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Aloysius F. Hepp and Maria T. Andras  
*Lewis Research Center  
Cleveland, Ohio*

Stan A. Duraj  
*Cleveland State University  
Cleveland, Ohio*

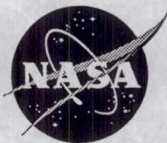
Eric B. Clark  
*Lewis Research Center  
Cleveland, Ohio*

David G. Hehemann  
*Kent State University  
Kent, Ohio*

Daniel A. Scheiman  
*Sverdrup Technology, Inc.  
Brook Park, Ohio*

Phillip E. Fanwick  
*Purdue University  
West Lafayette, Indiana*

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# SYNTHESIS AND DECOMPOSITION OF A NOVEL CARBOXYLATE PRECURSOR TO INDIUM OXIDE

ALOYSIUS F. HEPP,\* MARIA T. ANDRAS,\*‡ STAN A. DURAJ,\*\* ERIC B. CLARK,\*  
DAVID G. HEHEMANN,\*\*\*,‡ DANIEL A. SCHEIMAN,† AND PHILLIP E. FANWICK††

\*NASA Lewis Research Center, Photovoltaic Branch, M.S. 302-1, Cleveland, OH 44135

\*\*Department of Chemistry, Cleveland State University, Cleveland, OH, 44115

\*\*\*School of Technology, Kent State University, Kent, OH 44242

†Sverdrup Technology, Inc., 2001 Aerospace Parkway, Brook Park, OH 44142

††Department of Chemistry, Purdue University, West Lafayette, IN 47907

## ABSTRACT

Reaction of metallic indium with benzoyl peroxide in 4-methylpyridine (4-Mepy) at 25 °C produces an eight-coordinate mononuclear indium(III) benzoate,  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2 \cdot 4\text{H}_2\text{O}$  (I), in yields of up to 60%. The indium(III) benzoate was fully characterized by elemental analysis, spectroscopy, and X-ray crystallography; (I) exists in the crystalline state as discrete eight-coordinate molecules; the coordination sphere around the central indium atom is best described as pseudo-square pyramidal. Thermogravimetric analysis of (I) and X-ray diffraction powder studies on the resulting pyrolysate demonstrate that this new benzoate is an inorganic precursor to indium oxide. Decomposition of (I) occurs first by loss of 4-methylpyridine ligands (100°-200°C), then loss of benzoates with formation of  $\text{In}_2\text{O}_3$  at 450°C. We discuss both use of carboxylates as precursors and our approach to their preparation.

## INTRODUCTION

Our interest in oxygen-containing gallium and indium complexes derives from efforts to produce precursors for deposition of thin-film materials for solar cell fabrication [1]. Oxides are deposited or grown on solar cells to provide electrical insulation, to decrease surface recombination, or to produce anti-reflective coatings [2]. A major issue surrounding technological applications of InP is the deposition of insulators on the surface that are chemically stable with good electrical and interfacial properties. With this goal in mind, we are searching for easy to prepare and handle chemical vapor deposition (CVD) precursors to  $\text{In}_2\text{O}_3$ . Recently, we reported the preparation and characterization of the first main group oxo-centered trimeric carboxylate,  $[\text{Ga}_3(\mu_3\text{-O})(\mu\text{-O}_2\text{CC}_6\text{H}_5)_6(4\text{-Mepy})_3][\text{GaCl}_4]$  [3]. In an attempt to prepare a monomeric indium benzoate, we reacted indium metal with benzoyl peroxide in 4-methylpyridine at 25 °C and obtained  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$  (4-Mepy = 4-methylpyridine) (I) in good yields.

The existence of indium and gallium carboxylates is well-documented [4,5]. Numerous homoleptic polynuclear indium(III) carboxylates,  $[\text{In}(\text{O}_2\text{CR})_3]_x$  (R = H,  $\text{CH}_3$ ,  $\text{C}_2\text{H}_5$ ,  $n\text{-C}_3\text{H}_7$ ,  $(\text{CH}_3)_2\text{CH}$ , and  $(\text{CH}_3)_3\text{C}$ ) [6], as well as polynuclear organoindium(III) carboxylates,  $[\text{R}_2\text{In}(\text{O}_2\text{CR}')_3]_x$  (R =  $\text{CH}_3$ ,  $\text{C}_2\text{H}_5$ , R' =  $\text{CH}_3$ ,  $\text{C}_2\text{H}_5$  or R =  $n\text{-C}_4\text{H}_9$ , R' =  $\text{C}_2\text{H}_5$ ) are known [6-10]; however, there is a void in the literature on analogous indium(III) benzoates. To the best of our knowledge,  $\text{Cl}_2\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)\text{py}_2$  (py = pyridine), a six-coordinate mononuclear species, is the only structurally characterized indium(III) benzoato complex to date [11]. We describe the synthesis, structure and mass spectral analysis of the eight-coordinate mononuclear indium(III) benzoato complex (I). Thermogravimetric analysis (TGA) of (I) and X-ray diffraction powder (XRD) studies on the resulting pyrolysate demonstrate that this new benzoate is a precursor to indium (III) oxide,  $\text{In}_2\text{O}_3$ .

‡ - National Research Council/NASA Lewis Research Center Resident Research Associate.

‡‡ - Senior Research Fellow/NASA Lewis Research Center Resident Research Associate.

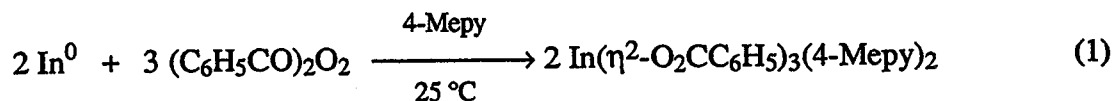
## EXPERIMENTAL

All operations of 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 deaerated under vacuum at room temperature. Elemental analyses were performed by Galbraith Microanalytical Laboratories, Inc. (Knoxville, TN). Thermogravimetric analyses were performed under an atmosphere of nitrogen using a Perkin Elmer TGS-II. Powder X-ray diffraction (XRD) data was collected using monochromated Cu K $\alpha$  radiation on a Scintag PAD V and a Phillips APD diffractometer. Electron impact mass spectra were recorded on a Finnigan TSQ-45 mass spectrometer. X-ray diffraction data were collected at 20  $\pm$  1  $^{\circ}$ C on a 0.38 x 0.38 x 0.31 mm crystal using an Enraf-Nonius CAD-4 diffractometer.

In( $\eta^2$ -O $_2$ CC $_6$ H $_5$ ) $_3$ (4-Mepy) $_2$  was prepared by reaction 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 at ambient temperature for several days. The mixture was filtered, and the resulting off-white solid washed with three 25 mL aliquots of hexane 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 fresh hexane and dried under vacuum for 2 h. The solids were combined, recrystallized from 4-methylpyridine/hexane (v/v 40/70) and dried under vacuum for 18 h, the yield is 53-60%. The analytical data was consistent with the single crystal X-ray structure and is detailed in a prior publication [12].

## RESULTS AND DISCUSSION

Oxidation of indium metal by benzoyl peroxide in 4-methylpyridine produces the first mononuclear indium(III) benzoate in yields of up to 60% (Eq. 1):



In( $\eta^2$ -O $_2$ CC $_6$ H $_5$ ) $_3$ (4-Mepy) $_2$  is very stable; it can be stored under an inert atmosphere at room temperature for extended periods of time. TGA studies show that it is thermally stable up to 100  $^{\circ}$ 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 [13].

Colorless single crystals, suitable for X-ray diffraction studies, were grown by slow interdiffusion of hexane into a 4-methylpyridine solution of (I), the compound crystallized as the tetrahydrate (I) $\cdot$ 4H $_2$ O, no doubt from trace amounts of water in the coordinating solvent. Crystallographic data are summarized in Table 1, a detailed description of the structural analysis is given in [12]. Single crystal X-ray diffraction analysis reveals that (I) $\cdot$ 4H $_2$ O is composed of an ordered array of discrete mononuclear eight-coordinate molecules positioned on a crystallographic two-fold rotation axis. The solid-state molecular structure of (I) 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) bidentate benzoate groups. The In-O bond distances range from 2.225(6) to 2.413(5)  $\text{\AA}$ . Within the symmetrically independent benzoate ligand, the In-O bond lengths are not equivalent. The In-O(22) bond length, 2.413(5)  $\text{\AA}$ , is slightly longer (by 0.19  $\text{\AA}$ ) than the In-O(21) bond length, 2.225(6)  $\text{\AA}$ . Unsymmetrical bonding of chelating carboxylate groups to an indium(III) center is not unusual [14].

TABLE 1.  
Crystallographic Data for  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2 \cdot 4\text{H}_2\text{O}$ .

chemical formula	$\text{InO}_{10}\text{N}_2\text{C}_{33}\text{H}_{37}$	formula weight	736.49
$a$	$11.7195(8) \text{ \AA}$	space group	$C2/c$ (No. 15)
$b$	$11.995(1) \text{ \AA}$	$T$	$20 \text{ }^\circ\text{C}$
$c$	$25.407(2) \text{ \AA}$	$\lambda$	$0.71073 \text{ \AA}$
$\beta$	$94.177(6)^\circ$	$\rho_{\text{calc}}$	$1.373 \text{ g cm}^{-3}$
$V$	$3562.0(8) \text{ \AA}^3$	$\mu(\text{Mo K}\alpha)$	$7.04 \text{ cm}^{-1}$
$Z$	4	$R(F_o)^a$	0.059
		$R_w(F_o)^b$	0.079

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

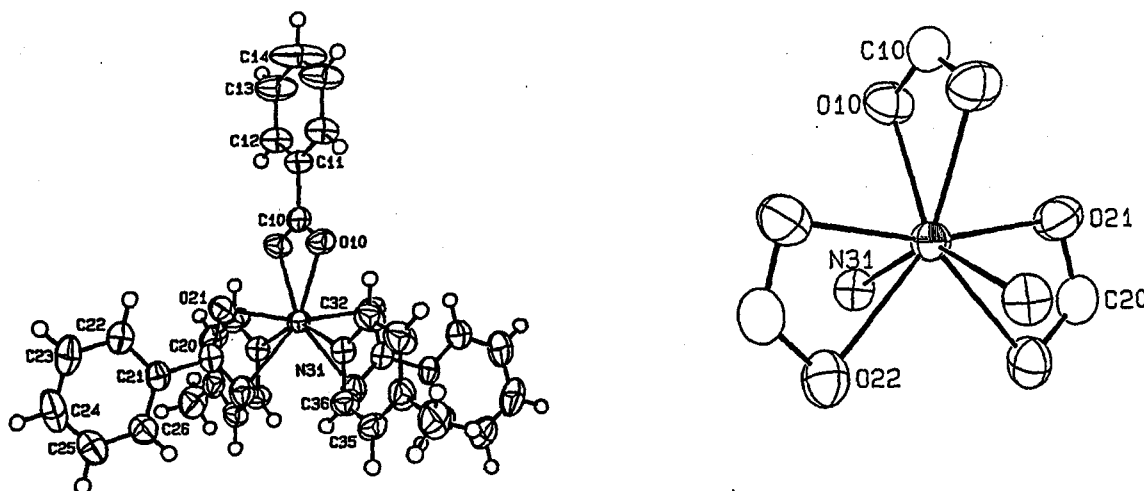


Figure 1. ORTEP drawing of the  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$  molecule showing 50% thermal ellipsoids and the atomic labeling scheme. Compound (I)  $\text{InO}_6\text{N}_2$  core on right.

A common reactivity/bonding characteristic of indium(III) complexes is the expansion of the indium(III) atom coordination sphere through polymerization or adduct formation [6-15]. In the case of indium(III) carboxylates (of which (I) is an example), the coordination number of the indium(III) atom generally increases to 6 or 8 via polymerization of the  $[\text{In}(\text{OOCR})_3]$  units - oxygen atoms from adjacent carboxylate molecules bridge the units creating infinite  $[\text{In}(\text{OOCR})_3]_n$  chains. In (I), the presence of the two methylpyridine ligands prevents such polymerization by coordinatively saturating the indium(III) atom, resulting in the formation of the first mononuclear eight-coordinate indium(III) benzoato species. The tendency to associate can be seen by the complete lack of parent ions in mass spectra with 25 and 70 eV ionization. The most intense peaks in the spectra, with  $m/e$  values: 105 and 77, correspond to loss of O and  $\text{CO}_2$  from the benzoate group to form  $\text{C}_6\text{H}_5^+$  and  $\text{C}_6\text{H}_5(\text{CO})^+$  ions. While much less intense, the strongest metal-containing ion peaks, in descending intensity order are  $m/e$  (for  $^{115}\text{In}$ ): 357, 313, 115, 269, 435, 479, these peaks can be assigned to, respectively:  $\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_2^+$ ,  $\text{C}_6\text{H}_5\text{In}(\text{O}_2\text{CC}_6\text{H}_5)^+$ ,  $\text{In}^+$ ,  $(\text{C}_6\text{H}_5)_2\text{In}^+$ ,  $(\text{C}_6\text{H}_5)\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_2^+$ , and  $\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_3^+$ . While the volatility of (I) is low at decomposition, the compound readily produces  $\text{In}_2\text{O}_3$ .

Thermal decomposition of  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$  was followed by TGA in both air and nitrogen, and the composition of the final pyrolysate 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 same results were obtained in air and nitrogen. This lack of effect on the formation of oxide is expected due to the  $\text{InO}_6\text{N}_2$  coordination environment around indium, it is also consistent with the ready decomposition of benzoate seen in the mass spectra.

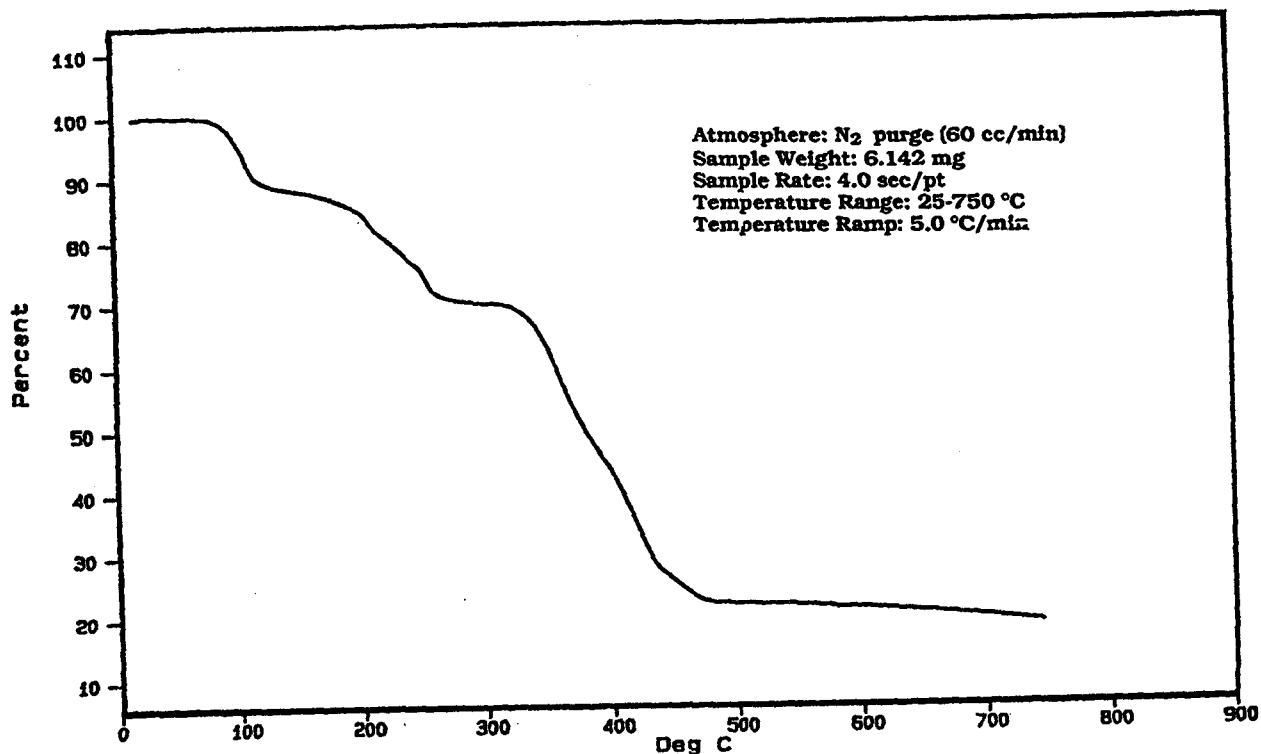


Figure 2. Thermogravimetric analysis of  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$  under an atmosphere of nitrogen. The theoretical values for weight changes are: 86%,  $[\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2 - (4\text{-Mepy})]$ ; 72%,  $[\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2 - 2(4\text{-Mepy})]$ ; 21%,  $\text{In}_2\text{O}_3/[\text{In}(\text{O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2]$ .

The final weight loss corresponds to complete decomposition of  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$  to  $\text{In}_2\text{O}_3$ , as demonstrated by weight loss and XRD pattern, Table 2 [16,17]. The most interesting aspect of the TGA data is the relatively low temperature (475 °C) for the stabilization of weight loss. By contrast, the polymeric  $\text{In}(\eta^2\text{-O}_2\text{CCH}_3)_n$  [13], did not reach a stable mass until between 1000 and 1100 °C, above the sublimation temperature of  $\text{In}_2\text{O}_3$  of 850 °C [18]. No attempt was made to characterize the intermediate materials. The morphology of material produced during a typical thermal analysis run is shown in a scanning electron micrograph, figure 3. As can be seen, melting has occurred in the material, as the temperature of the sample reached 750 °C; this is still 300 °C below the temperature needed to fully convert the polymeric acetate [13].

TABLE 2.

X-ray Diffraction (XRD) Powder Pattern for Pyrolysate,  $\text{In}_2\text{O}_3$ ,  
Between 1.20 and 5.00 Å.

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

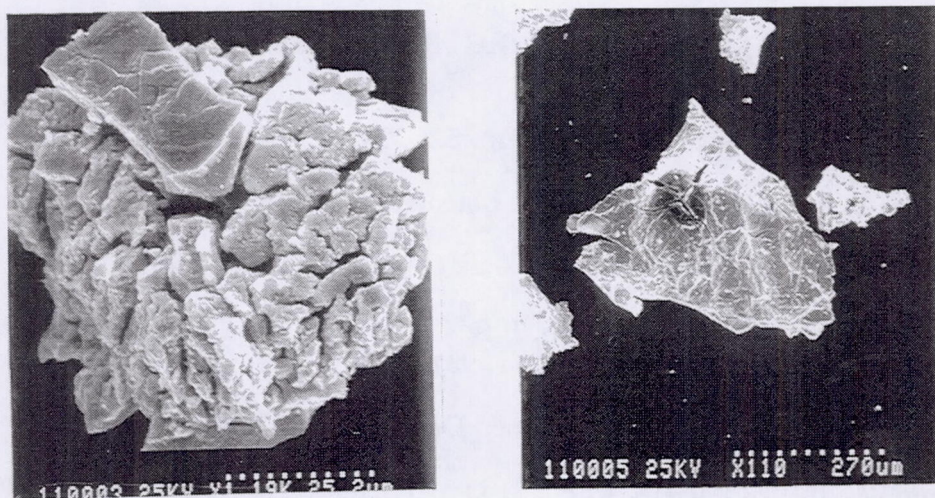


Figure 3. Scanning electron micrographs of typical samples of  $\text{In}_2\text{O}_3$  produced during TGA experiments and characterized by XRD, see text.

## CONCLUSIONS

We have demonstrated a simple and direct one-step route to  $\text{In}(\eta^2\text{-O}_2\text{CC}_6\text{H}_5)_3(4\text{-Mepy})_2$ , the first mononuclear eight-coordinate indium(III) benzoate. Our approach to the synthesis of indium(III) carboxylates differs significantly from previously reported methods [4-11]. The presence of 4-methylpyridine at the initial stages of reaction virtually eliminates all probability of  $\text{In}(\text{O}_2\text{CR})_3$  polymerization by coordinatively saturating the indium(III) center as it is formed. In addition, we have established that (I) is a stable inorganic precursor to indium oxide.

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14. For example,  $\text{In}(\text{O}_2\text{CMe})_3\text{L}$  where L is a bidentate N, N'-donor molecule, i.e., 2,2' bipyridine or 1,10-phenanthroline; see V.H. Preut and F.Z. Huber, *Anorg. Allg. Chem.* **450**, 120 (1979).
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17. Chemical analysis showed total C, H, and N content below detection limit.
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