Comprehensive Organic Chemistry Experiments for the Laboratory Classroom

Edited by Carlos A M Afonso, Nuno R Candeias, Dulce Pereira Simão, Alexandre F Trindade, Jaime A S Coelho, Bin Tan and Robert Franzén

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16.5. Polymerization of ε-Caprolactone Using a Ruthenium(II) Mixed Metallocene Catalyst

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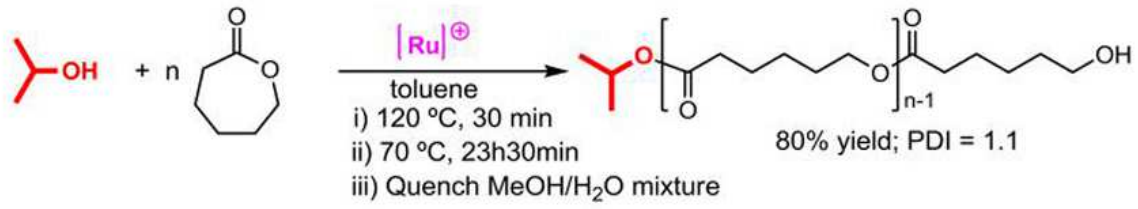
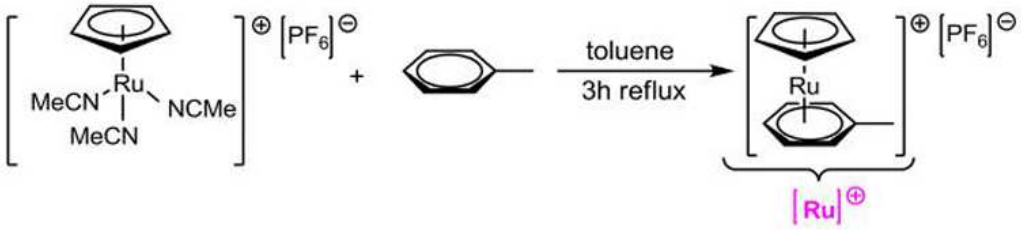


Synthesis of the Ru(II) catalyst:



Number of sessions (duration of each session)	Hazard level	Difficulty level	Level of study
2 (4 h + 2 h)	Moderate	High	Advanced

Class names Alcohol, ester

Concepts involved This experiment involves a method for the synthesis of a ruthenium(II) mixed metallocene compound and its use as catalyst in the polymerization of ε-caprolactone. This procedure is based on the ring-opening of the ε-caprolactone *via* an activated monomer mechanism (using isopropyl alcohol as initiator and the ruthenium(II) as catalyst)

Chemicals needed ε-Caprolactone, isopropyl alcohol, toluene, *n*-hexane, tris(acetonitrile)cyclopentadienylruthenium(II) hexafluorophosphate, deuterated acetone, deuterated benzene, methanol, dichloromethane, sodium metal, magnesium metal and calcium hydroxide as drying agents

Equipment and experimental techniques involved Apparatus for reaction with temperature (40–150 °C) and magnetic stirring, oil and sand baths, Schlenk type flasks, vacuum/nitrogen line, NMR tube equipped with a Young valve, desiccator, round-bottom flasks

Keywords Liquid–solid separation, NMR, polycaprolactone, ring-opening polymerization, ruthenium organometallic catalyst

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Published by the Royal Society of Chemistry, www.rsc.org