### Supporting Information for:

Conversion Between Doubly and Triply Carboxylate-Bridged Di(ethylzinc) Complexes and Formation of the (μ-Oxo)tetrazinc Carboxylate [Zn₄O(Ar_TolCO₂)₆]

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NMR Spectroscopic Data:

Figure S1: $^1$H-NMR spectrum (500 MHz) of crystals of 1 dissolved in C$_6$D$_6$. The asterisk indicates residual solvent. The ethyl group resonances at 1.11 and 3.26 ppm (highlighted by arrows) are due to a small amount of zinc ethoxide impurity.
Figure S2: $^1$H NMR spectrum (500 MHz) of 1·THF-$d_8$ in C$_6$D$_6$. Compound 1·THF-$d_8$ is the same as 1 except that the THF protons are all deuterons. Resonances at 3.49, 2.80, 1.37, and 1.14 ppm in the $^1$H NMR spectrum of 1 (Figure S1) have disappeared. This result confirms that the unusually broad resonances at 2.80 and 1.14 ppm arise from THF.
Figure S3: VT-\textsuperscript{1}H-NMR spectra (500 MHz) of the mixture of 1 and 2 produced by dissolving 1 in C\textsubscript{7}D\textsubscript{8}. The inset highlights the coalescence of the methyl groups, which is difficult to interpret owing to the overlap with the C\textsubscript{6}D\textsubscript{5}CD\textsubscript{2}H peak from the solvent. The expected quintet from C\textsubscript{6}D\textsubscript{5}CD\textsubscript{2}H is observed at high temperatures.
Figure S4: $^1$H-NMR spectra (500 MHz) of 1 dissolved in C$_6$D$_6$ and in the presence of increasing amounts of THF. Labels for resonances corresponding to complex 1 and 2 are labeled for selected peaks. The intensity of 2 decreases with increasing THF while the intensity of 1 increases. The chemical shifts of 1 also shift with the increase in THF concentration. The THF peaks have been removed for clarity.
Figure S5: $^1$H-NMR spectrum (400 MHz) of 2 dissolved in toluene-$d_8$. The peaks at 0.87 and 1.25 ppm correspond to pentane.
Figure S6: $^{13}$C-$^1$H-NMR spectrum (500 MHz) of 2 dissolved in C$_6$D$_6$. The inset provides a close-up of the peaks near the solvent residual peak.
Figure S7: $^1$H NMR spectra (500 MHz) of 2 in C$_6$D$_6$ including peak assignments and the same spectrum with addition of THF, Et$_2$Zn, or both. The blue arrows indicate the presence of 1. The peaks between 0.8-1.0 ppm and an underlying multiplet at 1.19-1.23 ppm correspond to hexane and methylcyclopentane impurities in the Et$_2$Zn solution.
Figure S8: $^1$H-NMR spectrum (500 MHz) of crystals of 3 (black) and Ar$_{\text{Tol}}$CO$_2$H (blue) in CD$_2$Cl$_2$ superimposed. The superimposition highlights the large difference in chemical shifts in the resonances in 3 from those of the free acid. The large shift of greater than 1.00 ppm of the methyl resonance from 2.47 ppm in the free acid to 1.43 ppm in 3 is explained by the $T_d$-symmetric shell of 3 that forces each Ar$_{\text{Tol}}$ methyl group to be positioned above the aromatic system of another Ar$_{\text{Tol}}$ phenyl ring.
NMR Spectroscopic and DOSY NMR Data:

*NMR spectroscopy.* Spectra were referenced to residual solvent peaks. ¹H, ¹³C, and VT-NMR data were obtained on 500 MHz Varian Inova spectrometers or a 400 MHz Bruker Avance spectrometer. The ¹H DOSY NMR data were recorded on a 400 MHz Bruker Avance spectrometer using the *ledbpgp2s* pulse program with sine shaped gradients. The gradient length was set to 1700 µs, the recovery delay to 0.1 ms, and the diffusion delay to 0.1 s. The data were collected at 298 K across 32 experiments ranging from 5% gradient strength to 95% gradient strength at linear increments.

*DOSY data analysis.* Diffusion coefficients from the DOSY data were determined from fitting the exponential decay of intensity for each peak using the relaxation module in Bruker TopSpin 3. The average of the diffusion coefficients of each well resolved peak for a particular compound was used as its diffusion coefficient. Hydrodynamic radii (R_H) were determined by the Stokes-Einstein relation:

$$R_H = \frac{kT}{6\pi\eta D} \quad (2)$$

Here, k is the Boltzmann constant, T is temperature, η is the viscosity of the solvent, and D is the measured diffusion coefficient. X-ray radii (R_X-ray) were determined by dividing the unit cell volume of the crystal structure by Z and computing that volume by using the equation of a sphere.

| Table S1: Summary of DOSY Data and Calculated Hydrodynamic Radii (R_H) Values. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Spectrum | Compound | Peaks Used (ppm) | Diff. Coeff. (avg, x 10⁻¹⁰ m²/s) | R_H Value (Å) |
| Fig. S9 | 1 | 2.09 | 6.18 | 6.09 |
| Fig. S9 | 2 | 1.97, 1.12, -0.44 | 5.43 | 6.93 |
| Fig. S9 | Et₂Zn | 1.19, 0.14 | **11.37** | **3.31** |
| Fig. S10 | THF | 3.44, 1.38 | 13.91 | 2.71 |
| Fig. S10 | 1 | 7.47, 2.16 | 5.90 | 6.38 |
| Fig. S10 | 2 | 1.97, 1.08, -0.48 | 5.54 | 6.80 |
| Fig. S10 | Et₂Zn | 1.33, 0.20 | **7.51** | **5.01** |
| Fig. S10 | THF | 3.55, 1.46 | 20.81 | 1.81 |
| Fig. S11 | 2 | 7.33, 7.23, 6.96, 2.75, 1.97, -0.43 | 4.82 | 7.81 |

*The resonances labeled as diethylzinc are in equilibrium with 1 and 2 (see text). Because the equilibrium is shifted toward compound 1 in Figure S10 from that in Figure S9, the diffusion coefficient from the “diethylzinc” peaks is significantly lowered.*
Figure S9: $^1$H DOSY NMR spectrum (400 MHz) of 1 dissolved in toluene-$d_8$. 
Figure S10: $^1$H DOSY NMR spectrum (400 MHz) of 1 and 37 equiv. of THF-$d_8$ dissolved in toluene-$d_8$. 
Figure S11: $^1$H DOSY NMR spectrum (400 MHz) of 2 dissolved in toluene-$d_8$. 
Miscellaneous Tables and Figures:

Table S2: Summary of Ethylzinc Carboxylates and their Carboxylate-to-Ethyl Ratios.

| Compound | RCO₂:Et | Solvent | Ref. |
|----------|---------|---------|------|
| [Zn₅(CH₃CO₂)₆(Et)₄] (9) | 3:2 | C₇H₈, C₆H₆ | 9 |
| [Zn(CH₃CO₂)(Et)]ₙ (10) | 1:1 | THF | 10 |
| [Zn₆(PhCO₂)₆(Et)₆] (11) | 1:1 | CH₂Cl₂, C₈H₁₄ | 8 |
| [Zn₂(PhCO₂)₃(Et)(THF)] (12) | 3:1 | THF | 11 |
| [Zn₂(ArMesCO₂)₂(Et)₂] (13) | 1:1 | CH₂Cl₂ | 7 |
| [Zn₂(ArTolCO₂)₃(Et)(THF)] (1) | 1:1 | THF | a |
| [Zn₂(ArTolCO₂)₃(Et)(THF)] (2) | 3:1 | C₇H₈, C₆H₆ | a |

*aThis work.

Figure S12: Structure of H₂PIM.

X-ray Data Collection and Refinement. Single crystals of 1, 2, and 3 were coated with Paratone oil and mounted on a Bruker SMART APEX CCD X-ray diffractometer using Mo Kα radiation (λ = 0.71073 Å). Data were collected at 100 K and the diffractometer was controlled by the APEX2 (v. 2010.1-2) software package. SAINT³ was used for data reduction and SADABS⁴ was used to apply empirical absorption corrections for 1 and 3 and TWINABS⁵ was used for 2. Initial solutions were determined by Patterson methods and were refined by full-matrix least squares based on F² of the data with the SHELXTL-97 software package for 1 and SHELX-2013 for 2 and 3.⁶ PLATON was used to check for higher symmetry.⁷ Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed at idealized positions and refined using a riding model with the thermal parameters set to 1.5 (methyl hydrogen atoms) or 1.2 (non-methyl hydrogen atoms) times the equivalent isotropic displacement parameters of the atom to which they are attached. Refinement details and selected structural parameters for 1-3 are provided in the tables below.
|                | 1                              | 2                              | 3                              |
|----------------|---------------------------------|---------------------------------|---------------------------------|
| Empirical formula | [Zn₂O₆C₅₄H₆₀]                  | [Zn₂O₇C₆₉H₄₄]                 | [Zn₄O₁₃C₁₂₆.₂₂H₁₀₂.₁₈]³     |
| Formula weight  | 935.76                          | 2412.14                        | 2088.34                        |
| Temperature (K) | 100(2)                          | 100(2)                          | 100(2)                          |
| Wavelength (Å)  | 0.71073                         | 0.71073                         | 0.71073                         |
| Crystal system  | Triclinic                       | Triclinic                       | Trigonal                        |
| Space group     | P1                              | P1                              | R₃                             |
| a               | 9.9717(7) Å                     | 15.384(2) Å                    | 24.9722(6) Å                   |
| b               | 10.6664(8) Å                    | 19.152(3) Å                    | 24.9722(6) Å                   |
| c               | 11.9804(9) Å                    | 23.061(3) Å                    | 14.2207(6) Å                   |
| α               | 114.4700(10) °                  | 68.151(2) °                    | 14.2207(6) Å                   |
| β               | 97.5130(10) °                  | 89.974(2) °                    | 14.2207(6) Å                   |
| γ               | 94.7570(10) °                  | 82.900(2) °                    | 14.2207(6) Å                   |
| Volume (Å³)     | 1136.50(14)                     | 6250.5(15)                     | 7680.1(5)                      |
| Z               | 1                              | 4                              | 3                              |
| Calculated density (g/mm³) | 1.367                          | 1.282                          | 1.355                          |
| Absorption coefficient (mm⁻¹) | 1.106                          | 0.822                          | 0.991                          |
| F(000)          | 492                            | 2522                           | 3250                           |
| Crystal size (mm³) | 0.10 x 0.08 x 0.05              | 0.55 x 0.31 x 0.20             | 0.30 x 0.20 x 0.12             |
| Θ range for data collection | 1.90 to 25.79 °                | 1.602 to 29.660 °              | 1.631 to 29.589 °              |
| Index ranges    | -12<=h<=12                      | -21<=h<=21                     | -34<=h<=34                     |
|                 | -13<=k<=13                      | -24<=k<=26                     | -34<=k<=34                     |
|                 | -14<=l<=14                      | 0<=l<=32                       | -19<=l<=19                     |
| Reflections collected | 18019                          | 155896b                        | 55471                          |
| Independent reflections | 4343                           | 44048b                         | 4795                           |
| Completeness to Θ | 99.7%                          | 100.0%                         | 100.0%                         |
| Absorption correction | Semi-empirical                 | Semi-empirical                 | Semi-empirical                 |
| Max. and min. transition | 0.7453 and 0.6630              | 0.745908 and 0.554282          | 0.7459 and 0.6883              |
| Data / restraints / parameters | 4343 / 0 / 282                 | 34565 / 366 / 1663            | 4795 / 2125 / 610              |
| Goodness-of-fit on F² | 1.037                          | 1.043                          | 1.258                          |
| Final R indices | R1 = 0.0314,                    | R1 = 0.0656,                   | R1 = 0.0811,                   |
|                 | [I>2σ (I)] wR2 = 0.0620         | wR2 = 0.1450                   | wR2 = 0.1866                   |
|                 | R1 = 0.0469,                    | R1 = 0.1198,                   | R1 = 0.0904,                   |
|                 | wR2 = 0.0671                    | wR2 = 0.1713                   | wR2 = 0.1906                   |
| Largest diff. peak and hole (eÅ³) | 0.384 and -0.393               | 0.994 and -0.784              | 0.409 and -0.634              |

³Error in the empirical formula results from the error in modeling a three part disorder using the SUMP command in SHELX.

bTotal reflections across both domains.
**Table S4**: Selected Bond Distances and Angles for Compound 1.

![Diagram of bond distances and angles](image)

| Bond Distances (Å)\(^a\) | Zn1-Zn1' | Zn1-O2 | Zn1-O3 |
|--------------------------|----------|--------|--------|
| Zn1-Zn1'                 | 4.0641(5)| 1.998(2)|        |
| Zn1-C1                   | 1.970(2) |        | 2.108(1)|
| Zn1-O1                   | 1.988(2) |        |        |

| Bond Angles (°)\(^a\) | C1-Zn1-O1 | O1-Zn1-O2 | O1-Zn1-O3 | O2-Zn1-O3 | 89.85(6) |
|-----------------------|-----------|-----------|-----------|-----------|----------|
| C1-Zn1-O2             | 121.74(8) | 108.43(7) | 91.58(6)  | 89.85(6)  |          |
| C1-Zn1-O3             | 114.44(8) | 88.71(6)  |           | 89.85(6)  | 89.85(6) |

\(^a\)The number scheme used matches the cartoon above and not necessarily the numbers assigned in the X-ray structure.

*Refinement details for 1*: Structure solution and refinement for 1 was straightforward. Half the molecule sits in the asymmetric unit and the full molecule is generated by an inversion center located in the center of the two zinc centers.

**Table S5**: Selected Bond Distances and Angles for Compound 2.

![Diagram of bond distances and angles](image)

| Bond Distances (Å)\(^a,\(^b\)| Zn1-Zn2 | Zn2-C1 | Zn2-O2 | Zn2-O4 | Zn2-O6 |
|------------------------------|--------|--------|--------|--------|--------|
| Zn1-Zn2                     | 3.326(2), 3.320(3) | 1.956(7), 1.942(8) |        |        |        |
| Zn1-O1                      | 1.915(3), 1.907(3) | 2.031(3), 2.072(3) |        |        |        |
| Zn1-O3                      | 1.939(3), 1.919(3) | 2.085(3), 2.080(3) |        |        |        |
| Zn1-O5                      | 1.923(3), 1.940(3) | 2.066(2), 2.039(3) |        |        |        |
| Zn1-O7                      | 2.013(7), 2.012(5) |        |        |        |        |

| Bond Angles (°)\(^a,\(^b\)| O1-Zn1-O3 | C1-Zn2-O2 | C1-Zn2-O4 | C1-Zn2-O6 | O2-Zn2-O4 | O2-Zn2-O6 | O4-Zn2-O6 | 97.0(1), 97.7(1) |
|-----------------------------|----------|-----------|-----------|-----------|-----------|-----------|-----------|------------------|
| O1-Zn1-O5                   | 125.2(1), 127.4(1) | 122.8(2), 120.2(2) |          |          |          |          |          |                  |
| O1-Zn1-O7                   | 112.7(1), 110.0(1) | 109.9(2), 108.6(2) |          |          |          |          |          |                  |
| O3-Zn1-O5                   | 96.1(2), 94.4(1)   | 122.7(2), 119.7(2) |          |          |          |          |          |                  |
| O3-Zn1-O7                   | 114.8(1), 114.7(1) | 102.6(1), 104.1(1) |          |          |          |          |          |                  |
| O5-Zn1-O7                   | 100.8(2), 97.3(1)  | 100.6(1), 100.5(1) |          |          |          |          |          |                  |

\(^a\)The numbering scheme matches the cartoon above and not necessarily the numbers assigned in the X-ray structure determination files.
The two metrics correspond to the two isomers located in the asymmetric unit respectively. Metrics for the minor components of the disordered atoms in 2 are not included.

**Figure S13:** View of a ball and stick model of 2 along the Zn-Zn vector. The Ar\textsubscript{Tol} terphenyl groups are colored to aid in visualizing the pseudo-C\textsubscript{3} axis which gives rise to mirror image isomers as shown.

**Figure S14:** View of chains of 2 along c in the crystal lattice. The three Ar\textsubscript{Tol} groups of each molecule conform in either a Δ or Λ configuration and the configuration alternates along the chain. The colored Ar\textsubscript{Tol} groups and arrows demonstrate how rotation into the alternative configuration would switch the configuration along the entire chain by a domino effect.

**Refinement details for 2:** In the solid state, compound 2 exists as mirror image isomers generated from a pseudo-C\textsubscript{3} axis along the Zn-Zn vector (Figure S13). The conformation of one isomer affects that of the neighbor and, to a first approximation, in the solid state 2 exists as chains of alternating mirror image isomers along the c axis (Figure S14). A twin-law was applied for a second domain due to non-merohedral twinning discovered by Cell\_Now\textsuperscript{8} (final contribution of the second domain was 26.2%). The space group is P\textoverline{1} and each asymmetric unit contains one of each enantiomer. Each molecule of 2
has two zinc environments. Each molecule of 2 has disorder that involves the exchange of the two zinc environments. This exchange creates an inversion of configuration. Thus, considering only the \(m\)-1,3-bis(\(p\)-tolyl)phenylcarboxylate portions of the molecules, the molecules align in chains of alternating configuration along \(c\). Due to the disorder that switches configuration on a local level, the chains of 2 along \(c\) are actually random sequences of mirror image isomers. The percent contributions of the main component of each molecule in the asymmetric unit are 78.3% and 80.5%. To model the ~20% smaller component, distance restraints were applied based on the metrics of the main component. During anisotropic refinement, the anisotropic parameters of the disordered components of 2 were restrained to be similar to prevent non-positive definite parameters. The large amount of disorder within the main component of the crystal structure resulted in a large variation in the anisotropic displacement parameters of non-solvent molecules. In the unit cell, a non-disordered benzene was found as well as a disorder between two pentane molecules and a benzene (45.3%, 38.9%, and 16.9% occupancy). The solvent molecule C-C distances were restrained to expected values and the benzene molecules were enforced to lay flat in the same plane. The anisotropic parameters of the solvent were set to be the same for each given solvent molecule.
Table S6: Selected Bond Distances and Angles for Compound 3.

| Bond Distances (Å) |       | Bond Angles (°)       |
|-------------------|-------|-----------------------|
| Zn1-Zn1’          | 3.187(2) | Zn1-O2               | 1.957(5) |
| Zn1-O1            | 1.948(7) | Zn1-O3               | 1.952(6) |
|                   |        | O1-Zn1-O2            | 108.2(3) |
|                   |        | O1-Zn1-O3            | 109.4(2) |
|                   |        | O1-Zn1-O4            | 109.3(2) |
|                   |        | O2-Zn1-O4            | 112.0(2) |
|                   |        | O3-Zn1-O4            | 109.0(2) |
|                   |        | Zn-O4-Zn             | 108.60(5), 110.33(5) |

The numbering scheme matches the cartoon above and not necessarily the numbers assigned in the X-ray structure determination files.

The remaining four Zn-O4-Zn angles are related by symmetry and take one or the other value.

Figure S15: Space filling models of 3 and cartoons of the Zn₄ core showing four faces of the Tₜ-symmetric shell aligning with zinc centers and four faces without zinc centers. Circular arrows show the designation of Λ and Δ faces. There are then two enantiomers, ΛLLLL and ΔΔΔΔ.
Refinement details for 3: Compound 3 crystalizes in the space group $R\overline{3}$ with $Z$ equal to 3. As discussed in the text, the structure of 3 is composed of a $T_h$-symmetric shell of Ar$^{Tol}$ groups and a $T_d$ basic zinc carboxylate core. The alignment of these symmetries creates a $T$-symmetric molecule to a first approximation in the solid state and 3 can be thought of as $\Delta\Delta\Delta\Delta$ and $\Lambda\Lambda\Lambda\Lambda$ isomers as shown in Figure S15. Because the generation of $T$ symmetry occurs at the intersection of the $T_d$ core and $T_h$ shell, the chirality is ‘hidden’ within the symmetric $T_h$ shell. Thus, for a racemic crystal, the orientation of the $T_d$ core is random throughout the lattice. This random distribution is observed in the Zn$_4$O$_{13}$ tetrahedral core of the molecule, which sits on an inversion center and thus renders both enantiomers on the same average position. Indeed, the electron density within the $T_h$ shell shows a chemically unreasonable Zn$_8$O cube surrounded by 24 oxygen atoms. The core disorder was modeled by setting the carboxylate oxygen atoms at half occupancy, the central oxygen on the inversion center at one sixth occupancy, the zinc atom on the three fold axis at one sixth occupancy, and the zinc atom that sits on a general position at half occupancy. The carboxylate -CO$_2^-$ carbon was set at full occupancy. The Ar$^{Tol}$ wing is disordered across three parts and required restraints to be placed on the anisotropic parameters of all Ar$^{Tol}$ carbon atoms to orient in similar direction (the SIMU card in SHELX). These anisotropic parameter restraints add greater than 2000 restraints to the model, which increases the data-to-parameter ratio, which was 7.86 before restraints were added and 11.34 after. This increase makes the model pass the recommendation that data to parameter ratios for structures exceed 10. In addition, similarity restraints were applied to the benzene rings of the Ar$^{Tol}$ wings and the seven atom sets of the tolyl groups were restrained to lie in the same plane. The final occupancies of the three parts of the disorder are 39.5%, 38.9%, and 21.9%. The benzene carbon attached to the carboxylate group (C301) is the atom that is closest to its disorder counterparts and thus, despite anisotropic parameter restraints, remains oblate in shape.

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