausible reaction mechanism.

Table 1. Intramolecular pinacol coupling of mono-$Cr(CO)_3$ complexes of 2,2′-dicarbonyl-1,1′-biphenyls (**1**, see Scheme 1).

Entry	R^1	R^2	R^3	**2**		**3**	
				yield [%]	$[\alpha]_D$ (c)[a]	yield [%]	$[\alpha]_D$ (c)[b]
1	H	H	H	81	+95.4 (0.44)	98	−81.9 (0.20)
2	OMe	H	H	85	+66.8 (0.38)	96	−80.0 (0.26)
3	H	Me	H	82	+200.9 (0.21)	97	−168.7 (0.20)
4	H	H	Me	82	+62.8 (0.24)	97	−167.9 (0.21)
5	OMe	Me	H	79	+235.3 (1.08)	97	−229.0 (0.42)

[a] In $CHCl_3$. [b] In EtOH.

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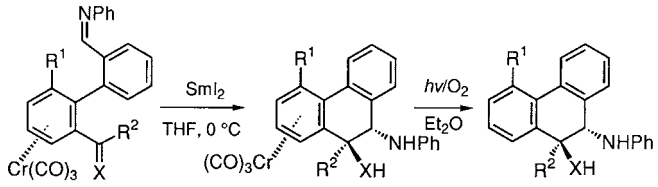
mediates with samarium metal chelating both carbonyl groups, the transition state **4** would be more favorable because the two arene rings are required to be nearly coplanar for formation of the alternative transition state **5**, in which severe steric hindrance between the substituents at the 6- and 6′-positions is present. Therefore, the *trans*-1,2-diol chromium complexes **2** would be formed by the coupling of biradical intermediate **6** generated by an attack of the samarium to both *re* faces of aldehyde carbonyl groups.

This intramolecular pinacol coupling can be further applied for the preparation of optically pure six-membered *trans*-1,2-diamines and amino alcohols (Scheme 3, Table 2). Thus, the



8 **9** **10**

Scheme 3. Synthesis of enantiomerically pure *trans*-1,2-diamines and amino alcohols **10** (X = NPh, O).

mono-$Cr(CO)_3$ complex of diiminobiphenyl **8** ($R^1 = R^2 = H$, X = NPh)[10] produced a single *trans*-1,2-diamine derivative **9** under the same reaction conditions (entry 1). The relative stereochemistry of the diaminochromium complex **9** (R^1 = OMe, $R^2 = H$, X = NPh) is shown in Figure 2. The benzylic

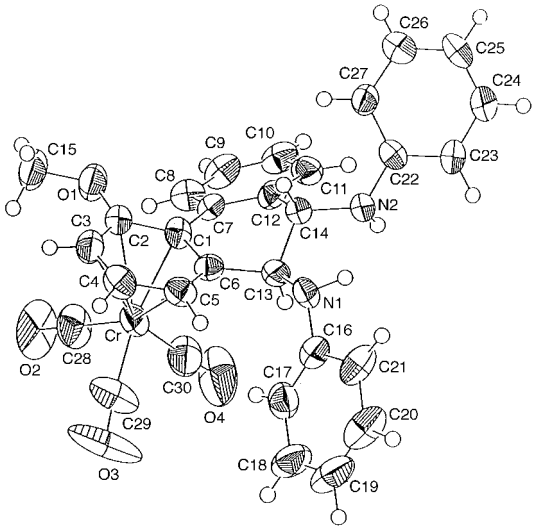


Figure 2. X-ray structure of racemic **9** (R^1 = OMe, $R^2 = H$, X = NPh).

nitrogen atom adjacent to the chromium-complexed arene is also *anti* to the $Cr(CO)_3$ moiety.[6] The diamino complex **9** was exposed to sunlight to give the corresponding chromium-free *trans*-1,2-diamine **10** ($R^1 = R^2 = H$, X = NPh). Similarly, the tricarbonylchromium complexes of 2-acyl-2′-phenylimino-1,1′-biphenyl produced stereoselectively 9-*exo*-hydroxy-10-*endo*-anilino chromium complexes **10** under the same reaction conditions (entries 3 and 4).

In conclusion, we have demonstrated that planar chiral mono-$Cr(CO)_3$-complexed biaryls with carbonyl or imino groups at the 2- and 2′-positions produced stereoselectively cyclic *trans*-1,2-diols, amino alcohols, or diamines in optically pure form by the samarium diiodide mediated intramolecular pinacol coupling. These enantiomerically pure 1,2-diols, diamines, and amino alcohols would be expected to be significant compounds for asymmetric reactions.

Experimental Section

Typical procedure for pinacol coupling: A solution of SmI_2 (0.1M, 5.7 mL, 0.57 mmol) in THF was added to a solution of **1** ($R^1 = R^2 = R^3 = H$; 80 mg, 0.23 mmol) in THF (0.5 mL) at 0 °C by syringe under argon. The reaction mixture was stirred for 30 min at 0 °C, and quenched with saturated aqueous NH_4Cl. The precipitate was filtrated, and the filtrate was extracted with Et_2O. The extract was washed with brine, dried over $MgSO_4$, and evaporated under reduced pressure. The crude product was purified by chromatography on SiO_2 to afford 65 mg (81 %) of the $Cr(CO)_3$-complexed *trans*-1,2-diol **2** ($R^1 = R^2 = R^3 = H$). M.p. 102 °C; $[\alpha]_D^{25} = +95.4$ ($c = 0.44$ in $CHCl_3$); ^1H NMR (270 MHz, $CDCl_3$): $\delta = 2.16$ (br, 2H, O*H*), 4.74 (br, 2H, ArC*H*), 5.43 (t, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 5.47 (d, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 5.89 (d, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 6.02 (t, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 7.34 – 7.60 (m, 4H, Ar*H*); IR ($CHCl_3$): $\tilde{\nu} = 3200, 1940, 1860\ cm^{-1}$; elemental analysis calcd for $C_{17}H_{12}O_5Cr$: C 58.63, H 3.47; found: C 58.90, H 3.76.

9 ($R^1 = R^2 = H$, X = NPh)**:** yellow liquid; $[\alpha]_D^{26} = +45.3$ ($c = 0.76$ in $CHCl_3$); ^1H NMR (270 MHz, $CDCl_3$): $\delta = 3.87$ (d, 1H, $J = 8.1$ Hz, N*H*), 4.20 (d, 1H, $J = 8.1$ Hz, N*H*), 4.64 (t, 1H, $J = 8.0$ Hz, Ar(Cr)C*H*), 4.80 (t, 1H, $J = 8.0$ Hz, ArC*H*), 5.31 (t, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 5.58 (t, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 5.80 (d, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 5.89 (d, 1H, $J = 6.0$ Hz, Ar(Cr)*H*), 7.11 – 7.65 (m, 14H, Ar*H*); IR ($CHCl_3$): $\tilde{\nu} = 3200$, 1960, 1870 cm^{-1}; elemental analysis calcd for $C_{29}H_{22}O_3N_2Cr$: C 69.87, H 4.45, N 5.61; found: C 69.70, H 4.16, N 5.52.

Photooxidative demetalation: A solution of **2** ($R^1 = R^2 = R^3 = H$; 100 mg, 0.29 mol) in Et_2O (5 mL) was exposed to sunlight for 1 h. The precipitate was filtrated, and the filtrate was reduced in vacuo. The residue was purified by column chromatography on SiO_2 (Et_2O/hexane 1/1) to give **3** ($R^1 = R^2 = R^3 = H$; 60 mg, 98 %). M.p. 155 °C; $[\alpha]_D^{27} = -81.9$ ($c = 0.20$ in EtOH); ^1H NMR (270 MHz, $CDCl_3$): $\delta = 2.58$ (br, 2H, O*H*), 4.75 (s, 2H, ArC*H*), 7.25 – 7.44 (m, 4H, Ar*H*), 7.67 – 7.77 (m, 4H, Ar*H*); IR ($CHCl_3$): $\tilde{\nu} = 3300\ cm^{-1}$; elemental analysis calcd for $C_{14}H_{12}O_2$: C 79.22, H 5.70; found: C 79.35, H 5.49.

10 ($R^1 = R^2 = H$, X = NPh)**:** colorless liquid; $[\alpha]_D^{26} = +163.2$ ($c = 0.30$ in $CHCl_3$); ^1H NMR (270 MHz, $CDCl_3$): $\delta = 3.88$ (br, 2H, N*H*), 4.78 (s, 2H, ArC*H*), 6.67 (d, 4H, $J = 8.2$ Hz, Ar*H*), 6.75 (t, 2H, $J = 8.2$ Hz, Ar*H*),

Table 2. Pinacol coupling of mono-$Cr(CO)_3$ complexes of diiminobiphenyl and iminocarbonylbiphenyl (**8**, see Scheme 3).

Entry	R^1	R^2	X	**9**		**10**	
				yield [%]	$[\alpha]_D$ (c)[a]	yield [%]	$[\alpha]_D$ (c)[a]
1	H	H	NPh	86	+45.3 (0.44)	97	+163.2 (0.20)
2	OMe	H	NPh	75	+58.1 (0.38)	97	+141.2 (0.26)
3	H	Me	O	77	+96.8 (0.21)	98	−70.0 (0.20)
4	OMe	Me	O	78	+169.1 (1.08)	98	−41.4 (0.42)

[a] In $CHCl_3$.

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7.16 – 7.45 (m, 10H, Ar*H*), 7.84 (d, 2H, $J = 8.2$ Hz, Ar*H*); IR ($CHCl_3$): $\tilde{\nu} =$ 3200, 1360 cm^{-1}; HR-MS ($C_{26}H_{22}N_2$): calcd: 362.1770; found: 362.1766.

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[10] The diimino complex **8** ($R^1 = R^2 = H$, X = NPh) was obtained from **1** and aniline.

Imprint Coating: A Novel Synthesis of Selective Functionalized Ordered Mesoporous Sorbents**

Sheng Dai,* Mark C. Burleigh, Yongsoon Shin, Charles C. Morrow, Craig E. Barnes, and Ziling Xue

Dedicated to Professor T. F. Williams

Polymerization of metal alkoxides in the presence of molecular assemblies of surfactants or related substances as structure directors has resulted in several novel classes of mesoporous and macroporous inorganic materials with extremely high surface areas and ordered mesostructure.[1–4] These materials have now found extensive applications as catalyst supports and chromatographic resins.[4] Recently, Feng et al.[5] and Mercier and Pinnavaia[6] have developed new, effective mesoporous sorbents based on mesoporous materials as supports for the removal of toxic metal ions. The essence of their methodology is to coat surfaces of hexagonally packed mesoporous silica with organic functional groups to enhance their affinities for the targeted metal ions. High capacities and fast kinetics have been observed for these new sorbents.[5–7] The selectivity of these materials relies solely on the affinity of the surface-coated functional ligand for a specific metal ion, with no consideration of the stereochemical interactions between the ligand and metal ion. However, the stereochemical arrangement of the ligand with respect to the targeted metal ion plays a key role in molecular recognition[8] and dative bond formation between the toxic metal ion and coordinating ligands.[5]

We have been interested in the development of sol – gel sorbent materials based upon the potential superior performance offered by molecular imprinting.[9] Herein, we describe a design strategy for imprint-coated, functionalized ordered mesoporous sorbents through surface molecular imprinting. This coating methodology allows precise control of the stereochemical arrangement of ligands on the surfaces of mesopores, which in turn optimizes the binding of a targeted metal ion.

Imprinting methods based on the template approach have been used in cross-linked polymers, as well as in silica gels, to prepare polymeric supports that possess organized solid-state

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