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Metal complexes of N-o-chlorobenzamido-*meso*-tetraphenylporphyrin: cis-Tl(N-NCO(o-Cl)C₆H₄-tpp)(OAc) and trans-Cd(N-NHCO(o-Cl)C₆H₄-tpp)(OAc) (tpp = 5, 10, 15, 20-tetraphenylporphyrinate)

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ABSTRACT

The crystal structures of diamagnetic (cis-acetato) (N-o-chlorobenzamido-meso-tetraphenylporphyrinato)thallium(III) 0.5 water solvate $[cis-Tl(N-NCO(o-Cl)C_6H_4-tpp)(OAc) 0.5 H_2O; 3.0.5 H_2O]$ and diamagnetic (trans-acetato) (N-o-chlorobenzamido-meso-tetraphenylporphyrinato)cadmium(II) methylene chloride solvate [trans-Cd(N-NHCO(o-Cl)C₆H₄-tpp)(OAc) CH₂Cl₂; 4 CH₂Cl₂] were determined. The coordination sphere around the Tl^{3+} (or Cd^{2+}) in **3** (or **4**) is a distorted square-based pyramid in which the apical site is occupied by a chelating bidentate OAc^{-} group. In **3**, TI^{3+} and N(5) are located on the same side at 1.18 and 1.26 Å from it 3N plane, but in 4, Cd^{2+} and N(5) are located on different sides at 1.06 and -1.55 Å from it 3N plane. The free energy of activation at the coalescence temperature T_c for the intermolecular acetate exchange process in **3** in CD_2Cl_2 solvent is found to be $\Delta G_{198}^{\neq} = 42.1$ kJ/mol through ¹H NMR temperature-dependent measurements. Likewise, the free energy of activation $\Delta G_{293}^{\neq} = 55.94 \text{ kJ/mol}$ is determined for the intramolecular exchange of the ortho protons between o'-H (34, 40) and o'-H (38, 44) in 3 in CD_2CI_2 . VT NMR (¹H and ¹³C) studies of 4 show that the acetate acts as a bidentate ligand and the OAc⁻ exchange does not occur in CD₂Cl₂. Moreover, the NH proton [*i.e.*, H(5)] of **4** in CD₂Cl₂ is observed as a singlet at δ –0.09 ppm with $\Delta v_{1/2}$ = 13 Hz at 20 °C indicating that the NH protons undergo intermediate intermolecular proton exchange with water at this temperature.

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Previously, we reported two-stage formation of (N-o-chlorobenzamido-*meso*-tetraphenylporphyrinato)(methanol)zinc(II) methanol solvate [Zn(*N*-NCO(o-Cl)C₆H₄-tpp)(MeOH)·MeOH; **1**. MeOH] [1]. Compound **1** is a zinc complex of *N*-NHCO(o-Cl)C₆H₄-Htpp) (**2**) (Chart 1). The absolute values of hardness η for Zn²⁺, Tl³⁺ and Cd²⁺ are 10.88, 10.4, and 10.29 eV, respectively [2]. It is observed that the effective ionic radius (*r*) for the metal ion increases from 0.82 Å for Zn²⁺ (*S* = 0) with coordination number (CN) = 5 [or 1.025 Å for Tl³⁺ (*S* = 0) with CN = 6] to 1.09 Å for Cd²⁺ (*S* = 0) with CN = 6 [3]. In these three metal ions the polarizing power z/r^2 (z = charge on the cation, r = ionic radius) decreases from 3.652 (Zn²⁺) [or (Tl³⁺)] to 2.041 (Cd²⁺) [4,5]. The metal cations of different polarizing power selected were Tl³⁺ and Cd²⁺. The soft acid Tl³⁺ with a larger polarizing power (2.855) similar to that of Zn²⁺ attacks the two N–H protons of **2** and lead to a six-coordinate

distorted trigonal prismatic Tl (III) derivative, that is, (cis-acetato)(N-o-chlorobenzimido-meso-tetraphenylporphyrinato)thallium (III) 0.5 water solvate $[Tl(N-NCO(o-Cl)C_6H_4-tpp)(OAc) \cdot 0.5 H_2O;$ $3.0.5 H_2O$ possessing a nitrene moiety inserted between the thallium atom and one nitrogen atom, N(4) (Scheme 1). During the metallation of free base **2** with $Cd(OAc)_2$, the soft acid Cd^{2+} with a lower polarizing power (2.041) prefers to retain one OAc⁻ ligand and coordinate to the N-H proton [i.e. H(2A)] of 2 also to form a six-coordinate distorted trigonal prismatic complex, that is, (trans-acetato)(N-o-chlorobenzimido-meso-tetraphenylporphyrinato)cadmium(II) methylene chloride solvate [trans-Cd(N-NHCO(o-Cl)C₆H₄-tpp)(OAc)·CH₂Cl₂; **4**·CH₂Cl₂] (Scheme 1). In this paper, we describe the X-ray structural investigation on the complexation of Tl³⁺ and Cd²⁺ classified as C acids (covalent acids) but with different polarizing power into **2** leading to mononuclear complexes of cis-3 and trans-4 [4,5]. In addition, the ¹H and ¹³C NMR spectra of cis-3 in CD₂Cl₂ at various temperatures are used to investigate the intermolecular apical ligand (OAc⁻) exchange process and in turn to determine the free energy of activation at the coalescence temperature, ΔG_{Tc}^{\neq} , for the exchange process.

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Using a d¹⁰ metal, namely, thallium(III) and cadmium(II), the new complexes **3** and **4** were synthesized. The synthetic strategy is outlined in Scheme 1. The complex cis-Tl(N-NCO(o-Cl)C₆H₄tpp)(OAc) (3) was produced in 63% yield by heating a N-NHCO(o-Cl) C₆H₄-Htpp) (**2**) solution in CH₂Cl₂/MeOH under aerobic conditions with an excess of Tl(OAc)₃ (Scheme 1). The complex trans- $Cd(N-NHCO(o-Cl)C_6H_4-tpp)(OAc)$ (4) was synthesized in 53% yield by reacting 2 with excess Cd(OAc)₂ in CH₃CN under aerobic conditions (Scheme 1). The molecular frameworks are depicted in Fig. 1a for 3.0.5H₂O and in Fig. 1b for 4.CH₂Cl₂. The cadmium-nitrogen bond distances are comparable to those of Cd(1)-N(p) =2.301(5) Å in Cd(N-NHCOC₆H₅-tpp)(OAc) [6]. The Cd \cdots N(4) distance of 2.600(4) Å for **4** is longer than 2.301(5) Å but is significantly shorter than the sum of the van der Waals radii of Cd and N (3.15 Å) [3]. This longer Cd \cdots N(4) contact in **4** may be viewed as a secondary intramolecular interaction. Most chemists seems to consider this secondary interaction between the metal ion and the fourth N as a weak bond in N-substituted porphyrin metal complexes [7,8].

We adopt the plane of three strongly bound pyrrole nitrogen atoms [*i.e.*, N(1), N(2) and N(3)] for **3** and **4** as a reference plane,

3N. The geometry around Tl (III) (or Cd (II)) is a distorted squarebased pyramid in which the apical site is occupied by a chelating bidentate OAc⁻ group in **3** (or **4**). In complex **3**, Tl (III) and N(5) are located on the same side at 1.18 and 1.26 Å from its 3N plane, but for complex **4**, Cd (II) and N(5) are located on different sides at 1.06 and -1.55 Å from its 3N plane. Apparently, chelating bidentate OAc⁻ in **3** is *cis* to the (*o*-Cl)BA group with O(2) and O(3) being located separately at 2.95 and 3.13 Å out of the 3N plane, and bidentate OAc⁻ in **4** is *trans* to the (*o*-Cl)BA group with O(2) and O(3) located at 3.21 and 2.85 Å out of the 3N plane.

The N(4) pyrrole rings bearing the (o-Cl)BA group in 3 and 4 deviate mostly from the 3N plane, thus orienting separately with a dihedral angle of 47.6° and 30.8°, whereas small angle of 7.5°, 13.5° and 9.8° occur with N(1), N(2) and N(3) pyrrole for **3** and the corresponding angles are 21.8°. 1.4 ° and 17.0° with N(1). N(2) and N(3) pyrrole for **4**. In **3**, such a large deviation from planarity for the N(4) pyrrole is also reflected by observing a 16.2-20.3 ppm upfield shift of the C_{β} (C17, C18) at 115.4 ppm compared to 134.1 ppm for C_{β} (C2, C13), 134.1 ppm for C_{β} (C3, C12) and 131.6 ppm for C_{β} (C7, C8). In **4**, a similar deviation is also found for N(4) pyrrole by observing a 7.4–10.1 ppm upfield shift of C_{B} (C17, C18) at 124.0 ppm compared to 134.1 ppm for C_{β} (C3, C12), 134.0 ppm for C_{β} (C7, C8) and 131.4 ppm for C_{β} (C2, C13). The dihedral angles between the mean plane of the skeleton (3N) and the planes of the phenyl groups are 88.1° [C(24)], 54.1° [C(30)], 42.7° [C(36)] and 36.2° [C(42)] for **3** and the corresponding angles are 61.1°, 51.7°, 39.9° and 36.4° for 4.

In solution, the molecule has effective C_s symmetry with a mirror plane running through the N(4)–N(5)–Tl(1)–N(2) unit for **3** and the N(2)–Cd–N(4)–N(5) unit for **4**. As a result, the ¹H NMR spectrum will exhibit four pyrrole resonances [H_β (2, 13), H_β (3, 12), H_β (7, 8), H_β (17, 18)] for these two complexes (Figs. 2 and 3). Fig. 2 depicts the representative ¹H spectra for **3** in CD₂Cl₂ at 20 and –90 °C. The NMR study of **3** showed three different types of Tl–H coupling constants for H_β. In **3** at 20 °C, the doublet at 9.30 ppm is assigned as H_β (2, 13) with ⁴J(Tl–H) = 10.8 Hz and the other doublet at 8.59 ppm is due to H_β (7, 8) with ⁴J(Tl–





(b) $\mathbf{4} \cdot \mathrm{CH}_2\mathrm{Cl}_2$

Fig. 1. (a) Molecular structure of *cis*-TI(N-NCO(*o*-CI)C₆H₄-tpp)(OAc)-0.5H₂O (**3**.0.5H₂O) and (b) *trans*-Cd(N-NHCO(*o*-CI)C₆H₄-tpp)(OAc)-CH₂Cl₂ (**4**-CH₂Cl₂), with 30% thermal ellipsoids. Hydrogen atoms, solvent H₂O for **3**.0.5H₂O and solvent CH₂Cl₂ for **4**-CH₂Cl₂ are omitted for clarity. Selected bond distances (Å): TI(1)–N(1), 2.382(3); TI(1)–N(2), 2.148 (3); TI(1)–N(3), 2.350(3); TI(1)–N(5), 2.131(3); TI(1)–O(2), 2.299(3); TI(1)–O(3), 2.432(3) for **3**.0.5H₂O; Cd–N(1), 2.294(4); Cd–N(2), 2.246(4); Cd–N(3), 2.331(4); Cd–O(2), 2.281(12); Cd–O(3), 2.344(10) for **4**?CH₂Cl₂.

H) = 75.0 Hz. The singlet at 8.95 ppm is assigned to H_β (3, 12) and the other singlet at 7.26 ppm is due to H_β (17, 18) (Fig. 2a). In **4** at 20 °C, the doublet at 8.77 ppm is assigned to H_β (2, 13) with ³*J*(H–H) = 4.2 Hz and the other doublet at 8.71 ppm with ³*J*(H–H) = 4.2 Hz is due to H_β (3, 12) (Fig. 3a). The singlet at 8.81 ppm is assigned to H_β (7, 8) and the other singlet at 8.64 ppm is due to H_β (17, 18) (Fig. 3a).

The signal arising from the NH proton of **4** in CD₂Cl₂ was observed as a singlet at δ –0.99 ppm ($\Delta v_{1/2}$ = 13 Hz) at 20 °C. This NMR data suggests that the NH protons of **4** undergo intermediate intermolecular proton exchange with water at 20 °C. At low temperature the chemical exchange slows down that allows the observation of a broad singlet for the NH proton at –1.08 ppm ($\Delta v_{1/2}$ = 17 Hz) for **4** at –90 °C. In this case, the NH proton signal for **4** at –90 °C is broadened by the quadrupolar interaction of the ¹⁴N nucleus.

Non-equivalence of the two sides of the macrocycle will cause each phenyl ring have two distinct *ortho* resonances, with one set of ortho protons, o-H (22, 32) [or o-H (26, 32)] and o-H (26, 28) [or o-H (22, 28)], for phenyl C(24) and C(30) and the other set of ortho protons, o'-H (34, 40) [or o'-H (34, 44)] and o'-H (38, 44) [or o'-H (38, 40)] for phenyl C(36) and C(42) of 3 (or 4), respectively. In **3** at 20 °C, the rotation of the phenyl group along C_1 – C_{meso} [C(5)-C(21) or C(10)-C(27)] bond is slow [9]. This slow rotation is supported by the two doublets at 8.25 and 8.12 ppm due to o-H (22, 32) and o-H (26, 28), respectively (Fig. 2a). Moreover, the rotation of phenyl group along C(15)-C(33) [or C(20)-C(39)] bond for 3 is at the near fast exchange region [9]. In this fast exchange region, the signals are observed as broad singlet at 8.47 ppm (Fig. 2a). At -90 °C, both rotations are extremely slow. Hence, the rate of intramolecular exchange of the ortho protons for 3 in CD₂Cl₂ is also extremely slow. The singlet at 8.16 ppm and the doublet at 8.13 ppm with ${}^{3}I(H-H) = 5.4$ Hz is assigned as ortho protons o-H(26) and o-H(28). The two sets of signals at 8.27 and 8.20 ppm is due to ortho protons o-H (22) and o-H (32) for 3 (Fig. 2b). Likewise, the two partially overlapping doublets at 8.72 ppm with ${}^{3}J(H-H) = 7.8$ Hz is



Fig. 2. ¹H NMR (599.95 MHz) spectra showing four different β -pyrrole protons H $_{\beta}$ and phenyl protons (o-H, m,p-H) for 3 in CD₂Cl₂: (a) 20 °C and (b) -90 °C.



Fig. 3. ¹H NMR (599.95 MHz) spectra showing four different β-pyrrole protons H_β and phenyl protons (o-H, m,p-H) for 4: (a) in CDCl₃ at 20 °C and (b) in CD₂Cl₂ at -90 °C.

due to *ortho* protons o'-H (34, 40). The same two sets of signals at 8.27 and 8.20 ppm is also due to ortho protons o'-H (38) and o'-H (44) for **3** (Fig. 2b). The free energy of activation $\Delta G_{293}^{\neq} = 55.94$ kJ/ mol is therefore, determined for the intramolecular exchange of the *ortho* protons between o'-H (34, 40) and o'-H (38, 44) in **3**.

In a similar fashion, the rotation of the phenyl group of **4** in CDCl₃ at 20 °C along C(10)–C(27) [or C(5)–C(21)] bond is slow and the rotation along C(20)–C(39) [or C(15)–C(33)] is at the intermediate exchange region [9]. Hence the ¹H resonances for the *ortho*

protons of **4** in CDCl₃ at 20 °C were observed as two sets of doublets: one doublet at 8.65 ppm is assigned to *ortho* protons *o*-H (26, 32) with ${}^{3}J(H-H) = 6.6$ Hz and the other doublet at 8.44 is due to *ortho* protons *o*-H (22, 28) with ${}^{3}J(H-H) = 6.6$ Hz (Fig. 3a). Likewise, two sets of broad singlet at 8.37 and 8.08 ppm is due to *ortho* protons o'-H (34, 44) and o'-H (38, 40) (Fig. 3a). However, for *ortho* protons of **4** in CD₂Cl₂ at -90 °C, the rotation of phenyl group along C₁-C_{meso} bond in **4** is extremely slow, which is evident from the appearance of the four doublets at 8.49 [*o*-H (26, 32)],

8.42 [o-H (22, 28)], 8.31 and 8.04 ppm for o'-H (34, 44) and o'-H (38, 40) due to four different *ortho* protons of the aromatic ring in **4** (Fig. 3b).

Due to the ring current effect, upfield shifts for the ¹H resonances of (o-Cl)BA-Ph-H₆, (o-Cl)BA-Ph-H₃, (o-Cl)BA-Ph-H₅ and (o-Cl)BA-Ph-H₄ for **4** in CDCl₃ at 20 °C are $\Delta \delta = -3.2$ [from 7.77 (obtained from o-chlorobenzamide) to 4.57 ppm], -1.46 (from 7.42 to 5.96 ppm), -1.34 (from 7.35 to 6.01 ppm) and -1.08 (from 7.40 to 6.32 ppm), respectively. As the distance between the geometrical center (C_t) of the 4N plane [*i.e.*, N(1), N(2), N(3), N(4) for 3 and 4] and axial protons gets smaller, the shielding effect becomes larger. In **4**, the distance for $C_t \cdot \cdot (o-Cl)BA-Ph-H_6$, $C_t \cdots (o-Cl)BA-Ph-H_3$, $C_t \cdots (o-Cl)BA-Ph-H_5$ and $C_t \cdots (o-Cl)BA-Ph-H_4$, increases from 5.419, 6.284, 7.057 to 7.396 Å. As the (o-Cl)BA- $Ph-H_6$ proton of **4** is closer to C_t , the shielding gets larger for this (o-Cl)BA-Ph-H₆ protons. A similar ring current effect is also observed for **3**. The average distance between $C_t \cdots (o-Cl)BA-Ph-H_6$, $C_t \cdots (o-Cl)BA-Ph-H_5$, $C_t \cdots (o-Cl)BA-Ph-H_3$ and $C_t \cdots (o-Cl)BA-Ph-H_4$ for **3** increases from 3.233, 4.866, 5.946 to 5.984 Å. The ¹H NMR spectra reveal that the aromatic protons of the (o-Cl)BA group appear as a doublet of triplets at 6.36 ppm [(o-Cl)BA-Ph-H₄], a doublet at 6.21 ppm [(o-Cl)BA-Ph-H₃], the triplet at 6.10 ppm [(o-Cl)BA-Ph-H₅] and the other doublet at 4.87 ppm [(o-Cl)BA-Ph- H_6 for **3**. The (o-Cl)BA bonding argument is further supported by the result that at 20 °C in ¹³C NMR the (o-Cl)BA-Ph-C₁ [*i.e.*, C(46)] and the C(45) peaks in **3** were observed at 132.6 ppm with $^{3}J(TI-C) = 20.1 \text{ Hz}$ and at 167.7 ppm with $^{2}J(TI-C) = 665 \text{ Hz}$, respectively.

The ¹H NMR spectrum for OAc⁻ of **4** in CD₂Cl₂ displays a sharp singlet for CH₃ at δ 0.06 ppm with $\Delta v_{1/2}$ = 3 Hz at 20 °C and remains a sharp singlet for the same methyl proton at δ –0.01 ppm with $\Delta v_{1/2}$ = 4 Hz at –90 °C. This minimum deviation in the value of line width ($\Delta v_{1/2}$) upon cooling indicates that OAc⁻ exchange does not occur in compound **4**.

Upon cooling of a 0.02 M CD₂Cl₂ solution of **3**, the methyl proton signal of OAc⁻, being a single peak at 20 °C (δ 0.17 ppm), first broadened (coalescence temperature $T_c = -75$ °C) and then split into peaks with a separation of 14.4 Hz at δ 0.08 ppm at -90 °C. As the exchange of OAc⁻ within **3** is reversible, the results at 599.95 MHz confirm the separation as a coupling of ⁴*J*(Tl–H) rather than a chemical shift difference [10,11]. The most likely cause of loss of coupling is due to the reversible dissociation of acetate with a small dissociation constant.

$$Tl(N-NCO(o-Cl)C_6H_4-tpp)(OAc)$$

$$\stackrel{\text{cb_2ch_2}}{\longleftarrow} \text{Tl}(N-\text{NCO}(o-\text{Cl})\text{C}_6\text{H}_4\text{-}\text{tpp})^+ + \text{OAc}^-$$
(1)

Such a scenario would lead to the change in the chemical shift with temperature and no detectable free OAc⁻ and Tl(*N*-NCO(*o*-Cl)C₆H₄-tpp)⁺ at low temperature, but would lead to the loss of coupling between acetate and thallium at higher temperature [10–12]. The chemical shift in the high-temperature limit is the average of the two species (*i.e.*, Tl(*N*-NCO(*o*-Cl)C₆H₄-tpp)(OAc) and OAc⁻) in Eq. (1) weighted by their concentration. The free energy of activation at the coalescence temperature T_c for the intermolecular exchange of OAc⁻ in **3** is determined to be $\Delta G_{198}^{\neq} = 42.1$ kJ/mol. At 20 °C, intermolecular exchange of the OAc⁻ group for **3** is rapid as indicated by the appearance of singlet signals due to carbonyl carbons at 175.0 ppm and methyl carbons at 18.5 ppm. At -90 °C, the rate of intermolecular exchange of OAc⁻ for **3** in CD₂Cl₂ is slow. Hence,

at this temperature, the methyl and carbonyl carbons of OAc⁻ are observed at 17.5 ppm [with ${}^{3}J(TI-C) = 200 \text{ Hz}$] and 174.7 ppm [with ${}^{2}J(TI-C) = 204 \text{ Hz}$] as doublets, respectively [11].

X-ray diffraction analysis unambiguously confirms that **3** and **4** have a chelating bidentate OAC^- ligand in the solid state. The ^{13}C NMR chemical shifts were shown to be a useful tool for diagnosing the nature of acetate ligands, whether unidentate or bidentate in diamagnetic complexes. Unidentate acetate ligands were located at 20.5 ± 0.2 and 168.2 ± 1.7 ppm and bidentate acetate ligands at 18.0 ± 0.7 and 175.2 ± 1.6 ppm [13]. The methyl and carbonyl chemical shifts of the acetate group in **3** (or **4**) at 20 °C in CDCl₃ are separately located at 18.5 (or 18.9) and 175.0 (or 176.4) ppm confirming that the acetate is chelating bidentately and is coordinated to the thallium (or cadmium) atom in **3** (or **4**) in the solution phase.

In conclusion, we have investigated two new, diamagnetic and mononuclear porphyrin complexes, namely, a thallium(III) complex $\mathbf{3}$ ·0.5H₂O and a cadmium(II) complex $\mathbf{4}$ ·CH₂Cl₂ and their X-ray structures are established. In $\mathbf{3}$, the N–H bond of the *o*-chlorobenzamido ligand is cleaved and the *o*-chlorobenzamido nitrogen participates in bonding to the thallium ion. Complex $\mathbf{3}$ is a bridged metalloporphyrins with a metal–N–N linkage. In $\mathbf{4}$, the (*o*-Cl)BA substituent is left intact and the cadmium(II) ion is coordinated to the four nitrogens [N(1)–N(4)] of the macrocycle core. Compound $\mathbf{4}$ is a cadmium(II) N-substituted-N-aminoporphyrin complex.

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Appendix A. Supplementary material

CCDC 695921 (for **3**·0.5H₂O) and 695922 (**4**·CH₂Cl₂) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.inoche.2010.01.022.

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