A Brief Summary of Work Done by Stewart during Your Trip

Previous Work

Prior to this meeting I had synthesized PIB with the following characteristics:

M_n = 9,333 g/mole (target was 10,000 g/mole)

PDI = 1.06

Conversion of single end-groups to double bond = 98-100%

This starting material was hydrosylilated to give PIB arms containing 98-100% triethoxysilyl end-groups. These triethoxysilyl-containing arms were then used for two things:

1. Star synthesis

1g of the above arm material was dissolved in 25 mL of THF and reacted at 70°C for 24 hours in the presence of 10 drops of 0.15 N HCL/H_2O catalyst. GPC and LS analyses showed that stars with 6 arms having a PDI = 1.07 were formed in a 20% yield.

2. New functionalized PIB synthesis

The formation of a new end-group, $Si(H)_3$, was attempted. This was done by reducing 1g of hydrosilylated arm material with $LiAlH_4$ in ether. Formation of this new group was accomplished; however, the exact percent conversion of $Si(OEt)_3$ to $Si(H)_3$ could not be determined accurately due to impurities.

Current Work

Increasing Catalyst Strength

Just before we had discussed the above results I wanted to investigate whether increasing the strength of the acid catalyst would increase the yield of star material. The star + arm mixture from the first reaction was dissolved in 25 mL of THF and reacted at 70°C for 24 hours in the presence of 10 drops of 0.5 N HCL/H_2O catalyst. GPC analysis showed that this mixture was essentially unchanged. LS analysis was not available.

We had decided that in order to increase the yield of star material we needed to try two things:

A. Increase the concentration of the arm material.

B. Use a more hydrophobic solvent with a higher boiling point.

More PIB was hydrosylilated and again 98-100% triethoxysilyl end-groups were obtained. As before this material was used for two things:

1. Star synthesis using o-dichlorobenzene as a solvent

1.1 About 0.8g of the star + arm mixture from the previous two star reactions was dissolved in 20 mL of
o-dichlorobenzene. This is close to the polymer concentration used in the reactions carried out in THF. This solution was heated to 147°C for 24 hours in the presence of 10 drops of 0.5 N HCL/H_2O catalyst. GPC showed that again the mixture was essentially unchanged. LS analysis was not available at the time.

1.2 2g of the hydrosylilated arm material were dissolved in 50 mL of o-dichlorobenzene. This solution was
heated to 147°C for 24 hours in the presence of 10 drops of 0.5 N HCL/H_2O catalyst. GPC showed that 5 armed stars with a PDI = 1.07 were formed in an 8% yield. LS analysis was not available at the time.

1.3 1g of the hydrosylilated arm material was dissolved in 5 mL of o-dichlorobenzene. This solution was
heated to 147°C for 24 hours in the presence of 10 drops of 0.5 N HCL/H_2O catalyst. GPC showed that 5 armed stars with a PDI = 1.06 were formed in a 14% yield. LS analysis was not available at the time.

2. New functionalized PIB synthesis

As was done before, the newly hydrosylilated PIB arm material was reduced with $LiAlH_4$ in ether.
This time the resultant polymer appeared to be of much higher purity than before. Analysis of this material was delayed due to the electrical shut-down

Future Work

I plan to do several things:

1. Hydrosylilate my remaining PIB and run the star syntheses at even higher polymer concentrations.

2. Polymerize more PIB for future use as arm material. I have been preparing for this reaction for several days.

3. Analyze my newly reduced PIB arms.

4. Analyze all previous star reactions with LS as soon as it is fixed.