

## Memo 1

**DIVERSIFIED CHEMICAL PRODUCTS****Specialty Products Division****Cambridge MA**

**TO:** U. R. Engineer  
**FROM:** I. M. Supervisor  
**DATE:** 2007 Sep 7  
**SUBJECT:** Lucretex Batch Process Development Project Description and Goals

Chemists in the Research and Product Development Division in Blacksburg VA have synthesized a promising new class of silicon-based polymers. These polymers have been under study for the past four years, and a series of pilot trials has recently been completed at the Blacksburg facility. Marketing executives at Corporate Headquarters in New York believe that the potential world market for this family of polymers (to be called Lucretex) exceeds \$200 million annually because of the exceptional strength and flow properties of the material.

Management has authorized the production of 310,000 lb of the new polymer for extensive test-marketing. The monomer will be produced here at our Cambridge plant because of the multiproduct nature of our facility. The polymerization step will subsequently be carried out by the Polymers Group in Worcester. Marketing would like to have the Lucretex as soon as possible in order to maximize market penetration. Pasadena Polymer Products is believed to have a competitive product under development.

You have been assigned the problem of developing the process to produce the monomer. A description of the pilot plant trials conducted by the Research and Product Development Division is attached in order to give you a starting point for your design. Additional data will be available from Blacksburg in the near future. Various equipment units will be made available here in the plant for your use. Existing equipment should be used wherever possible.

Management wants to know: (1) how much it will cost to make the monomer and (2) how soon the campaign can be completed. Cal Tech Industries (CTI) has just submitted a proposal to management that claims they can produce 310,000 lb of monomer in 25 days at a cost of \$9/lb. As your supervisor, I will see that suitable incentives are made available if we can beat the CTI performance figures.

You will prepare a detailed report to describe the proposed design, including the recommended equipment, operating procedures, safety analysis, and cost estimate. Corporate Headquarters indicates that no more than eight weeks should be taken for developing and optimizing your process design.

**Memo 1****PILOT PLANT RUN**

The following is a description of the recent successful pilot plant run for manufacturing the monomers used in production of Lucretex. Refer to Figure 1 for the molecular structure of the reactants and products, and Figure 2 for the stoichiometry of the main reactions taking place in the process.

The entire process was conducted in the single agitated glass-lined jacketed kettle of 150-gallon volume shown in Figure 3. The kettle was equipped with a 4-inch diameter overhead column packed with ceramic packing up to a depth of 48 inches. There was an overhead condenser with cooling water on the shell side. Steam and cooling water could also be passed through the kettle jacket. There were four receivers to receive the various products and byproducts.

The following amounts of reactants were measured out and stored in safe containers:

- 300 lb<sub>m</sub> tetramethyldisiloxane (TMDS)(R2)
- 261 lb<sub>m</sub> allyl alcohol (R1)
- 1 L Pt catalyst slurry (5 mol Pt per L toluene)
- 43.6 gal toluene
- 71.4 lb<sub>m</sub> methanol

All the toluene was charged into the clean kettle, followed by 20% of the catalyst. Steam passing through the jacket was used to bring the reactor to steady state under total reflux. The two reagents R1 and R2 were then added steadily at 0.6 - 0.7 lb<sub>m</sub>/min over about 10 hours. Additional catalyst was added every hour such that all the catalyst had been added at the end of 10 hours.

Vapor leaving the kettle bypassed the column into the overhead condenser, and condensate was returned to the kettle. The noncondensable hydrogen released in the reaction (Reaction 1) was diluted with nitrogen before exiting to the atmosphere. Temperature in the kettle was controlled by adjusting flow of cooling water/steam in the jacket. However, the temperature in the kettle dropped with time from an initial value of 110°C to a final value of 95°C.

After 10 hours, the reaction mass was further heated under reflux for an hour when an IR spectrum of a sample showed no residual Si-H bonds. The reactor was allowed to cool to 75 - 80°C, all the methanol charged into it, and refluxed for a couple of hours. During this time, Reaction 2 occurred, leading to formation of E.

The next step in the process was atmospheric distillation. Steam was passed through the jacket as the heating medium. Vapor from the kettle passed through the column into the overhead condenser, and the condensate was collected in Receiver 1. Rectification was conducted until the pot temperature reached 135°C. This extracted all the unreacted allyl alcohol, methanol, and bulk of the toluene. (Safety note: above 140°C, the Pt catalyzes highly undesirable exothermic side reactions, leading to the formation of impurities and/or thermal runaway due to the heat generated by reaction.)

**Memo 1**

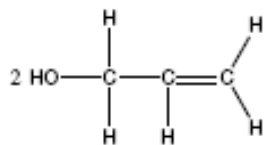
The kettle was then cooled in preparation for the vacuum distillation. A vacuum of 5 mm Hg was applied to the kettle and a second overhead cut was taken in Receiver 2. This was composed of the remaining toluene and most of the A and E. The pot residue was mostly Pt catalyst and some oligomers.

The residue from the kettle was removed, the kettle cleaned with toluene, and the A and E from Receiver 2 transferred back to the kettle. The hydrolysis reaction was carried out by adding excess water.

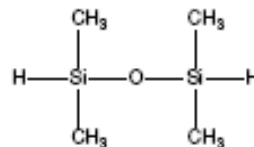
At the end of the hydrolysis reaction (Reaction 3), another atmospheric distillation was conducted. This time the overhead fraction was predominantly water, methanol, and toluene. The distillation proceeded until the pot temperature reached 160°C. A and D were left in the bottoms, and 434 lb<sub>m</sub> of this mixture was recovered for polymerization.

The total cycle time for the above process was 80 hours. The total yield of A+D was 92%. Figure 4 shows schematically the sequence of processing tasks to make A+D.

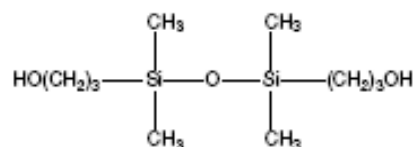
Allyl Alcohol (R1)



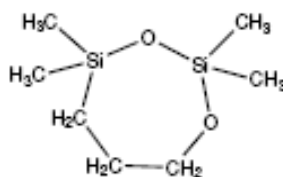
Tetramethyldisiloxane (R2)



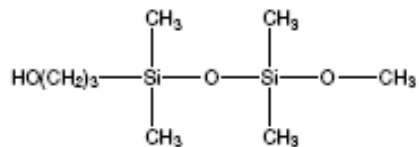
Molecule A



Molecule C



Molecule E



Molecule D

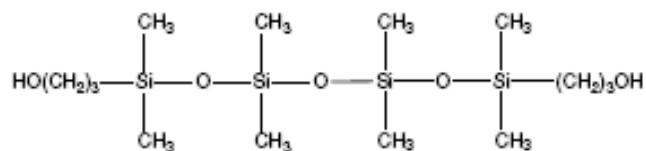
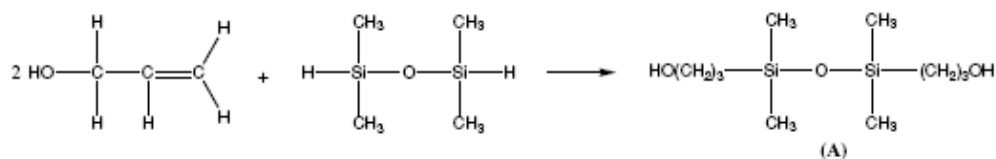
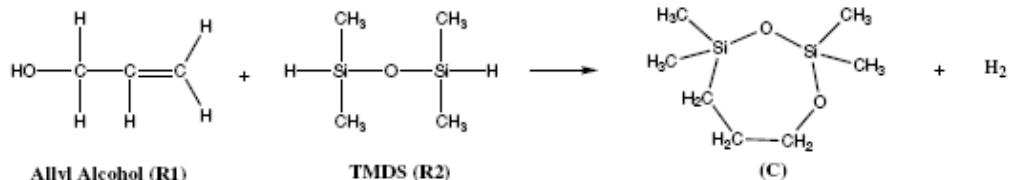


Figure 1: Structures of Chemical Species in the Lucretex Process

**REACTION 1:**

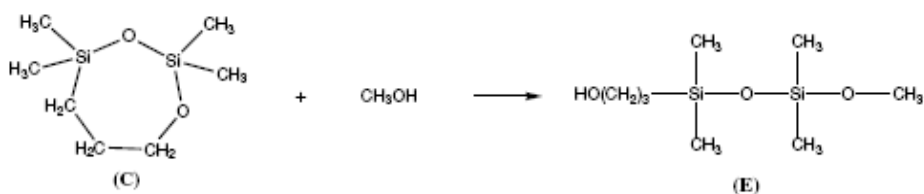
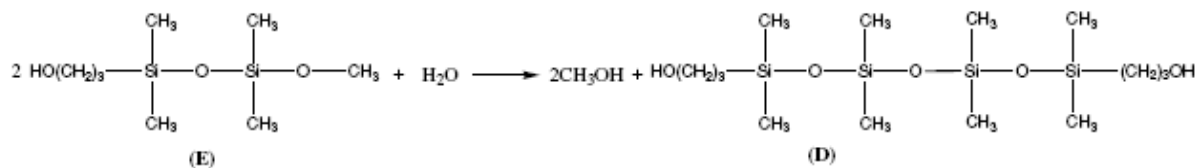
Allyl Alcohol (R1)

TMDS (R2)



Allyl Alcohol (R1)

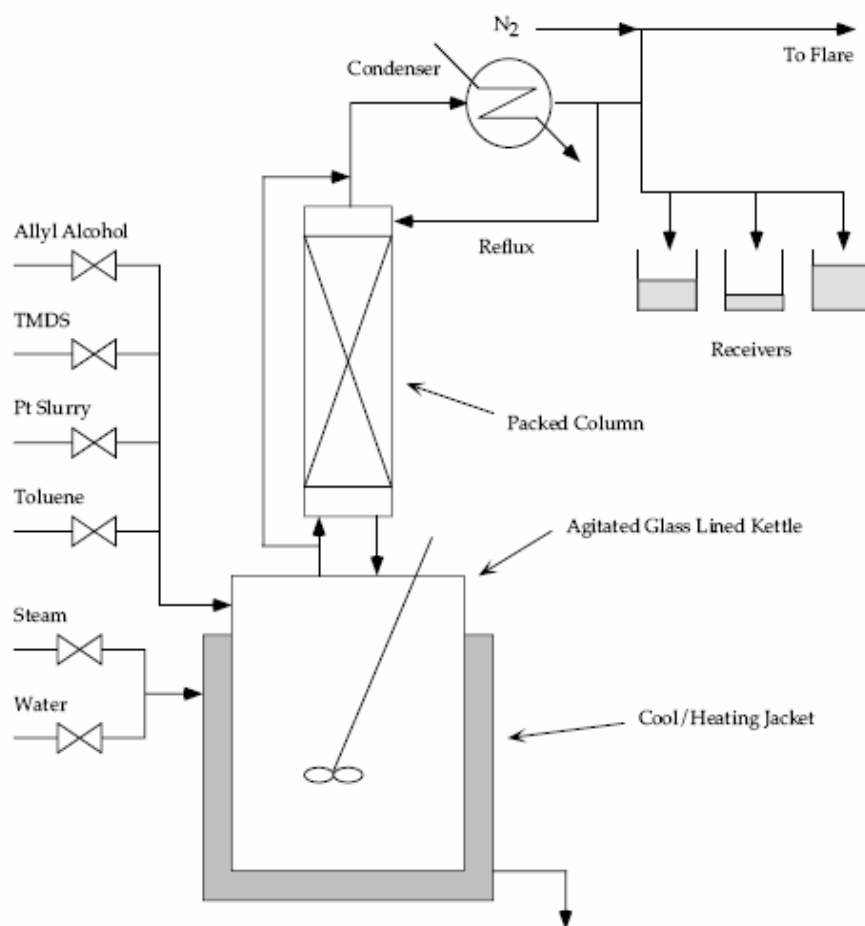
TMDS (R2)

**REACTION 2:****REACTION 3:**

(E)

(D)

**Figure 2: Reactions in the Lucretex Process**

**Figure 3: Pilot Plant Equipment Schematic**

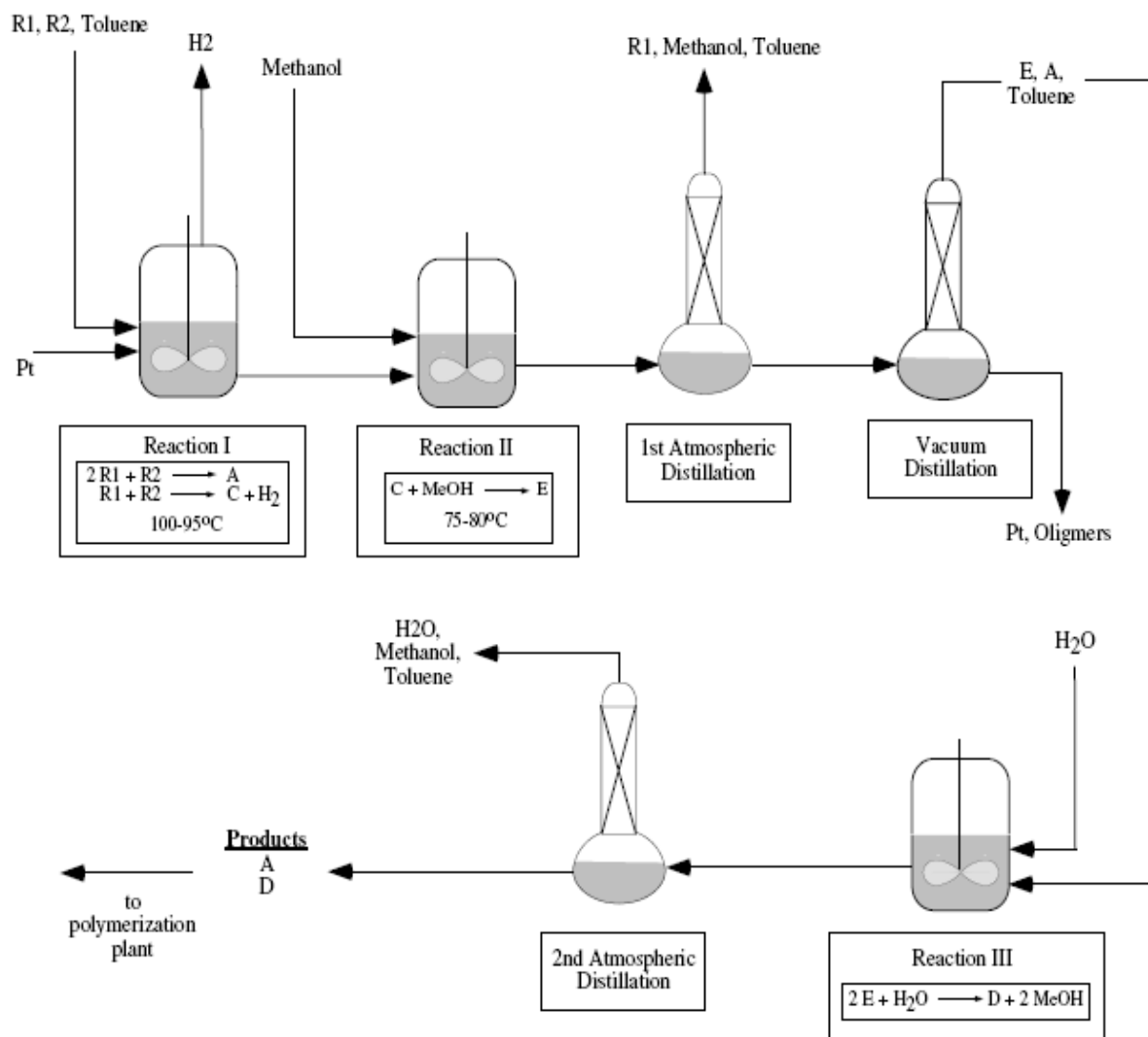


Figure 4: Pilot Plant Lucretex Task Network