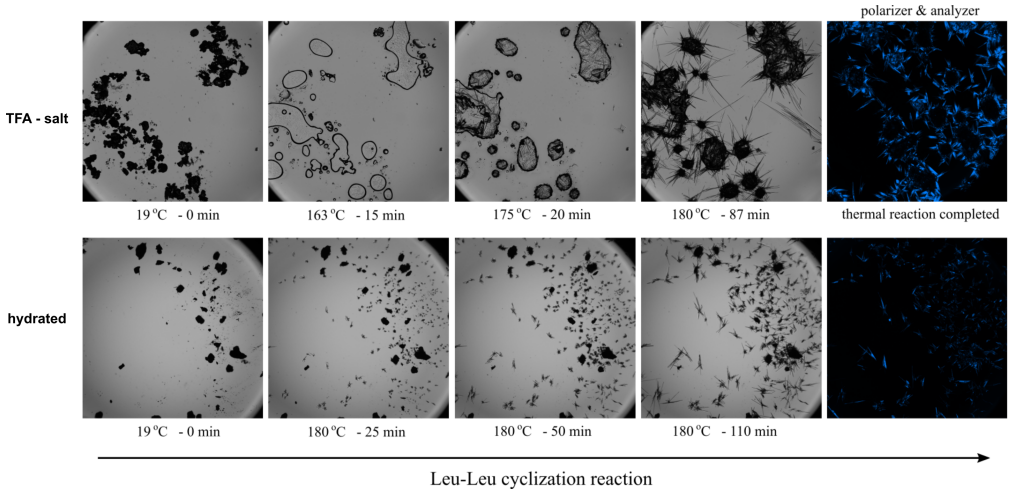
Unveiling the Impact of Trifluoroacetic Acid on Phase Transitions in Solid-State Synthesis of Cyclo Leu-Leu: Revelations and Implications

A. F. Perez Mellor[1], D. Rosa Gastaldo[1], C. Besnard[2], N. Giamboni[1], D. Chernyshov [3], X. Wang[1], J. Brazard [1], R. Cerny[2], T. Adachi[1], T. Buergi[1]*

[1]Department of Physical Chemistry, University of Geneva, Switzerland, [2]Laboratory of Crystallography, University of Geneva, Switzerland, [3]Swiss-Norwegian beamlines at The European Synchrotron Radiation Facility, Grenoble, France.

Oligopeptides, short chains of amino acids, have a unique ability for self-assembly, a feature widely exploited in the nanomaterials sector [1]. Their self-assembly into diverse nanostructures enables various applications, including biosensing technologies, organic semiconductor production, and targeted drug delivery systems [2-4]. The supramolecular structure and chirality of oligopeptides, which are intricately linked to the absolute configuration of the amino acid monomers, play a crucial role in this self-assembly [5]. Some oligopeptides, like the linear dipeptide (L)Leu-(L)Leu, can undergo a cyclization process when exposed to heat [6,7]. In this presentation, I will present a detailed analysis of cyclo Leu-Leu's solid-state synthesis, divided into three parts focusing on the role of the counterion during the synthesis, morphological changes, and the preservation of chirality.

Initially, we will focus on the role of the TFA counterion in the synthesis, using transient absorption infrared (IR) spectroscopy to analyze the dynamic changes over time. The second part will investigate the morphological changes during synthesis using optical microscopy (figure below), Raman spectroscopy, and X-ray powder diffraction for detailed structural analysis. Finally, we will conclude with an assessment of chirality conservation during the process using vibrational circular dichroism.



[1] Manchineella S, et al. Chempluschem (2017): 88-106. [2] Joon Seok Lee, et al. Angew. Chem. Int. Ed. (2011): 1196-1199. [3] Rondes F. Silva, et al. Langmuir (2013): 10205-10212. [4] S. Kogikoski, et al. Phys. Chem. Chem. Phys. (2016): 3223-3233. [5] Görbitz, et al. Chem. Eur. J. (2001): 5153-5159. [6] Ziganshin, M.A, et al. J. Therm. Anal. Calorim. (2016). [7] Marat A. Ziganshin, et al. J. Phys. Chem. B (2017).