

Supporting Information

Single-crystal X-ray diffraction. For crystallographic analysis of the ammonium $^{57}\text{Fe}(\text{II})$ sulfate, an Agilent Excalibur Gemini S single-crystal diffractometer, using mirror-monochromatic MoK_α -radiation ($\lambda=0.71073 \text{ \AA}$) at 293 K, was applied. Images were collected at a 65 mm fixed crystal-detector distance, using the oscillation method. Crystallographic calculations were carried out using CrysAlis CCD for data collection and CrysAlis RED for cell refinement and data reduction.ⁱ The crystal structures were solved by direct methods using SIR2008,ⁱⁱ and refined by full-matrix least squares on F^2 using SHELXL-97.ⁱⁱⁱ The X-ray powder diffraction pattern (XRPD) was recorded on the same diffractometer using $\text{CuK}_{\alpha 1}$ -radiation ($\lambda=1.5418 \text{ \AA}$). All data were collected at 293 K over the angular 2θ range $5\text{-}80^\circ$ with a step width of 0.057° . **Figure 1S** shows the unit cell composition of $(\text{NH}_4)_2^{57}\text{Fe}(\text{SO}_4)_2$ determined by X-ray diffraction. The melting point of the synthesized salt was 104.5°C which is in good agreement with the theoretical range $100\text{-}110^\circ\text{C}$.

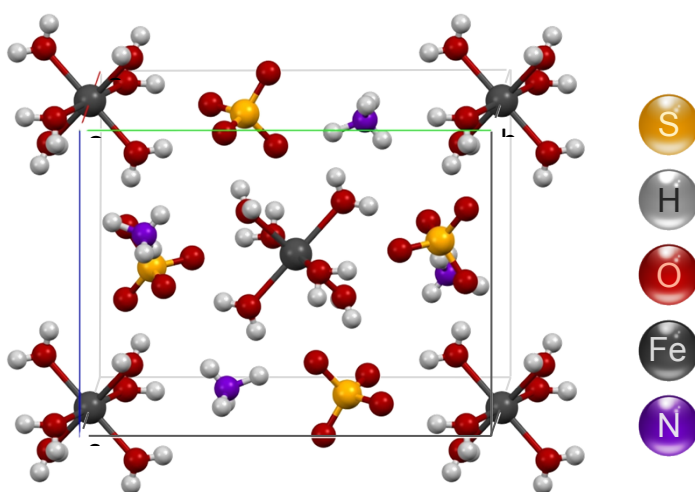


Figure 1S Unit cell composition of $(\text{NH}_4)_2^{57}\text{Fe}(\text{SO}_4)_2$ determined by X-ray diffraction.

Formation of the oligomers. The formation of oligomers occurred since the beginning of the synthesis providing chromatograms like those shown in Figure 3. The monitoring of the formation of the oligomers with time can be seen in Figure 2S when plotting the ratio monomer/oligomer. The product was chromatographically analysed to see the evolution of the product with time (1h, 2h, 3h, 7 d and 14d) in absence of Fe and oxygen (N_2 was bubbled through the solution). As can be seen, the ratio decreases (in

favour of the oligomer) by a factor of 2 in just three hours. This trend is maintained for the whole monitored period but the conversion slows down obtaining a final peak areas ratio of 1 after 14 days. The evolution of the complex after eliminating the Fe from the solution shows that the ultrafiltration was not sufficient to stop the oligomerization.

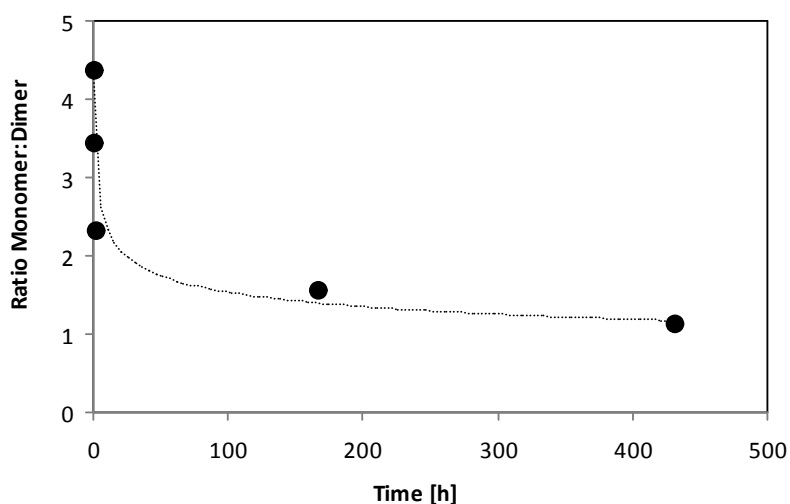
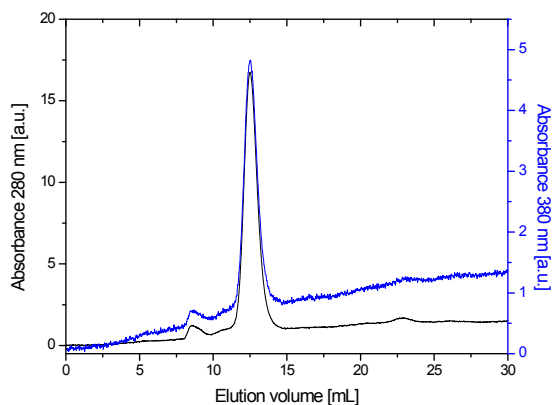


Figure 2S Evolution of the oligomers with time.

Stability of the guanidinium treated tracer. The tracer treated with guanidinium hydrochloride was stable for a month without apparent modification as can be observed in the two chromatograms of Figure 3S corresponding to the tracer analyzed by SEC-UV-VIS in the dates (A: 03/05/2013 and B: 27/05/2013).

A)



B)

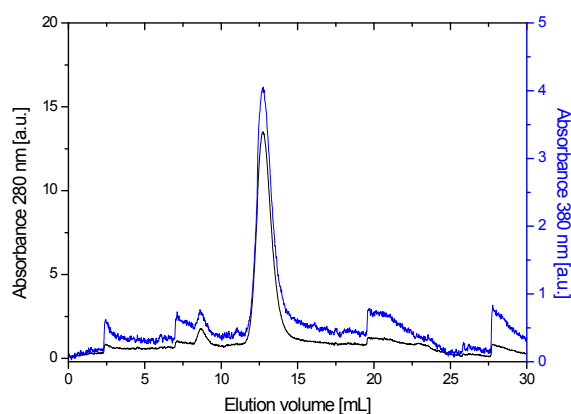


Figure 3S Stability of the tracer with time after treatment with guanidinium hydrochloride (injection of the same solution A: 03/05/2013 and B: 27/05/2013)

References

- ⁱ Oxford Diffraction Ltd., Abingdon, England, 2010, **Version 1.171.34.36**
- ⁱⁱ M. C. Burla, R. Caliandro, M. Camalli, B. Carrozzini, G. L. Cascarano,; L. De Caro, C. Giacovazzo, G. Polidori, D. Siliqi, R. Spagna, *J. Appl. Cryst.*, 2007, **40**, 609
- ⁱⁱⁱ G. M. Sheldrick, **SHELXS97 and SHELXL97**, University of Göttingen, Germany, 1997