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Notes

Synthesis, Characterization, and Decomposition of the First Mononuclear Eight-Coordinate Indium(III) Benzoate, $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$

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Introduction

The existence of indium and gallium carboxylates is well documented.[1] Numerous homoleptic polynuclear indium(III) carboxylates, $[In(O_2CR)_3]_x$ (R = H, CH_3, C_2H_5, n-C_3H_7, $(CH_3)_2CH$, $(CH_3)_3C$),[2] as well as polynuclear organoindium(III) carboxylates, $[R_2In(O_2CR'[Hl])]_x$ (R = CH_3, C_2H_5, R'[Hl] = CH_3, C_2H_5; R = n-C_4H_9, R'[Hl] = C_2H_5)[3] are known; however, there is a void in the literature on analogous indium(III) benzoates. To the best of our knowledge, $Cl_2In(\eta^2-O_2CC_6H_5)(py)_2$ (py = pyridine), a six-coordinate mononucler species, is the only structurally characterized indium(III) benzoato complex to date.[4]

In this note we report the synthesis and full characterization of the first eight-coordinate mononuclear indium(III) benzoato complex, namely, $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$ from the reaction of indium metal with benzoyl peroxide in 4-methylpyridine at 25 °C. The preparation of this indium(III) benzoate is significant in the development of indium(III) carboxylato chemistry since the occurrence of eight-coordinate *mononuclear* indium(III) carboxylates is rare.[5]

Experimental Section

Materials and General Procedures. All operations involving moisture- and air-sensitive materials were performed under an inert atmosphere using standard Schlenk techniques and a double-manifold vacuum line. Solids were manipulated in a Vacuum Atmospheres Co. drybox equipped with a HE-493 Dri-Train. Solvents were freshly distilled from sodium benzophenone ketyl prior to use. Solutions were transferred via stainless steel cannulae and/or syringes. Indium powder (Aldrich) was used without additional purification. Benzoyl peroxide was deareated under vacuum at room temperature. Elemental analyses were performed by Galbraith Microanalytical Laboratories, Inc., Knoxville, TN. NMR spectra were recorded on a Bruker AC300F magnetic resonance spectrometer. The infrared spectrum was obtained from a KBr pellet using a Galaxy FT-IR 4020 spectrophotometer. Thermogravimetric analyses were performed under an atmosphere of nitrogen using a Perkin-Elmer TGS-II. Powder X-ray diffraction (XRD) data were collected using monochromated Cu Kα radiation on a Scintag PAD V and a Phillips APD diffractometer.

Preparation of $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$. A mixture of 150-mesh indium powder (0.50 g, 4.35 mmol) and benzoyl peroxide (1.58 g, 6.52 mmol) in 35 mL of 4-methylpyridine was stirred at ambient temperature for 6 days. The mixture was filtered, and the resulting off-white solid was washed with three 25-mL aliquots of hexanes and dried under vacuum for 2 h. Hexane, 150 mL, was added to the bright yellow filtrate to further precipitate the white solid. The supernatant was decanted, and the white solid was washed with two 25-mL aliquots of hexanes and dried under vacuum for 2 h. The solids were combined, recrystallized from 4-methylpyridine/hexanes (v/v 40/70) and dried under vacuum for 18 h. Yield: 53–60%.

Anal. Calcd (found) for $C_{33}H_{29}InO_6N_2$: C, 59.66 (59.34); H, 4.4 (4.52). ^{1}H-NMR (DMSO-d_6): δ 2.33 (s, 6H, Me), 7.28 (d, 4H, β), 7.46 (t, 6H, meta), 7.56 (t, 3H, para), 8.00 (d, 6H, ortho), 8.50 (d, 4H, α). ^{13}C-NMR (DMSO-d_6): δ 20.5, 124.8, 128.2, 129.9, 131.9, 148.0, 149.1, 171.1. IR (KBr disk, cm^{-1}): 3091 (m), 3069 (s), 3058 (m), 3048 (m), 3031 (m), 2926 (m), 1654 (m), 1623 (vs), 1611 (vs), 1600 (vs), 1566 (vs), 1543 (vs), 1507 (vs), 1496 (vs), 1418 (vs, br), 1347 (vs, sh), 1314 (s), 1306 (s), 1234 (vs), 1220 (m), 1175 (s), 1068 (s), 1021 (vs), 868 (vs), 814 (vs), 810 (vs), 721 (vs), 716 (vs), 690 (vs), 642 (m), 542 (m), 492 (vs).

X-ray Crystallography. Colorless single crystals, suitable for X-ray diffraction studies, were grown by slow interdiffusion of hexanes into a 4-methylpyridine solution of $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$. Pertinent crystallographic data are summarized in Table I. X-ray diffraction data were collected at 20 ± 1 °C on a 0.38 × 0.38 × 0.31 mm crystal using an Enraf-Nonius CAD-4 diffractometer. Unit cell parameters were determined from least-squares refinement using the setting angles of 25 reflections in the range $20 < \theta < 23°$. Intensity data were collected in the range $4 \le 2\theta \le 45°$ by the ω–2θ scan technique. Within index ranges ($-12 \le h \le 12$, $0 \le k \le 12$, $0 \le l \le 27$), 2449 unique reflections were collected, of which 2067 were considered observed, $F_o^2 > 3\sigma(F_o)^2$. Intensities were corrected for Lorentz, polarization, and absorption effects. Systematic monitoring of three representative reflections at regular intervals showed no changes in diffraction intensity.

All calculations were performed on a VAX computer using Enraf-Nonius MolEN.[6] The In atom was located using the Patterson function and was found to reside on a 2-fold rotation axis. The positions of the remaining non-hydrogen atoms were located and refined through a series of difference Fourier maps and full-matrix least-squares refinements. Thermal parameters for all of the non-hydrogen atoms except O(W100) and O(W200) were refined anisotropically. Hydrogen atoms (except water hydrogens) were located from a difference Fourier map and included in the structure factor calculations without refinement. Atomic scattering factors were taken from ref 7. Least-squares refinement of 200 parameters resulted in residuals $R(F_o) = 0.059$ and $R_w(F_o) = 0.079$ and in a quality-of-fit of 2.6. Final positional and thermal parameters are listed in Table II.

Results and Discussion

Oxidation of indium metal by benzoyl peroxide in 4-methylpyridine produces the mononuclear indium(III) benzoate in

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(1) (a) Mehrotra, R. C.; Bohra, R. *Metal Carboxylates*; Academic Press: London, 1983; pp 121–129, 178–181. (b) Tuck, D. G. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: New York, 1982; Vol. 1, p 719. (c) Wade, K.; Banister, A. J. In *Comprehensive Inorganic Chemistry*; Bailar, J. C., Jr.; Emeleus, H. J., Nyholm, R., Trotman-Dickenson, A. F., Eds.; Compendium Publishers: New York, 1973; Vol. 1, pp 1105–1106.

(2) Lindel, V. W.; Huber, F. *Z. Anorg. Allg. Chem.* **1974**, *408*, 167.

(3) (a) Hausen, H. D. *J. Organomet. Chem.* **1972**, *C37*, 39. (b) Einstein, F. W. B.; Gilbert, M. M.; Tuck, D. G. *J. Chem. Soc., Dalton Trans.* **1973**, 248. (c) Habeeb, J. J.; Tuck, D. G. *Can. J. Chem.* **1974**, *52*, 3950. (d) Nomura, R.; Fujii, S.; Kanaya, K.; Matsuda, H. *Polyhedron* **1990**, *9*, 361.

(4) Khan, M. A.; Peppe, C.; Tuck, D. G. *Acta Crystallogr., Sect. C* **1983**, *C39*, 1339.

(5) For example, $In(O_2CMe)_3L$ where L is a bidentate N,N'[Hl]-donor molecule, i.e., 2,2'[Hl]-bipyridine or 1,10-phenanthroline: Preut, V. H.; Huber, F. *Z. Anorg. Allg. Chem.* **1979**, *450*, 120.

(6) *MolEN, an Interactive Structure Solution Procedure*; Enraf-Nonius: Delft, The Netherlands, 1990.

(7) Cromer, D. T.; Waber, J. T. In *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, U.K., 1974; Vol. IV, Table 2.2B.

0020-1669/93/1332-4150$04.00/0

Table I. Crystallographic Data for $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2 \cdot 4H_2O$

chem formula $InO_{10}N_2C_{33}H_{37}$	space group $C2/c$ (No. 15)
fw 736.49	$T = 20$ °C
$a = 11.7195(8)$ Å	$\lambda = 0.710\,73$ Å
$b = 11.995(1)$ Å	$\rho_{calc} = 1.373$ g cm^{-3}
$c = 25.407(2)$ Å	μ(Mo Kα) = 7.04 cm^{-1}
$\beta = 94.177(6)°$	$R(F_o)^a = 0.059$
$V = 3562.0(8)$ Å^3	$R_w(F_o)^b = 0.079$
$Z = 4$	

[a] $R(F_o) = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w(F_o) = [\sum w|F_o| - |F_c|]^2/\sum w|F_o|^2]^{1/2}$; $w = 1/\sigma^2(|F_o|)$.

Table II. Positional Parameters and Their Estimated Standard Deviations for $In(\mu-O_2CC_6H_5)_3(4-Mepy)_2 \cdot 4H_2O$

atom	x	y	z	B,[a] Å^2
In	0	0.10564(5)	$^1/_4$	2.52(1)
O(10)	0.0705(4)	0.2736(4)	0.2248(2)	3.6(1)
O(21)	0.1192(5)	0.1312(4)	0.3212(2)	4.0(1)
O(22)	−0.0841(4)	−0.0438(4)	0.1967(2)	3.8(1)
N(31)	0.1466(5)	0.0444(5)	0.1996(2)	3.3(1)
C(10)	0	0.3261(8)	$^1/_4$	3.3(2)
C(11)	0	0.4501(8)	$^1/_4$	3.0(2)
C(12)	−0.0792(6)	0.5077(6)	0.2769(3)	3.8(2)
C(13)	−0.0790(8)	0.6223(7)	0.2770(5)	5.8(2)
C(14)	0	0.679(1)	$^1/_4$	8.9(5)
C(20)	0.1329(6)	0.0289(6)	0.3322(3)	3.2(1)
C(21)	0.2045(6)	−0.0057(6)	0.3801(3)	3.2(1)
C(22)	0.2620(7)	0.0741(7)	0.4122(3)	4.3(2)
C(23)	0.3257(8)	0.0405(9)	0.4568(4)	5.6(2)
C(24)	0.3316(9)	−0.071(1)	0.4698(4)	6.2(2)
C(25)	0.2776(9)	−0.1494(8)	0.4381(4)	5.7(2)
C(26)	0.2139(8)	−0.1161(7)	0.3938(3)	4.4(2)
C(32)	0.2081(7)	0.1106(6)	0.1719(3)	4.1(2)
C(33)	0.2937(8)	0.0699(7)	0.1413(3)	4.9(2)
C(34)	0.3172(7)	−0.0400(7)	0.1392(3)	3.9(2)
C(35)	0.2518(7)	−0.1083(6)	0.1685(3)	3.9(2)
C(36)	0.1688(6)	−0.0645(6)	0.1973(3)	3.5(1)
C(37)	0.4099(9)	−0.0849(9)	0.1084(4)	5.7(2)
O(W100)	0.456(2)	0.330(2)	0.4959(9)	18.6(6)*
O(W200)	0.030(1)	0.001(2)	0.0365(6)	17.3(5)*

[a] Starred values indicate that atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $(4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$.

yields of up to 60% (eq 1). $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$ can be

$$2In^0 + 3(C_6H_5CO)_2O_2 \underset{25\ °C}{\overset{4\text{-Mepy}}{\longrightarrow}} 2In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-Mepy})_2 \qquad (1)$$

stored under an inert atmosphere at room temperature for extended periods of time. TGA studies show that it is thermally stable up to 100 °C; at this temperature loss of 4-methylpyridine occurs. In contrast, pyridine adducts of indium(III) acetate and formate are unstable, losing pyridine slowly at room temperature.[8]

Single-crystal X-ray diffraction analysis reveals that $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2 \cdot 4H_2O$ is composed of an ordered array of discrete mononuclear eight-coordinate molecules positioned on a crystallographic 2-fold rotation axis. Interatomic bond distances and angles are presented in Table III. The solid-state molecular structure of $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$ is shown in Figure 1.

The immediate coordination sphere around the central indium(III) atom is best described as a pseudo square pyramid with each bidentate benzoate assuming a single position. The In atom is bound to six oxygen atoms from three equivalent (vide infra) bidendate benzoate groups. The In–O bond distances range from 2.225(6) to 2.413(5) Å. Within the symmetrically independent benzoato ligand, the In–O bond lengths are not equivalent. The In-O(22) bond length, 2.413(5) Å, is slightly longer (by 0.19 Å) than the In-O(21) bond length, 2.225(6) Å. Unsymmetrical bonding of chelating carboxylate groups to an indium(III) center is not unusual. Slight deviations within bidentate carboxylato In–O bond distances are reported for the eight-coordinate complex $In(O_2CMe)_3L$ [L = phen: In–O(21) = 2.370(7) Å and In–O(22) = 2.213(6) Å; In–O(31) = 2.422(7) Å and In–O(32) = 2.224(7) Å. L = bipy: In–O(11) = 2.394(6) Å and In–O(12) = 2.222(6) Å].[5] More pronounced unsymmetrical bonding is found in (acetato)(porphyrinato)indium(III), $(OEP)In(CO_2CH_3) \cdot 2CHCl_3$ (OEP = 2,3,7,8,12,13,17,18-octaethylporphyrinate(2−)). The central indium atom is bound to the acetato group through both oxygen atoms; In–O(1) is 2.60(2) Å and In–O(2) is 2.14(1) Å.[9] Also, in the dimeric μ-oxo-bridged neutral complex $[L_2In_2(CH_3CO_2)_4(\mu-O)] \cdot 2NaClO_4$ (L = 1,4,7-triazacyclononane), each indium(III) atom is bound to one monodentate and one bidentate acetate group. The two chelating In–O bond

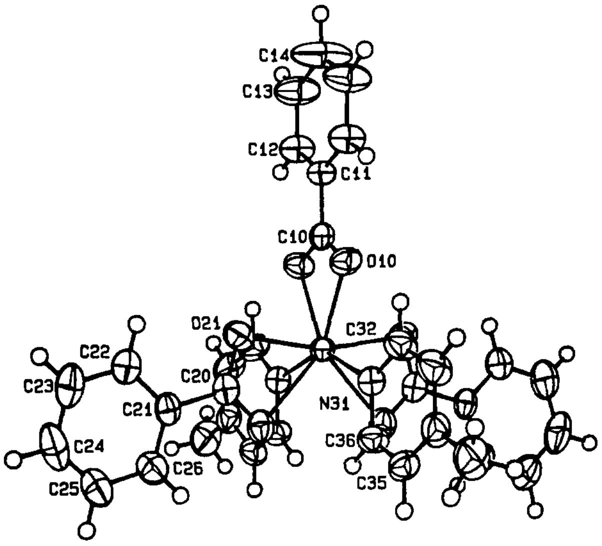

Figure 1. ORTEP drawing of the $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2$ molecule showing 50% thermal ellipsoids and the atomic labeling scheme.

Table III. Selected Bond Distances (Å) and Angles (deg) and Their Estimated Standard Deviations for $In(\eta^2-O_2CC_6H_5)_3(4-Mepy)_2 \cdot 4H_2O$

In–O(10)	2.286(5)	C(20)–C(21)	1.49(1)
In–O(21)	2.225(6)	C(21)–C(22)	1.40(1)
In–O(22)	2.413(5)	C(21)–C(26)	1.37(1)
In–N(31)	2.335(6)	C(22)–C(23)	1.37(1)
O(10)–C(10)	1.252(7)	C(23)–C(24)	1.37(2)
O(21)–C(20)	1.266(9)	C(24)–C(25)	1.37(2)
O(22)–C(20)	1.252(9)	C(25)–C(26)	1.36(1)
N(31)–C(32)	1.31(1)	C(32)–C(33)	1.40(1)
N(31)–C(36)	1.33(1)	C(33)–C(34)	1.35(1)
C(10)–C(11)	1.49(1)	C(34)–C(35)	1.38(1)
C(11)–C(12)	1.379(9)	C(34)–C(37)	1.49(1)
C(12)–C(13)	1.37(1)	C(35)–C(36)	1.36(1)
C(13)–C(14)	1.37(1)		
O(10)–In–O(10)	56.5(3)	N(31)–In–N(31)	143.3(3)
O(10)–In–O(21)	83.5(2)	N(31)–C(36)–C(35)	123.4(7)
O(10)–In–O(21)	82.5(2)	In–O(10)–C(10)	92.0(5)
O(10)–In–O(22)	129.7(2)	In–O(21)–C(20)	96.2(4)
O(10)–In–O(22)	132.1(2)	In–O(22)–C(20)	87.9(4)
O(10)–In–N(31)	80.1(2)	In–N(31)–C(32)	124.0(5)
O(10)–In–N(31)	136.6(2)	In–N(31)–C(36)	119.1(5)
O(21)–In–O(21)	164.1(3)	C(32)–N(31)–C(36)	116.9(7)
O(21)–In–O(22)	139.9(2)	O(10)–C(10)–O(10)	119 (1)
O(21)–In–O(22)	55.9(2)	O(10)–C(10)–C(11)	120.3(5)
O(21)–In–N(31)	92.6(2)	O(21)–C(20)–O(22)	120.0(7)
O(21)–In–N(31)	92.4(2)	O(21)–C(20)–C(21)	120.5(7)
O(22)–In–O(22)	84.1 (3)	O(22)–C(20)–C(21)	119.5(7)
O(22)–In–N(31)	75.2(2)	N(31)–C(32)–C(33)	122.0(7)
O(22)–In–N(31)	77.7(2)		

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(8) Habeeb, J. J.; Tuck, D. G. *J. Chem. Soc., Dalton Trans.* **1973**, 243.

(9) Cocolios, P.; Guilard, R.; Bayeul, D.; Lecomte, C. *Inorg. Chem.* **1985**, *24*, 2058.

lengths differ significantly, i.e., 2.14 and 2.87 Å.[10] It should be noted that, in most indium(III) bidentate carboxylato complexes, the In–O bond distances are equivalent within experimental error.[3,4,9] Within the benzoate groups, the bond distances and angles are in accordance with values reported for similar complexes.[4,11]

The central indium(III) atom is also coordinated to two symmetrically equivalent 4-methylpyridine ligands by their N-donor atoms. The In–N(31) bond distance of 2.335(6) Å is comparable to distances found in $In(O_2CMe)_3L$ (L = bipy, 2.325(6) Å; L = phen, 2.335(3) Å)[5] and is significantly longer than values reported for six-coordinate $Cl_2In(O_2CPh)(py)_2$, 2.28(2) Å.[4] Within the 4-methylpyridine ligands, the bond distances and angles are comparable to values reported for related complexes.[4]

The equivalency of the three chelating benzoates and the two 4-methylpyridine ligands is expected for a fluxional pseudo-square pyramidal arrangement. At room temperature, the ^{1}H-NMR spectrum shows a single pattern for the benzoate groups and 4-methylpyridine ligands (cf. Experimental Section).

A common reactivity/bonding characteristic of indium(III) complexes is the expansion of the indium(III) atom coordination sphere through polymerization or adduct formation.[2,3,8,12] In the case of indium(III) carboxylates (of which $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ is an example), the coordination number of the indium(III) atom generally increases to 6 or 8 via polymerization of the $[In(OOCR)_3]$ units—oxygen atoms from adjacent carboxylate molecules bridge the units, creating infinite $[In(OOCR)_3]_n$ chains. In $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ the presence of the two methylpyridine ligands prevents such polymerization by coordinatively saturating the indium(III) atom, resulting in the formation of a mononuclear eight-coordinate indium(III) benzoato species.

Thermal decomposition of $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ was followed by TGA, and the composition of the final pyrolysate was determined by XRD. No attempt was made to identify the intermediate pyrolysates produced during this analysis. The first two steps in the thermogram (see Figure 2) correspond to the sequential loss of the two 4-methylpyridine ligands. The final weight loss corresponds to complete decomposition of $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ to In_2O_3, as demonstrated by the X-ray powder diffraction pattern (Table IV).[13,14] The material is more than 90% crystalline In_2O_3, consistent with scanning electron microscopy, X-ray powder data, and thermal analysis.

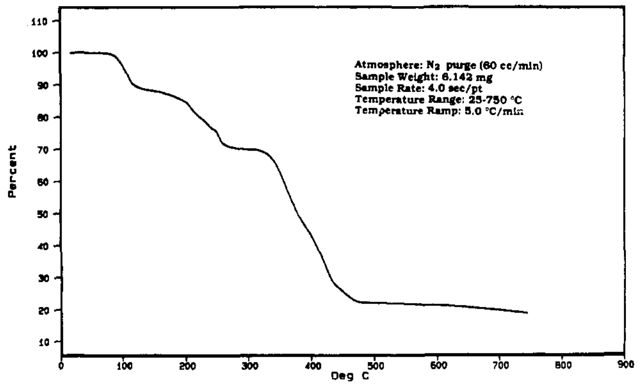

Figure 2. Thermogravimetric analysis of $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ under an atmosphere of nitrogen. Theoretical values for weight changes: 86%, $[In(O_2CC_6H_5)_3(4\text{-}Mepy)_2 - (4\text{-}Mepy)]$; 72%, $[In(O_2CC_6H_5)_3(4\text{-}Mepy)_2 - 2(4\text{-}Mepy)]$; 21%, $In_2O_3/In(O_2CC_6H_5)_3(4\text{-}Mepy)_2$.

Table IV. X-ray Diffraction (XRD) Powder Pattern for Pyrolysate, In_2O_3, between 1.20 and 5.00 Å

angle, 2θ, deg	d, Å	I/I_{max}, %	angle, 2θ, deg	d, Å	I/I_{max}, %
21.522	4.126	1.18	59.200	1.56	8.30
30.750	2.905	100.00	60.675	1.525	58.17
35.510	2.53	35.19	52.222	1.491	13.16
37.725	2.383	3.48	63.700	1.46	12.67
41.895	2.155	11.72	65.198	1.43	4.85
45.710	1.983	6.64	68.030	1.377	8.11
49.302	1.847	3.11	69.475	1.351	5.16
51.115	1.786	65.64	73.745	1.284	5.63
52.745	1.734	2.99	75.102	1.264	8.50
56.075	1.638	7.54	76.272	1.247	7.92

Concluding Remarks

In summary, we have demonstrated a simple and direct one-step route to $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$, the first mononuclear eight-coordinate indium(III) benzoate and precursor to indium(III) oxide. Our approach to the synthesis of indium(III) carboxylates differs significantly from previously reported methods.[1-12] The presence of 4-methylpyridine at the initial stages of reaction precludes $In(O_2CR)_3$ polymerization by coordinatively saturating the indium(III) center as it is formed. In addition, we have established that $In(\eta^2\text{-}O_2CC_6H_5)_3(4\text{-}Mepy)_2$ is a stable inorganic precursor to indium oxide. A thorough discussion of material and thin-film preparation and characterization is forthcoming.

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Supplementary Material Available: Tables giving crystal data and details of the structure determination, complete bond lengths and angles, anisotropic thermal parameters, torsion angles, and hydrogen atom locations (6 pages). Ordering information is given on any current masthead page. Supplementary material may also be obtained from A.F.H. of the NASA Lewis Research Center.

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(10) Wieghardt, K.; Kleine-Boymann, M.; Nuber, B.; Weiss, J. *Inorg. Chem.* **1986**, *25*, 1654.

(11) Andras, M. T.; Duraj, S. A.; Hepp, A. F.; Fanwick, P. E.; Bodnar, M. M. *J. Am. Chem. Soc.* **1992**, *114*, 786.

(12) Kumar, R.; Mabrouk, H. E.; Tuck, D. G. *J. Chem. Soc., Dalton Trans.* **1988**, 1045.

(13) The diffraction pattern was matched with that of In_2O_3 in the JCPDS International Centre for Diffraction Data, 1989: Powder Diffraction File No. 6-0416.

(14) Chemical analysis showed total C, H, and N content below the detection limit.