

# The Missing Link Crystallized from the Ionic Liquid 1-Ethyl-3-methylimidazolium tosylate: Bis-aqua-(p-toluenesulfonato-O)-europium(III)-bis-p-toluenesulfonate dihydrate

*Si-Fu Tang and Anja-Verena Mudring\**

Anorganische Chemie I – Festkörperchemie und Materialien, Ruhr-Universität Bochum, 44801,

Bochum, Germany

E-mail: anja.mudring@rub.de

Supporting Information

## Synthesis

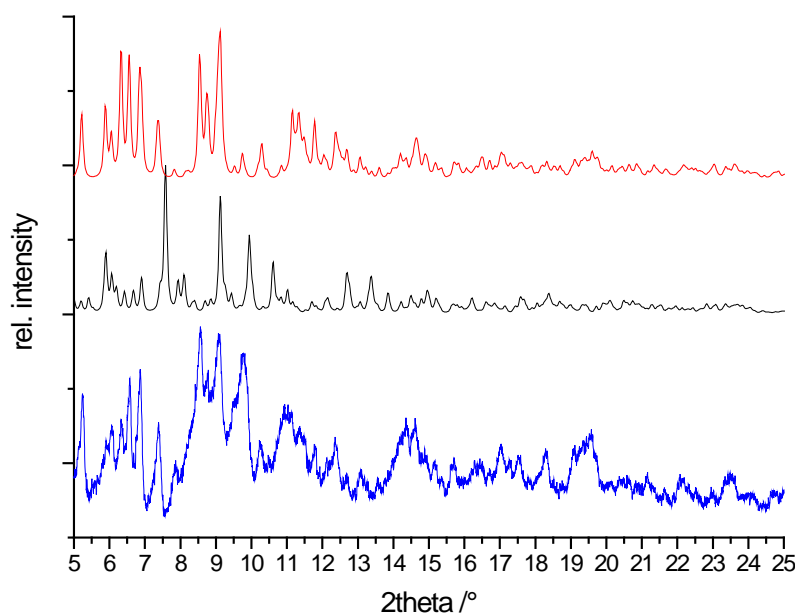
1-Ethyl-3-methylimidazolium tosylate ( $C_2mim$ )(p-Tos) was purchased from *Iolitec* (Denzlingen, D), p-toluenesulfonic acid monohydrate from ACROS.  $Eu_2O_3$  (99.99%) was purchased from Smart Elements.

All chemicals were used as received.

$Eu(p-Tos)_3$  was synthesized by dissolving a slight excess of europium oxide ( $Eu_2O_3$ ) in aqueous toluene-4-sulfonic acid and subsequent removal of the excess  $Eu_2O_3$  and liquid phase. The obtained salt was dried 48 hours under vacuum.

$[Eu(p-Tos)(H_2O)_7][p-Tos]_2(H_2O)_2$  was obtained from a solution of  $Eu(p-Tos)_3$  in  $(C_4mim)(p-Tos)$  (1:2 molar ratio) under ambient conditions. Needle shaped crystals of  $[Eu(p-Tos)(H_2O)_7][p-Tos]_2(H_2O)_2$  with sufficient quality for X-ray structure analysis precipitated from this solution after several months at room temperature.

## Powder X-ray diffraction analysis of the reaction product



**Figure SII.** PXRD pattern of the reaction product. Measured diffractogram of the product (bottom, blue), X-ray pattern of  $[\text{Eu}(\text{p-Tos})(\text{H}_2\text{O})_7][\text{p-Tos}]_2(\text{H}_2\text{O})_2$  (middle, black) and  $[\text{Eu}(\text{p-Tos})_2(\text{H}_2\text{O})_6][\text{p-Tos}](\text{H}_2\text{O})_3$  (top, red), simulated from single crystal structure data.

### Crystal structure determination of 1

Suitable crystals of  $[\text{Eu}(\text{p-Tos})(\text{H}_2\text{O})_7][\text{p-Tos}]_2(\text{H}_2\text{O})_2$  were selected, mounted in glass capillaries and checked for their quality on a single crystal X-ray diffractometer (IPDS II, Stoe, Darmstadt, D). A complete data set was measured in a cold nitrogen stream ( $T = 170(2)$  K) for the best specimen. Data reduction was carried out with the program package X-red<sup>1</sup> and numerical absorption corrections were carried out with the program X-Shape<sup>2</sup>. Crystal structure solution by direct methods using SIR92<sup>3</sup> yielded the heavy atom positions. Subsequent difference Fourier analyses and least squares refinement with SHELXL-97<sup>4</sup> allowed for the location of the remaining atomic positions. In the final step of the crystal structure refinement hydrogen atoms were added and treated with the riding atom mode. The hydrogen atoms on the water molecules are located from the difference Fourier maps and their isotropic

displacement factors were chosen as 1.2 times the preceding oxygen atoms. All non-hydrogen atoms were refined anisotropically.

(1)X-red. Stoe & Cie: Darmstadt, Germany, **2002**.

(2)X-Shape. Stoe & Cie: Darmstadt, Germany, **2002**.

(3)A. Altomare, G. Cascarano, C. Giacovazzo, *J. Appl. Crystallogr.* **1993**, 26, 343.

(4)W. S. Sheldrick, *SHELXL-97*; Universität Göttingen: Göttingen, Germany, **1997**.