Per your request, we performed a number of batch kinetics experiments in order to provide you with data to evaluate models for the kinetics of the first reaction in the production of the Lucretex monomers. The results of these 11 experiments are contained on the following two pages.

The experiments were carried out according to the following protocol:

1. Charge the 250 ml flask with 41.7 ml of toluene.
2. Charge the flask with 35 g of TMDS (R2).
3. Place the flask in a constant temperature bath for 1 hour.
4. Add 0.22 ml of platinum catalyst slurry at \( t = 0 \).
5. Add \( x \) g of allyl alcohol (R1) at \( t = 0 \).
6. Take 1 ml samples from the flask every 24 min. These samples are immediately quenched in ice water. Concentration analyses are done later to determine the concentrations of R1, R2, A, and C.
7. The reaction is allowed to run for 4 hours.

We were unable to accurately measure the hydrogen evolution rate as you requested. Everything had to be done with extreme care because the materials we were dealing with are highly toxic. In addition, the temperatures that were requested were above the boiling points of the initial reaction mixtures. Therefore, trials were done at lower temperatures.

The following runs were done: Three sets of experiments were done, each set at a particular temperature, leading to a total of nine runs. The temperatures investigated were 80, 85 and 90°C. In each set, the amount of R1 charged into the flask, \( x \), was varied.
$x$ took values of 15.135 g, 30.27 g, and 45.405 g. These values correspond to molar ratios of 1:1, 2:1, and 3:1 (R1:R2). Two final experiments were conducted at 85°C with $x = 30.27$ g with catalyst loadings of 0.11 ml and 0.33 ml.
# REACTION I KINETICS STUDY

Catalyst Loading: Base Amount: 0.22 ml

(Concentrations in Millimolar)

<table>
<thead>
<tr>
<th>Time, h</th>
<th>R1/R2=1/1</th>
<th>R1/R2=2/1</th>
<th>R1/R2=3/1</th>
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Catalyst Loading: Low: 0.11 ml

R1/R2 = 2/1

Temperature = 85°C

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Catalyst Loading: High: 0.33 ml

R1/R2 = 2/1

Temperature = 85°C

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## COMPARISON OF CONVERSIONS AND YIELDS AFTER 4.0 HOURS IN THE REACTOR

<table>
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<tr>
<th>R1/R2</th>
<th>TEMP, °C</th>
<th>CAT, ml</th>
<th>MOLS A/R2</th>
<th>MOLS C/R2</th>
<th>TOTAL YIELD</th>
<th>MOLS A/C</th>
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</thead>
<tbody>
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<td>1:1</td>
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<td>0.3116</td>
<td>0.0927</td>
<td>40.43%</td>
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<td>0.3009</td>
<td>0.1633</td>
<td>46.42%</td>
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<td>0.22</td>
<td>0.2825</td>
<td>0.2600</td>
<td>54.25%</td>
<td>1.087</td>
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<td>0.6678</td>
<td>0.0907</td>
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<td>0.6958</td>
<td>0.1559</td>
<td>85.22%</td>
<td>4.463</td>
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</tbody>
</table>

pib: August 29, 2005