THERMAL DECOMPOSITION OF SCANDIUM(III) 
o-NITROBENZOATE, o-CHLOROBENZOATE,
o-METHYLBENZOATE, 
o-HYDROXYBENZOATE AND o-AMINOBENZOATE 
IN AIR ATMOSPHERE

W. Brzyska and R. Kurpiel-Gorgol

DEPARTMENT OF INORGANIC AND GENERAL CHEMISTRY, 
INSTITUTE OF CHEMISTRY, MARIE CURIE SKŁODOWSKA UNIVERSITY, 
20-031 LUBLIN, POLAND

(Received September 18, 1986; in revised form, March 17, 1987)

The conditions of thermal decomposition of scandium o-nitrobenzoate, o-chlorobenzoate, o-
methylbenzoate, o-hydroxybenzoate and o-aminobenzoate were studied. On heating, the 
carboxylates decompose in two steps, only scandium anthranilate decomposes in one step. The 
hydrated complexes first lose water of crystallization and then are transformed to Sc₂O₃. The 
dehydration of the complexes is an endothermic process and the decomposition of anhydrous 
complexes is strongly exothermic. Scandium o-nitrobenzoate decomposes explosively.

Scandium(III) complexes with o-benzoic acids are little known. Crookes [1] has 
obtained basic scandium(III) o-methylbenzoate \( (\text{CH}_3\text{C}_6\text{H}_4\text{COO})_2\text{Sc(OH)} \cdot 3\text{H}_2\text{O} \) in the reaction of ammonium o-methylbenzoate and scandium(III) nitrate 
solutions. This compound loses water of crystallization at 150°. Crookes has 
prepared also scandium(III) o-methylbenzoate \( 2(\text{CH}_3\text{C}_6\text{H}_4\text{COO})\text{ScO} \cdot \text{Sc(OH)}_3 \) by adding o-methylbenzoic acid to a suspension of scandium(III) hydroxide. The 
prepared complex is sparingly soluble in water and ethanol, and soluble in dilute 
acids.

Prozorovskaya et al. [2, 3] have prepared scandium(III) salycilate 
\( \text{Sc}_2(\text{OHC}_6\text{H}_4\text{COO})_3\text{OH} \cdot \text{H}_2\text{O} \), recorded its IR spectra, studied the thermal 
decomposition and determined its density and solubility in water.

Scandium(III) anthranilate was prepared as an anhydrous neutral salt 
\( \text{Sc(NH}_2\text{C}_6\text{H}_4\text{COO})_3 \) [2, 4, 5] and its IR spectra and thermal decomposition were 
recorded. The compounds of scandium(III) with o-nitro- and o-chlorobenzoic acids are 
unknown.
The aim of our work was to obtain o-nitrobenzoate, o-chlorobenzoate, o-methylbenzoate (o-toluate), o-hydroxybenzoate (salicylate), and o-aminobenzoate (anthranilate) of scandium(III) and study their thermal decomposition in air.

**Experimental**

The o-nitro-, o-chloro-, o-methyl-, o-hydroxy- and o-aminobenzoate of scandium(III) were prepared in double decomposition reaction by adding equivalent amounts of 0.1 \( M \) solutions of ammonium o-nitrobenzoate \( (pH 3.5) \), o-chlorobenzoate \( (pH 4.5) \), o-toluate \( (pH 5.1) \), salicylate \( (pH 4.8) \) or anthranilic acid \( (pH 3.6) \) to a hot solution containing \( \text{Sc(NO}_3\text{)}_3 \) \( (pH 4.0) \). The precipitates formed were heated in the mother liquor for 1 h, filtered off, washed with water to remove \( \text{NH}_3^+ \) ions and dried at 30° to constant weight.

The carbon, hydrogen and nitrogen content of the prepared complexes was determined by elemental analysis using \( \text{V}_2\text{O}_5 \) as an oxidizing agent. The chlorine content was determined by the Schöniger method. The scandium(III) content was determined from the TG curves by converting the complexes to \( \text{Sc}_2\text{O}_3 \) at 900°. The water content was determined from the TG curves. The elemental analysis data are given in Table 1.

The obtained data indicate that the scandium(III) o-nitrobenzoate is a hemihydrated oxosalt with a metal to ligand ratio of 1:2, o-chloro- and o-methylbenzoate are hemihydrated salts with a metal to ligand ratio of 1:3, salicylate of scandium 3,5 hydrated salt with a metal to ligand ratio of 2:5 and anthranilate is an anhydrous salt with a metal to ligand ratio of 1:3.

The IR spectra recorded for prepared complexes over the range 4000–400 cm\(^{-1}\) confirmed the elemental analysis results. The prepared scandium o-benzoates are white solids, with the exception of scandium(III) o-nitrobenzoate which is cream coloured and o-aminobenzoate, which is brown coloured. The complexes are crystalline solids, sparingly soluble in water.

The thermal stability of the prepared complexes was studied. The TG, DTG and DTA curves were recorded. The measurements were made on a derivatograph at a heating rate of 9 deg·min\(^{-1}\) and sensitivity TG–100 mg. The samples were heated in air atmosphere in ceramic crucibles. The obtained results are given in Tables 2 and 3, and typical curves are illustrated in Fig. 1.

The studied scandium o-benzoates (with the exception of scandium o-aminobenzoate) on heating in air decompose in two steps. In the first step they are dehydrated endothermically in the temperature range 40–350°, yielding anhydrous salts. This is followed by ignition of organic anions, what is connected with exothermic effects. \( \text{Sc}_2\text{O}_3 \), which is formed at 480–690° is the final product of decomposition.

*J. Thermal Anal. 34, 1988*