Ring Opening Metathesis Polymerization of Cyclooctadiene

Introduction

The goal of the third part of our metathesis project is to demonstrate the effectiveness of your synthetic complex 2 for a ROMP reaction.

Experimental Section

This experimental section is based on work by Prof. Steve Nolan at the University of New Orleans, and Prof. Melanie Sanford at the University of Michigan, who are greatfully acknowledged for sharing this information with us.

Part B: RCM of diallylmalonate



Safety Recommendations: Cyclooctadiene is harmful if swallowed, inhaled, or absorbed through skin.

Chemical Data: Complete this table *before* running the experiment.

Compound	FW	Amount	Mmol	Mp [°C]	Density
cyclooctadiene					
Ru-catalyst 2					

Required Equipment: Glassware for this reaction -25 mL flasks containing a stir bar - should be dried in the oven for at least 8 h before starting the experiment. Glove bag. Septa. Magnetic stirrer. Centrifuge.

ROMP Reaction. A solution of catalyst **2** (8 mg, 0.01 mmol) in dry degassed CH_2Cl_2 (2 mL) was prepared under nitrogen. Cyclooctadiene (100 mg, 0.926 mmol) was added in one portion to the rapidly stirring reaction mixture, and the solution was stirred under nitrogen for 30 min at room temperature. After addition of 100 mg of ethyl vinyl ether, the mixture was stirred for an additional 60 min under nitrogen. The solution was added dropwise to ca. 30 mL of rapidly stirring methanol, which caused a white powder to precipitate. The precitipated was collected by filtration (a double or triple filtration might be necessary to collect all product) and dried under vacuum. A crude ¹H NMR spectrum was acquired.

Characterization of Product. ¹H NMR; IR. How do your data compare to the literature values of the product? Do you have any cyclooctadiene left?

Questions. **1.** How could the product be purified? **2**. What is the function of ethyl vinyl ether? **3**. What is a likely structure of the product? **4**. How would you determine the molecular weight of the product? **5**. Why is COD only a moderately active substrate for ROMP? What would be a more reactive substrate?

References:

Christopher W. Bielawski and Robert H. Grubbs, Macromolecules 2001, 34, 8838.