DATE: July 23, 2012

TO: DIERS Users Group

FROM: Joseph C. Leung Design/Testing Committee chair

cc: Harold G. Fisher DIERS Users Group chairman

SUBJECT: DIERS Round-Robin (RR) Testing (new testing plan)

The proposed RR system is a Vinyl Acetate solution polymerization in Toluene with LPO (dilauroyl peroxide, Laurox) as free-radical initiator. We have conducted a preliminary phase which gave us very useful information regarding the severity of the runaway and its peak pressure and temperature – some of these data have been presented at the last DIERS UG meeting in Kansas City this May. However we would like to extend this invitation to all who can participation in conducting runaway reaction experiments. When this first phase is completed, we will proceed to propose a design problem based on this RR data. Note that there are two scenarios we are examining:

Scenario #1 – adiabatic runaway starting at 50°C

Scenario #2 – runaway simulating external heating of 1.5 °C/min

The design problem (to be suggested later) will also be addressing these two scenarios.

Recipe information: vinyl acetate solution polymerization

60% wt. Vinyl acetate (VAM)

39% wt. Toluene

1% wt. Dilauroyl peroxide (99% assay, available from Sigma-Aldrich)

50°C onset temperature (note the onset self heat rate is in the vicinity of 0.1 C/min)

Note 1 – It should be okay to premix the monomer, the initiator and the solvent at ambient temperature, but the solution should be used immediately.

Note 2 – For a 120 ml test cell the suggested sample size is 70 grams. For other apparatus, please use the same charge loading - 58 grams per 100 ml test cell volume.

Note 3 – If the test apparatus can be used to simulate fire heating at the prescribed rate, it will further provide valuable data for modeling later on.

Note 4 - Be warned about potential decomposition of the polymer at high temperatures, leading to high pressure developed in a closed test cell.

Note 5 - Make sure the air is evacuated and/or replaced by nitrogen since VAM can form peroxide with air and alter the kinetics.

Note 6 – For Scenario #1, a certain amount of time (induction time) would be needed at 50°C before self heating is observed. This induction time is due to the presence of the inhibitor which comes with the VAM from the supplier. For those who has the ability to remove the inhibitor, please specify what method is used and this kind of uninhibited VAM data would also be useful for modeling comparison later.

Items to submit:

#1 - determine and record the ppm HQ(inhibitor) in the VAM , and mention whether the inhibitor was removed prior to conducting the test.

#2 - give details about the sample size, close or open system test, and whether the test cell was rid of nitrogen to start.

#3 – submit data in Excel format, showing time (min), T(°C),P(psia or bara), dT/dt(°C/min), dP/dt(psi/min, or bar/min) with clear header identifying each column.

#4 – submit data plots showing T(°C) and P(psia or bara) vs time(min), dT/dt(°C/min) and dP/dt(psi/min, or bar/min) vs -1000/TK Arrhenius type plots, and log P vs 1/TK chart display.

#5 - measure and record the percent solids as a percent of the available VAM after the test. This will be useful for the modeling effort later.

THANK YOU ALL.

Deadline for Submission: Sept 30, 2012

Send data or inquiry to: Joseph Leung at the following Email: leunginc@cox.net

JCL:lak