Chapter 2

APPLICATION AND CASE STUDIES

Outline

- SBR Semi-Batch Reactor System: Monitoring
- Batch Pulp Digester: Inferential Kappa Number Control
- Nylon 6,6 Autoclave: Monitoring & Inferential Control of Quality Variables
- Continuous Pulp Digester: Inferential Kappa Number Control

2.1 PCA MONITORING OF AN SBR SEMI-BATCH REACTOR SYSTEM

2.1.1 INTRODUCTION

Background

- In operating batch reaction systems, certain *abnormalities* (e.g., increased feed impurity level, catalyst poisonging, instrumentation malfunctioning) develop that eventually throw the quality completely off spec.
- It is desirable to catch these incipient faults quickly so that the problem can be rectified.
- It is desirable not to rely on lab measurements for this purpose since this will introduce significant delays.

Key Idea

- Use more easily measured process variable trends to classify between normal batches and abnormal batches.
- The key problem is to extract out the key identifying features (*finger prints*) from trajectories of large amount of variables.

Appication

- An SBR Polymerization Reactor.

2.1.2 PROBLEM DESCCRIPTION

Process / Problem Characteristics

- Reaction:

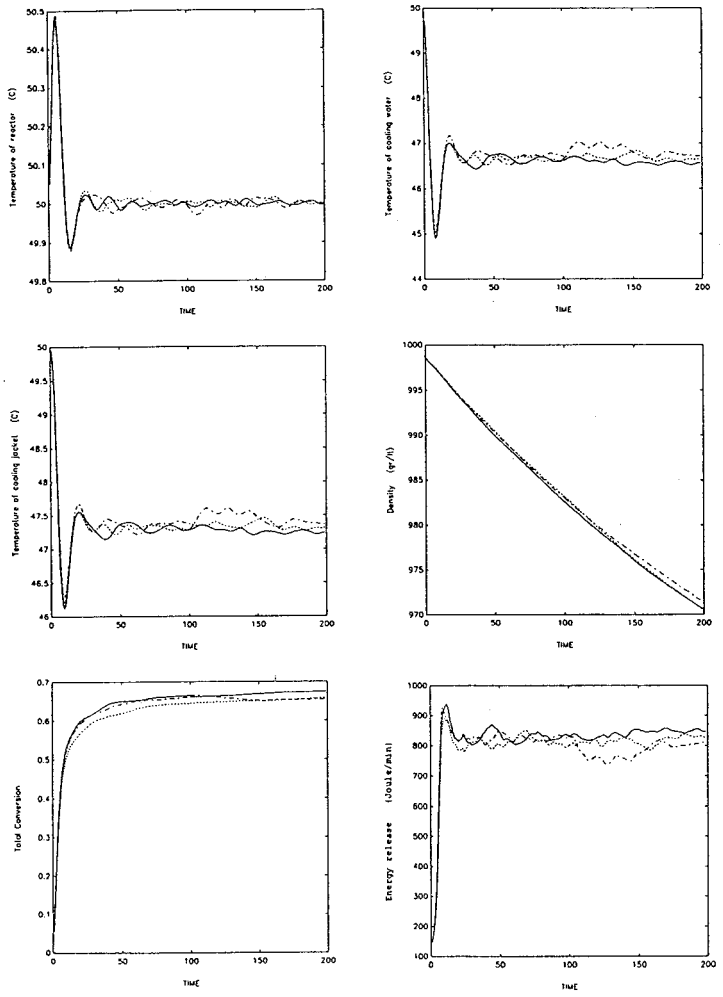
$$\text{Styrene} + \text{Butadiene} \longrightarrow^{\text{polymerization}} \text{Latex Rubber}$$

- Emulsion Polymerization
- The reactor is initially charged with seed SBR particles, initiator, chain-transfer agent, emulsifier, a small amount of styrene and butadiene monomers.
- Batch duration is 1000 minutes.
- The following measurements are available with 5 minute interval:
  - flow rates of styrene
  - flow rates of butadiene
  - temperature of feed
  - temperature of reactor
  - temperature of cooling water
  - temperature of reactor jacket
  - density of latex in the reactor
  - total conversion (an estimate)
  - instantaneous rate of energy release (an estimate)

Available Data

- 50 batch runs with typical random variations in base case conditions (such as initial charge of seed latex, amount of chain transfer agent and level of impurities).

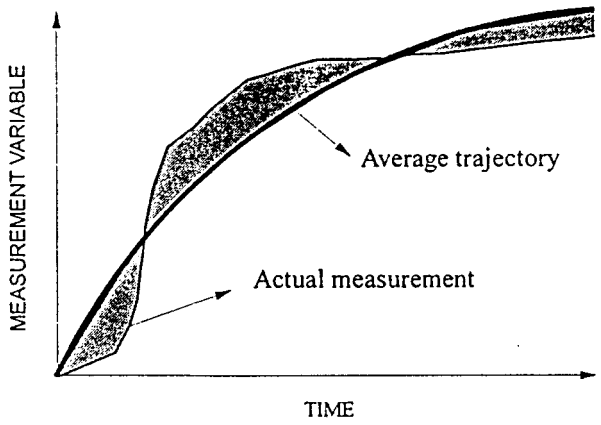
- Two additional batches with "unusual disturbances."
  - impurity of 30% above that of the base case was introduced in the butadiene feed at the beginning of the batch.
  - impurity of 50% above that of the base case was introduced in the butadiene feed at the halfway mark.



2.1.3 RESULTS

End-Of-Batch Principal Component Analysis

- Establish the mean trajetory for each variable and compute the deviation trajectory.



- Normalize each variable with its variance.
- Perform "lifting", that is, stack all the trajectories into a common vector to obtain a single vector $\mathcal{Y}$ for each batch. Then, form a matrix Y by aligning $\mathcal{Y}$ for the entire 50 batches.

  Note the dimension of $\mathcal{Y}$ is 9×200. Clearly there are only a few modes of variations in this vector.

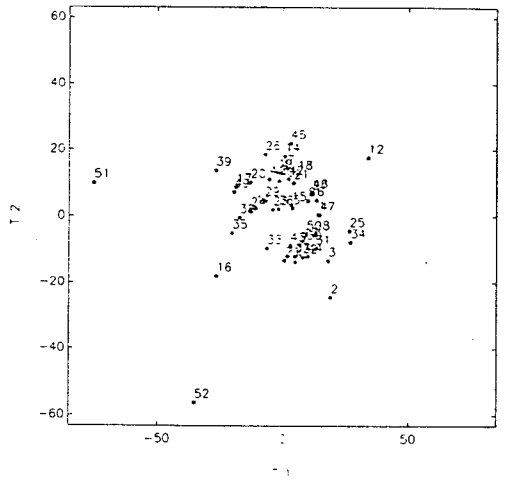
- Determine the principal component directions (eigenvectors of Y with significant eigenvalues). Three components were judged to be sufficient.

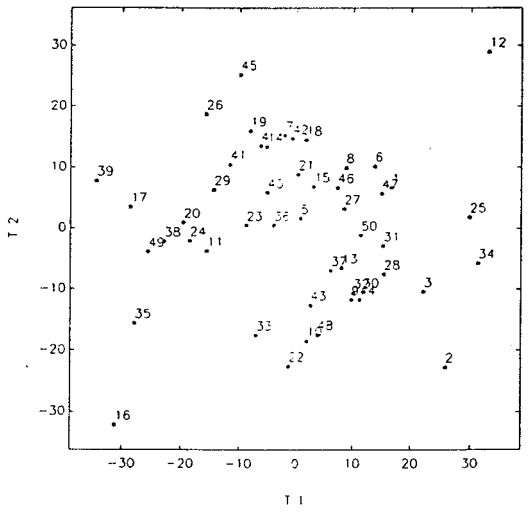
$$Y = \left[\begin{array}{ccc|ccc} v_1 & v_2 & v_3 & v_4 & \cdots & v_{1800} \end{array}\right] \left[\begin{array}{ccc|ccc} \sigma_1 & & & & & \\ & \sigma_2 & & & & \\ & & \sigma_3 & & & \\ \hline & & & \sigma_4 & & \\ & & & & \ddots & \\ & & & & & \sigma_{1800} \end{array}\right] \left[\begin{array}{c} v_1^T \\ v_2^T \\ v_3^T \\ \hline v_4^T \\ \vdots \\ v_{1800}^T \end{array}\right]$$

- Compute the principal component score variables for each batch:

$$t_i(j) = v_i^T \mathcal{Y}(j), \qquad i = 1, \cdots, 3 \quad j = 1, \cdots, 50$$

The first two P.C. scores for the 50 batches and the two bad batches are plotted below:





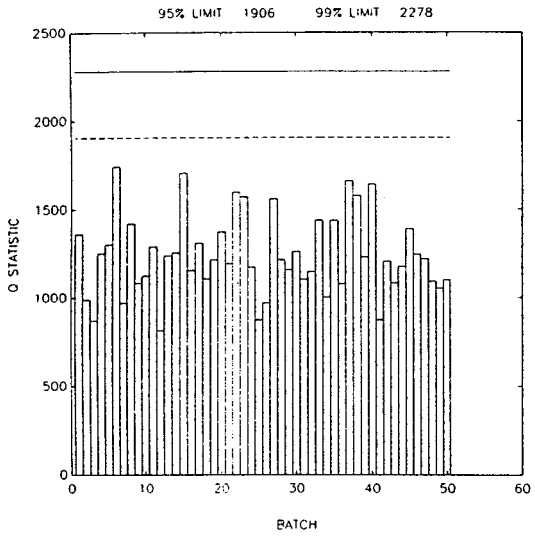
Compute the covariance matrix (diagonal) R_t for the P.C.'s. Establish the 95% and 99% confidence limits (ellipses) for the P.C.'s.

One can also use Hotelling Statistics:

$$D = t^T R_t^{-1} t \frac{N(N-m)}{m(N^2-1)} \sim F_{m,N-m}$$

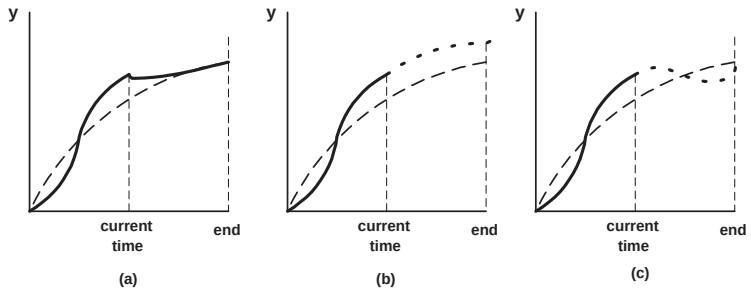
Here $t = [t_1,\ t_2,\ t_3]^T$ and $N = 50$ and $m = 3$.

- Compute the residuals and establish the 95% and 99% confidence limits for the square sum (assuming normality of the underlying distribution). The SPE (sum of the squares of the residuals) for each batch is plotted against the confidence limits:



During-Batch Principal Component Analysis

- The main issue in applying the PC monitoring during a batch is what to do with the missing future data.



Handling missing measurement

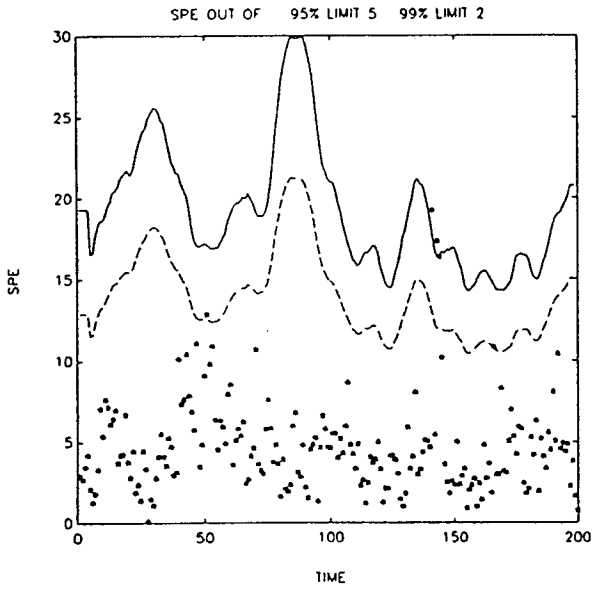
Options are:

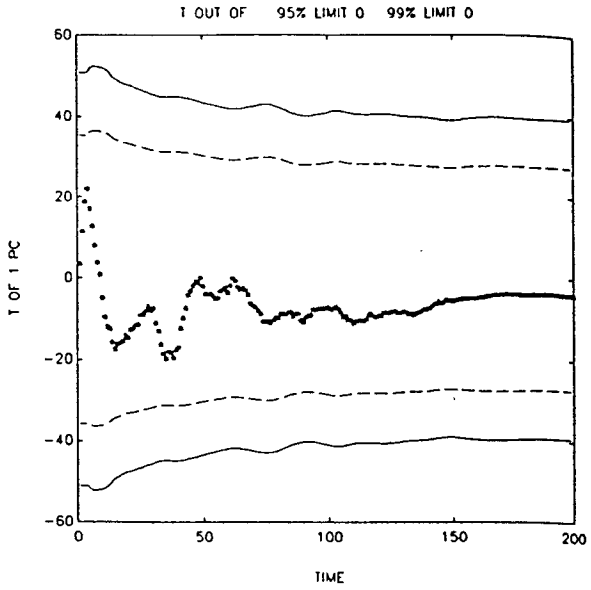
- Assume for all the variables that the future deviation will be zero.
- Assume for each variable that the current level of deviation will continue until the end of batch.
- Use statistical correlation to estimate the future deviation.

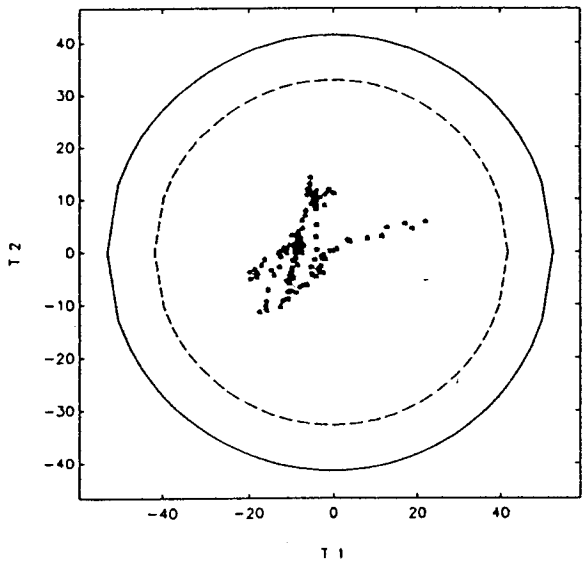
We will denote the lifted vector at time t with missing future measurements filled in as $\hat{\mathcal{Y}}_t(j)$, where t and j denote the time and batch index.

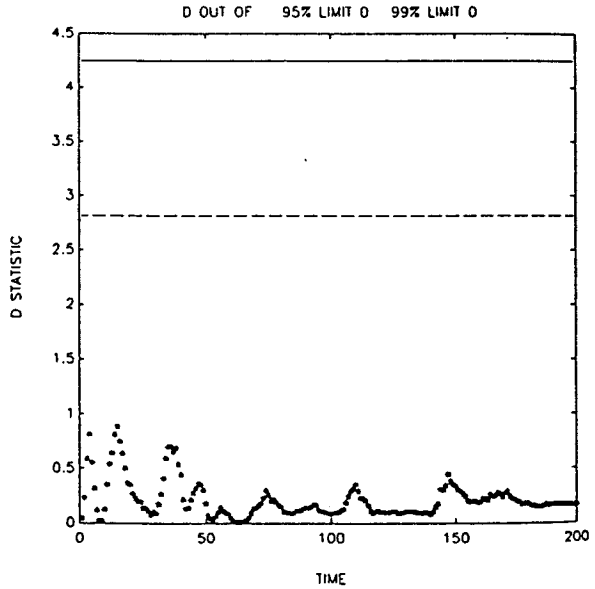
- For each time step, the confidence limits for the SPE and P.C.'s can be established.
- Now, at each time step for each batch, compute the P.C.s and SPE and compare against the confidence intervals.

Good Batch

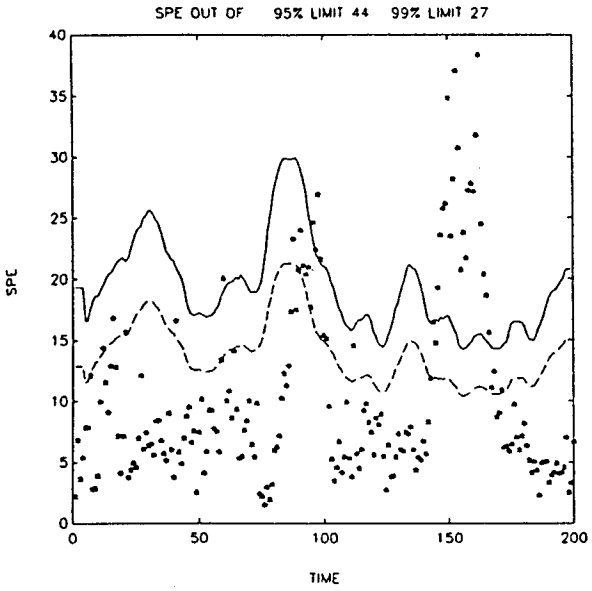


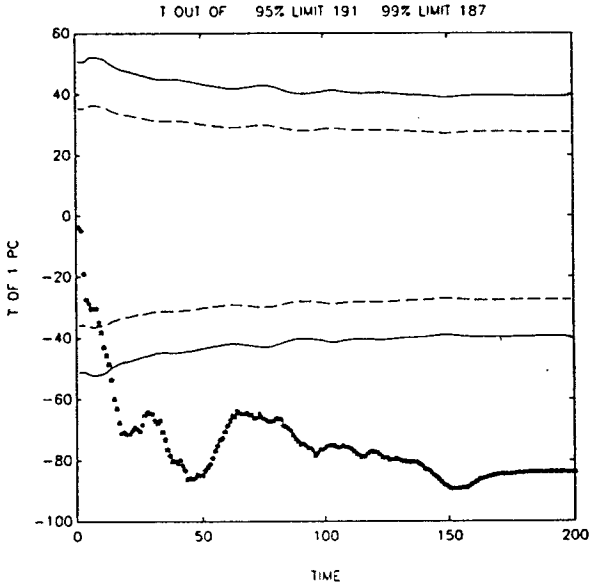


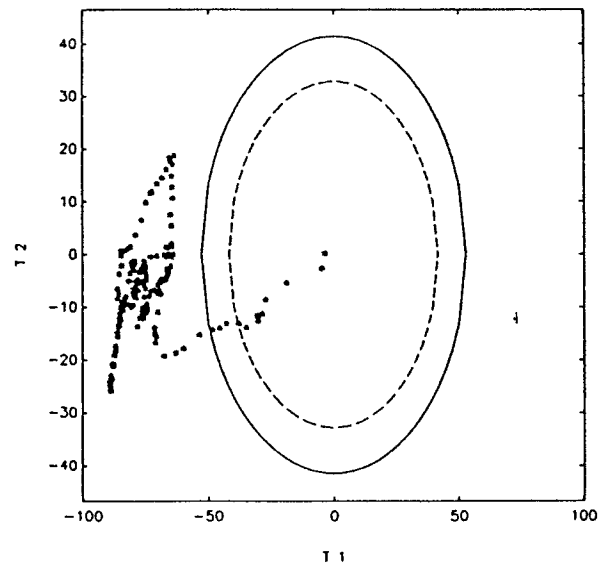


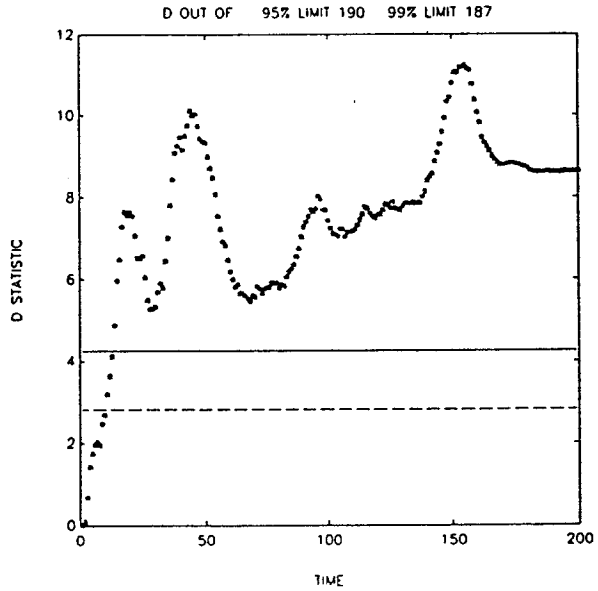


Bad Batch I









Bad Batch II