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# Mesomerization of $S_4$-Symmetric Tetrahedral Chelate Complex $[In_4(L^3)_4]$: First-Time Monitored by Temperature-Dependent $^1H$ NMR Spectroscopy[‡]

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*Dedicated to Professor Paul von Ragué Schleyer on the occasion of his 80th birthday*

**Keywords:** Bailar twist / NMR spectroscopy / Density functional calculations / Indium / N,O ligands / Supramolecular chemistry

VT $^1H$ NMR spectroscopy proved that a non-dissociative and reversible mesomerization process links the tetranuclear indium(III) complexes *meso*-(Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-**3** and *meso*-(Λ,Λ,Δ,Δ)(*M*,*M*,*P*,*P*)-**3′**. During this process four tandem Bailar twists, resulting in the (Δ)/(Λ) isomerization at the indium centers, and the (*P*)/(*M*) inversion of the four coordinating face-centered, helical ligands $(L^3)^{3-}$ are involved. In addition, gas-phase DFT calculations (B3LYP/LANL2DZp) revealed a $C_1$-symmetric transition state (+21.9 kcal mol$^{-1}$) for the mesomerization mechanism which connects **3** and **3′**.

## Introduction

Metal-assisted self-assembly[2] is a very convenient method for the synthesis of supramolecular aggregates. NMR spectroscopy was shown to be an effective tool for the investigation of these self-assembled coordination compounds and for the study of their dynamic behavior in solution.[3,4] In this context, we reported on variable-temperature (VT) $^1H$ NMR spectroscopy studies revealing tetrahedral, racemic, homochiral (Δ,Δ,Δ,Δ)-[$(NH_4)_4$∩{$Mg_4(L^1)_6$}] ⇄ (Λ,Λ,Λ,Λ)-[$(NH_4)_4$∩{$Mg_4(L^1)_6$}] ($NH_4$-**1**) to enantiomerize.[4a] This phenomenon can be explained by simultaneous Bailar twists associated with (Δ)/(Λ) isomerization at the four octahedrally coordinated magnesium centers synchronized with sterically unhindered (*R*)/(*S*) atropisomerization processes around the C–C single bonds of the six edge-bridging $C_2$-symmetric ligands $(L^1)^{2-}$. Supplementary support for this phenomenon is presented by additional studies of (Δ,Δ,Δ,Δ)-[$(EtNH_3)_4$∩{$Mg_4(L^1)_6$}] ⇄ (Λ,Λ,Λ,Λ)-[$(EtNH_3)_4$∩{$Mg_4(L^1)_6$}] ($EtNH_3$-**1**), where the diastereotopic methylene protons of the ethylammonium counterions of $EtNH_3$-**1** display similar temperature-dependent coalescence as the ligand vinylether methylene protons (Figure 1). On the contrary, racemic (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-[$(NH_4)_4$∩{$Mg_4(L^2)_6$}] ($NH_4$-**2**) showed to be kinetically and dynamically stable on the $^1H$ NMR timescale. Owing to steric hindrance by the extra $CO_2Et$ groups, rotation around the central C–C bond in $(L^2)^{2-}$ is blocked, which prevents $NH_4$-**2** from enantiomerization (Figure 1).[4a]

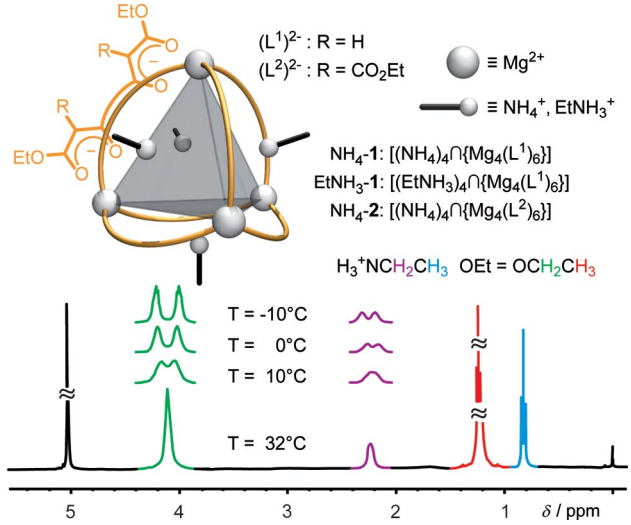


Figure 1. Top: Schematic representation of $NH_4$-**1**, $EtNH_3$-**1**, and $NH_4$-**2**. Bottom: VT $^1H$ NMR spectrum of $EtNH_3$-**1**.

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## Results and Discussion

These VT $^1$H NMR studies on the enantiomerization of $NH_4$-**1**, and $EtNH_3$-**1** prompted us to also study the potential dynamic behavior of tetrahedral *meso*-(Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-[$In_4(L^3)_4$] (**3**) and homochiral racemic (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-[$In_4(H^NL^3)_4$]($ClO_4$)$_4$ (**4**) (Figure 2).

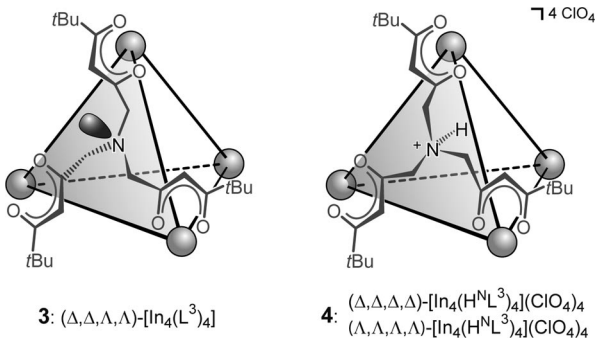


Figure 2. Schematic presentation of *meso*-(Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-[$In_4(L^3)_4$] (**3**) and (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-[$In_4(H^NL^3)_4$]($ClO_4$)$_4$ (**4**).

Based on an X-ray structural analysis, complex **3**[5] is composed of four indium ions, located at the apices of a tetrahedron linked by four ligands $(L^3)^{3-}$ across the tetrahedral faces. The distorted octahedrally coordinated indium ions have alternately (Δ) or (Λ) configuration. The $C_3$ symmetry of the ligands $(L^3)^{3-}$ is broken during complexation to the indium ions. However, despite the desymmetrized $C_1$-symmetric ligands $(L^3)^{3-}$, **3** is intrinsically achiral. This is due to the (*P*)/(*M*) helicity of the ligands $(L^3)^{3-}$ resulting in an overall $S_4$ molecule symmetry of *meso*-(Δ,Δ,Λ,Λ)-(*P*,*P*,*M*,*M*)-[$In_4(L^3)_4$] (**3**) (Figure 3).[6]

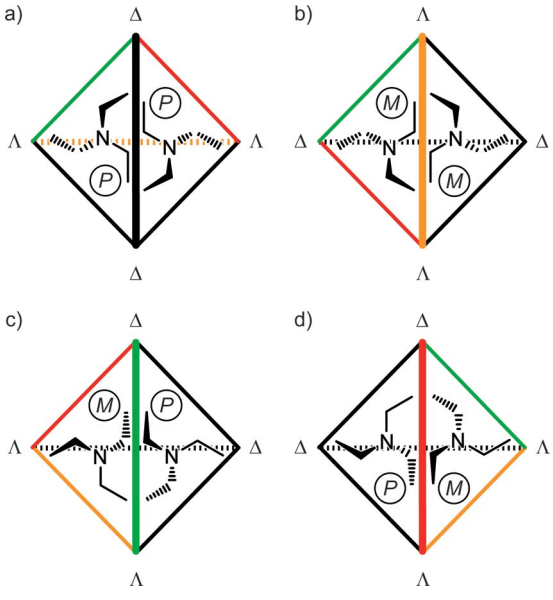


Figure 3. Schematic presentation of the (*P*)/(*M*) helicity of the $C_1$-symmetric ligands $(L^3)^{3-}$ of $S_4$-symmetric **3**. View towards the edges: (a) (Δ,Δ) black, (b) (Λ,Λ) ocher, (c) (Δ,Λ) green, (d) (Δ,Λ) red.

In total agreement with the X-ray data,[5] the $^1$H NMR spectrum of **3** in [$D_8$]toluene at 20 °C displays two sets of three signals for the olefinic protons and *t*Bu groups ($\delta$ = 6.42, 6.33, 5.26 ppm and $\delta$ = 1.32, 1.18, 1.00 ppm, respectively). The diastereotopic $CH_2$ protons appear as three simple, but different, AB systems [$\delta$ = 4.30 and 3.35 ppm, $|^2J_{H,H}|$ = 19.3 Hz; $\delta$ = 3.73 and 3.19 ppm, $|^2J_{H,H}|$ = 14.4 Hz; $\delta$ = 3.09 and 2.66 ppm, $|^2J_{H,H}|$ = 14.1 Hz]. This proves **3** to kinetically be stable on the NMR timescale. Most interestingly, and according to what we expected, the $^1$H NMR spectrum of *meso*-(Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-[$In_4(L^3)_4$] (**3**) revealed temperature dependence. In the range of 20–105 °C the signals of the olefinic protons (blue), the *t*Bu groups (red), and of the diastereotopic $CH_2$ protons (green) become broader and finally coalesce (Figure 4). The unique dynamic temperature-dependent $^1$H NMR spectroscopic behavior of *meso*-**3** can be explained by an unprecedented mesomerization of the identical twins (Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-**3** ⇄ (Λ,Λ,Δ,Δ)(*M*,*M*,*P*,*P*)-**3′** (Figure 4). This mesomerization process is the first of this type and requires *four* tandem Bailar twists, which result in the inversion of the chirality at the indium centers together with the (*P*)/(*M*) inversion of the *four* coordinated $C_1$-symmetric helical ligands $(L_3)^{3-}$.[7] It is worth to note, that **3** and **3′** are identical and would only be distinguishable in the case of two pairs of different metal ions. A prerequisite for the performance of the Bailar twists in **3** is the sufficiently flexible [$In_4(L^3)_4$] scaffold. This is guaranteed, since the coordinated and thus helical $C_1$-symmetric ligands $(L^3)^{3-}$ allow simultaneous ste-

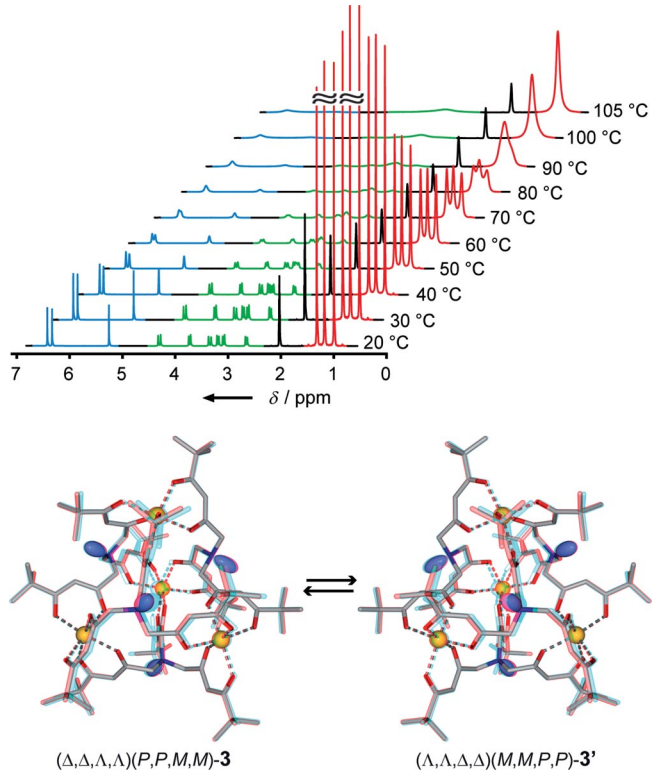


Figure 4. Top: VT $^1$H NMR spectra of *meso*-**3**. Bottom: 3D presentation (red/blue) of the mesomerization (Δ,Δ,Λ,Λ)(*P*,*P*,*M*,*M*)-**3** ⇄ (Λ,Λ,Δ,Δ)(*M*,*M*,*P*,*P*)-**3′**.

  

rically unhindered back and forth twists around the single bonds. Furthermore, this dynamic process goes along with concurrent helix (*P*)/(*M*) isomerization of the face-centered ligands and thus leads to the enantiotopization of the olefinic protons, *t*Bu groups, and diastereotopic $CH_2$ protons, as monitored by $^1$H NMR spectroscopy. This mesomerization process is reversible and occurs non-dissociative without the formation of diastereomers. The high-temperature $^1$H NMR spectra (Figure 4) indicate time-averaged pseudo-*T* molecular symmetry for **3** due to rapid, non-dissociative mesomerization (**3** ⇄ **3′**). As explicitly shown for the *t*Bu protons of **3**, coalescence is observed at 95 °C (400 MHz). The activation parameters for the molecular interconversion have been derived from line-shape analysis: $\Delta H^{\ddagger}$ = 11.8 kcal mol$^{-1}$, $\Delta S^{\ddagger}$ = –16 cal K$^{-1}$, $\Delta G^{\ddagger}_{368}$ = 17.7 kcal mol$^{-1}$ (see the Supporting Information).

Density functional calculations at B3LYP/LANL2DZp[8–11] on the model complex $[In_4(L^*)_4]$[12] suggest, that the mesomerization of $[In_4(L^*)_4]$ starts by (*P*)/(*M*) isomerization of a $C_1$-symmetric helical ligand $(L^*)^{3-}$ at the nitrogen center, initiating the (Δ)/(Λ) isomerization at the ligated indium(III) centers. This leads to the subsequent (*P*)/(*M*) isomerization of the other three helical ligands $(L^*)^{3-}$ and the Bailar twists at the remaining metal center, resulting in the mesomerization of the whole complex. This corresponds to an activation barrier of 21.9 kcal mol$^{-1}$. The imaginary mode indicates that the mesomerization proceeds in a concerted, yet asynchronous fashion (Figure 5).

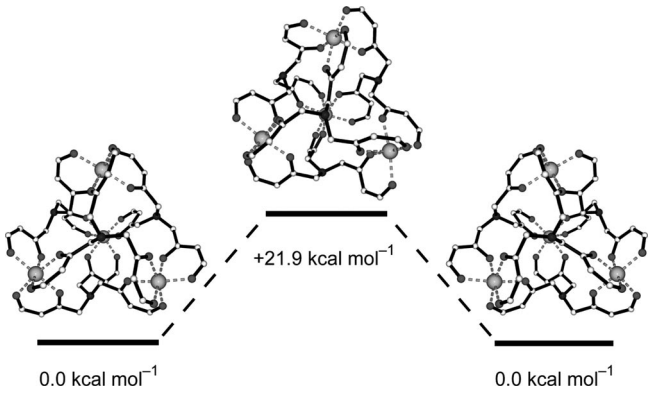


Figure 5. Energetic profile, illustrating the mesomerization of $[In_4(L^*)_4]$ in a concerted, yet asynchronous fashion. In gray, C white, O gray (small), N black.

Contrary to *meso*-**3**, the solid-state structure analysis of $[In_4(H^NL^3)_4](ClO_4)_4$ (**4**) shows that the tetracation $(\mathbf{4})^{4+}$ is present in the solid state as a racemic mixture of homochiral (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-*fac* stereoisomers with the protons at the nitrogen atom directed towards the center of the tetrahedron.[5] The $^1$H NMR spectrum of $[In_4(H^NL^3)_4]^{4+}$ $(\mathbf{4})^{4+}$ in [D$_6$]dmso at 23 °C reveals one set of signals for the four equivalent ligands, indicating *T* molecule symmetry. However, owing to the fixation of the protons at the nitrogen atom of the ligands $(H^NL^3)^{2-}$, enantiomerization of both enantiomers of racemic (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-$[In_4(H^NL^3)_4](ClO_4)_4$ (**4**) is blocked, which is documented by the temperature independence of the $^1$H NMR spectra of **4** (see the Supporting Information).

## Conclusions

We have shown that the complexes *meso*-(Δ,Δ,Λ,Λ)-(*P*,*P*,*M*,*M*)-**3** and *meso*-(Λ,Λ,Δ,Δ)(*M*,*M*,*P*,*P*)-**3′** are identical and are correlated with each other by four tandem Bailar twists, which results in the (Δ)/(Λ) isomerization at the indium centers and the (*P*)/(*M*) isomerization of the four coordinating helical ligands $(L^3)^{3-}$. This mesomerization process is non-dissociative and reversible as was demonstrated by VT $^1$H NMR spectroscopy. The mechanism connecting **3** and **3′** was elucidated by DFT calculations revealing a $C_1$-symmetric transition state. However, owing to steric fixation, (*P*)/(*M*) isomerization of the ligands $(H^NL^3)^{2-}$ of racemic (Δ,Δ,Δ,Δ)/(Λ,Λ,Λ,Λ)-$[In_4(H^NL^3)_4](ClO_4)_4$ (**4**) is blocked and thereby the enantiomerization of **4**, as shown by $^1$H NMR spectroscopy.

## Experimental Section

**General Methods:** All reagents and solvents employed were commercially available high-grade purity materials (Fluka, Aldrich), which were used as supplied without further purification. Synthesis and further spectroscopic data of **3** and **4** were reported previously.[5] Temperature-dependent $^1$H NMR spectra of **3** and **4** were recorded with a JEOL EX400 spectrometer (400 MHz) in indicated solutions. The residual solvent signal ($\delta$ = 2.03 and 2.49 ppm for [D$_8$]toluene and [D$_6$]dmso, respectively) was used as an internal standard. The numbers of the free activation enthalpies were derived by using standard equations or by complete line-shape analysis (software: gNMR, Cherwell Scientific) for complex **3**. The two methods yield similar results for $\Delta G^{\ddagger}$ within 0.1 kcal mol$^{-1}$.[13] All structures were fully optimized without any constraints by using B3LYP[9] and the LANL2DZ basis set augmented with polarization functions (denoted as LANL2DZp).[10,11] All structures were characterized as minima or transition states by computation of vibrational frequencies (*xyz* files of the calculated reactant and transition states are available in the Supporting Information). Energies are ZPE-corrected on the same level, and the Gaussian 03 suite of programs was used throughout.[8]

**Supporting Information** (see footnote on the first page of this article): Line-shape analysis of the *t*Bu signals of **3**, Eyring plot of the data obtained from the VT $^1$H NMR of **3**, linear regression for the Eyring plot, description of the $^1$H NMR spectrum of **4** in [D$_6$]dmso and picture of the temperature-independent $^1$H signals for the $H^{N+}$ proton and the diastereotopic $CH^AH^B$ proton, respectively, as well as the *xyz* files of the calculated (RB3LYP/LANLDZp) $[In_4(L^*)_4]$ complexes (reactant and transition states).

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[7] To the best of our knowledge, this is the first report on the interconversion of a tetrahedral complex with $[M_4L_4]$ stoichiometry (L = face-centered ligand). Analogous processes were observed for $[M_4L_6]$ systems (L = edge-bridging ligand).[4a,4b]

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