Dynamic Process Performance Monitoring
and
the Integration of Spectral and Process Data

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Towards Process and Product Consistency across the Supply Chain

Process Consistency

Product Consistency

This is now becoming a major driver in many biotechnological and related companies. The aim is to provide Quality Assurance on the plant, through:

- The wide adoption of Process Analytical Technologies
- Quality by Design
- Assured Process Manufacturing
- Real-time Release
Multidimensional Sensor Data Management, Interpretation and Visualisation – the Challenges

- Increasing availability of sensor based data – spectroscopic (e.g. IR, MIR, Raman, UVviz, acoustic, XRD, process, image, tomographic, ...)

- Major challenge is to go from data to information to knowledge and understanding.

- Data is disparate, dynamic, temporal, possibly non-linear, non-continuous, discrete, spot samples, ..... 

- Sensor data management and data fusion and integration still represent major challenges for chemists, biologists, engineers and statisticians.
Multivariate Statistical Analysis

- One approach, amongst many, to achieving a deeper understanding of the processes and systems is through the multivariate statistical data analysis and modelling methodologies.

- The simplest of these are based upon the statistical projection techniques of Principal Components Analysis (PCA) and Projection to Latent Structures (PLS).

  - **PCA** views the process or system through a single block of information - the process or quality variables.

  - **PLS** views the process or system through a model of the quality variables and/or chemical information developed from process information.

- and their ‘so-called dynamic’ variants.
Towards Process / Plant ‘Signatures’

- Stages of a product manufacturing process can be characterised and then described based on the use of a variety of diverse sensor-based measurement and ‘multivariate modelling’ techniques.

- This multi-dimensional profile can be used to produce a process / plant ‘signature’ which, in turn, offers a means of ensuring process reproducibility and robustness.

- The process/plant ‘signature’ may also be viewed as an end-point to work towards during scale-up or after equipment changes or site changes, for example.
Why Batch Monitoring?

- Continuous Process Performance Monitoring (MSPC) is traditionally based on PCA and PLS but can be extended to dynamic situations through dynamic modelling approaches such as time series modelling (e.g. AutoRegressive – AR, or Canonical Variate Analysis – CVA).

- Batch process performance monitoring (Multi-way MSPC) is traditionally based on MPCA and MPLS but can be extended to dynamic situations through dynamic modelling approaches.

- Question – why bother with ‘batch monitoring’ when the ‘trend’ is to move from ‘batch’ to ‘continuous’ processing?
  - Batch modelling / monitoring approaches can uniquely address transitions (e.g. grade changes, recipe changes, etc)
Batch Performance Monitoring

- Current state of the art techniques include:
  - Multiway principal component analysis
  - Batch dynamic principal component analysis
  - Moving window principal component analysis

- Challenges facing the current state of the art approaches:
  - Dynamics and non-linear behaviour ✓
  - Serial (auto correlated) data ✓
  - Unequal batch lengths ✓
  - Multi-scale nature of process data ✓
  - Integrating spectral and process data ✓

Addressed by CPACT R&D
Key References

- Nomikos and MacGregor (N&M) approach, 1995.

    - Unfolding in this direction (Route 2) followed by mean centering and scaling does not remove the mean trajectories from the data and hence only captures the covariance among the mean trajectories of the variables, which is not of interest in process performance monitoring.
    - Rather, it is the covariance structure of the deviations of the variables about these mean trajectories that is of interest.
    - This unfolding approach also only provides a ‘static’ model that captures only the local covariance structure amongst the variables and does not account for the dynamic behaviour of batch processes.
Dynamic Process Modelling
for Performance and Monitoring
Latent Variable and Subspace Methods

Latent Variable Techniques:
- PCA, PLS, Canonical Correlation Analysis (CCA)
  - Takes account of inter-variable correlation / covariance
- Dynamic PCA (DPCA); Dynamic PLS (DPLS)

Subspace Techniques:
- N4SID, MOESP, Canonical Variance Analysis (CVA)
  - Uses past / future correlations
  - More parsimonious than LV techniques
Past values of $u$ and $y$ are used by the state space model to predict the process outputs.

PCA is performed on the difference between the actual and predicted outputs.

Only works if residuals are white.
Super Model-Based Performance Monitoring

Process Inputs → Process

Process Model (Mechanistic or Time Series) → Error Model

Error Model → PCA Monitoring Statistics

Process Outputs
Dynamic Model-Based Statistical Batch Process Monitoring

A new batch process monitoring method based on a dynamic model and Mult-way Principal Component Analysis (MWPCA) is comprised of three model building steps.

Firstly, batch data set is centered and scaled - N&M MWPCA approach.

- removes major nonlinearities of variables against batch time and transient patterns.

A multivariate AutoRegressive dynamic model (AR) is then used to represent the dynamics of the variables about the mean trajectory.

- It is assumed that the dynamic behaviour remains the same throughout the batch run.
Dynamic Model-Based Statistical Process Monitoring

- A PCA model is then constructed using the model residuals after unfolding and preserving the variable direction.
  - The AR model plays the role of filtering the auto- and cross-correlation of the pre-processed data, and MPCA is used to describe the correlation among the filtered variables through the AR model.

Batch Data Unfolding (Nomikos & MacGregor)
Batch Data Unfolding (Wold et al)

MWPCA_w
The AR Modelling Approach

- After centering and scaling of the unfolded batch matrix, the multivariate dynamic patterns throughout the batch is modelled using an by AutoRegressive (AR) time series approach.

- The model built based on historical batch data which describes the whole batch dynamics.

- The AR model used here has the following structure:

\[ x_k^i = \sum_{l=1}^{L} C_l x_{k-l}^i + e_k^i \]

- where \( x_k^i \in \mathbb{R}^m \) and \( e_k^i \in \mathbb{R}^s \), and the measurement and residual vectors at time point \( k \) of the \( i \)th batch, respectively (\( L \) is the number of time lags).

- \( C_l \in \mathbb{R}^{(J \times J)} \) is the model coefficient matrix corresponding to the observation vector at time point \((k - l)\).
The concept of state space modelling is based on describing a system in terms of $k$ first-order difference equations that are combined into a first-order vector–matrix difference equation.

The stochastic state space model which forms the basis of this work is based on the continuous CVA modelling approach proposed by Larimore:

$$x_{t+1} = Cx_t + Gu_t + w_t$$

$$y_t = Hx_t + Au_t + Bw_t + e_t$$

where: $x_t =$ state vector, $u_t =$ process inputs, $y_t =$ process output and $w_t$ and $e_t =$ white noise
The AR Modelling Approach

- The system matrix $C$ can be determined by the ordinary least square approach.

- However, it often becomes very imprecise and unstable due to the existence of the auto- and cross-correlation of measurements.

- Thus, in this study, the AR model is derived from a PLS algorithm to avoid the rank-deficient problem.

- All the model matrices in PLS are calculated by the nonlinear iterative partial least squares (NIPALS) algorithm.
AR Model-based Dynamic Statistical Process Monitoring

**MODELLING**
- Historical Data

**MONITORING**
- Samples 
  - $k-p$ to $k$

**AR Model**
- Output Residuals

**PCA Modelling**
- Dynamic Modelling
- $x_{k-p:k}$

**PCA Model**
- $x_k$
- $e_k$
- $t_{t+1}$

**Contribution Plots**
- Fault
- Normal
CVA Model-based Dynamic Statistical Process Monitoring

- Canonical Variate Analysis (CVA) is designed for the optimal prediction of future behaviour using past observations.

- Canonical variables describe the directions of optimum correlation between future and past observation vectors.

- Canonical variables corresponding to past observations are taken to be the states of a system.

- The number of states used to describe the system are selected using Akaike’s Information Criterion (AIC) which takes into account both model fit and model complexity.
  - The CVA states are statistical states similar to PCA scores.
With the knowledge of the canonical states and the plant data, the state space matrices $C, G, H, A$ and $B$, can be computed using linear least-squares regression.

The states generated by this identification approach are not the true states, rather they are an optimal linear combination of the past inputs and outputs of the plants.

Larimore et al. showed that CVA provides near maximum likelihood estimates of the system parameters.
The first step in CVA is to build past and future matrices of the lagged process observations.

The past and future vectors take the form

\[ p_t = [y_{t-1}^T, \ldots, y_{t-j}^T, u_{t-1}^T, \ldots, u_{t-j}^T]^T \]

\[ f_t = [y_t^T, \ldots, y_{t+h}^T]^T \]

where: \( j = \) number of past lags, \( h = \) number of future lags
SVD is then performed on the product of the covariance matrices.

\[ \Sigma_{pp} \Sigma_{pf} \Sigma_{ff} = V_1 S V_2^T \]

where

\[ S = \text{diag}(\gamma_1, \ldots, \gamma_k, 0, \ldots 0) \]
It is now possible to calculate the state vector for time (t),

\[ J_k = V_{1k}^T \Sigma_{PP}^{-1/2} \]

\[ X_t = J_k p_t \]

where \( V_{1k} \) = first \( k \) columns of \( V_1 \)
Batch CVA State Space Modelling (1)

For each batch process data \((K \times J)\), the past and the future matrices are constructed as follows:

\[
\begin{align*}
P &= \begin{bmatrix}
y^i (l : 1) \\
y^i (l + 1 : 2)^T \\
\vdots \\
y^i (k - 1 : k - l)^T \\
\vdots \\
y^i (K - l : K - 2l + 1)^T
\end{bmatrix}
\quad \text{time} = l + 1 \\
\quad \text{time} = l + 2 \\
\quad \text{time} = k \\
\quad \text{time} = K
\end{align*}
\]

\[
\begin{bmatrix}
y^i (2l : l + 1)^T \\
y^i (2l + 1 : l + 2)^T \\
\vdots \\
y^i (k + l - 1 : k)^T \\
\vdots \\
y^i (K : K - l + 1)^T
\end{bmatrix}
\]

\[
\begin{align*}
F &= \begin{bmatrix}
y^i (2l : l + 1)^T \\
y^i (2l + 1 : l + 2)^T \\
\vdots \\
y^i (k + l - 1 : k)^T \\
\vdots \\
y^i (K : K - l + 1)^T
\end{bmatrix}
\end{align*}
\]
Batch CVA State Space Modelling (2)

Time

Batches

Variables

State Space Model

CVA

\[ S_{pp}^1 \quad S_{ff}^1 \quad S_{pf}^1 \]

\[ S_{pp}^2 \quad S_{ff}^2 \quad S_{pf}^2 \]

\[ S_{pp}^0 \quad S_{ff}^0 \quad S_{pf}^0 \]
Key References


Dynamic Process Monitoring
Case Study on Batch Polymerisation
Batch MMA Reactor

Process measurements
- Coolant flow rate
- Inlet jacket temperature
- Outlet jacket temperature
- Monomer conversion
- Reactor temperature

20 nominal batches to build the dynamic CVA Model
- Sampling time – 1 min
- Reaction time – 120 mins

Fault condition 1:
Reactor wall fouling from time 31 minutes

Fault condition 2:
Reaction Impurity Occurring at time 31 minutes
Univariate Control Charts Results – Reactor Fouling

Time Evolution of Coolant Flow Rate

Time Evolution of Inlet Jacket Temperature

Fault detected

Fault detected
AR Dynamic Monitoring
Based on the Observation Residuals

- Given a dynamic model which describes the auto- and cross-correlation of a batch process, an MPCA model for fault detection and diagnosis may be constructed based on the AR model residual (called the AR-residual).

- In particular, when the correlations among the residuals are not negligible and their variances are quite different, a MPCA model is needed for monitoring the AR-residuals efficiently.

- To avoid confusion with the score, loading, and residuals used in the PLS based monitoring the monitoring statistics developed for this new dynamic approach are termed the *M-score, M-loading, M-residual matrix*, respectively.
AR Dynamic Monitoring Fault Detection

The monitoring charts to detect abnormal behaviour are based on three monitoring statistics: Hotelling's $T^2$ based on the AR-score and the M-score, and the Q statistic based on the M-residual.

- The score vector from the multivariate AR model, $t$ (AR-score) and the score and residual vectors from the MPCA model, $t_M$ (M-score) and $f$ (M-residual), are used to monitor the evolving batch.

- They are calculated from the past $L$ measurements. Thus the on-line monitoring begins after $L$ time points from the start of the new batch.
MPCAW and MPCNM Monitoring Charts:
- Reactor Wall Fouling
AR Dynamic Performance Monitoring:
- Reactor Wall Fouling

Statistics at $t = 31$ min
CVA Based Performance Monitoring: Reactor Fouling

Fault detection delay: 6 min

(b) Fault detection delay: 6 min

(c)
MPCA\textsubscript{W} and MPCA\textsubscript{NM} Monitoring Charts: - Reaction Impurity
AR Dynamic Performance Monitoring:
- Reaction Impurity

Statistics at $t = 31$ min

Sample number

Variables
CVA Based Performance Monitoring:
– Reaction Impurity
MPCA$_W$ and MPC$_{NM}$ Monitoring Charts: - Control Valve Failure
AR Dynamic Performance Monitoring:
- Control Valve Failure

Statistics at $t = 31$ min
Comparison of Fault Detection Capabilities

![Detection Delay Graph]

- CVA
- PCA
- Adaptive PCA
- Dynamic PCA
- Moving Window PCA
- Shewhart
Integration of Process and Spectroscopic Data

Case Study – Batch Cooling Crystallisation
Case study: Crystallization of L-GA

- 10 good quality batches
  - Process data: reactor temperature, supersaturation and concentration

- Acoustic spectral data was available throughout the batch

- Two faults were examined:
  - Change in the cooling rate
  - Presence of an impurity

- Multiblock Multiway Principal Component Analysis was used for modelling and performance monitoring
The Multiblock Approach

Super Block T

Process

Scores

Spectroscopic

Scores

Super Level

Base Level

Scores

Super Scores
Multiblock and Wavelets Approach

Super Block $T$

Super Scores

Process Scores

Spectroscopic Wavelet coefficients Scores

Super Level

Base Level
MSPC In Crystallisation Performance Monitoring

T, pH  Enablir  PC  Turbidity

FTIR spectrometer  FTIR probe

N₂ purging tube  pH probe  Turbidity probe  Reactor

CSD

Spectra

USS

MSPC
Crystallisation Cooling Rate Fault: Time series Plots

Change in the cooling rate at time 100 mins
Crystallisation Cooling Rate Problem

\[ T^2_S \]

\[ Q_S \]

Time (mins)
Crystallisation Cooling Rate Problem

Variables Contribution for $T^2$

Variables Contribution for $Q$

Temperature
Crystallisation Impurity Problem: Time series plots

- Crystallisation Temperature
- Concentration
- Relative Supersaturation

Impurity introduced at time 100 mins
Crystallisation Impurity Problem – Process Data Block

![Graph showing T² and Q over time](image)

- **T²**: The graph shows the evolution of the Hotelling's T² statistic over time, with a threshold line at 0.02. The data points are plotted against time in minutes, ranging from 0 to 200.
- **Q**: Similarly, the Q statistic is plotted, with a threshold line at 1. The data points are also plotted against time in minutes, ranging from 0 to 200.

The graphs indicate variations in the impurity levels during the batch process, with notable peaks and troughs that may require further analysis or process adjustments.
Crystallisation Impurity Problem - Spectral Block

![Graph showing T2 and Q over time (min)]
Crystallisation Impurity Problem

![Graph showing process and acoustic data comparison]
Potential Industrial Impact

- Process Performance Monitoring is enhanced significantly by:
  - Dynamic process models (e.g. AR or CVA)
  - Using multi-block and multi-scale approaches.

- The proposed methods can be used to integrate more than one set of data – i.e. spectral and process data.

- Integrated dynamic data monitoring in many processes where both the chemical and physical conditions of a reaction are important, e.g. in crystallization, fermentation, polymerisation, etc., is now possible.

- The development of an integrated framework significantly enhances the process information and know-how.
Recipe Driven Multi-Product Models
There is an increasing need for monitoring models which encompass a range of products, grades or recipes.

Such process representations will enable the monitoring of both a number of different product types, products only produced in a few batches, as well as new product developments, using a single process representation.

Extensions to PCA and PLS through the Common Subspace Models (CSM) enables a number of similar products to be monitored through a single multi-group generic model.

In this way, products manufactured infrequently or in small quantities insufficient to construct an individual set of monitoring charts can be monitored through a pooled variance-covariance approach.
Recipe Driven Multi-Product Models

- Process manufacturing is increasingly being driven by market forces and customer needs and perceptions, resulting in the necessity for flexible manufacturing.

- This is the case in the manufacture of products such as food products, household goods, specialty chemicals, etc. where new product formulations require to be introduced to the market over a short time scale to ensure competitive advantage.

- There is a real need for models to enhance production and product changeover which can encompass a range of different product types, grades or recipes; or different operating parameters or different unit processes, using a single process representation.
The elimination of between group variation is a prerequisite for statistical process monitoring - the interest can then focus on within process (product) variability.

This can, to a greater or lesser extent, be achieved in a number of different ways:

- constructing separate control charts for each type of product or grade.
  - in many situations this may be impractical because of the large number of control charts required to monitor all the products being manufactured and the limited amount of data available from which to develop a process representation.

- Use of the lower latent variables where between group variability may be removed (masked).

- The subtraction of local or global ‘mean levels’
Case Study - Multi-Recipe Manufacturing

- Detergent manufacturing with Unilever Lever Fabergé.
- Approximately 50 different products and five main production units
  - Complicated by multiple recipes.
- Monitoring the process using standard MSPC would mean the building and maintenance of approximately 250 statistical monitoring models
- Too complicated for the operators
- High model-maintenance costs
- The study was carried out as part of a European Consortium project in collaboration with MDC Technology (Emerson Process Management).
Liquid Making Area
Plant Flow Diagram

Recipe
- Stir
- Dose RM1
- Dose RM2
- Dose RM3
- Dose RM4
- Delay

Pre weigher
- Stirrer power
- Dosing Time
- Main mixer
- Load cells

Pre mixer
- In flights
- Raw Materials

Load cells

Centre for Process Analytics and Control Technology (CPACT)
University of Newcastle, UK
A novel development allows generic multiple recipe, multiple product models to be constructed.

Consider a semi-batch manufacturing process manufacturing around 50 different products (recipes) on 5 main production units.

Monitoring the process using standard MSPC would mean the creation and maintenance of approximately 250 statistical monitoring models.

The multiple recipe modelling approach requires only a few monitoring models (in this application: 8).

In the application 88 variables, on each of two production units, are monitored on-line in real-time using a multiple recipe model.
The goal was to construct a multi-group process representation to monitor the production of a number of different recipes.

The recipes comprise both different types and different numbers of raw material additions.

Three contrasting approaches were considered:

- a separate MSPC representation for each recipe
- a MSPC representation based upon a global model (mixed or combined model) comprising all recipes
- a multi-recipe generic MSPC model.
The pooled variance-covariance matrix was calculated as:

\[ s_{jk} = \frac{(n_1 - 1)s_{1jk} + (n_2 - 1)s_{2jk} + \ldots + (n_g - 1)s_{gjk}}{(N - g)} \]

where \( s_{ijk} \) are the elements of the individual variance-covariance matrices for the \( i^{th} \) recipe \((i = 1, 2, \ldots, 4)\) comprising 88 variables.

\( n_i \) is the number of production runs associated with each recipe, 29, 19, 28 and 23 for recipes 1, 2, 3 and 4, respectively, N is the total number of production runs (here 89) and g is the total number of recipes (here 4).

The elements of the pooled sample variance-covariance matrix are therefore a weighted sum of the individual elements of the sample variance-covariance matrices.
Common Subspace (Multi-Group) Modelling

Group 1
- Scale data
- Correlation Matrix
- Pooled Correlation Matrix
- Scores Group 1
- Multiple Group Monitoring Chart

Group 2
- Scale data
- Correlation Matrix
- Pooled Correlation Matrix
- Scores Group 2
Generic Modelling

- Calculate a weighted average of individual covariance (correlation) matrices.
  - Calculate the covariance (correlation) matrices of each group.
  - Calculate the pooled covariance (correlation) matrix.
  - Obtain the common principal component loadings.
- The methodology is illustrated through the development of a multi-product model for the monitoring of a semi-discrete batch manufacturing operation, which involves the production of a variety of different products (recipes).
Two recipes are used to demonstrate the multi-group model, each recipe came in two distinct varieties, e.g. Pine and Lemon.

As a consequence there are four data sets to be included in the process representation.

<table>
<thead>
<tr>
<th>Recipe</th>
<th>Mixer</th>
<th>Batches</th>
<th>Raw Materials</th>
<th>Process Variables</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>29</td>
<td>17</td>
<td>88</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>19</td>
<td>17</td>
<td>88</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>28</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>23</td>
<td>13</td>
<td>68</td>
</tr>
</tbody>
</table>
An Individual Recipe Model

- An individual PCA representation was developed for one recipe.

![Graph showing principal component analysis for one recipe.](image-url)
Instead of building a separate model for each individual recipe, which would result in a large number of separate models, a mixed model representation might be considered.

The data from the four recipes was first combined into a single data set.

Data entries which are still empty, as a result of that particular class of raw materials not being present within a recipe, are in-filled using the mean value for that particular class of variable.
Impact of Different Recipes and Different Mixers – A Mixed (Combined) Recipe Model

- This model contains both within group and between group variation. The spread of the individual recipe clusters result in the statistical warning and action limits being far too wide to provide sensitive performance monitoring and the detection of subtle process faults.

- Subtle process events cannot be detected; the greater the number of distinct groups in the data set, the greater the impact of between group variation.
Common Sub-space Models
The Pooled-Variance Covariance Matrix

- A common set of principal component loadings can be extracted from the pooled variance-covariance matrix that allow a common set of monitoring charts to be constructed.

- This approach allows the monitoring of several different grades or types of product, or processes on different production sites, to be monitored using a single set of monitoring charts.

- By weighting the individual variance-covariance matrices to reflect the number of batches of products available, products manufactured in small quantities can also be monitored along with those manufactured more frequently.

- In this way, products manufactured infrequently or in small quantities insufficient to construct an individual set of monitoring charts can be monitored through a pooled variance-covariance approach.
The generic multi-recipe model shows the individual recipes all belonging to a single cluster which is sensitive to subtle process faults and abnormalities.
The monitoring chart shows the scores jumping outside the action and warning limits as the process malfunction impacts.
Variables (62 - 65) were identified as being related to raw material addition.

A faulty dosing-control valve was located as being the source of the problem.
The manufacturing plant produced around 50 different products using five main production units.

To demonstrate the multiple group algorithm, two product formulations were chosen.

<table>
<thead>
<tr>
<th>Recipe</th>
<th>Group</th>
<th>Mixer</th>
<th>Number of Batches</th>
<th>Raw Materials</th>
<th>Quality Variables</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1</td>
<td>19</td>
<td>23</td>
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</tr>
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<tr>
<td>4</td>
<td>2</td>
<td></td>
<td>29</td>
<td>17</td>
<td>1</td>
</tr>
</tbody>
</table>
Fault Detection

Latent Variable 3

Latent Variable 4
Fault Diagnosis

- Variable 4 (cold process water temperature) and variable 13 (batch temperature) were identified as being reflective of the malfunction in process operation.

- This resulted from a failure of the process-water cooling unit at the beginning of the manufacturing process.

- This was not observed by the plant operator and no corrective action was taken.

- This was one of the motivations for applying MSPC.

- Pooling of product monitoring models into product groups, using the synergy between the raw material structures in the different products enable the whole production process to be run using 18 process monitoring models updated for new recipes by the plant Quality manager.
Lever Fabergé Results

- Increased knowledge of batch-making process
  - Most critical parameters / relations
  - Awareness / understanding of process variability

- Reduced process variability
  - Increase % first-time-right
  - Increase process efficiency and output
  - Consistent operating procedures

- Novel tool for the operators to:
  - Anticipate process problems
  - Improve quality
  - Reduce batch times
Why are Multi-Group (Generic) Models Important?

- Minimal process data with which to model.
- Data from Products manufactured infrequently / small amounts.
- Impractical to generate a model for every product or process type.
- Method for monitoring a number of different processes using a single model.
  - Different product / recipe types or evolving recipes / strains.
  - Different ways of manufacturing a product.
  - Different plant and processing vessels.
  - Different numbers of measured variables.
  - Different manufacturing sites.
Potential Industrial Impact

- In most industrial situations the elimination of between group variation is a prerequisite for statistical process performance monitoring.

- To do this normally requires constructing separate control charts for each type of product or grade of product to be monitored, but in many process monitoring situations this will be impractical.

- As an alternative, a multi-group model has been proposed.

- The industrial manufacturing application has shown that it is possible to extend the methodology of MSPC to situations where a number of different production recipes or grades are manufactured using a single multi-group model.
Integration of Process and Spectroscopic Data

A major contribution to Batch Process Performance Enhancement and to PAT
Data Integration for Enhanced Performance Monitoring

- Kinetic Data
- Process Data
- Spectroscopic Data

Performance Monitoring Model
This study was carried out as part of a European project in 2001/2002 in collaboration with MDC Technology (Emerson Process Management).

The quality of the batch is assessed by considering the hydroxyl number and acid number of the reactor contents.

- Accurate knowledge of the hydroxyl number is required to fix the point at which the batch meets the reaction curve.

- The relationship between the hydroxyl number and viscosity is controlled to follow the path of the reaction curve, with upper and lower limits used to account for allowed process variability.

- When the trajectory enters the end zone, the batch is terminated.
Monitoring of the Hydroxyl Number

OH# vs Time

Start

End Zone

Ideal Reaction

Reaction with Correction

Corrective addition
Monitoring of Hydroxyl Number

- The path of a batch requiring a corrective addition is monitored.

- When the path leaves an upper confidence limit, an addition is made. This procedure adds about two hours to the total batch time.

- The red curve demonstrates how the corrective addition breaks up long established chains, increasing the hydroxyl number and decreasing the viscosity temporarily.

- The quantity of acid or glycol added is based on a calculation made by the process operator.

- Once the system stabilises, the reaction proceeds to the end zone.

- Samples are taken periodically to ensure that the batch is progressing to plan.
Process Measurements

- Process data is available at a frequency of five minutes.
  - Temperature Corrected Viscosity
  - Reactor Temperature
  - Reactor Vapour Temperature
  - Bottom Column Temperature
  - Middle Column Temperature
  - Top Column Temperature
  - Reactor Pressure / Vacuum

- NIR spectral data available throughout the batch.
The process comprised combinations of 5 different reactors processing 4 different viscosity groups.

Nominal batch data from two viscosity groups is discussed here.

Thirty batches were used in the development of the nominal models:
- 16 batches - viscosity group 2
- 14 batches - viscosity group 4

All seven process variables were included in the analysis.

A single, a combined and a common subspace model were investigated.

A ‘non-standard’ batch with a temperature control problem was investigated.
Different Production Reactors and Different Viscosity Groups

Bivariate Scores plot of production batches from five different reactors (PC1 versus PC2)

Bivariate scores plot of batches from four different viscosity groups (PC1 versus PC2)
Monitoring of Individual Viscosity Group 2

Principal Component 1

Principal Component 2
Process Fault Detection for Individual Viscosity Group 2

Principal Component 1

Principal Component 2
Combined Monitoring Chart

Principal Component 1

Principal Component 2
Process Fault Detection for a Mixed (Combined) Model

Principal Component 1

Principal Component 2
Pooled Covariance Model

Principal Component 1

Principal Component 2
Process Fault Detection for Pooled Covariance Model Approach

Principal Component 1

Principal Component 2
## Conclusions - Time to Fault Detection

<table>
<thead>
<tr>
<th>Model Type</th>
<th>PC1 (99% Limit)</th>
<th>PC2 (99% Limit)</th>
</tr>
</thead>
<tbody>
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<td>Single</td>
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<td>35</td>
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<td>Combined</td>
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<td>-</td>
</tr>
<tr>
<td>Pooled Covariance</td>
<td>23</td>
<td>36</td>
</tr>
</tbody>
</table>
Integration of Spectral and Process Data

- Thirty-seven batches were modelled from four viscosity groups.

- Analysis of the traditional process data from the thirty-seven available data sets revealed that thirty-two of the batches are considered to exhibit nominal operating conditions.

- NIR data was also available for these batches.

- A ‘non-standard’ batch was investigated – the duration of the batch was longer than desirable.
Batch Monitoring (Chemistry Problem) - Process Data Only

SPE

Hotelling’s $T^2$
The Conjunction of Spectral and Process Data

- A number of techniques have been investigated in a number of industrial studies including crystallisation, polymerisation, fermentation and speciality chemicals.
  - The conjunction of single-source spectral and process data using multi-block techniques.
  - The conjunction of multi-source spectral data (e.g. NIR, MIR, XRD, ultrasonic particle size spectra) with process data.
  - The use of wavelet transformations for the conjunction of spectral and process data.
  - The building of robust calibrations through bootstrap aggregation methods (bagging)
PCA of the raw, un-scaled spectral data can be used to identify the more significant wavelengths.

The wavelengths of bond frequencies identified are characteristic of the chemistry that exists within the resins polymerisation reaction.

Loadings plot of raw NIR data
In this particular application, a wavelet transformation of the NIR spectra was used to extract the important features that occurred at different frequencies within each spectrum.

NIR data was collected over the range 1078nm to 2100nm so that each spectrum now had a dyadic length of 1024 columns and a wavelet transformation was performed using a Symmlet #8 mother wavelet.

The approximation and detail coefficients were investigated at each level, with the most suitable group of coefficients being identified as the detail coefficients at level 5.
Conjunction of Process and Spectral Data

- The investigation was repeated to include a set of variables from the wavelet transformed NIR data.

- At level 5, the coefficients summarise information from around 22 wavelengths.

- The two areas of interest lie around 1450nm and 1900nm wavelengths.

- To capture all of the information from the transformed spectra, the two detail coefficients from either side of these important wavelengths are extracted for inclusion in the model.

- This leads to the addition of four extra variables to the nominal data set.
Batch Monitoring (Chemistry Problem)
- Process and Spectral Data

Principal Component 1

Principal Component 5
Batch Monitoring using Process and Spectral Data

SPE

Hotelling's $T^2$
Variable numbers 7 and 9 lie outside their 99% confidence limits.

These variables represent the wavelet detail coefficients of the OH and COOH bond frequencies.

The process variables (variables one to six) lie well inside their respective confidence limits.
The SPE contribution plot for time point thirty identifies variables seven, eight and ten as contributing to the batch trajectory.

Again, these variables are all associated with the quality information extracted from the NIR data set.
Potential Industrial Impact

- Where modelling traditional forms of process data in a MSPC monitoring scheme offers the potential for detecting faults in operating conditions, the inclusion of chemical information, in terms of wavelength data, allows for faster detection of abnormalities in the chemical make-up of the reactor contents.

- The inclusion of chemical information, even from a range of on-line real-time analysers when they can be justified on a business benefits basis, potentially offers significant advantages to using solely traditional process variables or single analysers.

- Integrating spectroscopic data with process data, properly, is not straightforward.

- Where modelling traditional forms of process data in a MSPC monitoring scheme offers the potential for detecting faults in operating conditions, the inclusion of chemical information allows for detection of abnormalities in the chemical make-up of the reactor contents.
Overall Conclusions

It has been shown that Process Performance Monitoring can be enhanced and made more applicable and fit-for-purpose across the process manufacturing industries through enhanced MSPC methodologies which include:

- Dynamic process models (e.g. CVA or AR)
- Multi-group models
- Integrated spectroscopic and process data
- ‘Intelligent’ Performance Monitoring (integrate with an Expert System such as G2)

These provide major contributions to the realisation of the FDA PAT Initiative
Some Business Benefits from the Technologies

- Savings in catalyst usage in a commercial scale fluidised bed reactor ~ £1M / annum (BASF, UK).
- Development of a Constrained Optimisation based PLS algorithm (BASF, Germany) – enabled application of MSPC to a multi-product commercial scale fluidised bed reactor.
- Assured monitoring of chemical reactions ~ £800,000.
- Early detection of faulty reactor dosing ~ £250K / annum (Unilever).
- Modified recycling of Catalytic Cracker Beds ~ £250K per Bed (Shell).
- Unexplained variability in fermentation production processes, expected benefits ~ £4.5M / annum.
- Performance Monitoring of a High Speed Polymer Sheet Production (Du Pont Films and UCB Films) ~ significant reductions in off-spec material.
- Application of Intelligent MSPC with Corus – significant operational cost savings.
Data Integration and Analysis for HTT
Micro plants and Intensified Process Development?
Acknowledgements

- EPSRC (The UK Engineering and Physical Sciences Research Council).
- The Centre for Process Analytics and Control Technology (CPACT).
- BASF, DSM, GSK and Unilever for access to their plant data and expertise.
- Other R&D is addressing the development of GRID and High Throughput (Experimentation) technologies:
  - EPSRC “Grid Orientated Full Lifecycle Chemicals Development”
  - EU “Sustainable Microbial and Biocatalytic Production of Advanced Functional Materials: BIOPRODUCTION – Process Intensification”