the OH functionality and the high electron density inside the β-CD cavity also cannot be ruled out. However, the absence of charge transfer between aromatic donors and acceptors,[17] separated by a saturated hydrocarbon bridge in the β-CD inclusion complex does not favor this proposition. Further, Kaim and co-workers have demonstrated[18] that a low lying π^* LUMO of BLs with higher molecular orbital (MO) coefficient at the coordinating center is crucial in effecting stronger metal–metal coupling in dinuclear complexes.

In conclusion, we have synthesized two mixed valent [2]rotaxane complexes with Ru^{III} and Fe^{II} units as the two molecular heads. Inclusion of a saturated bridging ligand (bpe or bpp for complex **5** and **6**, respectively) into the β-CD cavity has initiated optical electron transfer from the Fe^{II} to the Ru^{III} center, which is not observed in the absence of inclusion.

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[7] Formation of 1:1 inclusion complex, as proposed for complexes **3**–**6** was ascertained by FAB and ES mass spectrometry and analytical data. Complex **3**: FAB m/z: 1710 [M^+]; elemental analysis: calcd with $2\,H_2O$ (%): C 44.9, H 5.55, N 3.28; found C 44.1, H 5.7, N 3.2; UV/Vis (H_2O): 269 nm (sh); Complex **4**: FAB m/z: 1722 [M^+]; elemental analysis: calcd with $2\,H_2O$ (%): C 44.7, H 5.6, N 3.28; found: C 43.6, H 5.6, N 3.2; UV/Vis (H_2O): 270 nm (sh); Complex **5**: ES-MS m/z: 1963 [M^+], elemental analysis: calcd with $2\,H_2O$ (%): C 41.7, H 4.8, N 6.34; found: C 41.0, H 4.9, N 6.2; UV/Vis/NIR (ε, H_2O): 296 (7.3×10^3), 352 (5.3×10^3), 998 nm (1.46×10^3); diffused reflectance spectra ($BaSO_4$ matrix): 376, 1042 nm (br); Complex **6**: ES-MS m/z: 1977 [M^+], elemental analysis: calcd with 0.07β-CD and $2\,H_2O$ (%): C 42.0, H 4.85, N 6.3 (0.07 mole β-CD, in addition to the included one, is trapped with Complex **6**); found: C 41.6, H 4.9, N 6.2; UV/Vis/NIR (ε, H_2O): 300 (2.4×10^3), 356 (1.6×10^3), 985 nm (1.36×10^3), diffused reflectance spectra ($BaSO_4$ matrix): 380, 1050 nm (br) (for detailed synthetic procedure see the Supporting Information).

[8] Complex **5** or **6** was dissolved in a 1.0 mM aqueous solution of β-CD (0.1 mM) to which DMSO was added to give a final DMSO concentration of 0.2 M.

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Synthesis and Self-Assembly of Monodisperse Indium Nanoparticles Prepared from the Organometallic Precursor [In(η^5-C_5H_5)]**

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The current interest in studying elements of nanometer size[1] stems from the structural changes that can arise from size reduction, from the physical properties which can be modified (melting point, band gap of semiconductors, magnetism), from the expected enhanced chemical properties, and from the possibility to use organized nanoparticles for various applications, for example, in microelectronic devices.[2] Hence, much attention has been devoted during the past few years to precise spatial arrangement in two- and three-dimensional superlattice structures[3] of metals (Au, Ag, Cu, Pt, Pd, Co),[4] semiconductors (CdSe, CdS, CdTe),[5] sulfides (Ag_2S)[6] and oxides (CoO, Fe_2O_3, $BaCrO_4$).[7] However, ordering and exploitation of the nanomaterials necessitates the synthesis of monodisperse individual particles, for which no general method is presently available. Low-temperature decomposition of organometallic precursors produces monodisperse nanoparticles of noble or magnetic metals.[8] This method was recently extended to the synthesis of alloys and of semiconducting oxides for gas sensors.[8b, 9]

To prove the generality of the method and its potential use for preparing particles that can be organized in superlattices, we considered indium, for which to our knowledge no organized nanoparticles have been reported. Indium, a low-melting metal, is a likely candidate for such studies; it adopts a tetragonal structure which may or may not be affected by size reduction[10] and can be of various practical interests. It is easily alloyed and converted, for example, to InP[11] and InAs,[12] which are III–V semiconductors that display interesting optoelectronic properties. Indium is easily oxidized to In_2O_3, a transparent semiconducting material (band gap: 3.6 eV), which forms with SnO_2 the mixed indium tin oxide (ITO), a conducting glass.[13] However, studies on indium nanoparticles are very rare. Indium particles 8–15 nm in size were prepared by metal vapor deposition.[10] In addition, indium nanoparticles were prepared, among other colloidal metals, by evaporation of metal into a polymerizable monomer in order to study the thermal stability of the resulting composite material.[14] Indium particles displaying an oxidized surface were synthesized by reduction of $InCl_3$ by alkalides or

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electrides.[15] The literature concerning nanoparticles of indium compounds (InP, InAs) is more extensive, for example recent reports on the formation of semiconducting nanofibers.[16] In contrast, the literature concerning In_2O_3 remains limited, although applications in optoelectronic devices are expected; for example, nanoparticles of this oxide embedded in mesoporous silica display photoluminescence.[17] Here we describe the synthesis of monodisperse indium nanoparticles by an organometallic route and their arrangement in two- and three-dimensional networks.

The precursor chosen was $[In(\eta^5\text{-}C_5H_5)]$ ([InCp]). This indium(I) complex is easily prepared from InCl and LiCp in diethyl ether at room temperature,[18] and its decomposition is expected to be facile. In fact, [InCp] decomposes spontaneously at room temperature in dry anisole containing polyvinylpyrolidone (PVP) as stabilizer. The weight ratio of indium to PVP was fixed at 33%, in which case the reaction produces nanoparticles with size centered near 5 ± 3 nm and a broad size distribution (colloid **1**; Figure 1a). However, in the presence of a small quantity of water (e.g., 225 ppm, as determined by the Karl Fischer technique; $[In]:[H_2O] \approx 4$) the reaction proceeds under the same conditions to yield monodisperse particles with a mean diameter of 6 ± 0.7 nm (colloid **2**; Figure 1b). The size of the particles is independent of the In:PVP ratio (5 or 33 wt%), but the presence of PVP is necessary, since in its absence, even under the same conditions, agglomerated large particles are produced. The particles in colloid **2** consist essentially of indium metal, as demonstrated by X-ray diffraction (XRD) and wide-angle X-ray scattering (WAXS) studies, yet peaks attributable to In_2O_3 can also be observed by XRD. They presumably result from oxidation during preparation of the XRD sample. A high-resolution electron microscopy (HREM) study showed particles of uniform size, most of which did not present any fringe pattern and therefore appear amorphous (Figure 2), but did not provide evidence for the presence of an oxide shell. A few crystalline particles were visible and were identified as In_2O_3, the presence of which presumably results again from exposure of the sample to air prior to introduction into the microscope; further exposure to air increases the number of oxidized particles. The WAXS studies do not indicate the occurrence of any oxidation but clearly demonstrate the presence of indium metal. The radial distribution function (RDF) shows the presence of an amorphous phase, structurally related to the expected bct phase of bulk indium, in agreement with HREM observations. The presence of this amorphous phase may account for the large discrepancy between the coherence length obtained from WAXS studies (ca. 3.5 nm) and the size (6 nm) found by transmission electron microscopy (TEM). The lack of evidence for an oxide shell by any physical method, even if the presence of water clearly controls the growth of the particles, suggests the presence of a thin oxide surface layer which prevents further growth of the particles.

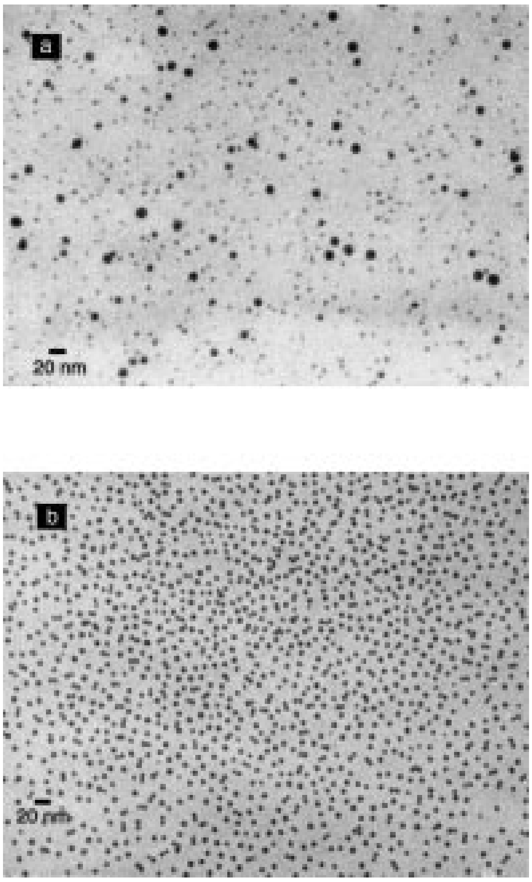

Figure 1. Transmission electron micrographs of indium particles in PVP. a) In the absence of water (colloid **1**). b) In the presence of traces of water (colloid **2**).

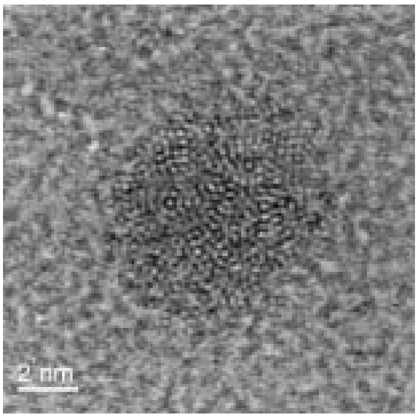

Figure 2. High-resolution electron micrograph of one particle of colloid **2** showing the amorphous (or disordered) structure.

Having demonstrated the possibility of synthesizing particles of regular size embedded in a polymer, it was of interest to undertake a similar study in the absence of a polymer to test the suitability of these particles for use in electronic devices. For this study, we chose trioctylphosphane oxide (TOPO), since this ligand was previously used for stabilization and organization of nanoparticles.[19] The reaction between [InCp] and TOPO (0.25 or 0.5 equiv) at room temperature or 50 °C in toluene containing about 50 ppm H_2O in the absence of light produced a stable brown solution (colloid **3**). This solution consists of well-separated, monodisperse indium particles of 5.2 ± 0.5 nm mean size. The

WAXS studies provide evidence for the presence of amorphous In with a similar structure to that observed in PVP for colloid **2**. The same conclusions regarding the structure can be deduced from HREM studies as for colloid **2**. Upon exposing the samples to air, we observed both a modification of the WAXS spectrum and of the HREM micrographs, which display crystalline features associated with the presence of In_2O_3. The solution can easily be deposited as monolayers or multilayers on a carbon grid, as evidenced by TEM and HREM studies (Figure 3). It is apparent that the particles tend to assemble to form regular networks. When more of the colloidal solution was deposited on the carbon grid, spontaneous self-organization of the particles into a monolayer displaying an hexagonal arrangement was observed, in which the distance between the particles from center to center is only 8.4 ± 0.7 nm. When at least four layers are present, crystallization of the particles into a compact structure (Figure 4) is observed. Given the recent increase in interest in nanoparticle self-assembly, the ease of these synthesis and assembly procedures make this system an attractive candidate for testing electrical properties. It is also noteworthy that when these particles are left in air in the solid state for several days, no change occurs in their self-organization, but HREM analysis clearly shows the presence of the cubic lattice of In_2O_3 and hence demonstrates the oxidation of the particles.

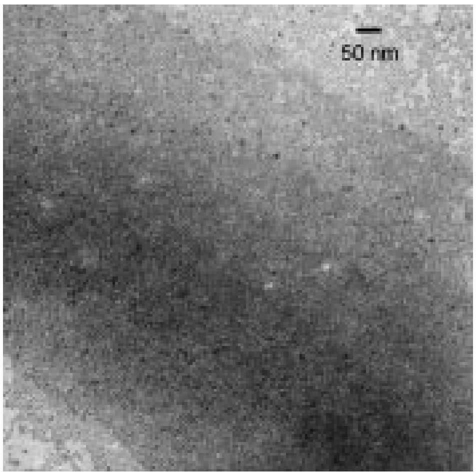

Figure 3. Mono- and multilayers of colloid **3** showing TOPO-protected In particles of narrow size dispersity.

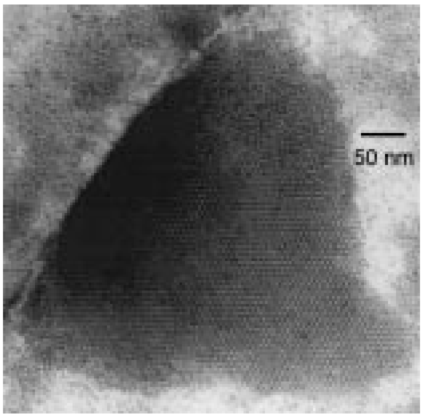

Figure 4. Ordered arrays of colloid **3**.

In conclusion, we have achieved the first synthesis of indium nanoparticles under mild conditions using the organometallic precursor [InCp]. The particles can be stabilized by a polymer or by ligands and, whatever the synthesis and stabilization method, the particles generally have a uniform size. They also adopt an amorphous (or disordered) structure derived from the bct structure of bulk indium. The particles prepared in wet anisole in the presence of PVP or in toluene in the presence of TOPO are monodisperse, and this clearly suggests the generality of the synthesis mode involving decomposition of organometallic precursors under mild conditions. The low size dispersity of the TOPO-stabilized particles allow their crystallization in a compact superlattice. These particles can be oxidized easily in air into cubic In_2O_3, a semiconducting oxide, without changing their organization. This is therefore a material of interest for applications involving electron transfer and electroluminescence.

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Induction of a Preferred Twist in a Biphenyl Core by Stereogenic Centers: A Novel Approach to the Absolute Configuration of 1,2- and 1,3-Diols**

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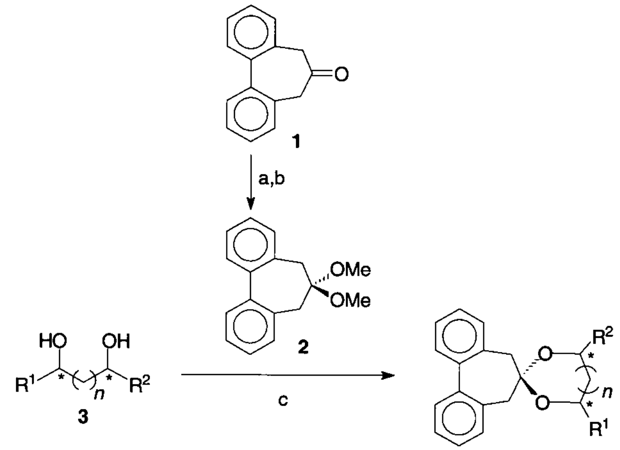
2,2′,6,6′-Tetrasubstituted biphenyls and 2,2′-disubstituted-1,1′-binaphthyls are the paradigms of atropisomeric[1] chiral substances because the steric hindrance to rotation around the C_{Ar}–C_{Ar} bond means that they can exist as a stable pair of enantiomers *P* and *M*.[1, 2] These configurationally stable biaryls have enjoyed a lot of attention in natural products chemistry,[3] asymmetric synthesis,[4] materials science,[5] and molecular recognition,[6] while only very recently nonatropisomerically stable, flexible biaryl compounds have also been used in asymmetric catalysis[7] and molecular recognition.[8] These latter compounds are usually present as racemic (*P*,*M*) mixtures because the free rotation around the C_{Ar}–C_{Ar} bond does not allow the isolation of a single enantiomer. A preferred enantiomeric conformation can be, however, stabilized through interaction with chiral metal complexes[7a] and/or substitution with chiral auxiliaries.[7b–e]

We present herein a novel application of flexible biphenyl compounds for the nonempirical determination of the absolute configuration of 1,2- and 1,3-diols by CD spectroscopy. This is a task of great importance considering the wide use of such diols as chiral building blocks[9] and chiral controllers in asymmetric processes.[4a, 10] Although CD spectroscopy has been already employed for this purpose, such as in the dibenzoate-[11] and in the metal-induced CD methods,[12] the former nonempirical approach often involves difficult conformational assignments, while the latter, being an empirical method, does not allow an immediate and completely reliable correlation between the CD spectrum and configuration. For these reasons we recently developed alternative nonempirical CD approaches to the assignment of the absolute configuration of 1,2-diarylethane-[13] and 1-arylethane-1,2-diols,[14] in which the problem of the conformational determination is overcome by transforming the original flexible diols into their 2,2-dimethyl-1,3-dioxolanes or 4-biphenylboronates, respectively (that is, cyclic, conformationally defined derivatives).

We were looking for a more general method that would also be suitable for nonchromophoric alkyl-substituted diols, and have developed the approach described herein which is based on the formation of conformationally defined derivatives between a diol and a pro-atropisomeric[7a] flexible biphenyl moiety. In order to determine the absolute configuration of aliphatic (and hence UV transparent) diols by CD analysis it is necessary to introduce suitable chromophores which are sensitive to the chirality of the diol and give rise to Cotton effects in the CD spectrum. Moreover, the mechanism by which the diol stereogenic centers induce optical activity in the chromophore must be known in order to obtain a nonempirical correlation between the CD spectrum and the absolute configuration of the diol.

Following our previous approaches,[13, 14] we thought that the dioxolanes **4** (Scheme 1), derived from chiral diols **3** and the dimethylacetal **2** (which in turn is obtained from the 2,2′-bridged biphenyl ketone **1**)[15] would constitute suitable



Scheme 1. Synthesis of biphenyldioxolanes **4**. a) $CH(OCH_3)_3$, *p*TsOH, MeOH, RT; b) NH_3 2 M in EtOH; c) $CHCl_3$, 4-Å MS, RT. Ts = toluene-4-sulfonyl; MS = molecular sieve.

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