REDUCING PROCESS VARIABILITY
IN CHEMICAL BATCH MANUFACTURING

by

Theresa Lai-Hing Mock

B.S., Chemical Engineering, Massachusetts Institute of Technology (1984)

Submitted to the Department of Chemical Engineering and
to the MIT Sloan School of Management
in Partial Fulfillment of the Requirements for the Degrees of

MASTER OF SCIENCE IN CHEMICAL ENGINEERING
and
MASTER OF SCIENCE IN MANAGEMENT

In conjunction with the Leaders for Manufacturing Program
at the
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ABSTRACT

Today's competitive environment is placing heavy emphasis on quality efforts to attain manufacturing excellence. A major focus is directed towards reducing process variability, and keeping processes in control. This thesis examines variability reduction through process understanding as a key business priority for chemical batch manufacturing. The objective of this research is to investigate and develop a systematic methodology for variability reduction, along with specific recommendations on the technical tools and organizational changes required for successful implementation.

I emphasize an integrated sociotechnical perspective, recognizing the importance of both the technical and organizational issues. The central thesis of my research is stated as follows: A data-driven operating strategy will lead to reduced process variability. To prove the validity of the hypothesis, I show how data can be used to improve process understanding, and how better process understanding can drive efforts towards variability reduction. My research methodology includes a series of case studies and field visits conducted during a seven-month internship in the Synthetic Chemicals Division of the Eastman Kodak Company.

My results provide guidelines for data collection, including identification of what data is needed for process analysis, and selection criteria for data collection methods. I also present an evaluation of data analysis techniques and tools, namely the Kodak Data Analysis Tool, univariate diagnostics, MacroManager, Principal Components Analysis and Partial Least Squares multivariate techniques, decision trees, Design of Experiments, and Evolutionary Operation. To identify the key factors impacting variability reduction efforts, I conducted an organizational factors survey.

I synthesize the learning from the research experience into a systematic approach to guide future process improvement efforts. The general methodology for variability reduction provides specific action steps, along with suggestions for the application of appropriate tools. I conclude with recommendations for a one-year implementation plan.

Advisors: George Stephanopoulos, Professor, Department of Chemical Engineering
Stephen C. Graves, Professor, MIT Sloan School of Management
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1. Introduction

1.1. Problem Statement

1.1.1. Reducing Process Variability as a Business Priority

In today's competitive environment, manufacturing companies put heavy emphasis on quality efforts to reduce process variability, and to keep processes in control. A major focus of these variability reduction efforts is directed towards achieving statistical process control. Critical process parameters are tracked on control charts to monitor variability. Out-of-control conditions are reviewed for assignable causes. The collection and analysis of operating data then becomes an integral part of the production process. The success of such an operating philosophy often depends on accompanying changes in the organizational culture. Both organizational and technical considerations are important in realizing quality improvements. A priority on variability reduction can lead to economic benefits, increased process knowledge, greater worker involvement, better quality products, cycle time advantages, and an improved competitive position.

In chemical batch manufacturing, chemicals are made in batches following a recipe of steps in the production process. Batch to batch variability may be introduced from a variety of sources that can be classified into five categories:

- **Materials**, i.e. quality of raw materials, cross-mixing of solvent delivery lines, seasonal variations in cooling water temperatures
- **Manpower**, i.e. different interpretations of the manufacturing recipe by operators, different levels of operator experience

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• *Methods*, i.e. heat sensitive chemistry, time-sensitive process steps, difficult phase separations

• *Machine*, i.e. mechanical failure, equipment performance in different production bays

• *Measurement*, i.e. calibration of scales, repeatability of analytical tests

While the effect of variability due to a specific process parameter is difficult to isolate, the resulting variability in product quality can be measured. The cost of bad quality can be quantified from measures such as yield loss, amount of rework, extra inventory, and additional analytical tests needed. Reducing process variability thus becomes critical in improving product quality. After all, the business priority is to meet customer needs for a quality product in the most cost-effective manner.
1.1.2. The Quality Imperative in Manufacturing

The quality movement has spread from automobile and electronics makers to pervade into every sector of manufacturing. The global marketplace of the 1990s requires managing for quality that takes unwavering leadership, commitment to goals, and corporate organizational change. Quality management can be applied to everything from cost-cutting and capital investment to environmental concerns. Done right, quality can become a potent competitive weapon. The benefits can be translated into increased market share, cost reductions, quicker customer responsiveness, flatter organizations, improved communications, and an empowered workforce.

*Managing for Quality.* The literature on managing quality improvement emphasizes five strategic actions:

- Pursue quality improvement as a sound business strategy.
- Manage quality as the organization's other most important tasks are managed.
- Establish specific management direction and goals for results.
- Build upon the existing organizational structure and involve everyone.
- Use a systemic organizational change process adapted to the culture of the firm.

The factors contributing to the success of a quality improvement process can be put into four categories: culture, leadership, process initiation, and training.

- *Dealing with Corporate Culture:* The organization's culture must be prepared to accept a quality process as an integrated social and technical system. The process

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should be spread throughout the organization to reap the benefits of total employee involvement.

• *The Need for Leaders’ Commitment:* The organization has a champion who is a persuasive spokesperson for the process. The leader must be powerful enough to make things happen and smart enough to solicit the input of others. The process should be done with a long-term perspective.

• *Getting Started:* The initial corporate quality policy must be followed up with divisional statements that detail how each division will contribute to the process.

• *Training for Everyone:* Both managers and hourly workers must be provided with training that is balanced between technical and social skills. An emphasis should be placed on the application of the learning as part of daily production activities.

*Cultural Contradictions of Quality Improvement.* The cultural impact of quality initiatives has been recognized in the literature. In a recent study of the introduction of statistical process control (SPC) into a manufacturing plant, three cultural themes were found to impede the implementation:7

• *Learning Versus Performing:* Plants are managed in ways that maximize performance at the expense of change, adaptation, and learning. However, all innovation involves a period of learning, and as a consequence, runs into the cultural impediment of performance-oriented norms. SPC, is particularly

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countercultural because implementation involves not only a period of learning about SPC itself, but a period of learning about every manufacturing process in which it is to be implemented. In order for SPC to be successful, learning must be as highly valued as performing.

• *The Value of Information*: Information in a manufacturing operation usually exists in the context of defining expectations for future performance, reporting on past performance, or assigning responsibility for handling problems. Such data typically describes deviations of actual performance from expected, and is of little use for problem analysis. In contrast, SPC uses information to highlight and fix problems. It works against a culture with strong status hierarchies where who is right is often determined by the position of the person giving the information. Instead, SPC gives powerful information to operators, who have the least status. SPC is countercultural in valuing a different form of information, and in placing it in the hands of production workers.

• *Holism Versus Segmentalism*: Work in a manufacturing organization is typically divided up into operations and distributed to individual workers. The tendency is to compartmentalize problems and information, and deal with each in isolation. SPC, on the other hand, takes a holistic approach. It requires multivariate thinking and works by managing interdependencies among all variables in a process.

The existence of these cultural contradictions highlight the organizational change that is needed for quality initiatives to take root.
Controlling Process Variation is Key to Manufacturing Success. A major objective of quality efforts is towards controlling process variation. The key to the best possible quality and lowest manufacturing cost starts in product design and process development.\textsuperscript{8} Once in production, process control is established from a technical understanding of each operation.\textsuperscript{9} A process flow diagram is used to identify the unit operations for analysis. The data requirements define the parameters to measure, the tools for analysis, and the methods for data collection. Process variability is monitored with statistical control charts.\textsuperscript{10} Thus, controlling process variation requires the integrated efforts of design, development, and production.

\textsuperscript{8} Don Clausing and Bruce H. Simpson, "Quality by Design," Quality Progress, January 1990, pp.41-44.
\textsuperscript{9} Larry H. Anderson, "Controlling Process Variation is Key to Manufacturing Success," Quality Progress, August 1990, pp.91-93.
1.1.3. Data Analysis in the Process Industries

*The Transformation of the Chemical Industry.* In the past twenty years, the chemical industry has undergone an extensive restructuring. Many of the major American chemical firms have reshaped their businesses by reducing the scope of their commodity chemical operations while diversifying into such areas as specialty chemicals, pharmaceuticals, biotechnology, and advanced materials. Global competition is heating up as diversification continues and technological alliances are formed. Against this backdrop, the chemicals sector has been transformed into a more research-intensive, market-driven industry with an emphasis on product innovation.¹¹

*Focus on Data-Driven Operations.* The reshaping of the chemicals sector, along with other environmental factors, have compelled the process industries to follow a data-driven operating strategy. The literature provides examples of companies, such as Air Products Limited,¹² who has implemented a data management and data reconciliation tool to collect and analyze plant data. The benefits of providing an integrated data collection and management system include savings in energy consumption, the formulation of improved control and optimization strategies, and quicker response to plant problems. The system's data analysis capabilities are valuable for the graphical reporting of data, exception reporting, process monitoring and trend analysis, detection of faulty instruments, and detection of leaks.

Much can also be learned from the pharmaceutical industry which must collect and interpret process data to determine whether a process is operating within predefined


specifications for quality control. For example, Pfizer, Inc. has developed the Quality Analyst,\textsuperscript{13} and Miles, Inc., the health care business of Bayer USA, Inc. has created the Application Productivity Tool\textsuperscript{14} to automate data collection and analysis.

\textit{Requirements for Batch Tracking.} The needs of pharmaceutical manufacturers provide insights into the collection and analysis of data which is applicable for chemical batch manufacturing. Batch production may involve many ingredients and many discrete processing stages. Tracking information for batch processes is much more complicated than for continuous processes. A batch historian is a computerized system for batch tracking which can have the following capabilities:\textsuperscript{15}

- \textit{Batch End Data}: Summary of historical data for a specific batch.
- \textit{Snapshot Data}: Summary of the process at the time of a process milestone.
- \textit{Event Tracking}: Logging of significant events during the operation of the batch process.
- \textit{Batch Material Genealogy}: Record of the history of all feedstocks, intermediate production, and end products.
- \textit{Operator Comments}: Comments and explanations manually entered by the operator.
- \textit{Continuous Data}: Continuous collection of process data, including time stamps and batch identification, at variable sample rates based upon the process sequence.
- \textit{Data Archival and Retrieval}: Storing of data for review at a later date.

• **Graphical Trend Plots:** Ability to view and compare tabular continuous data from different batches in the form of a two-dimensional trend.

The batch historian can also have options for data review:

• **Manual Analysis:** Pre-formatted reports are generated at a specified frequency.

• **Automatic Exception Analysis:** Raw data are used to identify exceptions to predetermined limits.

• **Statistical Quality Analysis:** Quality control software is applied on the data from the batch historian for dynamic, real-time, higher-level interpretation of whether the process is under control.

• **Artificial Intelligence Analysis:** Computer is used to analyze relationships between operating parameters based on programmed knowledge from process supervisory personnel.
1.1.4. Chemical Batch Manufacturing

Chemical batch manufacturing occurs as a series of discrete unit operations in which the raw materials are converted to the final product. The unit operations involved include chemical addition, reaction, filtration, crystallization, distillation, centrifuging, and drying. The manufacturing procedure, often referred to as the batch recipe, describes the requirements to manufacture one batch of a chemical. The recipe lists the material requirements, equipment usages, processing sequence, and process parameters, i.e. reaction time, agitation speed, vessel pressure, and contents temperature. A campaign of several batches is typically scheduled to meet order quantities, increase operating efficiency, and reduce setup and cleanup costs.

Production areas are differentiated by the volume capacity of the equipment. Production bays follow a standard equipment layout composed of reactor vessels, associated receivers, liquid-solid separation units, and ancillary equipment. The equipment is controlled with pneumatic, electronic, or hybrid control systems. As a result of incremental improvements over time, equipment variation is a possible source of process variability.
1.2. Company Background

This research was carried out during a seven-month internship in the Synthetic Chemicals Division (SCD) of the Eastman Kodak Company (Kodak). SCD is an internal supplier of synthetic photographic chemicals, such as hardeners, sensitizing dyes, and couplers, used in Kodak's film and paper products. The division is a batch manufacturing operation with a product list of over 1,000 chemicals. Process variability reduction is a recognized priority within SCD. The complexity of the processes and the turnover of chemicals make the efforts on variability reduction especially challenging.

1.2.1. Kodak's Response to the Competitive Environment

*Imaging Market Overview.* Kodak is responding to the challenges of a competitive environment with a corporate vision to be the world leader in imaging. In the photosensitized goods market, Kodak's biggest competitors are Fuji, Agfa, and 3M. As an example of the market competition, Kodak has struck back with an aggressive strategy to attack Fuji in its home market. Since re-entering the Japanese market in 1984, Kodak's sales have soared sixfold, to an estimated $1.3 billion in 1990. Kodak's share of sales to amateur photographers has grown to 15%. The tactics for this success has been local investment to nurture supplier relations, and heavy advertising to establish brand awareness.¹⁶

Electronic imaging is also a developing threat to the traditional film market. Kodak is currently spending 25% of its $1.7 billion research and development budget on electronic imaging.¹⁷ The battle for the electronic imaging market include Kodak and Polaroid, whose strengths lie in silver halide chemistry, and electronic giants, such as Sony, Cannon, and Fuji.

However, according to Kodak CEO Kay Whitmore: "Our forecast well beyond 2000 is for traditional photography to continue growing, although electronic imaging may grow faster. We don’t think traditional photography will shrink and die. That’s one thing that I don’t worry about."  

Emphasis on Manufacturing Excellence. To meet the market pressures, the company has mobilized under Kodak’s Perfect Product, Perfect Process (KP4) quality initiative. In the manufacturing sector, KP4 has emphasized variability reduction and lead time reduction. While improving process control is not a new idea within Kodak, the KP4 initiative has led to a renewed emphasis on manufacturing excellence.

However, the application of quality control methods is not new in the manufacture of photosensitized materials. Over a decade ago, Fuji Photo Film Co., Ltd. discussed its approach to quality control. The methods were aimed at producing consistent and reliable photographic materials. As part of Fuji’s company philosophy and policy, priority was given to the assurance of quality over other goals such as productivity or cost savings. Fuji believed that such efforts would result in minimal variation within and between lots, which would ultimately improve consumer reliance and company profits. Fuji’s production workers understood the significance and necessity of control charts, used them everyday, and above all, became deeply interested in the quality of the products they manufactured.

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18 Ibid., p.39.
Kodak's KP4 initiative is a direct challenge and response to the increasing importance of quality and customer responsiveness in the photosensitized goods market.
1.2.2. The KP4 Initiative

Kodak's corporate vision is to be the world leader in imaging materials, technology, and manufacturing. Kodak's KP4 quality initiative is the driving force to achieve this vision. Each business unit must align its objectives within the strategic intent of the corporation. KP4 deployment is pursued via specific measures, objectives, and projects directed towards improving quality, delivery, cost, benchmarking, and new product development. The Quality Leadership Process (QLP) is the means to achieve KP4 objectives. QLP stresses a customer focus and application of the Plan-Do-Check-Act (PDCA, also referred to as Assess-Plan-Do-Verify) process model to drive continuous improvement.

SCD's business focus is on achieving customer satisfaction without compromising safety and environmental responsibility. Within SCD, the KP4 directive is translated as "Customer Satisfaction Through Process Control." The emphasis is on variability reduction and cycle time reduction based on a fundamental understanding of the process. The management strategy to achieve this consists of the following elements:

- **Visible Management Leadership**: To provide constancy of purpose
- **Customer Focus**: To meet customer needs
- **Teamwork**: To drive incremental improvements
- **Analytical Approach**: To make data-driven decisions
- **Training**: To educate and empower workers
- **Quality Incident Reviews**: To eliminate root causes of variability

My research conducted in SCD was focused on the priority of reducing process variability. The insights gained from my field experience is summarized in a general methodology for variability reduction for chemical batch manufacturing. Specific recommendations are proposed for implementation of variability reduction efforts in a production environment.
1.2.3. SCD Organizational Structure

The SCD organizational structure consists of three levels - divisional, departmental, and areal. (Figure 1). SCD is headed by a division manager who leads a business team composed of the department managers and staff support managers. Area managers and process chemists report to department managers. Area managers handle the business aspects of the day-to-day operations, including cost and delivery responsibilities. Process chemists handle the technical issues involving the manufacture of the chemicals, including responsibilities for product quality. Group leaders, who lead a team of operators, report to area managers. The divisional staff functions hold responsibility for materials management, quality, and safety and environmental issues.

The functional support units are coordinated with SCD's manufacturing operations via a matrix structure. The support unit managers have a dotted-line relationship to the division manager. Communication between SCD management and support unit management provide the basis for obtaining support staff involvement in divisional goals. The support functions cover analytical services, engineering and maintenance, finance, health, safety, and environmental, human resources, purchasing and planning, and research and development.

Production operates on a rotating seven-day, 24-hour, three-shift system. Daily production area staff meetings during shift changes are the most common forum for staff communications. Additional production meetings are scheduled to review manufacturing protocols and troubleshoot process problems. Cross-functional process management teams (PMTs) are formed to address production problems and to carry out specific KP$^4$ improvement projects. Most interaction is with the development group responsible for improving existing processes and transferring new ones to production. Typically, new technology, such as data acquisition systems and data analysis tools, is introduced into
production by engineering and research. The functional groups "sell" the new tools, assist in implementation, and provide technical support.
Figure 1. SCD Organizational Structure

Division Manager

Support Functions
Analytical
Engineering & Maintenance
Finance
Human Resources
Purchasing & Planning
Research & Development

Department Manager

Staff Functions
Quality
Materials Management
Safety & Environmental

Process Chemist

Area Manager

Group Leader

Operator
2. Research Goals

2.1. Taking a Sociotechnical Perspective

Variability reduction through process understanding is a key business priority for chemical batch manufacturing. The objective of this research is to investigate and develop a systematic methodology for variability reduction, along with specific recommendations on the technical tools and organizational changes required for successful implementation. I emphasize an integrated sociotechnical perspective, recognizing the importance of both the technical and the organizational issues. Within this context, this research addresses the following critical issues:

- What data is needed for process analysis?
- What is the best way to collect the necessary data?
- What techniques and tools are applicable for process analysis?
- What are the organizational factors impacting variability reduction efforts?
- What is a general methodology for implementation of variability reduction efforts?
- What are the policy guidelines for variability reduction efforts?

2.2. Hypothesis Statement

The central thesis of my research is stated as follows: A data-driven operating strategy will lead to reduced process variability. To prove the validity of the hypothesis, I show how data can be used to improve process understanding, and how better process understanding can drive efforts towards variability reduction. The desirable outcome is process control based on a basic understanding of the chemical manufacturing process. The mechanism to achieve this goal is a team approach to integrate the efforts between production and the supporting functional units.
2.3. Thesis Scope

My thesis scope consists of four components focusing upon the key issues for implementing variability reduction efforts in batch chemical manufacturing.

2.3.1. Component 1: Assessment of Data Needs for Process Analysis

Process data can come in the form of either qualitative or quantitative information. According to this broad definition of data, I explored possible modes of data collection and investigated the following three methods in depth:

- *Data Acquisition Systems* which automatically log quantitative data of specified process parameters, i.e. temperature profiles.

- *Operator Batch Sheets* which include both quantitative and qualitative data manually collected on data sheets, i.e. pH, clarity of solution.

- *PMT Meetings*, in which the team of operators, group leader, process chemist, and development chemist assigned to a given chemical, review the results of past batches, and discuss improvements for future runs.

In assessing the data needs for process analysis, I addressed the following key questions:

- *What are the important process data to be collected?* Can the data requirements be classified according to unit operations?

- *How can the data be collected?* What kind of information is best captured by data acquisition systems versus batch sheets versus PMT meetings?

- *How can the data be used?* How can the data be processed to gain the most insights for reducing process variability?
2.3.2. Component 2: Evaluation of Data Analysis Techniques and Tools

My objectives in evaluating data analysis techniques and tools were three-fold:

- To develop a step-by-step guide to performing process analysis
- To identify what types of analysis are useful to achieving process understanding
- To demonstrate when to apply which technique or tool

I reviewed available data analysis techniques and tools, and evaluated the application of the following ones in my research:

- **Kodak Data Analysis Tool (KDAT).** KDAT is an Kodak internally developed software tool designed for analyzing time series profiles of batch process data. It is especially useful for comparing analogous process points (called events) across different runs. KDAT's data management and data manipulation capabilities aid the graphical and statistical analysis of process data.

- **Statistical Analysis Using Univariate Diagnostics.** Univariate diagnostics are useful for reducing the data represented by a time series profile into a few key variables. The diagnostics to be calculated are selected to summarize the key features of the data, i.e. the temperature slope to represent the reaction heating profile. These univariate tests can then be control charted for statistical analysis of batch to batch variability.

- **MACroMANager (MACMAN).** MACMAN is a Kodak internally developed front-end interface to KDAT that contains a package of macros for statistical process analysis. The software package is especially useful for calculating univariate diagnostics of the time series batch profiles for control charting.
• Principal Components Analysis (PCA) and Partial Least Squares (PLS) Multivariate Techniques. PCA and PLS are statistical multivariate techniques which reduces a data set with a large number of physical variables into a data set with a small number of derived latent variables. The multivariate statistics can then be used to build predictive models that relate process variables to quality variables.

• Decision Tree Analysis. The decision tree analysis technique can be used to process both quantitative and qualitative information. The approach classifies batches into good versus bad by systematically categorizing the discriminating effects of the process variables. A branching pattern can be induced to identify the critical limits of key process parameters for acceptable operation. The operating envelope identified can be used to guide production activities.

• Design of Experiments (DOX) / EVolutionary OPeration (EVOP). DOX and EVOP are systematic approaches for experimentation that emphasize the statistical analysis of data. DOX can be especially useful in identifying the interactions among key operating variables during process development. EVOP leverages fundamental process understanding to guide production operations to systematically move towards optimum conditions.
2.3.3. Component 3: Organizational Factors Analysis

The success of variability reduction efforts is as much influenced by organizational factors as by technical issues. I conducted a division-wide survey to measure the existing barriers to variability reduction within SCD. My purposes for performing an organizational factors analysis were:

- To identify the organizational factors impacting variability reduction efforts
- To diagnose the current perceptions of existing barriers
- To recommend suggestions to improve organizational effectiveness

The survey was formulated as an exploratory analysis tool, and distributed to all work centers. I applied the following methods in reviewing the survey data:

- **Oblique Principal Components Cluster Analysis.** The SAS VARCLUS hierarchical clustering procedure was used to analyze the underlying structure of the survey data. Based on the survey responses, the multivariate algorithm sorts the survey questions into the most appropriate clusters.

- **Chronbach Alpha Reliability Test.** This internal consistency was used to verify the identification of the survey question clusters.

- **Graphical Data Analysis.** Statistics of the means, ranges, and standard deviations were calculated for each question. The results were plotted by each cluster to identify the most significant organizational barriers.
2.3.4. Component 4: Policy Strategy for Variability Reduction

A major goal of this research is to synthesize the findings of the case studies and field experience into a systematic approach for variability reduction. Towards this end, I propose a structured methodology for variability reduction following the Plan-Do-Check-Act process model. My purposes in taking a systemic perspective were:

- To develop the general principles for implementation of variability reduction efforts
- To identify the specific actions for each phase of the improvement process
- To highlight the applicability of specific tools for each action step

In conclusion, I propose a policy strategy for variability reduction which consists of the following components:

- A set of general policy recommendations for variability reduction efforts
- Specific management directives for a one-year implementation plan to address the key barriers within SCD
- Recommendations for future work

The policy strategy again follows the framework of the Plan-Do-Check-Act process model. Both organizational and technical issues are addressed relevant to the people-systems-technology triad:

- **People**: Changing roles and responsibilities of managers, technical staff, production staff, and support staff
- **Systems**: Requirements of systems for team infrastructure, performance appraisal, and information technology
- **Technology**: Application of technology, including the evaluation of and training in new tools
2.4. Research Methodology

*Interactive Approach.* I worked closely with both the production and research staffs in order to learn the manufacturing processes and the analytical tools. Our teamwork helped to improve communications and facilitate technology transfer.

*Case Studies.* To evaluate methods of data collection and techniques for data analysis, I conducted a series of case studies. Table 1 provides an overview of the six cases. The selected chemicals covered a range of processes differing in the following characteristics:

- Manufacturing work center
- Mode of data collection
- Techniques applied for data analysis
- Type of process control system
- Availability of an integrated information system
- Level of process automation

I performed the cases according to the following methodology:

- Collect data to quantify sources of variability and monitor process parameters.
- Analyze data using process characterization techniques and data analysis tools.
- Identify the key process parameters.
- Identify the major sources of variability.
- Recommend process improvements based on findings.
- Iterate through methodology for further reduction of process variability.

*Field Visits.* In addition to the case studies, I made field visits to other Kodak manufacturing sites. The scope of the thesis research covered operations in Rochester,
<table>
<thead>
<tr>
<th>Chemical</th>
<th>Data Collection</th>
<th>Data Analysis</th>
<th>Control System</th>
<th>Information System</th>
<th>Process Automation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical A</td>
<td>- Automatic data acquisition.</td>
<td>- KDAT.</td>
<td>- Electronic.</td>
<td>- No integrated IS.</td>
<td>- No automation.</td>
</tr>
<tr>
<td></td>
<td>- Batch sheets.</td>
<td>- PMT meetings.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical B</td>
<td>- Automatic data acquisition.</td>
<td>- KDAT.</td>
<td>- Electronic.</td>
<td>- No integrated IS.</td>
<td>- Programmed temperature ramp.</td>
</tr>
<tr>
<td></td>
<td>- Batch sheets.</td>
<td>- Spreadsheet.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical C</td>
<td>- Automatic data acquisition.</td>
<td>- KDAT.</td>
<td>- Electronic.</td>
<td>- No integrated IS</td>
<td>- Pilot batch process automation project.</td>
</tr>
<tr>
<td>Chemical D</td>
<td>- Batch sheets.</td>
<td></td>
<td>- Pneumatic.</td>
<td>- No integrated IS</td>
<td>- No automation.</td>
</tr>
<tr>
<td>Chemical E</td>
<td>- Automatic data acquisition.</td>
<td>- KDAT; univariate diagnostics; MACMAN; decision trees; PCA/PLS multivariate analysis.</td>
<td>- Distributed control system.</td>
<td>- IS for analytical data.</td>
<td>- Full automation.</td>
</tr>
<tr>
<td>Chemical F</td>
<td>- Automatic data acquisition.</td>
<td>- KDAT.</td>
<td>- Distributed control system.</td>
<td>- IS for analytical data.</td>
<td>- Full automation.</td>
</tr>
<tr>
<td></td>
<td>- EVOP/DOX.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
England, Arkansas, and Tennessee. The purpose of the field visits were to observe and benchmark current practices within Kodak. Table 2 lists the field study sites with information on the process characteristics. My field observations helped to confirm the research findings of the detailed case studies.

Survey. To identify the impact of organizational factors on variability reduction efforts, I developed a survey which was distributed to the production staff in the Synthetic Chemicals Division. I use the survey results as a basis for recommending priorities for implementing organizational change.
<table>
<thead>
<tr>
<th>Chemical</th>
<th>Data Collection</th>
<th>Data Analysis</th>
<th>Control System</th>
<th>Information System</th>
<th>Process Automation</th>
</tr>
</thead>
</table>
| Chemical G | • Manual logging of process data points on batch data sheets.  
• Manual entry of data into IS.  
• Strip charts. | • Data reviewed in meetings 3X/week.  
• SPC analysis performed using IS. | • Pneumatic. | • IS on mainframe. | • No automation; older equipment. |
| Chemical H | • Automatic collection using control system. | • Sporadic troubleshooting of problem batches.  
• Use KDAT for time series analysis. | • Distributed control system. | • IS on mainframe. | • Full automation. |
| Chemical I | • Manual logging of process data points on batch data sheets.  
• Manual entry of data into IS. | • Data reviewed in team meetings.  
• SPC analysis performed using IS. | • Pneumatic. | • IS on mainframe with PCs networked. | • No automation; older equipment. |
| Chemical J | • Automatic collection using control system. | • Still in plant start-up. | • Distributed control system. | • IS on mainframe with PCs networked. | • Full automation; brand new plant. |
3. Results

3.1. Guidelines for Data Collection

The key results presented in this section address the following questions:

- What data should be collected?
- Which data collection method should be used?
- How can current data collection activities be evaluated?

A case study is presented in section 3.1.4 to illustrate the application of the recommended guidelines for data collection.

3.1.1. Data Collection Matrix

The data collection matrix was developed to provide a systematic identification of what data should be collected for process analysis. The data requirements are characterized by unit operations. Each batch recipe can be broken down into a sequence of basic unit operations typical of all chemical batch manufacturing. For the type of processing done by each unit operation, a list of required and optional process parameters are identified. The required parameters are deemed to be the key process variables for that type of unit operation. The optional parameters are additional data that may be useful depending on the specific requirements of the chemical manufacturing process.

Classification of Data Requirements by Unit Operations. Table 3 shows the data collection matrix for the unit operations encountered in SCD processes. The various unit operations and key process variables are described below:

- *Chemical Addition*: Addition of a liquid or solid material into a reaction vessel. The rate of addition, contents temperature, and agitator speed are among the key process variables.
| Process Variable | Unit Operation | Rate of Addition | Vessel Temperature | Vapor Temperature | Agitator Speed | Nitrogen Purge | Elapsed Time | Rate of Reflux | In-Process Tests | Vessel Pressure | Driving Force | Pressure/Vacuum | Weight | Purge Time | Stirring Time | pH | Amount of Non-Product Layer | Volume per Load | Centrifuge RPM | Number of Washes | Amount of Wash | Centrifuge Temperature | Condenser Type | Dryer Type | Dryer RPM |
|------------------|----------------|------------------|-------------------|------------------|----------------|----------------|--------------|--------------|----------------|----------------|--------------|---------------|--------------|--------|------------|--------------|----|--------------------------|----------------|-------------|----------------|--------------|----------------|--------------|-----------|----------|
| Chemical         | Addition       | R R R R R R R     | O R               | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
|                  | Reaction       | R O R R O O O R   | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
|                  | Filtration/    | R R R R R O O     | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
|                  | Pressing       |                  |                   |                  |                |                |              |              |                |                | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
| Washes           |               | O R R R R O R O R | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
| Crystallization  | Precipitation  | O R R R R R R    | R                | R                | R              | R              | R            | R            | R              | R              | R            | R R           | R            | R                   | R             | R          | R          | R            | R              | R R R R      | R R R R   | R R         |
|                  | Distillation   | R R R O O R R R   | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
| Centrifuging     |                | R R R R R O       | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
| Refluxing        |                | R R R R O O R O R | O                | O                | O              | O              | O            | O            | O              | O              | O            | O R           | O            | O                   | O             | O          | O          | O            | O              | O R          | O R O R   | O R         |
| Drying           |                | R R R R O R R     | R                | R                | R              | R              | R            | R            | R              | R              | R            | R R           | R            | R                   | R             | R          | R          | R            | R              | R R R R      | R R R R   | R R         |
• **Reaction**: Chemical reaction of reactants to form a product. The time-temperature profile, agitator speed, and in-process analytical tests are important variables for monitoring the progress of the reaction.

• **Filtration/Pressing**: Solid-liquid separation of solids from solution. The filter mechanism either collects the solid contaminants from solution, as in a filter press, or collects the solid product, as in a membrane press. Filtering yields are a function of the pressure driving force, solution temperature, and the mechanical efficiency of the equipment setup.

• **Washes**: Physical separation of two liquid phases upon the addition of a wash, such as water. Critical process parameters in decanting include the vessel temperature, wash temperature, agitator speed, stirring time, and settling time.

• **Crystallization/Precipitation**: Formation of solid crystals from solution due to solubility relationships. The controllable parameters for crystal properties and yield are the contents temperature and rate of cooling. For downout operations, the rate of pumping is also important.

• **Distillation**: Removal of the more volatile material from a solution mixture. The vessel time-temperature and pressure profiles determine the phase equilibrium and rate of distillation. Nitrogen purge is sometimes applied to control foaming.

• **Centrifuging**: Liquid-solid separation by applying a centrifugal force to remove the liquor to form a solid cake. The centrifuging operation is dependent on the techniques used to load and wring the cake, including the rate of loading, volume per load, centrifuge speed, wring time, number of washes, and amount per wash.
• *Refluxing*: Similar to distillation, except the solvent is condensed and returned back to the reaction vessel.

• *Drying*: Removal of moisture content from a wet feed into a solid product via heat input. Important process parameters include dryer temperatures and drying time.

*Collecting Data for Systems with Batch Process Control.* Monitoring the process variables listed in the data collection matrix is the first step in identifying batch process variability. However, for systems with batch process control, faults or disturbances affecting key process variables may be masked. The process variables become independent, *controlled parameters*. Because the disturbances are compensated for by feedback control, the controlled process parameters will remain at their setpoints and no effects will be detected.

Instead, additional data on the controller outputs and for equipment calibration, which are the uncontrolled manipulated variables, should be collected. These uncontrolled variables are referred to as *verification parameters*, and are useful in identifying changes in the process or equipment masked by batch process control.

Examples of process faults that may not be immediately recognized in the controlled variables are sensor calibration shifts, piping leaks, equipment degradation, and actuator malfunctions. Verification parameters expose the disturbances of process faults that are hidden by process control. Table 4 lists the verification parameters for the corresponding controlled process variable in the data collection matrix.
Table 4. Controlled Parameters Versus Verification Parameters

<table>
<thead>
<tr>
<th>Controlled Parameters</th>
<th>Verification Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate of Addition</td>
<td>• Flow controller output (valve position, pump speed)</td>
</tr>
<tr>
<td></td>
<td>• Temperature controller output (for temperature-controlled additions)</td>
</tr>
<tr>
<td>Vessel Temperature</td>
<td>• Temperature controller output (valve position)</td>
</tr>
<tr>
<td></td>
<td>• Steam flow rate, pressure</td>
</tr>
<tr>
<td></td>
<td>• Tempered water flow rate</td>
</tr>
<tr>
<td>Vapor Temperature</td>
<td>• Thermocouple calibration</td>
</tr>
<tr>
<td>Agitator Speed</td>
<td>• Current/power draw at motor</td>
</tr>
<tr>
<td>Nitrogen Purge</td>
<td>• Nitrogen flow rate, pressure, valve position</td>
</tr>
<tr>
<td>Elapsed Time</td>
<td></td>
</tr>
<tr>
<td>Rate of Reflux</td>
<td>• Condenser water outlet temperature</td>
</tr>
<tr>
<td></td>
<td>• Condenser temperature controller output (valve position)</td>
</tr>
<tr>
<td></td>
<td>• Vapor temperature</td>
</tr>
<tr>
<td></td>
<td>• Temperature controller output</td>
</tr>
<tr>
<td></td>
<td>• Heat flux (steam flow rate, tempered water flow rate)</td>
</tr>
<tr>
<td>Analytical In-Process Tests</td>
<td>• Equipment calibration</td>
</tr>
<tr>
<td>Vessel Pressure</td>
<td>• Ejector states for multistaged ejectors (on/off)</td>
</tr>
<tr>
<td></td>
<td>• Vent valve position (block versus bleed pressure control)</td>
</tr>
<tr>
<td></td>
<td>• Nitrogen purge valve position (block versus bleed pressure control)</td>
</tr>
<tr>
<td></td>
<td>• Pressure controller output (valve position)</td>
</tr>
<tr>
<td>Jacket Temperature</td>
<td>• Jacket controller output (valve position)</td>
</tr>
<tr>
<td></td>
<td>• Steam flow rate, pressure</td>
</tr>
<tr>
<td>Driving Force Pressure/Vacuum</td>
<td></td>
</tr>
<tr>
<td>Weight</td>
<td>• Scale calibration</td>
</tr>
<tr>
<td>Purge Time</td>
<td></td>
</tr>
<tr>
<td>Wash Temperature</td>
<td>• Thermocouple calibration</td>
</tr>
<tr>
<td>Stirring Time</td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>• pH meter calibration</td>
</tr>
<tr>
<td>Amount of Non-Product Layer</td>
<td>• Drum volume calibration</td>
</tr>
<tr>
<td>Volume per Load</td>
<td>• Vessel volume calibration</td>
</tr>
<tr>
<td>Centrifuge RPM</td>
<td>• Current/power draw at motor</td>
</tr>
<tr>
<td>Number of Washes</td>
<td></td>
</tr>
<tr>
<td>Amount of Wash</td>
<td>• Solvent meter calibration</td>
</tr>
<tr>
<td>Centrifuge Temperature</td>
<td></td>
</tr>
<tr>
<td>Condenser Temperature</td>
<td>• Condenser temperature controller output (valve position)</td>
</tr>
<tr>
<td></td>
<td>• Brine flow rate, temperature</td>
</tr>
<tr>
<td>Type of Dryer</td>
<td></td>
</tr>
<tr>
<td>Dryer RPM</td>
<td>• Current/power draw at motor</td>
</tr>
</tbody>
</table>
An Example. A process flow diagram represents the sequence of unit operations required in a batch recipe. For the reaction unit operation, the following process variables are important to monitor:

- **Vessel Temperature**: To indicate the rate of reaction
- **Agitator Speed**: To determine the degree of mixing
- **Elapsed Time**: To estimate the extent of reaction
- **Jacket Temperature**: To measure the amount of heating or cooling

Depending on the specific batch recipe, other optional data may be important to collect, such as:

- **Rate of Reflux**: If the reaction occurs with refluxing of the solvent.
- **Analytical Tests**: If in-process tests, such as liquid chromatographs, gas chromatographs, or thin-layer chromatographs, are used to check for full conversion of a key reactant.
- **pH**: If proper pH adjustment is critical for downstream process steps.

The presence of a temperature control loop can mask the degree of fouling in the vessel jacket. Monitoring of the contents temperature, which is a controlled parameter, reveals no indication of the problem. However, tracking of the temperature controller output provides additional information. This verification parameter reveals that the valve position has widened. Because the reaction is exothermic, additional cooling water was required to keep the contents temperature at the setpoint.
3.1.2. Data Collection Methods Guide

Three possible methods of data collection are data acquisition systems, operator batch sheets, and PMT meetings. The selection of an appropriate data collection method depends on the following factors:

- Existing equipment, including the availability of sensors and a data acquisition system
- Type of information that needs to be gathered, quantitative or qualitative
- Usage of the data
- Current organizational infrastructure

In general, integrating the use of the three proposed data collection methods maximizes the quantity and quality of the data collected, as well as the level of production staff involvement.

Data Acquisition Systems: Data acquisition systems can be stand-alone units or part of the functionality of a batch control system. Desirable features of a data acquisition system include:

- **Automatic Logging of Data:** Scan data at a defined rate, including the time stamp and batch identification.
- **Transport of Data:** Interface with other computing environments to transfer data for further manipulation and analysis. Integrate data from other sources into the batch record.
- **Event Tracking Capability:** Mark events, or key process points, to enable the comparison between different batches.
- **Graphical Analysis:** Graph trend plots for batch tracking and basic data review; create batch reports.
- **Data Archival and Retrieval:** Store data for later retrieval; protect data integrity.
• *Automatic Exception Analysis*: Monitor key process variables and flag abnormal process conditions.

*Operator Batch Sheets*. Operators use BATch MANagement (BATMAN) sheets to manually log critical process data points. BATMAN sheets are easy to implement, and should be tailored to collect the needed data for a given batch chemical process. The exercise of identifying the critical few parameters for manual data collection generally provides important insights into the process.

*PMT Meetings*. The most effective PMTs are production-driven, cross-functional teams composed of operators, process chemists, and support staff from the research, development, engineering, analytical, and quality functions. PMT meetings facilitate communications and the sharing of experiential data amongst the production and support staffs. The meetings also provide a forum for group problem-solving and data analysis. PMTs play a key role in the analysis of data to drive process understanding and process improvements.

*Selecting a Data Collection Method*. Specific suggestions on the usage of each data collection method are given below.

**Use Data Acquisition Systems:**
- whenever available to collect key process parameters. The automatic collection of batch data reduces labor requirements and eliminates manual transcription errors.

**Use Operator Batch Sheets:**
• when the unit operation or process variable is not amenable to automatic data collection, i.e. centrifuge, washes, pH.

• when the capital expenditure for a data acquisition system is not available. A preliminary cost-benefit analysis with BATMAN sheets can be used to evaluate the capital expenditure for a data acquisition system.

• when the goal is to highlight operator attention to important process steps.

Use Process Management Team Meetings:

• when teamwork and operator involvement are important.

• when the goal is to improve communications amongst production staff and functional support groups.

• when the review of data is used to develop common understanding and standardize production procedures.
3.1.3. Data Collection Evaluation Grid

Process analysis is the means by which process understanding is derived from data analysis. The Data Collection Evaluation Grid (Figure 2), which relates the level of data content to the level of participation, illuminates the two key factors in the transformation of data into information. The level of data content refers to both the amount and quality of information, and is a function of the available hardware systems (data acquisition systems, process controllers) and operational protocols (operator logs, batch sheets) in place to gather data.

The level of participation is a measure of production and support staff involvement in reviewing and analyzing the data. The success of variability reduction efforts lies in getting people involved in understanding and improving their processes. Data is useless unless it is used. The effectiveness of data collection activities is measured by its impact on day-to-day operating decisions.

Figure 2. Data Collection Evaluation Grid

![Data Collection Evaluation Grid Diagram](image-url)
Mapping Out the Evaluation Grid. The case studies and field study sites are mapped onto the Evaluation Grid in Figure 2. Processes with high levels of participation and data content are represented in quadrant 1. The examples in this quadrant represent sites with participative management and modern equipment. Chemical E is the best case, with a close-knit production and support staff, automatic data acquisition, distributed process control, and full process automation.

Quadrant 2 represents processes with high participation, but low data content. The typical characteristics are sites with participative management, but old equipment. Chemicals G and I are two examples where data was manually collected on batch sheets. The distinguishing trait of these two operations was the pervasive believe in a data-driven operating strategy. A well-defined set of critical process data points was identified, collected, and regularly reviewed by the production staff. The availability of an information system encouraged operator involvement by making data accessible and easy to analyze. Although the lack of a data acquisition system can restrict the amount of data that is collected, a great deal of process improvements can be made with a limited, yet meaningful data set.

The chemical J site is located in quadrant 3, represented by low participation and high data content. Operations in quadrant 3 typically have traditional management and new equipment. Although data acquisition systems may be used to collect data, review of the data by production staff is done infrequently or only for troubleshooting. Chemical J is made in a new plant with a distributed control system. The plant is currently undergoing start-up operations and should soon phase-in a participative management structure. With an available data acquisition system, the key challenge is in training. Operators,
engineers, and managers need to know how to analyze and interpret the results drawn
from available data.

Quadrant 4 represents sites with low participation and low data content. Operations are
run with traditional management and old equipment. Little to limited data may be
available from existing modes of data collection, but not much effort is put into reviewing
the data. Chemical D is an example of a chemical manufactured in older, pneumatically
controlled equipment. A data acquisition system is not in place to collect process
measurements. Some key process data are manually logged on operator batch sheets.
However, not enough analysis is done with the collected data. Without more data and
more staff involvement, suspected sources of process variability are hard to quantify.

The case studies and field experience indicate that the most effective data collection
activities are those that have both high levels of participation and data content, represented
in quadrant 1. For these processes, the large amount of available data are effectively
used. In instances with a limited data set, a high level of involvement from the
production staff seems to be the key criterion of success, represented in quadrant 2. For
operations represented in quadrant 3, the potential benefits from a high level of data
content have yet been realized due to a low level of participation. Processes represented
in quadrant 4 suffer from setbacks in both low data content and participation.
3.1.4. Chemical A Case Study

Background. The chemical A case study highlights the role of data collection activities in variability reduction efforts. Chemical A is made in a production bay upgraded with an electronic control system. Although, the process controller was equipped with data acquisition and limited process automation capabilities, the full potential of the system has not yet been exploited. The upgraded control system was still used in a manner similar to the older pneumatic system it had replaced. Although the process controller was able to archive up to seven days of process data, the production staff needed to be trained in using the system. The new technology had been pushed-in, rather than pulled-in.

As standard procedure, the process chemist met with the operators in the production area at the beginning and end of a campaign. The group leader responsible for the job played a key role at the PMT meetings to review the manufacturing procedure. In addition, some data that was manually collected would be reviewed. Some emphasis was made to monitor key process variables to target and quantify the sources of process variability.

The Strategy. As part my thesis research, I selected chemical A to demonstrate the value of available data acquisition systems for variability reduction efforts. To accomplish this, I teamed up with the process chemist and group leader. Our strategy was to:

- Review the data currently collected by the process control system
- Share the data and involve operators in developing a better understanding of the process
- Implement recommendations for variability reduction
- Determine additional data needs and methods of data collection
Figure 3 shows the process flow diagram of the batch recipe. Most of the unit operations occur in two reactor vessels which are monitored by the process controller. The data acquisition system was used to collect time series profiles of the following variables:

- Contents temperature setpoint, process value, and controller output
- Jacket temperature setpoint, process value, and controller output
- Vapor temperature and pressure

![Figure 3. Chemical A Process Flowsheet](image)

Although some data had been manually recorded in the past, a systematic review of data needs was conducted. Operator batch sheets were used to manually collect data on unit operations, such as centrifuging, and process variables, such as volumes and pH, not tracked by the control system.
PMT meetings were held to bring the production staff up-to-speed with the new technology in data acquisition and analysis. KDAT demonstrations were used to show quantifiable batch-to-batch variability in the process data. The PMT meetings were organized to review the process data, analyze the causes of process variability, and solicit suggestions for variability reduction. A critical success factor was the involvement of the operators, who were closest to the process. Research and development staff also participated.

The Results. During my research internship, we were only able to collect data for one production campaign. However, even with the limited data set, examples of our findings include the following:

• Clarified Operator Interpretations. In reviewing the time-temperature series data, differences in operator interpretation of the manufacturing procedure in ramping the distillation temperature setpoint were addressed.

• Standardized Operating Procedures. Some key process steps were standardized based on a review of historical data manually logged on operator batch sheets. Instead of directions for incremental solvent additions to obtain phase separation, manufacturing procedures were updated to use standard amounts, eliminating variability in the process step.

• Quantified Equipment Differences. Equipment differences were quantified in time-temperature profiles resulting from exothermic heat effects of different rates of chemical addition. Batch-to-batch variability were traced to the use of different pumps in chemical addition.
The status of our efforts is best summarized in the chemical A process variability cause-effect diagram shown in Figure 4. Most of the learning came in the process of reviewing the data and of sharing operational insights from amongst the production staff.

Next Steps. Our plan of action based upon the initial analysis includes the following steps:

- *Implement Variability Reduction Suggestions.* We have proceeded to implement the suggestions discussed in our PMT meetings. At this point, most of the improvement will come from clarification and standardization of operating procedures. We will follow-up with data from additional runs.

- *Collect Additional Data.* Review of the currently available data revealed the need for more information. In line with the guidelines of the data collection matrix, we have identified additional process variables for data collection. Two new BATMAN sheets were developed to collect the extra data, including additional gas chromatograph tests to check for the presence of water at the end of the distillation steps.

- *Perform Further Analysis.* We recognize that the data collection and analysis activities are a process of continuous improvement. We plan to collect process data for future batches in order to establish a bigger data set for further data analysis. Future possibilities include extending the use of the process controller to handle additional feedback control, automate setpoint changes, and sequence process operations.

Lessons Learned. The chemical A case study illustrates some of the important lessons learned in implementing data collection and analysis activities:
Figure 4. Chemical A Process - Sources of Variability

**Materials**
- Melted raw material quality
- Contamination of solvent C wash
- Cross-mixing of solvent delivery lines

**Measurement**
- GC
- pH
- Brine temperature
- Acid assay
- Calibration of platform scales

**Method**
- Concentrate solution
- Add Super Cel until solution is clear
- Control foaming during solvent E distillation
- Incremental salt additions to get phase separation
- Make-up of salt solution
- Reaction hold time
- Settling time for phase separation

**Chemical A Process Variability**
- Rate of water addition
- Rate of solvent D addition
- Rate of base addition
- Rate of salt addition
- Rate of solvent C drawout for crystallization

**Machine**
- Vessel vacuums
- Filter press efficiency
- Vacuum oven temperature
- Drying time
- Vacuum break procedure to add solvent E during vacuum distillation

**Manpower**
- Centrifuge loads
- Centrifuge washes
- Centrifuge speed and wring time
- Loading of vacuum oven trays

* Italics indicates current variability reduction efforts.*
• *Get Buy-in to New Technology*. The successful introduction of data acquisition systems and data analysis tools must be accompanied by an understanding and acceptance of that technology by the production staff who will use it.

• *Involve the Right People*. The involvement of operators closest to the process, and the functional support staff most knowledgeable in addressing specific technical issues, is a key criterion for success. Also, the implementation of variability reduction activities should be production-driven. Commitment, consistency, and ownership from the production staff should be cultivated.

• *Emphasize Continuous Learning*. Reducing process variability is a process of continuous improvement to develop a better understanding of the process. If the process is not automated, the key benefits will come from clarifying, standardizing, and quantifying operating procedures. If the process is automated, the key benefits will come from the detection of process faults by monitoring both the controlled parameters and the verification parameters.
3.2. A Guide to Data Analysis Techniques and Tools

The key results presented in this section address the following questions:

- What is a systematic procedure for process analysis?
- What types of analysis should be performed?
- When should different data analysis techniques and tools be applied?

A case study is presented in section 3.2.3 to illustrate the proposed step-by-step procedure for process analysis.

3.2.1. Data Analysis Techniques

Process Analysis as Hypothesis Testing. Process analysis involves iterations of hypothesis testing by which fundamental understanding of a process is drawn from the analysis of data. Process characterization is the pattern recognition of data which establishes a reference or good data set, based on the quality of the output. Process variability is a measure of the differences in comparing batches of good and bad data. To determine the sources of variability, suspected explanations need to be tested. The key to hypothesis formulation is consistency with knowledge of the physical phenomenon, manufacturing procedures, and engineering principles. The exercise of formulating and proving such hypotheses is the central element of process analysis. Insights that are gained lead to actions to reduce variability. The end result is better process understanding and a more repeatable process.

A Formalized Procedure. The proposed procedure is applicable for the analysis of multivariate batch process data. Typical batch data are time series traces of key process variables (independent, controlled parameters), such as temperature, pressure, and weight, and verification parameters (uncontrolled, manipulated variables), such as the
temperature controller output, flow controller output, and steam flow rate, collected by a data acquisition system.

The methodology outlined below progresses through increasingly rigorous techniques for process analysis. The procedure was developed based on the learning from the case studies.

Step 1: Compare by Graphical Analysis Good Versus Bad Batches

The first step begins with an identification of a reference or good batch based on process or product quality measures. The main purpose is to try to visually detect patterns that differentiate good batches from bad ones. A good batch represents a standard run or the mean of a group of typical runs (as calculated by the Process AVerager utility program). Before evaluating all the available data, focus on a sampling of 4-10 runs which include both good and bad batches. An initial graphical analysis should be performed to highlight differences between good and bad runs. KDAT is a useful tool for the graphical analysis of batch time series data files.

Step 2: Formulate Univariate Diagnostics of Key Process Characteristics

The next step is to formulate univariate diagnostics to test for the causes of the bad batches. The diagnostics quantify process variability identified from graphical analysis. A univariate diagnostic is a single calculated variable which summarizes a major characteristic of a batch process profile. For the time-temperature profile, two examples of univariate diagnostics include: the slope of the curve and the area under the curve, which relates to the enthalpy of the system, i.e. heat of reaction, heat input, or heat removal. The diagnostics may be specific to key process steps or events. For example, separate values of the slope and area of a time-temperature profile may be calculated for the reaction and distillation unit operations. In this way, the diagnostic tests can be used to highlight batch-to-batch differences in each unit operation.
Step 3: Perform Statistical Analysis with Univariate Diagnostics

Inductive reasoning is used to make general conclusions based on the analysis of a limited data sample. The hypothesis formulated from the initial analysis is tested against a larger data set. Statistical analysis using univariate diagnostics is performed to confirm the hypothesis. MACMAN is a useful tool for calculating and control charting the univariate diagnostics. The actual value of the diagnostic or its standard deviation from the mean is plotted on the y-axis; the batch number is plotted on the x-axis. Iterations of the first three steps of the procedure often occur before a final set of univariate diagnostics is defined. Once the critical diagnostic tests have been identified, process analysis can be automatically calculated for additional batches.

Step 4: Apply Multivariate Analysis

Statistical multivariate methods are useful for the analysis of large amounts of correlated process data monitored for a batch process. Often, due to correlation between different variables, a statistical technique which looks at multiple effects is needed to discriminate good batches from bad ones. While univariate techniques (one variable at a time) depend upon the visual inspection of control charts for each diagnostic test, multivariate techniques compress the total set of univariate diagnostics into fewer latent or derived variables, which represent the underlying physical phenomena. Principal Components Analysis and Partial Least Squares techniques use statistical algorithms to detect process faults, equipment malfunctions, and other occurrences of special variability. The results are automatically computed in control charts and other reports for the reduced set of latent variables. The statistical methods can also be used to build predictive process models.

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PCA and PLS are two powerful multivariate analysis techniques to increase process understanding.

**Step 5: Capture Learning in Decision Trees**

To derive the most benefit from process data, the results from the analyses need to be summarized into a meaningful and usable format. A decision tree portrays the acceptable range of operating conditions for the critical process variables. The information from univariate or multivariate analysis is captured in terms of the original, measured process parameters. Each branch of the decision tree represents a unique set of process conditions that leads to a good or bad batch. Cause-effect diagrams are also useful for summarizing the sources of process variability that have been identified.

**Addendum: Apply Design of Experiments / EVolutionary OPeration**

DOX and EVOP are statistical methods of orthogonal experimentation analysis. Process parameters and settings, referred to as factors and levels, respectively, are identified and systematically perturbed over a set of experiments. DOX is typically applied in process development where controlled laboratory experiments are run. EVOP is applied in production where small changes in the process variables are made that do not significantly interfere with the ongoing process. The results of the changes in response are statistically analyzed to quantify the effects and interactions between several process parameters. Although not specified as one of the steps for process analysis, DOX and

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EVOP are statistical experimentation techniques that can be called upon at any point. The benefits of the two approaches include:

- Fundamental understanding of the key process parameters
- Better process control from start-up
- Shorter learning curve for process certification
- Guidelines for optimal operating conditions
- Lower production costs
3.2.2. Data Analysis Tools

Process Analysis Applications. The benefits of process analysis are most apparent when it is applied as a routine part of daily production operations. Both production staff and technical support play an integral role in implementing data analysis techniques and tools. Production staff use data analysis to quantify process variability, to identify improvement opportunities, and as a basis for establishing statistical process control. Technical support provides technical expertise, training, and insights for applications in other functional areas. For example, process analysis can be useful for preventive maintenance, in identifying process steps requiring development efforts, and as part of the transfer package to introduce a new chemical process from research to manufacturing.

The data analysis tools evaluated in this research were available from Kodak's research and engineering group. Descriptions and suggestions for their use are provided below: Kodak Data Analysis Tool.25 KDAT is a Kodak internally developed software tool which analyzes process data collected by multiple sensor channels. Time series profiles of monitored process variables are stored in batch data files. Data manipulation and management features are used to perform calculations and generate graphs for batch-to-batch comparison. Three important KDAT features include:

- **Graphical Analysis with Event Markers.** KDAT was designed for batch process data analysis. Events, or key process operations, can be marked for synchronization at analogous points across batches. An important application of KDAT is the graphical analysis of process profiles by comparing good versus bad batches. Figure 5 shows a graphical comparison of the crystallization time-temperature profiles for four batches.

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Figure 5. Sample KDAT Graphical Analysis
X-axis: Time (hr); Y-axis: Temperature (C)
• *Data Manipulation.* KDAT includes a set of standard mathematical functions and statistical commands. In addition, a series of KDAT commands can be programmed as a macro. As an example, a macro can be written to retrieve the time-temperature profiles for all batches in a campaign, load the data segment for the distillation process step, calculate the temperature ramp rate and maximum temperature for each batch, and present the results in control charts. In addition, KDAT data files can be interfaced with the MACMAN, PCA, and PLS software packages. A library of KDAT SPC macros have been written for MACMAN.

• *Data Management.* KDAT is set up to retrieve data files, other KDAT programs, store intermediate calculations, and generate output reports. KDAT commands can be used to load entire data files, specific data segments, or data points at selected intervals.

KDAT is a powerful tool for quantitative data analysis. It has advantages for data management, flexible graphical analysis, data manipulation using macros, and interfacing with other data analysis packages. The most appropriate users are process chemists, manufacturing engineers, quality statisticians, development engineers, and group leaders. Training and continued technical support are necessary for effective technology transfer and implementation.

*MACroMANager.* MACMAN is a front-end interface to KDAT that enables the user to build, test, and review a template or set of univariate diagnostics. The diagnostic tests are run for selected batch data files, and the resulting calculations are control charted. MACMAN's user-friendliness is due to the following characteristics:

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• Easy Diagnostic Testing. MACMAN facilitates the hypothesis testing stage of process analysis. Univariate diagnostics are formulated based on a library of available KDAT SPC macros. For example, a general KDAT SPC macro which calculates the slope of a curve can be used to build a univariate diagnostic of the temperature ramp rate of the distillation process step. This capability makes it easy to test for key process characteristics in the data.

• Control Charting of Univariate Diagnostics. The calculated diagnostics are presented graphically in control charts. Visual inspection for out-of-control data points is used to identify univariate diagnostics which cause bad batches. A statistical summary of the mean and 3-sigma control limits is also provided. Figure 6 shows a control chart of the amount of solvent removed during a distillation operation for 37 batches.

• Easy to Use Interface. MACMAN uses pull-down menus, pop-up windows, graphical displays of data and results, user annotation in reports, and built-in statistics functions.

MACMAN is a useful tool for the statistical analysis of univariate diagnostics. The development of the test template is an iterative process best accomplished in a group with a process chemist, group leader, and technical staff support from the research labs. After a set of critical diagnostics have been identified, operators can be given the responsibility to review the control charts as a routine production activity. Out-of-control conditions would be a signal of a process fault or an operator error. Over time, the goal is both to eliminate special cause variability, and to narrow the range of normal process variability. A basic understanding and working knowledge of KDAT concepts is a prerequisite for using MACMAN.
Figure 6. Sample MACMAN Control Chart

X-axis: Batch Number; Y-axis: Weight (kg)
Principal Components Analysis²⁷,²⁸/Partial Least Squares Toolbox.²⁹ The use of PCA and PLS for process analysis is a recently developed application of two established multivariate analysis techniques.³⁰ PCA is a procedure used to explain the variance in a single data matrix of process variables only. PLS is a method which uses the information contained in the process variables to predict changes in the output process performance or product quality variables. Descriptions of the PCA/PLS models are provided in Appendix 1. The mathematical algorithms are described by Jackson, and Kresta et al. The basic principles of these two statistical techniques are:

• Both PCA and PLS are suitable for analyzing large sets of correlated data.

• The multivariate techniques compress a large set of physical process variables into a small set of derived latent variables.

• A latent variable is the linear combination of the set of process variables multiplied by the corresponding loading factors. The first latent variable (or principal component) describes the greatest variability in the process variables. The second latent variable explains the greatest amount of the remaining variability, and so on.

• For PLS, the latent variables are selected to be highly predictive of the output variables.

The input for PCA/PLS analysis is a data file containing the calculated values of the univariate diagnostics for a set of batches. The multivariate techniques use a statistical regression algorithm to regress the product quality variables onto the process variables.

The data is mean-centered and scaled to account for differences in the units of the variables. For PCA, a complete decomposition of the data matrix shows the percent variance explained by each latent variable. The user is then asked to select the number of latent variables to retain in the model. For PLS, a cross-validation procedure is performed to determine the number of latent variables corresponding to the minimum cumulative predicted error sum of squares.

The essential information generated by the PCA/PLS model is provided in the following results. A case study example is discussed in section 3.2.3:

• **Loadings:** Weightings of the original process variables with respect to the latent variables. The bigger the loading, the greater the contribution of that process variable to the latent variable. (See Table 6 in section 3.2.3 for an example.)

• **Control Charts of Latent Variable Scores:** Plots of the latent variable score (y-axis) versus the batch number (x-axis). Out-of-control batches highlight variability that cannot be accounted for by the multivariate model. For PCA, a control chart of the $T^2$ statistic is also generated. The $T^2$ value, which is the sum of squares of all the scores of the latent variables, is a single quantity that represents the entire batch. (See Figure 16 in section 3.2.3 for an example.)
• **Score Plots**: Graphical representation of the batches onto a plane defined by two latent variables. Look for outliers, clusters, or other patterns in a scatterplot of the latent variables. (See Figure 17 in section 3.2.3 for an example.)

• **Contribution to Scores**: The contribution of each process variable to the latent variable scores for a given batch. This analysis highlights the process variables which cause a latent variable score to go out-of-control. Look for patterns of score contributions for bad batches to develop a decision tree. (See Figure 18 in section 3.2.3 for an example.)

MACMAN calculations of the univariate diagnostics can be interfaced for PCA/PLS analysis. The run procedure for the PCA/PLS algorithms can be standardized and automated. The important step is the review of the results by the production staff. PCA/PLS produces a more manageable set of outputs than the large number of control charts generated by MACMAN. Process chemists and operators need to be trained to interpret the PCA/PLS results.

The routines for the PCA/PLS Toolbox must be used with the MATLAB software package. MATLAB provides the environment for the necessary numerical methods and algorithms. A working knowledge of MATLAB is a prerequisite for using the PCA/PLS Toolbox.

*Selecting a Data Analysis Technique or Tool.* Specific recommendations for applying each of the techniques and tools are provided below:

*Use KDAT:*

• To perform graphical analysis of a limited data set, up to 12 batches at one time
• To compare key process operations or events across batches

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• To prototype univariate diagnostics

*Use Univariate Diagnostics:*
• To quantify key process characteristics
• As measures of process variability

*Use MACMAN:*
• To build and test a template of univariate diagnostics
• For hypothesis testing of data files
• For control charting of univariate diagnostics

*Use PCA/PLS:*
• To analyze large amounts of correlated data, i.e. process data, product quality data
• For statistical multivariate analysis of batch data
• For easy-to-interpret reports for process analysis

*Use Decision Trees:*
• To document the results of univariate and multivariate analysis into an easy-to-use format
• To classify the set of process conditions leading to a good versus bad batch
• To quantify the operating range of critical process parameters

*Use DOX/EVOP:*
• As statistical methods of experimentation
• To quantify the effects and interactions among process variables
• To determine the optimal settings of the process variables
3.2.3. Chemical E Case Study

*Problem Definition.* The chemical E case study provides a comprehensive example of the application of the various data analysis techniques and tools. Chemical E is used in Kodak's sensitized-goods. It is made in manufacturing equipment with a distributed control system. The scope of this case study was focused on the solvent A distillation operation.

Solvent A is removed from the chemical E product solution by vacuum distillation. Solvent B is then added to the stripped melt to crystallize out the product. It is desirable to remove as much solvent A as possible in the distillation operation. The presence of solvent A impairs crystal formation and adversely affects product quality. However, if too much solvent A is removed, the melt viscosity will rise rapidly resulting in the solidification of the melt.

Endpoint detection of the solvent A removal is difficult. As solvent A is removed, the viscosity increases, and the power draw of the electric agitator rises. Operators visually monitor the agitator kilowatt reading. When a value of about 8 kilowatts is reached, the operator stops the distillation. However, the endpoint can be missed because a sharp rise in the kilowatts can occur over a period of less than one minute at the end of the distillation.

Bad batches that solidify require repeats of the distillation sequence in which solvent is added back to dissolve the melt. Because a rework procedure exists, bad batches do not affect final product quality. Instead, melt solidification can potentially damage the agitator blades in the reactor vessel. In addition, rework adds to the manufacturing cost, causes variability in the cycle time, and requires additional equipment time, labor, and solvent.
**Action Plan.** For this case study, I worked closely with the site technical manager, the chemical E process chemist, and support staff from the research and engineering and quality assurance groups. The complete analysis is based on 37 batches of historical data. Of the 37 runs, 4 batches set-up, i.e. the melt solidified in the reactor vessel at the end of the distillation sequence. The process variables collected by the control system included the reactor mass, reactor temperature, reactor pressure, vapor flow, receiver mass, and kilowatt readout. Our action plan was to:

- Identify the process conditions leading to melt solidification
- Define the operating conditions which optimize the removal of solvent A
- Demonstrate the applicability of available data analysis techniques and tools by following the proposed procedure for process analysis
- Implement recommendations to avoid future set-ups

**Distillation Control Strategy.** Because chemical E is an automated process, the process controller plays a critical role in controlling the sequence of manufacturing steps. The removal of solvent A by vacuum distillation is divided into two steps in the manufacturing control sequence.

DIST: In the DIST step, the bulk of the solvent A is removed. Solvent A is distilled off under reduced pressure (setpoint at 225 mmHg). Initially, the reactor is under temperature control with a setpoint at 20°C above the boiling point. The temperature control manipulates the jacket steam rate. The vapor flow is under scan to check when the vapor flow reaches 10% of the scale, an indication that a good distillation rate has been reached. When this occurs, the temperature loop is turned off, and the system is put under vapor flow control. The vapor flow loop maintains the flowrate between 10-50%. However, there are time lags in the feedback loop, and the vapor flow can fluctuate
outside the desired range. To keep the process in control, the pressure setpoint is
increased, which increases the boiling point of the mixture, and drops the vapor flow.
When the vapor flow returns back to the desired range, the pressure setpoint is decreased
to the initial value. The DIST operation is stopped when the receiver weight reaches
1000 kg, or either the reactor or vapor temperatures hit upper limits.

STRIP: In the STRIP step, the remaining solvent A is removed. The vessel is put under
temperature control by manipulating the steam rate to the jacket. The pressure setpoint is
put at 200 mmHg. During the distillation, the temperature is kept within specified
ranges. The vapor flow is also scanned to keep it under 50%. The reactor weight is
continuously monitored at 20 minute intervals to check if a specification of 2800 kg has
been reached. If not enough solvent A has been stripped off during that time, the
pressure setpoint is increased by 50 mmHg, and the STRIP is continued. When the
specified reactor weight is reached, the STRIP is stopped, and the pressure setpoint is
ramped to 770 mmHg over one minute.

In reality, the 2800 kg reactor mass specification is set at an artificially low figure.
Instead, the endpoint of the STRIP step is determined manually by the control room
operator monitoring the kilowatt reading of the electric agitator. As solvent A is
removed, the power draw of the agitator rises. When a value of about 8 kilowatts is
reached, the operator stops the STRIP.

We performed the process analysis as described below:

*Step 1: Compare by Graphical Analysis Good Versus Bad Batches.* We compared two
good and two bad batches as a first step in diagnosing the causes of melt solidification.
The most revealing KDAT graphs, shown in Figures 7 and 8, point out differences in the
pressure and temperature profiles, respectively. The bad batches tend to operate at higher
Figure 7. Chemical E KDAT Pressure Profiles - Original

X-axis: Time (min); Y-axis: Pressure (mmHg)
Figure 8. Chemical E KDAT Temperature Profiles - Original

X-axis: Time (min); Y-axis: Temperature (°C)
pressures and temperatures.

Based on this preliminary KDAT analysis, we formulated the following hypothesis. Time lags in the controller feedback loops can cause the vapor flow to fluctuate above the desired range of 10-50% of the scale in the DIST step. When this happens, solvent A removal occurs at a higher rate. The result is higher pressures and temperatures throughout the DIST step. The higher temperature and the quick drop in the pressure setpoint to 200 mmHg at the beginning of the STRIP step causes more of the solvent A to flash off. If too much solvent A is stripped off, and at a faster rate, the melt solidifies.

*Step 2: Formulate Univariate Diagnostics of Key Process Characteristics.* We formulated a number of univariate diagnostics to assess the key process characteristics shown in the graphical analysis. A sampling of the diagnostic tests we developed include:

- **KW END**: KW reading at the end of the STRIP step
- **PMAX DIST**: Maximum pressure of the DIST step
- **TMAX DIST**: Maximum temperature of the DIST step. The DIST step is under vapor flow control, with the pressure setpoint as a manipulated parameter. The pressure and temperature profiles are closely correlated due to phase equilibrium relationships. The control charts for PMAX DIST and TMAX DIST exhibit similar behavior.
- **PSPDEV DIST**: Deviation between the reactor pressure and pressure setpoint profiles for the DIST step
- **RATE DIST**: Average rate of distillation for the DIST step
- **PRANGE STRIP**: Pressure range for the first 10 readings at the start of the STRIP step. This univariate quantifies the pressure drop between the end of DIST and the beginning of STRIP.
- **PEND STRIP**: Pressure at the end of the STRIP step
• TAREA STRIP: Area under the temperature curve for the STRIP step
• VF DELTA: Smoothed function of the difference in the vapor flow profiles between the current reading and the immediately preceding reading. This is a measure of the fluctuations in the vapor flow.

Step 3: Perform Statistical Analysis with Univariate Diagnostics. We used MACMAN to build and test the template of univariate diagnostics for all 37 batches. We made several iterations in order to identify the final set of 23 diagnostics. In the end, we were able to confirm the hypothesis with diagnostic tests that statistically detected out-of-control conditions for the bad batches.

Out of the 37 batches, the four bad batches are runs 3, 5, 14, and 23. A brief review of the MACMAN results is given below:

• KW END: The control chart of the final KW reading (Figure 9) shows that most batches are stopped at or below 8 kilowatts. Of the four bad batches, batches 3, 14, and 23 are out-of-control with high readings near 15 kilowatts. Batch 5 also set-up, but with a normal reading at about 8 kilowatts. These results indicate that visual monitoring of the agitator kilowatt reading may not be the best method for endpoint detection of the distillation operation. Sometimes the kilowatt rises so sharply that the endpoint can be missed, as in batches 3, 14, and 23. Other times, the 8 kilowatt stop criterion is not sufficient. During the time interval that it takes to add solvent B to the melt, the viscosity can continue to rise and result in melt solidification, as in batch 5. This analysis results in two recommendations: 1) Translate the stop criteria conditions in terms of the key process variables, i.e. pressure and temperature. 2) Automate the endpoint detection function.
Figure 9. Chemical E MACMAN KW END Control Chart

X-axis: Batch Number; Y-axis: KW END (kilowatt)
• Bad batches 3, 5, and 14 show similar process characteristics prior to batch set-up. These batches have higher PMAX DIST, TMAX DIST, and PRANGE STRIP, as shown in Figures 10, 11, and 12, respectively. The diagnostics confirm the hypothesis that bad batches reach a higher pressure and temperature at the end of DIST. When pressure is dropped at the beginning of the STRIP step, a larger amount of solvent A is flashed off which causes the melt to solidify.

• Bad batch 23 displays a different phenomenon from the other bad batches. The only diagnostic to explain reactor set-up is PEND STRIP, shown in Figure 13. The pressure profile for batch 23 steps down to 180 mmHg, about 20 mmHg lower than any other batch. The explanation is that with a better vacuum, more solvent A may be removed in the STRIP step, causing the melt to solidify.

• Batch 6 is identified as a marginal batch. From the diagnostic tests, batch 6 shows all the process characteristics similar to bad batches 3, 5 and 14. At the end of STRIP, a rising KW reading can be seen. The diagnostics show batch 6 on the boundary of conditions for reactor set-up. However, solidification of the melt is avoided.

• Batches 16 and 35 are good batches that exhibit some characteristics of a bad batch in PMAX DIST and TMAX DIST, as shown in Figures 10 and 11, respectively. However, these two diagnostics are necessary, but not sufficient evidence of a bad batch.

Step 4: Apply Multivariate Analysis. We conducted both PCA and PLS analysis using the final set of univariate diagnostics. We wanted to check whether the two techniques gave comparable results to the analysis performed via visual inspection of the MACMAN
Figure 10. Chemical E MACMAN PMAX DIST Control Chart

X-axis: Batch Number; Y-axis: PMAX DIST (mmHg)
Figure 11. Chemical E MACMAN TMAX DIST Control Chart

X-axis: Batch Number; Y-axis: TMAX DIST (C)
Figure 12. Chemical E MACMAN PRANGE STRIP Control Chart

X-axis: Batch Number; Y-axis: PRANGE STRIP (mmHg)
Figure 13. Chemical E MACMAN PEND STRIP Control Chart

X-axis: Batch Number; Y-axis: PEND STRIP (mmHg)
control charts. PCA and PLS both give similar conclusions to the MACMAN analysis described above. The additional information provided by the product quality variable, i.e. kilowatt reading, result in a slightly better PLS model over the PCA model.

**Principal Components Analysis.** Table 5 shows the percent variance explained by the first four latent variables, also referred to as principal components. We performed PCA with two principal components, which was able to account for about 50% of the variance in the process data.

<table>
<thead>
<tr>
<th>PC#</th>
<th>% Variation</th>
<th>% Total Variation</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>30.6336</td>
<td>30.6336</td>
</tr>
<tr>
<td>2</td>
<td>18.5070</td>
<td>49.1406</td>
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<tr>
<td>3</td>
<td>13.5057</td>
<td>62.6463</td>
</tr>
<tr>
<td>4</td>
<td>9.7783</td>
<td>72.4246</td>
</tr>
</tbody>
</table>

Table 6 shows the loadings of the original process variables with respect to the two latent variables. For principal component 1, the key contributors are PMAX DIST, TMAX DIST, PEND DIST, and TEND DIST. PCA accounts for the fact that these four univariate diagnostics are highly correlated. In fact, PMAX DIST and PEND DIST, and TMAX DIST and TEND DIST, are equivalent tests because the maximum pressure and temperature usually occurs at the end of the DIST step. PEND DIST and TEND DIST are slightly better diagnostics because they have higher loadings than PMAX DIST and TMAX DIST.

TMAX STRIP and PSLOPE STRIP also have relatively high loadings. TMAX STRIP is correlated to TEND DIST because the maximum temperature in the STRIP step is usually at the beginning. PSLOPF STRIP is a calculation of the change in pressure during the
Table 6. Chemical E PCA Loadings

<table>
<thead>
<tr>
<th>#</th>
<th>Variable Name</th>
<th>PC1</th>
<th>PC2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pmax Dist</td>
<td>0.3387</td>
<td>-0.1125</td>
</tr>
<tr>
<td>2</td>
<td>Tmax Dist</td>
<td>0.3348</td>
<td>-0.2037</td>
</tr>
<tr>
<td>3</td>
<td>Rate Dist</td>
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<td>-0.0864</td>
</tr>
<tr>
<td>4</td>
<td>Pmax Strip</td>
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<td>0.1060</td>
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<td>5</td>
<td>Pslope Strip</td>
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<td>Prange Strip</td>
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<td>Rate Strip</td>
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<td>9</td>
<td>Mrange Strip</td>
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<td>10</td>
<td>Mass Delta</td>
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<td>11</td>
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<td>0.1450</td>
</tr>
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<td>12</td>
<td>Mloss Dist</td>
<td>-0.0506</td>
<td>-0.1549</td>
</tr>
<tr>
<td>13</td>
<td>Mloss Strip</td>
<td>0.0283</td>
<td>0.3689</td>
</tr>
<tr>
<td>14</td>
<td>Vf Delta</td>
<td>-0.0046</td>
<td>-0.1021</td>
</tr>
<tr>
<td>15</td>
<td>Pend Dist</td>
<td>0.3617</td>
<td>0.0498</td>
</tr>
<tr>
<td>16</td>
<td>Tend Dist</td>
<td>0.3406</td>
<td>-0.1861</td>
</tr>
<tr>
<td>17</td>
<td>Pend Strip</td>
<td>-0.0784</td>
<td>-0.0091</td>
</tr>
<tr>
<td>18</td>
<td>Tend Strip</td>
<td>0.0814</td>
<td>-0.2460</td>
</tr>
<tr>
<td>19</td>
<td>Rate Abatic</td>
<td>-0.0267</td>
<td>0.1663</td>
</tr>
<tr>
<td>20</td>
<td>Pmin Dist</td>
<td>0.0959</td>
<td>0.1228</td>
</tr>
<tr>
<td>21</td>
<td>Tmin Dist</td>
<td>0.0130</td>
<td>-0.0336</td>
</tr>
<tr>
<td>22</td>
<td>Pmin Strip</td>
<td>0.0762</td>
<td>-0.2963</td>
</tr>
<tr>
<td>23</td>
<td>Tmin Strip</td>
<td>0.0868</td>
<td>-0.4332</td>
</tr>
</tbody>
</table>

The first ten readings of the STRIP step. It is equivalent to the PRANGE STRIP diagnostic which gives the pressure drop over the first ten readings.

The important process variables factored into principal component 1 is PEND DIST, TEND DIST, and PSLOPE STRIP, which are equivalent to the PMAX DIST, TMAX DIST, and PSLOPE STRIP diagnostics identified earlier in the MACMAN analysis. The loadings for principal component 2 do not provide additional information to the analysis.

The control charts for the two principal components are shown in Figures 14 and 15.

Bad batches 3, 5, and 14, and marginal batch 6, which show similar process
characteristics, are highlighted as out of control in principal component 1. The control chart for principal component 2 does not provide additional insight.

Figure 14. Chemical E PCA Control Chart for Principal Component 1

Figure 15. Chemical E PCA Control Chart for Principal Component 2
Figure 16 shows batches 3, 5, 6, and 14 with high values on the $T^2$ statistic control chart.

**Figure 16. Chemical E PCA $T^2$ Control Chart**

Value of $T^2$ with 95 Percent Limit Based on 2 PC Model

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>Value of $T^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>14</td>
</tr>
<tr>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>8</td>
<td>11</td>
</tr>
<tr>
<td>9</td>
<td>15</td>
</tr>
<tr>
<td>10</td>
<td>17</td>
</tr>
<tr>
<td>12</td>
<td>20</td>
</tr>
<tr>
<td>13</td>
<td>22</td>
</tr>
<tr>
<td>15</td>
<td>24</td>
</tr>
<tr>
<td>16</td>
<td>27</td>
</tr>
<tr>
<td>18</td>
<td>29</td>
</tr>
<tr>
<td>19</td>
<td>30</td>
</tr>
<tr>
<td>20</td>
<td>32</td>
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<td>33</td>
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<td>22</td>
<td>34</td>
</tr>
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<td>23</td>
<td>36</td>
</tr>
<tr>
<td>24</td>
<td>37</td>
</tr>
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<td>25</td>
<td>30</td>
</tr>
<tr>
<td>26</td>
<td>32</td>
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<td>27</td>
<td>33</td>
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<tr>
<td>28</td>
<td>34</td>
</tr>
<tr>
<td>29</td>
<td>36</td>
</tr>
<tr>
<td>30</td>
<td>37</td>
</tr>
</tbody>
</table>
The score plot for principal components 1 and 2 is shown in Figure 17. We notice a cluster of outliers for batches 3, 5, 6, and 14.

Figure 17. Chemical E PCA Score Plot for Principal Components 1 and 2

Figures 18 a-d present the contribution to the scores to latent variable 1 for batches 3, 5, 6, and 14, respectively. As expected, bad batches 3, 5, and 14, and marginal batch 6, show similar patterns with high contributions by PEND DIST, TEND DIST, PSLOPE STRIP, PRANGE STRIP, PMAX DIST, and TMAX DIST. Batch 23 does not exhibit this pattern, confirming the MACMAN analysis that the melt solidification was due to another phenomenon.

The PCA analysis give comparable results to the MACMAN analysis. With the first principal component, the PCA model is able to identify bad batches 3, 5, and 14, and marginal batch 6. However, even with two principal components, the model is not able to detect bad batch 23. We suspect that batch 23 is not a statistically significant data point to be captured by the PCA model.
Figure 18. Chemical E PCA Contribution to Scores
(a) Batch 3 in Principal Component 1, (b) Batch 5 in Principal Component 1,
(c) Batch 6 in Principal Component 1, (d) Batch 14 in Principal Component 1

(a) Contribution of batch 3 in dimension 1

(b) Contribution of batch 5 in dimension 1
Figure 18. (continued)

(c) Contribution of batch 6 in dimension 1

(d) Contribution of batch 14 in dimension 1
Partial Least Squares Analysis. The PLS model incorporates data for both the process variables (X-block), and the product quality variable (Y-block). The KW END reading is used as the product quality variable in the sense that high kilowatts are representative of bad batches.

Table 7 shows the cumulative predicted error sum of squares for the first four latent variables. The cross-validation program found the minimum cumulative predicted error sum of squares with one latent variable. However, we used two latent variables in order to capture batch 23 in the PLS model.

<table>
<thead>
<tr>
<th>LV#</th>
<th>Cum PRESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>32.9241</td>
</tr>
<tr>
<td>2</td>
<td>39.2662</td>
</tr>
<tr>
<td>3</td>
<td>39.4315</td>
</tr>
<tr>
<td>4</td>
<td>37.6377</td>
</tr>
</tbody>
</table>

Minimum Cumulative Predicted Error Sum of Squares is at 1 LV.

Table 8 shows the percent variance captured by each of the two latent variables to explain the X-block and Y-block variables.

<table>
<thead>
<tr>
<th>LV#</th>
<th>X-Block This LV</th>
<th>X-Block Total</th>
<th>Y-Block This LV</th>
<th>Y-Block Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>10.9098</td>
<td>39.4875</td>
<td>18.2023</td>
<td>44.4858</td>
</tr>
</tbody>
</table>

Table 9 presents the loadings for the two latent variables with PEND DIST, TEND DIST, PSLOPE STRIP, and PRANGE STRIP as the key contributors to latent variable 1. PMIN STRIP is the key contributor to latent variable 2. Because the minimum pressure
typically occurs at the end of the STRIP step, PMIN STRIP is equivalent to the PEND STRIP diagnostic highlighted in the MACMAN analysis.

Table 9. Chemical E PLS Loadings

<table>
<thead>
<tr>
<th>#</th>
<th>Variable Name</th>
<th>LV1</th>
<th>LV2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pmax Dist</td>
<td>0.2968</td>
<td>-0.2976</td>
</tr>
<tr>
<td>2</td>
<td>Tmax Dist</td>
<td>0.2825</td>
<td>-0.2987</td>
</tr>
<tr>
<td>3</td>
<td>Rate Dist</td>
<td>-0.3009</td>
<td>0.0725</td>
</tr>
<tr>
<td>4</td>
<td>Pmax Strip</td>
<td>0.1991</td>
<td>-0.0760</td>
</tr>
<tr>
<td>5</td>
<td>Pslope Strip</td>
<td>-0.3514</td>
<td>-0.0098</td>
</tr>
<tr>
<td>6</td>
<td>Prange Strip</td>
<td>0.3374</td>
<td>0.0299</td>
</tr>
<tr>
<td>7</td>
<td>Tmax Strip</td>
<td>0.2784</td>
<td>-0.2362</td>
</tr>
<tr>
<td>8</td>
<td>Rate Strip</td>
<td>0.1693</td>
<td>-0.2386</td>
</tr>
<tr>
<td>9</td>
<td>Mrange Strip</td>
<td>0.3007</td>
<td>0.1073</td>
</tr>
<tr>
<td>10</td>
<td>Mass Delta</td>
<td>-0.0154</td>
<td>0.1015</td>
</tr>
<tr>
<td>11</td>
<td>Mloss Abatic</td>
<td>-0.0572</td>
<td>-0.1267</td>
</tr>
<tr>
<td>12</td>
<td>Mloss Dist</td>
<td>-0.0817</td>
<td>-0.0507</td>
</tr>
<tr>
<td>13</td>
<td>Mloss Strip</td>
<td>0.1141</td>
<td>0.2561</td>
</tr>
<tr>
<td>14</td>
<td>Vf Delta</td>
<td>-0.0388</td>
<td>-0.1099</td>
</tr>
<tr>
<td>15</td>
<td>Pend Dist</td>
<td>0.3504</td>
<td>-0.2332</td>
</tr>
<tr>
<td>16</td>
<td>Tend Dist</td>
<td>0.2922</td>
<td>-0.2892</td>
</tr>
<tr>
<td>17</td>
<td>Pend Strip</td>
<td>-0.0918</td>
<td>0.0049</td>
</tr>
<tr>
<td>18</td>
<td>Tend Strip</td>
<td>0.0519</td>
<td>-0.0270</td>
</tr>
<tr>
<td>19</td>
<td>Rate Abatic</td>
<td>-0.0462</td>
<td>-0.1636</td>
</tr>
<tr>
<td>20</td>
<td>Pmin Dist</td>
<td>0.1326</td>
<td>0.0984</td>
</tr>
<tr>
<td>21</td>
<td>Tmin Dist</td>
<td>-0.0573</td>
<td>-0.3901</td>
</tr>
<tr>
<td>22</td>
<td>Pmin Strip</td>
<td>-0.0297</td>
<td>-0.4664</td>
</tr>
<tr>
<td>23</td>
<td>Tmin Strip</td>
<td>0.0131</td>
<td>-0.1854</td>
</tr>
<tr>
<td></td>
<td>Regression Coeff</td>
<td>0.2000</td>
<td>0.2693</td>
</tr>
</tbody>
</table>

Figure 19, the X-score control chart for latent variable 1, highlights bad batches 3, 5, and 14, and marginal batch 6. Figure 20, the chart for latent variable 2, highlights bad batch 23.
Figure 19. Chemical E PLS X-Score Control Chart for Latent Variable 1

Plot of the X-scores of Dimension 1

Figure 20. Chemical E PLS X-Score Control Chart for Latent Variable 2

Plot of the X-scores of Dimension 2
Figure 21 presents the score plot of the X-scores and Y-scores for latent variable 1. Bad batches 3, 5, and 14, and marginal batch 6, are shown as outliers in the X-score. PLS is able to pick-out the four batches although not all of them showed bad product quality, i.e. high KW END in the Y-score. The first latent variable is also able to model the high Y-score for bad batch 23.

Figure 21. Chemical E PLS Score Plot for Latent Variable 1

The contribution to the scores in latent variable 1 for batches 3, 5, 6, and 14, shown in Figures 22 a-d, respectively, follows patterns similar to those identified from PCA and MACMAN analysis.
Figure 22. Chemical E PLS Contribution to Scores
(a) Batch 3 in Latent Variable 1, (b) Batch 5 in Latent Variable 1,
(c) Batch 6 in Latent Variable 1, (d) Batch 14 in Latent Variable 1

(a) Contribution of batch 3 in dimension 1

(b) Contribution of batch 5 in dimension 1
Figure 22. (continued)

(c) Contribution of batch 6 in dimension 1

(d) Contribution of batch 14 in dimension 1

98
In Figure 23, batch 23 shows a strong contribution from PMIN STRIP. The PLS analysis confirms the MACMAN findings which support the occurrence of a different phenomenon for the melt solidification of batch 23.

Figure 23. Chemical E PLS Contribution to Scores - Batch 23 in Latent Variable 1
The score plot for latent variable 2 in Figure 24 clearly reveals batch 23 as an outlier.

Figure 24. Chemical E PLS Score Plot for Latent Variable 2
In Figure 25, the contribution to the scores in latent variable 2 for batch 23 again highlights the PMIN STRIP diagnostic.

Figure 25. Chemical E PLS Contribution to Scores - Batch 23 in Latent Variable 2

Using two latent variables, the PLS model is able to identify all four bad batches 3, 5, 14, and 23, and marginal batch 6. We feel that the specification of the PLS algorithm to pick latent variables predictive of the product quality variable, i.e. KW END, results in a better predictive model than PCA. The PLS model gives comparable results to the MACMAN analysis, but in a more efficient manner. The PLS Toolkit automates and quantifies the manual analysis required for MACMAN.
**Step 5: Capture Learning in Decision Trees.** Based on the process analysis described above, the results of the case study is summarized in the final induced classification decision tree presented in Figure 26. The branches of the decision tree represent unique sets of process characteristics which result in a class 1, 2, 3, or good, marginal, and bad batch (Figure 26 inset). The nodes are represented by a critical process variable and its associated threshold value. The branch proceeds along either an upper or lower branch depending upon whether the process parameter is greater or less than the threshold value. By iteratively splitting the data set at the nodes, an entire branch can be traced until only one class is represented in each terminal node.

For chemical E, the total data set of 37 runs is represented by 32 good batches, 1 marginal batch, and 4 bad batches. Only four key process parameters are needed to completely classify the 37 batches. Two branches of the decision tree characterize bad batches, one branch for the marginal batch, and two branches for good batches. Three bad batches are characterized by high PMAX DIST, high TMAX DIST, and high PRANGE STRIP. The fourth bad batch is represented by low PEND STRIP. Thirty of the good batches show no signs of abnormal process conditions. Two good batches and the one marginal batch show some but not all the characteristics of a bad batch.

**Summary of Results.** The purpose of this case study is to demonstrate the application of the proposed procedure for process analysis. A summary of results for the chemical E distillation study is listed below:

- Identified the process conditions leading to melt solidification based on 37 batches of historical data.

- Demonstrated the usefulness of KDAT and MACMAN as data analysis tools.
PMAX STRIP: Maximum pressure during STRIP.
TMAX STRIP: Maximum temperature during STRIP.
PEND STRIP: Pressure at the end of STRIP.
PRANGE STRIP: Pressure drop in the first 2 minutes of STRIP.
> Threshold value is the lower limit of upper branch.
< Threshold value is the upper limit of the lower branch.

C1: Good batch
C2: Marginal batch
C3: Bad batch
• Developed univariate diagnostic tests to statistically detect out-of-control conditions.

• Applied Principal Components Analysis and Partial Least Squares multivariate techniques.

• Applied decision tree analysis to classify status of batches based on operating parameters. The decision tree is able to discriminate between good and bad batches based on four key process parameters.

Implementation Plan. Based on the process analysis, several modifications to the control strategy were implemented to prevent future melt solidifications.

• Revised the Temperature Control Logic in the DIST Function. During the DIST step, modify the control logic to keep the process steady with less fluctuations in the vapor flow. Keeping vapor flow in control will prevent distillations from occurring at higher pressures and temperatures.

• Added Checks Before Proceeding to the STRIP Function. At the end of the DIST step, check temperature and pressure before proceeding to STRIP. If the temperature or pressure is outside an acceptable window, delay the sequence until the process variables fall inside the specified range.

• Implemented a Two-Part STRIP Sequence. Divide the STRIP operation into two steps - STRIP1 which removes half of the remaining solvent A after DIST, and STRIP2 which removes the rest of the solvent A, but at a slower rate. The endpoint of STRIP1 can be defined by a reactor weight specification, but the endpoint of STRIP2 will be based on the kilowatt readout. In the STRIP2 operation, distill solvent A at a lower temperature to get a slower rate of removal.
• Plans to Add a New Control Function to Automate Endpoint Detection.

Decrease the scan time interval during STRIP2. Automate monitoring of the kilowatt readout, with a lower limit on reactor weight, and upper limits on temperature and pressure.

Figures 27 and 28 show KDAT pressure and temperature profiles for three new batches after modifications to the control strategy. Pressure is steady at its setpoint; temperature is under control with maximum values well within the operating window; endpoint readings are typically below 8 kilowatts. These changes have resulted in improved process control, and a more repeatable process. Since implementation of the process control modifications, we have made over 25 batches with no incidence of melt solidification.

Quantification of Benefits. The estimated savings and costs of the chemical E distillation study are quantified below.

• Melt solidification increases cycle time on all linked vessels of the process. Reprocessing of the batches added a few hours to the cycle time, and affected equipment and labor. The annual savings without any bad batches is estimated at $70,000.

• Reactor set-ups can cause agitator blades to bend backwards at 90 degrees requiring cleaning for vessel entry, maintenance to reshape the blades, and reinstatement of the equipment. This typically occurred a limited number of times a year increasing cycle time on all vessels along the process chain. The annual savings is estimated at $30,000. The total annual savings sums to approximately $100,000.
Figure 27. Chemical E KDAT Pressure Profiles - Modified

X-axis: Time (min); Y-axis: Pressure (mmHg)
Figure 28. Chemical E KDAT Temperature Profiles - Modified

X-axis: Time (min); Y-axis: Temperature (°C)
• The chemical E case study took a few man-weeks to complete including communications, data analysis, and implementation. The total cost of the study is estimated at $20,000. Clearly, there is a significant quantifiable return, in addition to increased process understanding and reduced process variability.
3.3. Organizational Factors Survey

An organizational factors survey was conducted to identify the key factors impacting variability reduction efforts. The survey was used to evaluate organizational issues of top management support, operator empowerment, team learning, organizational infrastructure, incentives, and training. The goal was to recommend realistic organizational changes to cultivate an environment for continuous improvement.

The key results presented in this section address the following questions:

- What are the organizational factors impacting variability reduction efforts?
- What are the current perceptions of existing barriers?
- Where should improvement efforts be targeted?

3.3.1. Categorization of the Organizational Barriers

The survey asked respondents to evaluate a set of 33 posited barriers based on their actual experience in working on variability reduction. A copy of the survey is shown in Figure A-1. The SAS VARCLUS hierarchical clustering procedure\(^{31}\) was applied to identify the underlying structure of the survey data. Eight clusters were formulated to categorize the list of 33 barriers.

- *Top Management Support for Variability Reduction*. Top management support is an essential element of successful quality initiatives. Without a long-term commitment by the division manager and the department managers, variability reduction will be viewed as just another management perogative that comes and goes. Also, top management must communicate its support not only in words, but in actions such as the funding of variability reduction projects and approval of process improvement changes.

---

• Variability Reduction as a Production Priority in Practice. Although variability reduction is a top divisional goal, barriers can keep it from being a priority in practice. Evidence of variability reduction as a production priority includes whether it:
  - is a part of day-to-day activities,
  - is viewed as a job responsibility,
  - is supported by a person's immediate supervisor,
  - is incorporated in departmental or individual performance evaluations, or
  - involves the operators closest to the process

• Belief in the Benefits of Variability Reduction. In order for variability reduction to be an integral part of SCD's culture and production philosophy, people need to be convinced that it works. However, the current status shows the lack of a team infrastructure, lack of guidelines for data collection, and lack of data analysis tools. These are valid barriers that need to be addressed with a systematic approach to variability reduction. However, excuses that variability reduction does not benefit the bottom-line, is not applicable to infrequent runners, and is not worthwhile until processes are in control are not valid. Training, sharing success stories, and learning-by-doing can lead people to see the benefits of variability reduction.

• Variability Reduction Requires Time and Effort. Production is measured by how well it meets cost, delivery, quality, and safety specifications. Efforts to reduce process variability can be seen to compete with the overriding priority to get product out the door, at least in the short-term. However, time and resources must be allocated to balance between immediate needs and potential benefits.
Also, additional time and effort may be expended initially to move down the learning curve for new data acquisition systems and analysis tools. Management needs to recognize that variability reduction does require time, effort, and manpower, but that it is resources well spent.

• *Training for Variability Reduction*. The lack of training hinders the participation of the workforce. Training is needed in quality concepts, team problem-solving, computer skills, and process analysis. In addition, a systematic methodology for variability reduction should be documented into a set of practical guidelines.

• *Information System for Variability Reduction Efforts*. The effectiveness of variability reduction efforts is greatly enhanced by a computer system that collects, stores, integrates, and analyzes process data. An information system acts as a communication tool, database, and data analysis package. Ease of use and ease to access are the key design criteria.

• *Analytical and Development Support for Variability Reduction*. Variability reduction should be an integrated effort between production and the support functions. The analytical group is responsible for analyzing in-process and final product samples, and ensuring the repeatability of analytical tests. The development group is responsible for the technology transfer of a new process into production, and performs on-going process improvement studies in the laboratory. Their involvement helps to identify and eliminate suspected sources of process variability.

• *Equipment and Materials Support for Variability Reduction*. Engineering has an important role in the design and maintenance of plant equipment. The use of
preventive versus reactive maintenance cuts down the number of process
shutdowns due to unexpected equipment failure. Materials support is responsible
for the quality of raw materials from internal and external suppliers, and the
delivery of product to internal and external customers.
3.3.2. Major Findings

The overall survey response rate was 62% based on the actual number of SCD personnel. In general, the survey results reveal that SCD has made encouraging progress from current variability reduction efforts. In revealing current barriers, the survey provides direction for areas of improvement. The major findings from the survey analysis are described below. Details of the analysis are included in Appendix 2.

- **Perceptions Differed by Job Position.** Survey respondents were asked to identify their job position and work center. Analysis of the survey revealed that the best breakdown of the data was by job position. In general, supervisors indicated the existence of fewer organizational barriers than respondents from any of the other job position. Differences between work centers were nominal.

- **Knowledge and Work in Variability Reduction Lag Behind Rating of Importance.** Across all job categories, most people agreed that variability reduction was important to SCD's business. However, the pattern in decreasing order of knowledge and work in variability reduction followed a linear trend (Figure 29) according to job position. Supervisors have the most knowledge and involvement in variability reduction, then process chemists, then group leaders, and then operators (excluding staff which was hard to classify because it included a mixture of job responsibilities). Most people do not know about or work on variability reduction as much as their rating of its importance would seem to indicate.

- **Need for a Better Information System.** Process chemists and supervisors rated the lack of an adequate information system as a major barrier to variability reduction. They recognized the need for data acquisition systems to collect process data, and for a computerized system to store and integrate different data
Figure 29. Survey Data:
Knowledge about, Importance of, and Current Work in Variability Reduction

Sort by Job Position

How much do you know about variability reduction?
How important do you think variability reduction is to SynChem's business?
How much do you currently work on variability reduction in your job?
sources. Operators and group leaders expressed little opinion on this issue. Two possible explanations for this are either they felt that information technology was not a key factor, or they were not yet aware of the usefulness of such a system.

• *Too Many Other Competing Priorities.* The existence of too many other competing priorities was a frustration faced by respondents in all job positions, except supervisors. Process chemists, group leaders, operators, and staff felt that time and resource constraints adversely affected variability reduction efforts. Variability reduction was seen as another responsibility in addition to cost, quality, delivery, and safety performance goals.

• *Lack of Team Infrastructure.* Process chemists, group leaders, and operators recognized the lack of teams organized to work on variability reduction. Although PMTs existed, they were not formally charged with the responsibility. Special project teams were set up to work on targeted processes. On the whole, however, most production staff were not part of an organized variability reduction effort.

• *Lack of Operator Involvement.* Process chemists, group leaders, operators, and staff noted the lack of operator involvement in variability reduction. Operator participation had not been fully mobilized, although efforts were underway to increase awareness and provide training. The current operating philosophy did not strongly encourage operator involvement in problem-solving tasks.
3.3.3. Interpretation and Recommendations

Based on the survey results, my interpretation and recommendations are summarized below:

• *Internalize the Vision.* Management needs to continue providing direction to all levels of the organization. From the survey results and discussions with production staff, I noticed some hesitancy as to the acceptance of variability reduction as a real fundamental change in SCD’s mode of operation. While the good news is that variability reduction has a high level of awareness, the bad news is the lack of implementation efforts to make variability reduction a part of the day-to-day activities of production staff. The relative importance of stated management priorities can be clarified. Variability reduction as a strategic business objective requires the commitment of human and capital resources. In the short-term, variability reduction efforts may adversely affect bottom-line business results. Besides strictly financial criteria, metrics to measure the process of organizational change will also be useful. Accountability is needed to measure performance beyond the traditional cost, quality, delivery, and safety specifications.

• *Drive the Implementation from the Bottom-Up.* An important weakness of the current efforts is the lack of production-driven initiatives. A key measure of success is the participation level of production staff who are closest to the process. Process chemists, group leaders, and operators need to be trained, motivated, and equipped for their vital roles. Operators are especially valuable with their wealth of practical knowledge of the plant processes and equipment. The effect of driving implementation from the bottom is to create a grassroots change in the organizational culture for continuous improvement.
• Support the Vision with the Appropriate Organizational Infrastructure. Teams should be set up to support the implementation of variability reduction efforts, and to mobilize production staff participation. A team infrastructure organized by work center provides a vehicle for alignment of divisional goals with specific team projects. Such an infrastructure is also effective for sharing experiences across teams and within the division. Led by the process chemist and group leader, team meetings can provide conducive environments for group problem-solving and communication. To implement this recommendation, I propose that the existing role of PMTs be expanded to include all production staff, and become the focal point for variability reduction efforts. In this way, SCD can build upon an existing organizational infrastructure, rather than develop a new one.

• Invest in Information Technology. Information systems for collecting, storing, integrating, and analyzing process and production data are important productivity improvement tools. Investment in information technology today will result in significant long-term benefits in the future. The costs associated include financing a computer system and new equipment, and training of production staff in the use of the computers and software tools. The benefits are better operating decisions and improved processes based on accurate, reliable operating data. However, there will be a learning curve effect in the early stages of investing in such new engineering tools. Building upon successive pilot studies and leveraging support from functional groups will help to ease the transition.

• Leverage the Support of Functional Groups. Coordinated efforts with the functional groups can go far in addressing the need for additional resources. Technical support staff can provide the expertise, training, and tools to support the efforts of the production teams. As needed, they should become an integral
member of the teams. For example, a development chemist may be part of a team evaluating alternative solvents for a reaction step. Or engineers from the engineering and research functions may be involved in selecting a data acquisition system for a pilot study. The support groups play a key role in assisting production staff to pull-in new technology for process improvement efforts.

• *Measure Progress in Follow-up Surveys.* SCD is in the early stages of implementing the corporate KP4 quality initiative. The survey provided valuable feedback relative to perceptions of barriers to the variability reduction efforts within the division. Another major objective was to provide direction for improvement. Follow-up surveys would provide valuable insights in measuring the division's progress in overcoming the identified organizational barriers.
4. Methodology for Variability Reduction

The methodology for variability reduction synthesizes the learning from the case studies and field experience into a systematic approach to guide future process improvement efforts.

The key results presented in this section address the following questions:

- What are the general principles for implementation of variability reduction efforts?
- What are the specific steps in the proposed methodology?
- What are the appropriate tools to apply for each action step?

An illustrative example is presented in section 4.2 to demonstrate the application of the step-by-step methodology for variability reduction.

4.1. Process Improvement by PDCA

Guiding Principles. The proposed methodology for variability reduction is based on the Plan-Do-Check-Act process improvement cycle. Kodak’s KP^4 initiative emphasizes the use of the PDCA model as part of QLP. The general principles to be applied in any implementation plan include:

- Teamwork. A key ingredient for success is the involvement and coordination of the right people in the PMT. Team effectiveness is a function of the leadership, perspectives, and resources applied by its members. Management support is important to align team objectives with broader divisional goals, and to provide the necessary manpower and capital resources. Production staff are responsible to drive the process, while functional staff plays an integral support role. Operator involvement is also critical in leveraging operational knowledge of the production process. Together, the team provides results that exceed the additive contributions of its individual members.
• *Communication.* Variability reduction efforts promote a process of information sharing which builds organizational learning. As team members gather information, solve problems, standardize procedures, implement improvements, and document changes, they communicate experiences, observations, and ideas. As a result, the entire team benefits from the group problem-solving process. Increased communication increases a sense of teamwork, and raises the level of commitment and involvement of each individual.

• *Data-Based Analysis.* While much of the implementation issues for variability reduction efforts address changes in the organizational culture, group problem-solving should be based on analytical approaches. In the methodology described below, a range of analytical tools, ranging from simple control charts to multivariate analysis techniques are suggested for appropriate action steps. The focus is on the use of a data-driven operating strategy for variability reduction.

• *Continuous Improvement.* At all levels of the organization, a philosophy of continuous improvement should be enforced. The goal is not only to achieve a defined set of objectives, but to foster a culture of quality and excellence throughout the organization. The challenge is to effectively empower each individual to be an integral part of the continuous improvement process.

*Action Steps and Tools.* The methodology of the PDCA process cycle is further defined into specific action steps below. The appropriate tools to apply for each step are also discussed. The methodology is outlined in Figure 30.
Figure 30. Methodology for Variability Reduction

**Plan**

Identify a Process Improvement Opportunity

Form a Cross-Functional Process Management Team

Develop a Process Flowchart

Identify Key Process Parameters

Collect Data

**Do**

Analyze Data

Implement Process Changes

Is Process in Control?

- Yes
  - B
- No
  - Identify Sources of Variability

**Check**

**Tools**

Prioritize chemicals using Cost of Quality model.

Communicate common understanding.

Data Collection Matrix. EVOP/DOX.

Data Collection Methods Guide. Data acquisition systems; batch sheets; PMTs.

KDAT; univariate diagnostics; MACMAN; decision trees; PCA/PLS; OCMIS.

Control charts of univariate diagnostics. Cause-effect diagram.
Figure 30. (continued)

TOOLS

Update process write-ups & SOPs.
Batch process automation.

Incorporate into process write-ups.
Batch process control.
Knowledge-based expert systems.

Exception reports.
Alarm limit management in control systems.
**PLAN**

*Step 1: Identify a Process Improvement Opportunity*

The first step of the methodology is to identify the problem to be addressed. Because of limited resources, the idea is to focus on improvement opportunities with the highest expected returns. To make the selection, a useful tool for prioritization is the cost of quality\(^{32}\) model. Total cost of quality is composed of three components: prevention costs, appraisal costs, and failure costs. Table 10 outlines the calculation for total cost of quality and cost of quality per kilogram which takes into account all other measures of poor quality such as yield loss, amount of rework, inventory, and extra analytical tests.

<table>
<thead>
<tr>
<th>Table 10. Calculation of Cost of Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Total Cost of Quality:</strong></td>
</tr>
<tr>
<td>Total $COQ = $Prevention + $Appraisal + $Failure</td>
</tr>
<tr>
<td>$Emergency   $Analytical   $Rework</td>
</tr>
<tr>
<td>$Cleaning     $Testing      $Development</td>
</tr>
<tr>
<td>$Sample       $Yield Loss   $Inventory</td>
</tr>
<tr>
<td>$Blend Loss</td>
</tr>
</tbody>
</table>

**Cost of Quality per kg:**

\[
\frac{\text{$COQ}}{\text{kg}} = \frac{\text{Total $COQ - $Emergency}}{\text{Total kg}}
\]

The largest contributors are yield loss and inventory holding costs which fall under failure costs. A Pareto analysis based on the cost of quality model will help identify the top chemicals for focusing variability reduction efforts. The total cost of quality is useful for evaluating heavy runners, while cost of quality per

\(^{32}\) Koetje, *op. cit.* pp.44-58.
kilogram is useful for evaluating infrequent runners. The cost of quality analysis is most appropriate at the divisional or departmental level to provide an overview of likely improvement projects.

Step 2: Form a Cross-Functional Process Management Team

The next step is the assignment of improvement projects to specific PMTs. This step is designed to align team objectives with broader divisional goals. PMTs in appropriate production areas are matched with the identified high priority chemicals. Each PMT, made up of operators, and jointly led by a process chemist and group leader, then solicits functional support from development, engineering, maintenance, research, or analytical services as required. The goal is to build a cross-functional team with the expertise and resources to tackle the quality problems for the targeted chemical.

Step 3: Develop a Process Flowchart

The last step of the Plan phase is the development of a process flowchart. The main purpose for this is to clarify current operating procedures and establish common understanding about the process. Such a discussion can then lead to hypotheses of the key causes of variability and a plan to collect the appropriate data.

DO

Step 4: Identify Key Process Parameters

To further define the problem, the key unit operations and process parameters should be identified. The data collection matrix discussed in section 3.1.1 is a useful tool to determine what data should be collected. Additionally, DOX and

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EVOP experiments can be commissioned to quantify the effects and interactions between several process parameters.

**Step 5: Collect Data**

Data collection is a prerequisite to further analysis. A sufficient data set should be collected to confirm or reject hypothesis of suspected causes of variability. The data will either lead to further questions to clarify process understanding or result in recommendations for process improvement. The data collection methods guide discussed in section 3.1.2 is a useful tool in highlighting the use of data acquisition systems, batch sheets, and PMTs.

**Step 6: Analyze Data**

A data-driven approach is recommended as a basis for process analysis. The three steps in the Do phase embodies the systematic procedure of process analysis discussed in section 3.2.1. The formulation and testing of hypotheses using process data result in improved process understanding. Analytical tools provide substantial benefits in data analysis. Section 3.2.2 discusses the use of univariate diagnostics, KDAT, MACMAN, PCA/PLS multivariate analysis techniques, and decision trees.

**CHECK**

**Step 7: Check If Process is In Control**

The Check phase incorporates a feedback loop to evaluate the data analysis performed. The status of a process can be checked using control charts of univariate diagnostics. Out-of-control conditions require further investigation and analysis into the sources of variability. The iterative process continues until process control is established.
Step 8: Identify Sources of Variability

Process variability comes from sources which can be categorized into five categories: materials, manpower, methods, machine, and measurement. A cause-effect diagram is a useful tool to represent the relationship between an effect and its possible causes. Identification of the causes can lead to suggestions for process improvement, or gathering of additional data.

Step 9: Implement Process Changes

The implementation of process changes comes as a result of improved process understanding. Suggestions from PMT members are reviewed for the best solution to the quality problem at hand. The implementation requires monitoring of the key process parameters to check for the effect of the process changes. After confirmation that the process is in control, the recommended changes can be approved as part of standard operating procedures.

ACT

Step 10: Standardize Process Changes

In order to sustain gains from process improvements, the changes need to be documented and standardized. The changes should be communicated to the production staff in process reviews. The process write-ups and standard operating procedures need to be updated. Modifications to the controller sequence may be required for automated batch processes.

Step 11: Document Control Strategies

A control strategy provides information on the symptoms, causes, and action steps to preempt conditions leading to a process upset. The learning gained from
the process analysis studies need to be preserved. This data can be incorporated into the process write-ups to avoid future occurrences of the same phenomenon. For operations running under batch process control, protocols can be set up to detect and take the appropriate action. As an advanced approach, the insights gained can be built into a knowledge-based expert system.

**Step 12: Identify Plan for Periodic Review**

A key goal of variability reduction efforts is the effective long-term application of the organizational learning. After establishing that the process is stable and in control, exception reports can be used to monitor for abnormal conditions. Unless problems are detected, the PMT can move on to other variability reduction projects. For batch control systems, this process can be automated using the alarm limit management feature.

**Step 13: Continuous Improvement**

Continuous improvement is an ongoing process. The learning and benefits of the current effort can lead to other improvement ideas, or be extended to other processes. The methodology described provides a structured approach to group problem-solving which makes effective use of available tools and resources.
4.2. An Illustrative Example with Chemical D

Background. The chemical D example provides a current application of the proposed methodology for variability reduction. Chemical D is made in a production bay with pneumatic control and no process automation capabilities. Current studies are underway to upgrade the equipment with data acquisition and process control. The chemical D reaction step is fairly well-understood from DOX experiments conducted during process development. The difficult steps of the process are in the work-up operations, such as the crystallization and isolation of the final product. Suspected sources of variability are in the equipment, manual work-up operations, and difficulty of the isolation procedure. Gains have been made with recent efforts to standardize procedures, but yield variability continue to be a concern.

Stepping Through the Methodology. The purpose of the chemical D example is to test the proposed methodology. The illustration is an incomplete case study because efforts are ongoing. The action steps applied are described below:

PLAN

Step 1: Identify a Process Improvement Opportunity

The cost of quality model was used to target chemical D as a process improvement opportunity. Figure 31 shows chemical D as eighth in a Pareto analysis of the top twenty chemicals ranked by total cost of quality. Chemical D was chosen because the other seven chemicals were either:

- already a focus of current variabiility reduction efforts,
- undergoing certification for process improvements, or
- targeted for major process development efforts (shown in prevention cost component)
The cost of quality data backed up general opinion that chemical D should be a high priority chemical.

**Step 2: Form a Cross-Functional Process Management Team**

Although a formal PMT was not set up, discussions were held with the group leader, process chemist, operators, development chemist, and engineering staff. The different perspectives provided information on suspected sources of variability, key unit operations, important process parameters, and suggestions for additional data.

**Step 3: Develop a Process Flowchart**
The causes of variability were further defined in a step-by-step review of each unit operation in the process write-up with the group leader. The production staff provided operational feedback on the manufacturing procedure. Equipment differences, raw material variability, differences in operator interpretation, and sensitivity of the work-up were discussed.

**DO**

*Step 4: Identify Key Process Parameters*

The data collection matrix, described in section 3.1.1, provided guidelines in identifying the key process parameters. For the various process steps, the variables monitored included the amount of reagent charged, vessel temperature, vessel vacuum, distillation time, and pH of solution.

*Step 5: Collect Data*

The data collection methods guide, described in section 3.1.2, provided insight into the use of operator batch sheets. The chemical D process was not equipped with a data acquisition system. Instead, BATMAN sheets, shown in Figure 32, were developed as a preliminary step to build a process database. The collected data would be used to begin process analysis, and justify the need for a data acquisition system. In addition, the BATMAN sheets were useful because several unit operations in the work-up steps were not amenable to automatic data collection. The manual collection of data also highlighted operator attention to important process steps.

*Current Status:* The BATMAN sheets will be used to collect data on the next campaign of runs. Some suggestions have been brought forth as to suspected sources of variability. Data analysis will be performed to quantify and identify
Figure 32. Chemical D BATch Management Sheet - Select Sections

Chemical D BATMAN Sheet
(BATch MANagement)

Chem # __________ Mfg Supply # __________ Run # __________ Date __________

Record and report to the group leader or process chemist any abnormality in the process and equipment.

<table>
<thead>
<tr>
<th>PD</th>
<th>Step</th>
<th>Process Variable</th>
<th>Data</th>
<th>Remarks</th>
<th>Oper.</th>
</tr>
</thead>
<tbody>
<tr>
<td>...</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>10</td>
<td>TLC reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>34</td>
<td>11</td>
<td>Start time of distillation</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>11</td>
<td>Start vessel temp of distillation</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>36</td>
<td>11</td>
<td>Start vapor temp of distillation</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>37</td>
<td>11</td>
<td>End time of distillation</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>38</td>
<td>11</td>
<td>End vessel temp of distillation</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>39</td>
<td>11</td>
<td>End vapor temp of distillation</td>
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<td>...</td>
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</tr>
<tr>
<td>43</td>
<td>17</td>
<td>Start time of reaction</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>44</td>
<td>17</td>
<td>Start vessel temp of reaction</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>45</td>
<td>17</td>
<td>End time of reaction</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>46</td>
<td>17</td>
<td>End vessel temp of reaction</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>...</td>
<td></td>
<td></td>
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</table>
the causes. The current efforts are just the first steps along the proposed methodology. However, the structured approach was useful in guiding the sequence of action steps needed.
5. Conclusions

The key results presented in this section address the following questions:

- What are the policy guidelines for variability reduction efforts?
- What is a one-year implementation plan which addresses the key barriers within SCD?
- What are the recommendations for future work?

5.1. Policy Guidelines

The general conclusions from this thesis work are summarized by the following policy recommendations for variability reduction:

Strategic Thinking

- Guide Implementation from a Sociotechnical Perspective

A basic principle stemming from this thesis work is: A sociotechnical perspective is essential in understanding real-world manufacturing issues. A recurring theme has been the necessity of an integrated perspective in addressing both the organizational and technical challenges of implementing organizational change.

- Follow a Data-Driven Operating Strategy

The central thesis of my research is: A data-driven operating strategy will lead to reduced process variability. The case studies provide evidence to support how data can be used to improve process understanding, and how better process understanding can drive efforts towards variability reduction. This research supports a management philosophy of decision-making based on relevant operating data.

Tactical Implementation
• Success = Right People + Right Systems + Right Technology

The success of a business enterprise is built upon the people-systems-technology triad. The lack of an integrated perspective to evaluate the changing roles of people, systems, and technology will hamper progress in variability reduction. For each area, issues to address include:

People: What must managers do to communicate the vision of the enterprise to all levels of the organization? What can be done to leverage the time and resources of the process chemists? How should the involvement of operators be expanded to include new responsibilities? How can support staff be involved to play an integral role? How should teams be set up?

Systems: What organizational infrastructure is needed to support production-driven teams? What training is required for team-building and group problem-solving? What changes are required in the performance appraisal system to motivate individual excellence and team efforts? What are the requirements for a computer system and the needs for an information system?

Technology: How should equipment be upgraded with process control and data acquisition capabilities? What data analysis tools are useful for process analysis? What training is required to get staff up-to-speed on the new tools? How should technology be introduced into the production environment?

• Addressing the Critical Issues

The research goals of this thesis were framed to answer the following key questions:

1) What data is needed for process analysis?
The data collection matrix, discussed in section 3.1.1, provides a systematic identification of what data should be collected for process analysis as characterized by unit operations.

2) What is the best way to collect the necessary data?

The data collection methods guide, discussed in section 3.1.2, provides selection criteria for the three data collection methods reviewed - data acquisition systems, operator batch sheets, and process management team meetings.

3) What techniques and tools are applicable for process analysis?

A systematic procedure for process analysis is discussed in section 3.2. A review of the available data analysis techniques and tools, and recommendations for their application are also included. The specific techniques and tools evaluated are KDAT, univariate diagnostics, MACMAN, PCA/PLS, decision trees, and DOX/EVOP.

4) What are the organizational factors impacting variability reduction efforts?

The results of an organizational factors survey, conducted to identify the key factors impacting variability reduction efforts, are presented in section 3.3. An analysis of the current barriers, and recommendations for realistic organizational change are also discussed.

5) What is a general methodology for implementation of variability reduction efforts?

A step-by-step methodology for variability reduction, based on the Plan-Do-Check-Act process improvement cycle, is discussed in section 4. The
methodology provides a systematic approach for problem-solving, along with suggestions for the appropriate tools to apply at each step.
5.2. Implementation Plan

Basis of Recommendations. A one-year action plan to address the key barriers to variability reduction within SCD is described. The foremost considerations in the formulation of the implementation plan are:

• *Communicating the Vision*. The vision must be clearly communicated to all levels of the organization. Management’s job is to set the direction, commit the resources, and provide the necessary support. Implementation is most effective from a grassroots level on the production floor.

• *Soliciting Total Involvement*. For effective implementation of the vision, total employee involvement is desirable. Changing roles and responsibilities need to be clarified to enable participation of the entire production staff. Variability reduction needs to become a part of each person’s day-to-day activities.

• *Equipping for Action*. Training and new tools are needed to fully equip the production staff to handle new responsibilities. Support from functional groups is also essential. The essence of the implementation philosophy is to show people where the organization is heading, show them their role in that plan, and equip them to be an effective contributor.

• *Building on Successive Case Studies*. The use of case studies is recommended to build a repertoire of successes. The benefits of variability reduction can be quantified to maximize acceptance and learning for expanded implementation. Case studies also leverage current resources and work within the existing organizational structure.
**Proposed Organizational Structure.** Based on the above considerations, a modified organizational structure for SCD is shown in Figure 33. The changes are directed in creating a more participative, team-oriented environment to carry out the implementation plan. The SCD business team is led by the division manager, and includes the department managers, health, safety, and environmental manager, and materials manager. They are the management group responsible for setting the vision, strategic goals, and key quality initiatives of the division. Examples of quality initiatives may include process variability reduction, lead time reduction, a quality information system, or a total employee involvement program.

Department managers have line responsibilities for the manufacturing operations. PMTs are organized within the departments to focus on specific chemical processes. Each PMT is composed of the process chemist, group leader, operators, and staff from the appropriate support groups. The matrix structure aligns the activities of each PMT with a key division-wide quality initiative.

A quality steering committee is responsible for coordinating, communicating, and supporting the implementation efforts of the individual PMTs. The committee is led by the division quality advisor, and includes representatives from the SCD business team, division technology team, and support groups. The steering committee may set divisional standards for hardware platforms for new engineering software tools, set up training sessions, or organize a division quality day for sharing team experiences. While each PMT reports along departmental lines, its progress in meeting division-wide quality objectives will also be tracked by the quality steering committee.
One-Year Action Plan. With the proposed organizational structure in place, the details of the implementation plan are described below. The one-year action plan follows the framework of the Plan-Do-Check-Act process model.

**PLAN: Establish a Process Control Mindset**

- Link variability reduction with concrete goals for every level of the organization.

**Recommendations:**

1) The vision for variability reduction should be communicated top-down through departments, and supported across the division in project teams.

2) Form a quality steering committee which meets monthly to set the direction, coordinate, and review the status of division-wide initiatives.

3) In 1992, for every work center, form PMTs on two chemicals targeted in division-wide projects.

**DO: Make Variability Reduction a Practical Reality**

- Empower PMTs and provide them with the support and tools to be the driving force for variability reduction.

**Recommendations:**

1) Give the process chemist and group leader shared responsibility to lead the PMTs.

2) Follow the proposed step-by-step methodology for variability reduction.

3) Use the division technical staff to do the supporting data analysis until an information system, with KDAT, is in place.

4) Provide a PC for each work center, with KDAT and MACMAN installed, for data analysis and archival.

5) Create a focal point to evaluate and pull-in new technology. This can be accomplished through a division technology team, more active involvement of
the department managers in technology development, or a new position of a
division technology development coordinator.

**CHECK: Establish Metrics to Measure Performance**

- Incorporate variability reduction efforts as part of the performance appraisal system.

*Recommendations:*

1) Report progress of PMTs through departments by measuring - % of batches of data collected, % PMT meetings in which data is reviewed, # of suggestions implemented, % yield improvement, # reworks, kg inventory, as well as cost, delivery, and quality.

2) Incorporate SPC understanding as a component for skill-based operator certification.

3) The division quality steering committee should set up financial as well as process-related metrics to measure progress on division-wide initiatives.

**ACT: Strive for Continuous Improvement**

- Standardize improvements and share insights in order to reset goals.

*Recommendations:*

1) Document process changes into write-ups and standard operating procedures.

2) Schedule a division quality day for PMTs to present variability reduction experiences within their own departments.

3) At the end of the year, set up a one-day conference with representatives from each PMT to refine the variability reduction methodology based on case experiences.

4) The division quality steering committee should set new goals for next year based on the SCD business plan.
5.3. Future Work

Recommendations for future work stemming from this thesis work are discussed below:

People

1) Follow-up surveys should be conducted to measure progress on reducing existing organizational barriers.

2) Evaluate the skill set needs of production staff in light of increased employee participation, and the introduction of new technology. Promote a selective hiring policy to screen for people with the right knowledge base and work ethic.

3) Further research is recommended for improved understanding of the implementation of organizational change. For example, SCD faces the challenge of managing the cultural transition of increased employee involvement, the introduction of new technology, and the requirements for staff with a different skill set.

4) In the long-term, SCD may want to expand the role of PMTs into product business teams. PBTs will be business units organized along product lines with many of the traditional supervisory responsibilities for business performance. This will eliminate layers of middle management, and increase employee accountability for bottom-line business results.

Systems

1) A reevaluation of the performance appraisal system is suggested. Skill-based operator certification should be considered for SPC knowledge, computer proficiency, and use of data analysis tools. Team objectives also need to be incorporated as a component of individual and PMT performance appraisal.
2) A cost-benefit analysis should be conducted on the requirements to upgrade the information system and computer hardware. Both current and future projected needs should be considered in establishing division-wide computing standards. The key criteria for an information system are accessibility and ease of use.

3) Exploratory studies should be conducted on the application of computer integrated manufacturing (CIM) for chemical batch manufacturing. The use of CIM to integrate all aspects of the manufacturing process, from shop floor process control to production planning and scheduling, has significant implications of a new way of doing business.

Technology

1) The methodology for variability reduction should be reviewed and refined after learning from additional case studies.

2) The development of the data analysis tools and techniques should be advanced to make them more user-friendly, and to test them for a greater variety of processes.

3) A cost-benefit analysis should be performed for installing automated batch process control. SCD should establish vendor relationships to evaluate state-of-the-art flexible batch control systems with capabilities for process automation, data acquisition, on-line data analysis, and data archival.
References


Appendix 1. Descriptions of the PCA/PLS Models

Principal Components Analysis. PCA works on a single data matrix and attempts to explain the structure of the variation. Generally the $X$ matrix is mean centered and scaled before PCA is applied. The methodology of PCA is to decompose the data matrix into the following bilinear form:

$$X = \sum_a^A t_ap_a^T + E_a$$

Mathematically $TP^T$ is indeterminant; to obtain uniqueness one can impose various conditions on $t_a$ and $p_a$. One way to do this is to force orthogonality among the $t_a$'s, and among the $p_a$'s. If this is done, and it is further assumed that they are orthogonal to the rows/columns of $E$, then it can be shown that the $t_a$'s are the eigenvectors of $XX^T$, and $p_a$'s are the eigenvalues of $X^TX$. The correct number of principal components can be determined from a number of stopping criterion, one of the more popular techniques being cross validation.

Partial Least Squares. PLS is a regression technique, regressing $Y$ onto $X$, where $X$ is an $n \times k$ matrix, and $Y$ is an $n \times m$ matrix. This method is especially useful when the variables within $X$ and within $Y$ are correlated. The $Y$ matrix can be decomposed in a similar manner as the $X$ matrix into:

$$Y = \sum_a^A u_aq_a^T + F_a$$

Conceptually, PLS is similar to PCA except that it simultaneously reduces the dimensions of the $X$ and $Y$ spaces to find the latent vectors for the $X$ and $Y$ which are most highly correlated. The method can be described by the following algorithm:

1. Start: set $u$ equal to a column of $Y$.
2. $w^T = u^TX / u^Tu$ (regress columns of $X$ on $u$).
3. Normalize $w$ to unit length.

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33 Kresta, op. cit., pp.35-47.
4. \( t = Xw / w^Tw \) (calculate the scores).
5. \( q^T = iTY / iTt \) (regress columns of \( Y \) on \( t \)).
6. Normalize \( q \) to unit length.
7. \( u = Yq / q^Tq \) (calculate new \( u \) vector).
8. Check convergence: if YES to 9, if NO to 2.
9. \( X \) loadings: \( p = X^Tt / iTt \).
10. Regression: \( b = u^Tt / iTt \).
11. Calculate residual matrices: \( E = X - tp^T \) and \( F = Y - btq^T \).
12. To calculate the next set of latent vectors, replace \( X \) and \( Y \) by \( E \) and \( F \), and repeat.

**Nomenclature.**

- \( A \) = optimal number of latent vectors
- \( b_a \) = regression coefficient for the \( a \)th latent vectors of \( X \) and \( Y \) measurements
- \( B \) = diagonal matrix of regression coefficients
- \( E \) = residual matrix of \( X \)
- \( F \) = residual matrix of \( Y \)
- \( k \) = number of process/operational/input measurements
- \( m \) = number of quality/output measurements
- \( n \) = number of observations
- \( p_a \) = loading vector for the \( a \)th latent vector of \( X \) calculated by PCA or PLS
- \( P \) = loading matrix for \( X \) calculated by PCA or PLS
- \( q_a \) = loading vector for the \( a \)th latent vector of \( Y \)
- \( Q \) = loading matrix for \( Y \)
- \( t_a \) = scores associated with the \( a \)th latent vector of \( X \)
- \( T \) = matrix of scores associated with \( X \)
- \( T^T \) = transpose of the matrix
- \( u_a \) = scores associated with the \( a \)th latent vector of \( Y \)
- \( U \) = matrix of scores associated with \( Y \)
- \( w_a \) = loading vector used for prediction, calculated from PLS, for the \( a \)th latent vector
- \( W \) = matrix of prediction loadings for \( X \)
- \( X \) = \((n \times k)\) matrix of the process/operational/input measurements
- \( Y \) = \((n \times m)\) matrix of the process performance/product quality/output measurements
Appendix 2. Survey Analysis

Survey Formulation. The survey served as an exploratory analysis tool to assess the current perceptions of SCD personnel on variability reduction. Respondents were asked to indicate their job position and work center. The three opening questions asked respondents to rate their knowledge about, importance of, and current work in variability reduction. Respondents were then asked to evaluate a set of 33 posited barriers based on their actual experience in working on variability reduction. The 33 barriers were identified as potential barriers from field observations. The ratings were based on a five-point Likert scale. Respondents were also asked to identify any other barriers and propose suggestions for improvement. A copy of the survey is included as Figure A-1.

Six respondents were asked to fill-out the survey in a pretest. Four reviewers provided additional feedback. The survey was revised to clarify points of confusion before final roll-out. Survey distribution to division personnel was coordinated via production staff meetings at each work center. In general, respondents were asked to fill-out and return the surveys immediately.

Response Rate. The response rate was calculated as the number of respondents as a percentage of the actual number of SCD personnel. The distribution rate indicates the percentage of SCD personnel who received the survey. Table A-1 shows the response rates by job position and work center.

<table>
<thead>
<tr>
<th>Work Center</th>
<th>Operators</th>
<th>Group Leaders</th>
<th>Process Chemists</th>
<th>Technical Staff</th>
<th>Supervisors</th>
<th>Total</th>
<th>% Distributed</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC1</td>
<td>53%</td>
<td>63%</td>
<td>50%</td>
<td>-</td>
<td>75%</td>
<td>56%</td>
<td>92%</td>
</tr>
<tr>
<td>WC2</td>
<td>33%</td>
<td>33%</td>
<td>35%</td>
<td>-</td>
<td>67%</td>
<td>38%</td>
<td>100%</td>
</tr>
<tr>
<td>WC3</td>
<td>71%</td>
<td>75%</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
<td>80%</td>
<td>100%</td>
</tr>
<tr>
<td>WC4</td>
<td>57%</td>
<td>60%</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
<td>67%</td>
<td>89%</td>
</tr>
<tr>
<td>WC5</td>
<td>55%</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
<td>63%</td>
<td>77%</td>
</tr>
<tr>
<td>WC6</td>
<td>69%</td>
<td>83%</td>
<td>75%</td>
<td>67%</td>
<td>33%</td>
<td>73%</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>58%</td>
<td>69%</td>
<td>53%</td>
<td>80%</td>
<td>71%</td>
<td>62%</td>
<td></td>
</tr>
</tbody>
</table>
Hi! My name is Terri Mock. This survey is part of a Master's thesis research conducted in the Synthetic Chemicals Division. The purpose of this survey is to develop a better understanding of the existing barriers to reducing process variability within SynChem. The collection and analysis of process data can lead to a better understanding of the process and result in reduced variability. The goal is to establish process control by identifying and eliminating the sources of variability. I would greatly appreciate it if you would take a few minutes to fill out this survey. Your input is valuable and will be kept strictly confidential. Please return the completed surveys to me via Kodak mail by October 18th. My address is: 303/2 KP MC 23618. The results of the survey will be distributed to the individual departments through their Quality Facilitator. If you have any questions, you can reach me at x8-0867. Thanks!!

Optional Information:
Name: ____________________________ Address: ____________________________ Phone: ______

Job Position:
_____ Operator
_____ Group Leader
_____ Materials Services
_____ Staff
_____ Process Chemist
_____ Supervision

Building: Please indicate the building in which you work. Building _____.

How much do you know about variability reduction?
_____ Not at all
_____ Not very
_____ Somewhat
_____ Knowledgeable
_____ Very
knowledgeable
knowledgeable
Knowledgeable
knowledgeable

How important do you think variability reduction is to SynChem's business?
_____ Not at all
_____ Not very
_____ Somewhat
_____ Important
_____ Extremely
important
important
important
important

How much do you currently work on variability reduction in your job?
_____ Never
_____ Infrequently
_____ Only for
_____ Regularly
troubleshooting

Based on your actual experience in working on variability reduction, how would you evaluate each of the following statements?
1=Strongly disagree, 2=Disagree, 3=Neither agree nor disagree, 4=Agree, 5=Strongly agree.

1. I don't know what process data are important to collect for variability reduction. 1 2 3
2. We are not organized into teams to work on variability reduction. 1 2 3
3. There are too few batches on the same process to be useful for data analysis. 1 2 3
4. I don't see a correlation between reduction efforts and the business bottom-line. 1 2 3
5. I lack the tools required to analyze process data. 1 2 3
6. I have too many other competing priorities that take up my time. 1 2 3
7. Top management does not support my efforts on variability reduction. 1 2 3
8. Variability reduction is not part of my performance evaluation. 1 2 3
9. There are too many chemicals to analyze. 1 2 3
10. It takes too much effort to collect data manually. 1 2 3
Figure A-1. (continued)

Based on your actual experience in working on variability reduction, how would you evaluate each of the following statements?
1=Strongly disagree, 2=Disagree, 3=Neither agree nor disagree, 4=Agree, 5=Strongly agree.

11. Variability reduction is not a responsibility assigned to me. 1 2 3
12. It is too difficult to get approval for process changes to reduce variability. 1 2 3
13. Variability reduction is not part of my day-to-day activities in production. 1 2 3
14. I have no practical guidelines on how to start. 1 2 3
15. We do not have an adequate computerized system for storing historical data. 1 2 3
16. I don’t know how to analyze process data. 1 2 3
17. I don’t have my immediate supervisor’s support to work on variability reduction. 1 2 3
18. I lack the computer skills needed to analyze process data. 1 2 3
19. It is too difficult to get funding for variability reduction efforts. 1 2 3
20. It is too difficult to communicate process changes to other operators. 1 2 3
21. I don’t have enough training on how to reduce variability. 1 2 3
22. There is a lack of operator involvement in variability reduction. 1 2 3
23. It takes too much effort for me to analyze process data. 1 2 3
24. We do not have adequate data acquisition systems to collect process data. 1 2 3
25. SynChem does not have a long-term commitment to variability reduction. 1 2 3
26. There is not enough control of the process to make reduction efforts worthwhile. 1 2 3
27. I do not have easy access to a computer for data analysis. 1 2 3
28. We do not have an adequate computerized system for integrating different data sources. 1 2 3
29. ATD does not support SynChem enough in variability reduction. 1 2 3
30. CDD/CERL does not support SynChem enough in variability reduction. 1 2 3
31. CQS does not support SynChem enough in variability reduction. 1 2 3
32. MEMO does not support SynChem enough in variability reduction. 1 2 3
33. MSD does not support SynChem enough in variability reduction. 1 2 3

Please identify any other barriers you can think of.

What are your suggestions to overcome these barriers?
Survey Structure. An oblique principal components cluster analysis was performed to identify the underlying structure of the survey data. The SAS VARCLUS hierarchical clustering procedure\(^{34}\) was used to identify groups of questions that belonged together. Using this multivariate algorithm, eight clusters were identified to represent the 33 barriers (Figure A-2). To verify the question groupings, the 33 barriers was correlated with each other to form a 33 by 33 matrix of correlation coefficients. Intragroup and intergroup mean correlation coefficients were calculated. In addition, the Chronbach alpha reliability test was calculated as a measure of the internal consistency of the survey. Table A-2 shows the group correlation coefficients matrix and the Chronbach alphas.

Table A-2. Cluster Correlation Matrix

<table>
<thead>
<tr>
<th>Cluster</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.3593</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.3025</td>
<td>0.3970</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.1841</td>
<td>0.2113</td>
<td>0.2503</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.2412</td>
<td>0.2152</td>
<td>0.2025</td>
<td>0.3730</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.1783</td>
<td>0.3237</td>
<td>0.2363</td>
<td>0.2113</td>
<td>0.4471</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.1854</td>
<td>0.0410</td>
<td>0.0553</td>
<td>0.1784</td>
<td>0.0534</td>
<td>0.3172</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.1885</td>
<td>0.1606</td>
<td>0.1427</td>
<td>0.1258</td>
<td>0.1758</td>
<td>0.2095</td>
<td>0.5323</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.2187</td>
<td>0.1239</td>
<td>0.1504</td>
<td>0.1490</td>
<td>0.0654</td>
<td>0.1672</td>
<td>0.3124</td>
<td>0.5835</td>
</tr>
<tr>
<td>N</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>4</td>
<td>4</td>
<td>5</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>alpha</td>
<td>0.6916</td>
<td>0.7670</td>
<td>0.6670</td>
<td>0.7041</td>
<td>0.7639</td>
<td>0.6990</td>
<td>0.7735</td>
<td>0.7369</td>
</tr>
</tbody>
</table>

Intragroup R\(^2\) greater than intergroup R\(^2\).

Chronbach alpha reliability test for internal consistency.

\[
\alpha = \frac{N \times \bar{R}^2}{1 + (N-1) \times R^2}
\]

\(^{34}\) SAS Institute Inc., *op. cit.*, pp.1641-1659.
Figure A-2. Organizational Factors Survey Clusters

Cluster 1: Top Management Support for Variability Reduction
- Top management does not support my efforts on variability reduction. 7
- It is too difficult to get approval for process changes to reduce variability. 12
- It is too difficult to get funding for variability reduction efforts. 19
- SynChem does not have a long-term commitment to variability reduction. 25

Cluster 2: Variability Reduction as a Production Priority In Practice
- Variability reduction is not part of my performance evaluation. 8
- Variability reduction is not a responsibility assigned to me. 11
- Variability reduction is not part of my day-to-day activities in production. 13
- I don’t have my immediate supervisor’s support to work on variability reduction. 17
- We do not have enough operator involvement in variability reduction. 22

Cluster 3: Belief in the Benefits of Variability Reduction
- I don’t know what process data are important to collect for variability reduction. 1
- We are not organized into teams to work on variability reduction. 2
- There are too few batches on the same process to be useful for data analysis. 3
- I don’t see a correlation between reduction efforts and the business bottom-line. 4
- I lack the tools required to analyze process data. 5
- There is not enough control of the process to make reduction efforts worthwhile. 26

Cluster 4: Variability Reduction Requires Time and Effort
- I have too many other competing priorities that take up my time. 6
- There are too many chemicals to analyze. 9
- It takes too much effort to collect data manually. 10
- It takes too much effort for me to analyze data. 23

Cluster 5: Training for Variability Reduction
- I have no practical guidelines on how to start. 14
- I don’t know how to analyze process data. 16
- I lack the computer skills needed to analyze process data. 18
- I don’t have enough training on how to reduce variability. 21

Cluster 6: Information System for Variability Reduction Efforts
- We do not have an adequate computerized system for storing historical data. 15
- It is too difficult to communicate process changes to other operators. 20
- We do not have adequate data acquisition systems to collect process data. 24
- I do not have easy access to a computer for data analysis. 27
- We do not have an adequate computerized system for integrating different data sources 28

Cluster 7: Analytical and Development Support for Variability Reduction
- ATD does not support SynChem enough in variability reduction. 29
- CDD/CERL does not support SynChem enough in variability reduction. 30
- CQS does not support SynChem enough in variability reduction. 31

Cluster 8: Equipment and Materials Support for Variability Reduction
- MEMO does not support SynChem enough in variability reduction. 32
- MSD does not support SynChem enough in variability reduction. 33
Response Analysis. For each question, the mean, standard deviation, and range was calculated. The survey responses were analyzed by work center and job position. The best breakdown of the data for analysis was by job position. Differences between work centers were nominal. Graphs of the survey responses by job position and question cluster are shown in Figures A-3-10.
Figure A-3. Survey Data: Cluster 1 - Top Management Support for Variability Reduction

Sort by Job Position

Question 7: Top management does not support my efforts on variability reduction.
Question 12: It is too difficult to get approval for process changes to reduce variability.
Question 19: It is too difficult to get funding for variability reduction efforts.
Question 25: SynChem does not have a long-term commitment to variability reduction.
Figure A-4. Survey Data: Cluster 2 - Variability Reduction as a Production Priority In Practice

Sort by Job Position

Question 8: Variability reduction is not part of my performance evaluation.
Question 11: Variability reduction is not a responsibility assigned to me.
Question 13: Variability reduction is not part of my day-to-day activities in production.
Question 17: I don't have my immediate supervisor's support to work on variability reduction.
Question 22: We do not have enough operator involvement in variability reduction.
Figure A-5. Survey Data: Cluster 3 - Belief in the Benefits of Variability Reduction

Sort by Job Position

Question 1: I don't know what process data are important to collect for variability reduction.
Question 2: We are not organized into teams to work on variability reduction.
Question 3: There are too few batches on the same process to be useful for data analysis.
Question 4: I don't see a correlation between reduction efforts and the business bottom-line.
Question 5: I lack the tools required to analyze process data.
Question 26: There is not enough control of the process to make reduction efforts worthwhile.
Figure A-6. Survey Data: Cluster 4 - Variability Reduction Requires Time and Effort

Sort by Job Position

Question 6: I have too many other competing priorities that take up my time.
Question 9: There are too many chemicals to analyze.
Question 10: It takes too much effort to collect data manually.
Question 23: It takes too much effort for me to analyze data.
Figure A-7. Survey Data: Cluster 5 - Training for Variability Reduction

Sort by Job Position

Question 14: I have no practical guidelines on how to start.
Question 16: I don't know how to analyze process data.
Question 18: I lack the computer skills needed to analyze process data.
Question 21: I don't have enough training on how to reduce variability.
Question 15: We do not have an adequate computerized system for storing historical data.
Question 20: It is too difficult to communicate process changes to other operators.
Question 24: We do not have adequate data acquisition systems to collect process data.
Question 27: I do not have easy access to a computer for data analysis.
Question 28: We do not have an adequate computerized system for integrating different data sources.

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Figure A-9. Survey Data: Cluster 7 - Analytical and Development Support for Variability Reduction

Sort by Job Position

Question 29: ATD does not support SynChem enough in variability reduction.
Question 30: CDD/CERL does not support SynChem enough in variability reduction.
Question 31: CQS does not support SynChem enough in variability reduction.
Figure A-10. Survey Data: Cluster 8 - Equipment and Materials Support for Variability Reduction

Sort by Job Position

Question 32
MEMO does not support SynChem enough in variability reduction.

Question 33
MSD does not support SynChem enough in variability reduction.