

# Preparation of metallocene complexes of titanium and zirconium with pendant nitrile groups

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## **Abstract:**

Two cyclopentadienide salts with pendant nitrile groups were prepared in the form of their corresponding lithium salts and these salts were utilized for the preparation of titanium and zirconium metallocene complexes. One of the ligands was already described in the literature, while the other as well as all the prepared group 4 metallocene complexes are new compounds. Starting materials for these syntheses were 6,6-dimethylfulvene and spiro[2.4]hepta-4,6-diene which, upon a nucleophilic attack by 2-lithio-2-methylpropanenitrile, afforded the corresponding substituted lithium cyclopentadienides, which were characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy and infrared spectroscopy. Then lithium salts were reacted with  $\text{Cp}^*\text{TiCl}_3$  and  $\text{Cp}^*\text{ZrCl}_3$  to give the corresponding metallocene dichlorides. Solid-state structures of the prepared titanocene dichlorides were elucidated by means of single crystal X-ray diffraction analysis. The obtained structures allowed a comparison with the known structures of the previously prepared homologous derivative. Unfortunately the corresponding zirconocene dichlorides did not crystallize. Moreover, all the newly prepared metallocenes were characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR, and IR spectroscopies and mass spectrometry. Melting points were determined and the elemental analysis performed for crystalline titanocene dichlorides. The prepared compounds, especially the lithium salts of substituted cyclopentadienes may be utilized in the follow-up work. They may serve as precursors for new bifunctional ligands or alternatively for complexes of other transition metals.

**Keywords:** substituted cyclopentadienyl ligands, nitrile, lithium salt, titanocene, zirconocene, X-ray structural analysis, nuclear magnetic resonance, mass spectrometry, infrared spectroscopy