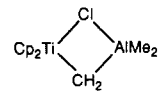
the α-glucopyranosyl ester 4[11] (Scheme I). This transesterification[12,13] proceeded with good α-stereoselectivity at −30 °C (α:β = 9:1) and superior selectivity at −78 °C *when only the α-anomer was produced* (α:β > 50:1). Much to our chagrin, attempts to convert the ester **4** into the vinyl ether **5** by methylenylation[5] using the Tebbe reagent **9** were unsuccessful. However methylenylation using the Nozaki–Takai protocol[14] readily afforded alkene **5**. Alkene **5** was desilylated with tetrabutylammonium fluoride on silica and the resultant alcohol cyclized by using iodine and base *in the presence of silica to provide only the β-D-fructofuranosyl system* **6**. This remarkable and fortuitous diastereoselectivity requires further comment. When the iodoetherification was carried out without silica present both **6** (36%) and **10** (34%) were formed. Additionally, in a blank experiment, **10** was not destroyed under the iodoetherification conditions in the presence of silica. It appears, therefore, that the silica kinetically biases the stereochemistry of cyclization. This effect of silica may be of use in other iodoetherification reactions.



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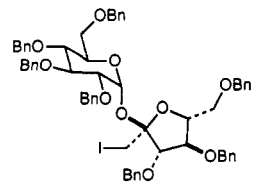

10

Much to our disappointment the iodide **6** proved to be refractory toward S_N2 displacement by oxygen-centered nucleophiles even under forcing conditions. However radical-mediated substitution[15] gave the hydroxylamine **7**. Global deprotection via dissolving metal reduction gave sucrose, which was conveniently isolated as the octaacetate **8**.[16] Finally Zemplen methanolysis regenerated sucrose **1**.[17]

These results clearly demonstrate the validity of redox glycosidation for the highly stereoselective elaboration of sucrose. The application of the chemistry to more complex systems and mechanistic studies of the origin of stereocontrol are currently under investigation.

Acknowledgment. We thank the National Institutes of Health (Grant GM-40949) and G.D. Searle and Company for support of our research; the National Institutes of Health (Grants RR-02314 and RR-03245) for the purchase of a 400-MHz NMR spectrometer and a high resolution mass spectrometer used in these studies; FRD DSIR, Pretoria for financial support to B.C.B.B.; and Drs. Amy R. Howell and Mark A. Russell for preliminary studies on the preparation of **4**.

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(7) For a discussion of alternative nonclassical approaches to prepare glycosides, see: Briner, K.; Vasella, A. *Helv. Chim. Acta* **1989**, *72*, 1371 and references therein.
(8) For earlier studies on the synthesis of sucrose, see: Lemieux, R. U.; Huber, G. *J. Am. Chem. Soc.* **1953**, *75*, 4118. Tsuchida, H.; Komoto, M. *Agric. Biol. Chem.* **1963**, *29*, 239. Ness, R. K.; Fletcher, H. G., Jr. *Carbohydr. Res.* **1971**, *17*, 465. Fraser-Reid, B. *Int. Symp. Carbohydr. Chem. VIIth, Bratislava*, **1974**. Khan, R. *Adv. Carbohydr. Chem. Biochem.* **1976**, *33*, 235. Iley, D. E.; Fraser-Reid, B. *Can. J. Chem.* **1979**, *57*, 645.
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(11) All new compounds were fully authenticated by spectroscopic data and microanalyses or HRMS.
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(16) The product was identical with authentic sucrose octaacetate (Aldrich) (TLC, mp, $[\alpha]_D$, IR, 400-MHz ^{1}H NMR, 101-MHz ^{13}C NMR, HRMS).
(17) The product was identical with authentic sucrose (TLC, mp $[\alpha]_D$, IR, 400-MHz ^{1}H NMR, 101-MHz ^{13}C NMR, HRMS).

$SnCl_4$ Chelation of an *N*-Acyloxazolidinone: An NMR Investigation[1]

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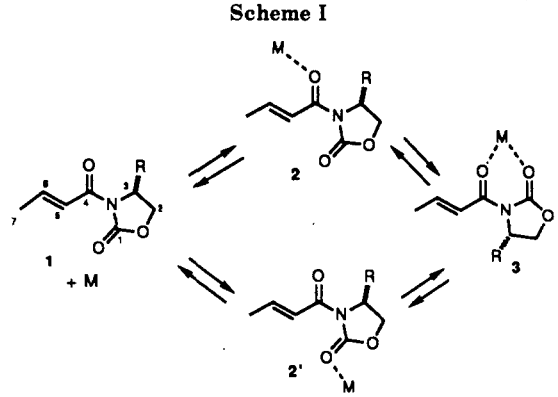
Summary: The $SnCl_4$ chelate of oxazolidinone, **1** (R = isopropyl), has been characterized by ^{119}Sn, ^{13}C, and ^{1}H NMR.

The *N*-acyloxazolidinones, **1**, pioneered by Evans as an asymmetric template, incorporate all the important design features necessary for efficient asymmetric synthesis.[2] The use of neutral Lewis acids to enhance reactivity and stereoselectivity in Diels–Alder additions between **1** and cyclopentadiene has also been examined by Evans.[2b] Our interests in the structure and reactivity of Lewis acid

Scheme I



(1) Presented at the 199th National Meeting of the American Chemical Society, Organic Division, paper 25, Boston, April 22, 1990.
(2) (a) Evans, D. A.; Clark, J. S.; Metternich, R.; Novack, V. J.; Sheppard, G. S. *J. Am. Chem. Soc.* **1990**, *112*, 866. (b) Evans, D. A.; Chapman, K. T.; Bisaka, J. *J. Am. Chem. Soc.* **1988**, *110*, 1238. (c) Evans, D. A.; Chapman, K. T.; Hung, D. T.; Kawaguchi, A. T. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 1184. (c) Evans, D. A. *Aldrichim. Acta* **1982**, *15*, 23 and references therein.

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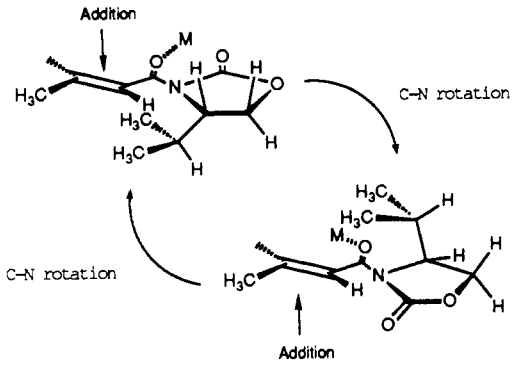
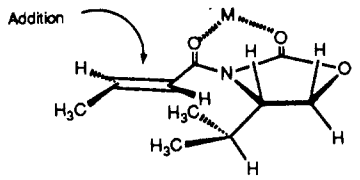
Scheme II



Scheme III



complexes have lead us to examine spectroscopically the intermediates formed between Lewis acids and *N*-acyloxazolidinones as a function of temperature and stoichiometry. In this paper, we report the results of variable-temperature ^1H, ^{13}C, and ^{119}Sn NMR studies between $SnCl_4$ and **1** (R = isopropyl).[3]

Since there are two key Lewis base sites in **1**, several 1:1 and 2:1 solution complexes are possible (Scheme I). Intermediates **2** and **3** are of particular interest since both should exhibit enhanced electrophilic character at the exocyclic α,β-unsaturated functionality.[4]

The impact of substrate conformation on product stereochemistry, as proposed by Evans,[2b] is illustrated in Schemes II and III. For species **2** (Scheme II), the observed facial selectivity should correlate with the preferred orientation of the acyl moiety with respect to the oxazolidinone ring. Conformations intermediate between those shown in Scheme II are expected to show less facial selectivity.

The chelated intermediate **3** should provide maximum stereofacial differentiation due to the fixed orientation of the isopropyl group (Scheme III). In addition to the 1:1 complexes between **1** and $SnCl_4$ shown in Scheme II, several 2:1 complexes of **1** and $SnCl_4$, which are activated toward addition, are also possible. The facial selectivities for these octahedral complexes may be governed by C–N conformation and steric interactions between other ligands in the complex.[5]

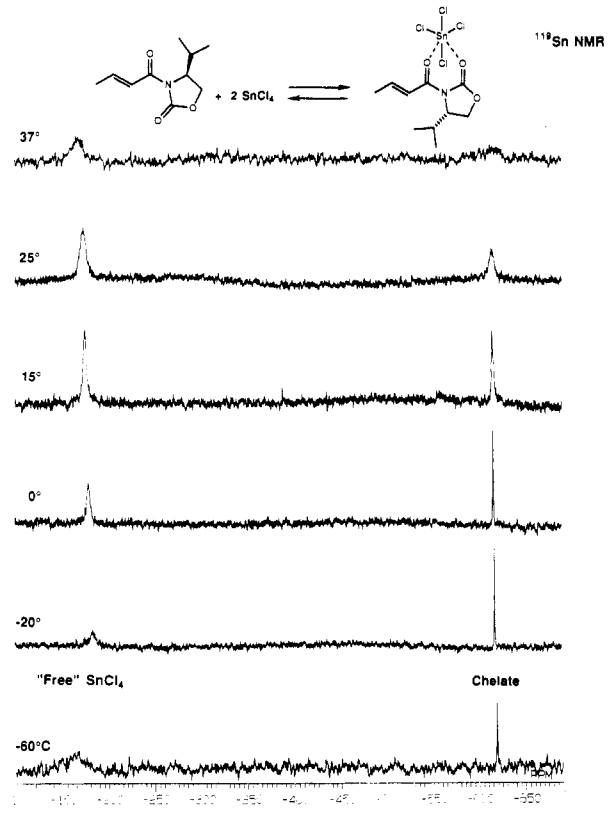


Figure 1.

Table I. ^{13}C Chemical Shift Data for 1 and $SnCl_4$ Chelate[a]

C no.	1, δ	$SnCl_4$ chelate, δ[b]	$\Delta\delta$
C-1	154.6	159.9	+5.3
C-2	63.8	66.9	+3.1
C-3	59.0	62.2	+3.2
C-4	165.2	169.2	+4.0
C-5	122.3	118.1	-4.2
C-6	146.5	164.9	+18.4
C-7 (Me)	18.6	20.5	+1.9
C-8	29.2	31.0	+1.8
C-9 (Me)[c]	18.0	18.0	0.0
C-10 (Me)[c]	14.9	14.0	-0.9

[a] Data collected at -20 °C. [b] One equivalent of $SnCl_4$; chelate is in the slow exchange limit. [c] Assignments interchangeable.

The ^{119}Sn NMR spectra of a 1:1 mixture of **1** (R = isopropyl) and $SnCl_4$ in CH_2Cl_2 at various temperatures (supplementary material, Figure 1).[6] A *single* sharp resonance at −619 ppm is observed from −90 to −20 °C.[7] Based on the chemical shift, we assign this resonance as a six-coordinate tin species.[8] We suggest that this species corresponds to a chelate since the alternative six-coordinate species, a 2:1 complex of **1** and $SnCl_4$, would leave un-

(3) For recent leading references on the conformation and structure of Lewis acid complexes see: (a) Review: Shambayati, S.; Crowe, E. W.; Schreiber, S. L. *Angew. Chem., Int. Ed. Engl.* **1990**, *29*, 256. (b) Denmark, S. E.; Wilson, T. M.; Almstead, N. G. *J. Am. Chem. Soc.* **1989**, *111*, 9258. (c) Keck, G. E.; Andrus, M. B.; Castellino, S. *J. Am. Chem. Soc.* **1989**, *111*, 8136. (d) Cruzado, C.; Bernabe, M.; Martin-Lomas, M. *J. Org. Chem.* **1989**, *54*, 465. (e) Denmark, S. E.; Wilson, T.; Wilson, T. M. *Tetrahedron* **1989**, *45*, 1053. (f) Reetz, M. T.; Harms, K.; Reif, W. *Tetrahedron Lett.* **1988**, *29*, 5881. (g) Denmark, S. E.; Wilson, T.; Wilson, T. M. *J. Am. Chem. Soc.* **1988**, *110*, 984. (h) Keck, G. E.; Castellino, S. *Tetrahedron Lett.* **1987**, 281. (i) Denmark, S. E.; Henke, B. R.; Weber, E. *J. Am. Chem. Soc.* **1987**, *109*, 2512. (j) Frye, S. V.; Eliel, E. L.; Cloux, R. *J. Am. Chem. Soc.* **1987**, *109*, 1862. (k) Reetz, M. T.; Hullmann, M.; Seitz, T. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 477. (l) Keck, G. E., Castellino, S.; Wiley, M. R. *J. Org. Chem.* **1986**, *51*, 3847. (m) Keck, G. E.; Castellino, S. *J. Am. Chem. Soc.* **1986**, *108*, 3847.

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(5) See refs 3a,c,i, also: Reetz, M. T.; Jung, A.; Bolm, C. *Tetrahedron* **1988**, *44*, 3889.

(6) Spectra were recorded on a JEOL GSX-400 spectrometer operating at 400, 100, or 149 MHz for ^1H, ^{13}C, or ^{119}Sn, respectively, using either a 5- or 10-mm broadband probe. The variable temperature control unit was calibrated for both probes against a methanol standard. ^1H and ^{13}C chemical shifts are referenced to methylene chloride. ^{119}Sn chemical shifts are referenced to tetramethyltin (0.0 ppm) as a secondary standard. Solutions were typically 10 mM in chelate in order to maintain complete solubility at low temperatures. ^1H and ^{13}C chemical shifts were assigned based on coupling constant analysis, selective decoupling experiments, and homo- and heteronuclear correlation experiments. All materials were carefully purified and handled by techniques appropriate for air- and moisture-sensitive materials.

(7) A slight temperature dependence is observed for the ^{119}Sn resonance of the chelate: between −90 and 25 °C there is a $\Delta\delta$ of +3.

(8) Wrackmeyer, B. *Annual Reports on NMR Spectroscopy: ^{119}Sn NMR Parameters*; Academic Press: London, 1985; p 73. Also see refs 3c,f,h,k,l.

complexed $SnCl_4$ (-155 ppm). "Free" $SnCl_4$ is not observed. At temperatures above 0 °C, this resonance broadens, indicating dynamic exchange of the tin on the NMR time scale.[9]

When 2 equiv of $SnCl_4$ are added to the oxazolidinone, the variable-temperature ^{119}Sn NMR data reveal two equilibrium processes. The ^{119}Sn NMR spectrum at -60 °C shows two resonances: a very broad peak at approximately -158 ppm and a sharp resonance at -619 ppm (Figure 1). As the temperature is elevated above -60 °C, the downfield peak at -158 ppm shifts upfield to -175 ppm ($\Delta\delta$) = 17, sharpens, and then broadens again above 0 °C, while the chelate resonance at -619 ppm remains narrow up to 0 °C and then broadens. The chemical shift of $SnCl_4$ in CH_2Cl_2 without additional ligands is at -155 ppm and is sharp. The broad "free" $SnCl_4$ resonance observed at low temperatures indicates a weak association between the excess tin and other Lewis base sites such as the ring oxygen in **1**. This interaction is in the intermediate time regime at -60 °C. As the temperature is increased, this equilibrium moves to the fast exchange limit, resulting in a narrowing of the line and a chemical shift that is a population-weighted average. At 0 °C the "free" $SnCl_4$ and the chelated $SnCl_4$ begin to exchange, and *both* resonances broaden with increasing temperature. This equilibrium reaches the intermediate exchange regime on the ^{119}Sn NMR time scale as the temperature reaches 40 °C.

Experiments with 0.5 equiv of $SnCl_4$ provide support for our chelate assignment. At low temperatures only a narrow single resonance is observed at -619 ppm despite the optimum stoichiometry for a 2:1 complex. When the temperature reaches 0 °C, the line begins to broaden as the chelate becomes dynamicaly unstable (supplementary material, Figure 2).

The ^{13}C NMR chemical shift data also support complete chelate formation upon the addition of $SnCl_4$ to **1**. Table I lists chemical shifts of the uncomplexed and chelated oxazolidinone at -20 °C. Both the acyl and the ring carbonyl carbons in the chelate are shifted downfield by 5 and 4 ppm respectively, relative to free **1**. The chemical shift perturbations in the acyl fragment are consistent with those expected for an enolate type intermediate: downfield shifts for the carbonyl and β-carbons and an upfield shift for the α-carbon. The largest change in chemical shift upon chelation is for the β-carbon of the acyl fragment. We observe a $\Delta\delta$ of about 17 ppm downfield. The β-carbon is therefore a highly activated electrophilic site.[4]

The ^{13}C NMR spectra for the chelate ($SnCl_4$:**1**) have also been examined as a function of temperature. At temperatures below 0 °C, all the resonances are sharp; as the temperature is increased the resonances broaden. This broadening is especially evident for the carbonyl and β-carbons. At 45 °C the resonances sharpen as the time scale moves into the fast exchange limit on the ^{13}C NMR time scale. These chemical shifts then represent population-weighted averages. A comparison of the chemical shifts from the slow exchange (-20 °C) and the fast exchange (45 °C) regions ($\Delta\delta \approx 0$) shows the equilibrium favors chelate formation even at this elevated temperature (supplementary) material, Figure 3).

The exclusive formation of the chelate intermediate and its dynamic stability at temperatures below 0 °C is clearly demonstrated in Figure 2. Spectrum A consists of sharp resonances for both the chelate and the uncomplexed oxazolidinone (0.5 $SnCl_4$:**1**, -60 °C). Spectra B and C are of

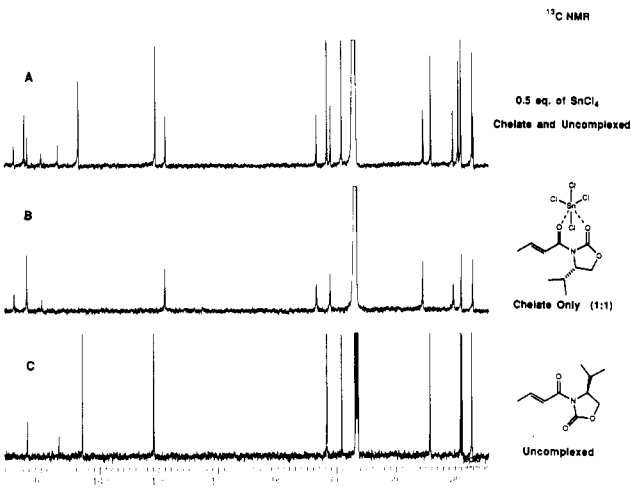

Figure 2.

Table II. ^1H Chemical Shift Data for 1 and $SnCl_4$ Chelate[a]

H no.	1, δ	$SnCl_4$ chelate, δ^b	$\Delta\delta$
H-2 (trans)[c]	4.19	4.68	+0.49
H-2′ (cis)[c]	4.26	4.84	+0.58
H-3	4.46	4.74	+0.28
H-5	7.24	6.25	-0.99
H-6	7.12	7.94	+0.82
H-7 (Me)	1.94	2.19	+0.25
H-8	2.36	2.30	+0.09
H-9 (Me)[d]	0.88	1.06	+0.18
H-10 (Me)[d]	0.82	1.06	+0.24

[a] Data collected at -20 °C. [b] One equivalent of $SnCl_4$; chelate is in the slow exchange limit. [c] Cis/trans with respect to H_3. [d] Assignments interchangeable.

the chelate ($SnCl_4$:**1**) and the uncomplexed oxazolidinone, respectively.

^1H NMR data are consistent with ^{119}Sn and ^{13}C NMR data. The ^1H chemical shifts for the chelated and uncomplexed oxazolidinone are summarized in Table II. Upon chelate formation, the direction of the chemical shift perturbations at H_5 and H_6 in the ^1H data mimics the trend observed in the ^{13}C data, upfield and downfield, respectively. However, there is a large difference in the magnitude of the chemical shift changes for the chelate when comparing ^1H and ^{13}C data. The ratio of the chemical shift perturbations for H_5 and H_6 ($\Delta\delta H_5/\Delta\delta H_6$) is 1.20 while the ratio for C_5 and C_6 in the ^{13}C NMR data is 0.23 (Tables I and II).

This data is consistent with the model for diastereofacial selection proposed by Evans (Scheme III). The 3:1 diastereofacial selectivity observed in the cycloadditions[2b] to the $SnCl_4$:**1** complex suggest that the rigidity imparted by chelation is not enough to provide optimum facial differentiation.[10] Other sources of conformational mobility such as the s-cis/s-trans isomerization of the exocyclic moiety may play a role in reducing selectivity. The conformation of the chelate ring and/or the location of the Lewis acid and the chlorine ligands may also influence stereoselectivity.[11] Studies are in progress to further explore the three-dimensional structure, dynamics, and

(9) Sandstrom, J. *Dynamic NMR Spectroscopy*; Academic Press: New York, 1982.

(10) For a general review, see: *Selectivities in Lewis Acid Promoted Reactions*; Schinzer, D., Ed.; NATO ASI Series, Series C, Vol. 289; Kluwer Academic Publishers: Dordrecht, 1989.

(11) It could be argued that relating the structural features of observed intermediates to product stereochemistry violates the Curtin-Hammet principle. However, these suggestions are chemically "reasonable" and supported by a recent report on the relative reactivities of $SnCl_4$ chelates and complexes.[3c]

thermodynamics of similar systems with various Lewis acids.

Acknowledgment. We wish to thank Professor Evans for supplying us with oxazolidinone samples. Financial support was provided by the EPSCoR program of NSF (RII861075) and the NDSU Grant-in-Aid. The NMR spectrometer was supported by a grant from the Air Force of Scientific Research (AFOSR-87-0036).

Supplementary Material Available: ^{119}Sn NMR stack plot of chelate between −60 and 40 °C, ^{119}Sn NMR stack plot of 1 and 0.5 equiv of $SnCl_4$ between −60 and 40 °C, and ^{13}C NMR stack plot of chelate between −20 and 45 °C (3 pages). Ordering information is given on any current masthead page.

A New Macrobicyclic Hard and Soft Ligand Built on Phenanthroline

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Summary: The synthesis of a new macrobicyclic hard and soft ligand containing an *o*-phenanthroline subunit and an aza-crown ether is described.

There is currently a growing interest in hard and soft heteroditopic ligands, which are able to complex soft transition metals as well as hard alkali cations.[1,2] Several of these ligands have been reported, and they emerge from three main approaches at the molecular level. Macrocyclic compounds combining Schiff bases and polyoxyethylene moieties have been described recently[3-6] and complete the important work on macrocyclic crown ethers incorporating pyridines and thioethers as soft sites.[7-9] Among the laterally branched compounds that have been described some years ago, phosphino-crown ethers have proven their ability to form heterodinuclear complexes.[10-12] Besides the macrocyclic and laterally branched macrocyclic approaches, the cryptate area has provided the concept of hard and soft macrobicycles combining crown ethers as hard binding sites and N or S heterocycles as sift sites.[13]

We report the synthesis of a new macrobicyclic ligand (Figure 1) which is built on a 2,9-diphenyl-1,10-phenanthroline unit used as the soft coordinating site.

Sterically hindered phenanthrolines have been extensively studied,[14-16] and their capability of complexing and stabilizing soft cations such as copper(I) has been demonstrated.[17-19] The aza-crown ether unit [18]N_2O_4 serves as the hard binding site for an alkali cation. The biphenyl spacer ensures the rigidity of the molecular framework and, consequently, the high degree of preorganization of the ligand.

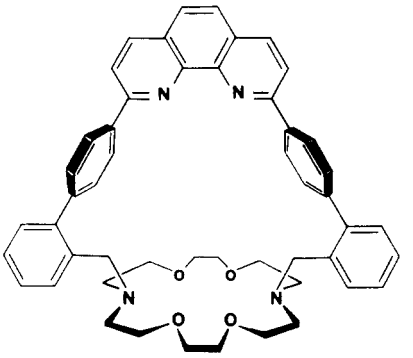

Figure 1. Rigid molecular framework incorporating both hard and soft coordination sites.

The 2,9-bis(bromophenyl)-1,10-phenanthroline (1) was obtained by previously described methods[20] by stepwise introduction of (*p*-bromophenyl)lithium at the 2- and 9-positions of *o*-phenanthroline. The diaryl bromide 1 (Figure 2) was a good precursor to form biphenyl units by "Suzuki" type cross-coupling with arylboronic acids.[21] The quantitatively protected *o*-bromobenzaldehyde 2 obtained by standard methods[22] was treated with Mg to give the Grignard reagent 3 in 95% yield. The Grignard was then slowly added to an excess of trimethylborate (THF/−78 °C/argon). After acidic workup (5% HCl/Et_2O) and purification by acid/base extraction, the deprotected boronic acid 4 was obtained in overall 65% yield as white crystals from CH_2Cl_2/hexane (mp 108–110 °C).

Reacting the boronic acid with the 2,9-bis(*p*-bromophenyl)-1,10-phenanthroline (1) in the remarkable "Suzuki" cross-coupling conditions[23] ($Pd(PPh_3)_4$/toluene/MeOH/aqueous Na_2CO_3/argon/80 °C/12 h) afforded, after chromatography (SiO_2/CH_2Cl_2/1–2% ethyl acetate),

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