

**Experiment title:**

Well Defined Silica-supported transition Metal Oxo Methyl synthesized by surface alkylation and their application in methane activation and metathesis reactions

Experiment number:

30-02 1082

Beamline: 30B	Date of experiment: from: 19 Nov 2014 to: 25 Nov 2014	Date of report:
Shifts: 18	Local contact(s): Isabelle Llorens	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

Samy Ould-Chikh, Anissa Bendjeriou,* Manoja Samantaray, Baraa Werghi, Julien Sofack-kreutzer,* Raju Dey, Ali Hamieh, Isabelle Llorens**

KAUST Catalysis Center (KCC), King Abdullah University of Science and Technology, Thuwal, Saudi Arabia

Report:**SOMC-designed silica supported tungsten Oxo imidazolin-2-iminato methyl precatalyst for olefin metathesis reactions**

Qureshi, Z.S., Hamieh, A., Barman, S., Maity, N., Samantaray, M.K., Ould-Chikh, S., Abou-Hamad, E., Falivene, L., D'Elia, V., Rothenberger, A., Llorens, I., Hazemann, J.-L., Basset, J.-M, *Inorganic Chemistry* (2017), 56(2), 861-871

Abstract

Synthesis, structure, and olefin metathesis activity of a surface complex [(Si-O)W(=O)(CH₃)₂-Im^{Dipp}N] (4) (Im^{Dipp} = 1,3-bis(2,6-diisopropylphenyl)-imidazolin-2-iminato) supported on silica by a surface organometallic chemistry (SOMC) approach are reported. The reaction of N-silylated 2-iminoimidazoline with tungsten- (VI) oxytetrachloride generated the tungsten oxo imidazolin-2-iminato chloride complex [Im^{Dipp}NW(O)Cl₃] (2). This was grafted on partially dehydroxylated silica pretreated at 700 °C (SiO₂₋₇₀₀) to afford a well-defined monopodal surface

complex $[(\text{Si-O})\text{W}(=\text{O})\text{Cl}_2\text{-Im}^{\text{DippN}}]$ (3). 3 underwent alkylation by ZnMe_2 to produce $[(\text{Si-O})\text{W}(=\text{O})(\text{CH}_3)_2\text{-Im}^{\text{DippN}}]$ (4). The alkylated surface complex was thoroughly characterized by solid-state NMR, elemental microanalysis, Raman, FT-IR spectroscopies, and XAS analysis. 4 proved to be an active precatalyst for self-metathesis of terminal olefins such as propylene and 1-hexene.