USING SIX SIGMA TO OPTIMIZE A CONTINUOUS CHEMICAL PROCESS AT ALLIED SIGNAL INC.

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Abstract
This thesis provides an overview of the Six Sigma methodology and its use in optimizing a
continuous chemical process at AlliedSignal’s Hopewell plant. In 1995, AlliedSignal
initiated its corporate-wide Six Sigma program to increase production and yields and reduce
waste through statistical process improvement methods. The Six Sigma methodology uses
statistical tools such as gauge studies, design of experiments, and analysis of variance
(ANOVA) to reduce process variability and optimize existing processes using minimal
capital dollars.

The Hopewell plant is part of AlliedSignal’s Chemical Intermediates business unit, which is
part of the larger Polymers business unit. As a vertically integrated business, the Hopewell
plant supplies caprolactam, the raw material for nylon-6, to downstream fibers, plastics and
film plants. Over the past few years, the Hopewell plant has been the bottleneck in the
Polymers nylon chain and has been unable to meet the caprolactam demand of the
downstream businesses. As a result, Polymers has had to purchase more expensive
caprolactam on the spot market to meet production needs.

Within the Hopewell Plant, the Hydrox production area is the bottleneck during a majority of
the year. To reduce reliance on outside caprolactam purchases, a team was chartered to
increase Hydrox production using Six Sigma methods. This thesis describes the Six Sigma
methodology, how it was applied to the Hydrox production process to increase capacity, and
opportunities for further improvement. Although the area of focus was a continuous
chemical process, it could be easily applied to any type of process. This includes
manufacturing processes such discrete or batch, as well as non-manufacturing activities such
as supply chain management, distribution, or process development.

Thesis supervisors:
Roy Welsch, Professor of Management Science
Paula Hammond, Professor of Chemical Engineering
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# Table of Contents

1. THESIS OVERVIEW ................................................................. 11
   1.1 COMPANY BACKGROUND .................................................. 11
   1.2 COMPANY CULTURE ...................................................... 12
   1.3 THESIS OBJECTIVES ..................................................... 13
   1.4 TERMS ........................................................................... 13
   1.5 THESIS OUTLINE .......................................................... 15

2. CAPROLACTAM AND THE NYLON SYSTEM ................................ 17
   2.1 PLANT BACKGROUND ...................................................... 17
   2.2 HYDROX PROCESS BACKGROUND .................................... 18
   2.3 IMPROVEMENT GOALS ................................................... 20
      2.3.1 Carbonate system ..................................................... 20
      2.3.2 Nitrite System .......................................................... 20
      2.3.3 Disulfonate System ................................................... 20
      2.3.4 Refrigeration ........................................................... 20
   2.4 REVIEW OF THE LITERATURE ........................................ 21

3. SIX SIGMA ............................................................................ 23
   3.1 SIX SIGMA: BUSINESS GOALS ....................................... 23
      3.1.1 Capacity Productivity ............................................... 23
      3.1.2 Rolled Throughput Yield ......................................... 24
      3.1.3 Cost of Poor Quality .............................................. 24
   3.2 SIX SIGMA METHODOLOGY ........................................... 24
      3.2.1 Measure ................................................................. 25
      3.2.2 Analyze ................................................................. 25
      3.2.3 Improve ................................................................. 26
      3.2.4 Control ................................................................. 26
   3.3 SIX SIGMA HISTORY ....................................................... 26

4. SIX SIGMA: MEASURE .......................................................... 29
   4.1 PURPOSE ...................................................................... 29
   4.2 TOOLS .......................................................................... 29
      4.2.1 Team Charter ........................................................... 29
      4.2.2 Process Map ............................................................ 30
      4.2.3 Gauge Studies ........................................................ 31
<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.2.4</td>
<td>Short Term Capability Studies</td>
<td>32</td>
</tr>
<tr>
<td>4.3</td>
<td>SUMMARY OF MEASURE</td>
<td>33</td>
</tr>
<tr>
<td>5.</td>
<td>SIX SIGMA: ANALYZE</td>
<td>35</td>
</tr>
<tr>
<td>5.1</td>
<td>PURPOSE</td>
<td>35</td>
</tr>
<tr>
<td>5.2</td>
<td>TOOLS</td>
<td>35</td>
</tr>
<tr>
<td>5.2.1</td>
<td>Failure Modes and Effects Analysis (FMEA)</td>
<td>35</td>
</tr>
<tr>
<td>5.2.2</td>
<td>Multi-variable Study</td>
<td>38</td>
</tr>
<tr>
<td>5.3</td>
<td>SUMMARY OF ANALYZE</td>
<td>44</td>
</tr>
<tr>
<td>6.</td>
<td>SIX SIGMA: IMPROVE</td>
<td>45</td>
</tr>
<tr>
<td>6.1</td>
<td>PURPOSE</td>
<td>45</td>
</tr>
<tr>
<td>6.2</td>
<td>TOOLS</td>
<td>45</td>
</tr>
<tr>
<td>6.2.1</td>
<td>Design of Experiments I</td>
<td>45</td>
</tr>
<tr>
<td>6.2.2</td>
<td>Design of Experiments II</td>
<td>49</td>
</tr>
<tr>
<td>6.3</td>
<td>SUMMARY OF IMPROVE</td>
<td>51</td>
</tr>
<tr>
<td>7.</td>
<td>SIX SIGMA: CONTROL</td>
<td>53</td>
</tr>
<tr>
<td>7.1</td>
<td>PURPOSE</td>
<td>53</td>
</tr>
<tr>
<td>7.2</td>
<td>TOOLS</td>
<td>53</td>
</tr>
<tr>
<td>7.2.1</td>
<td>Process Support Systems</td>
<td>53</td>
</tr>
<tr>
<td>7.2.2</td>
<td>Long-term capability study</td>
<td>54</td>
</tr>
<tr>
<td>7.3</td>
<td>SUMMARY OF CONTROL</td>
<td>57</td>
</tr>
<tr>
<td>8.</td>
<td>CONCLUSION/RECOMMENDATIONS</td>
<td>59</td>
</tr>
<tr>
<td>8.1</td>
<td>CONCLUSIONS</td>
<td>59</td>
</tr>
<tr>
<td>8.2</td>
<td>RECOMMENDATIONS</td>
<td>60</td>
</tr>
<tr>
<td>8.3</td>
<td>CLOSING COMMENTS</td>
<td>61</td>
</tr>
<tr>
<td>9.</td>
<td>REFERENCES</td>
<td>63</td>
</tr>
</tbody>
</table>
## Table of Figures

<table>
<thead>
<tr>
<th>Figure</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>ALLIED SIGNAL BUSINESS UNITS</td>
<td>11</td>
</tr>
<tr>
<td>2</td>
<td>NYLON VALUE CHAIN</td>
<td>12</td>
</tr>
<tr>
<td>3</td>
<td>HOPEWELL PLANT PROCESS FLOW</td>
<td>17</td>
</tr>
<tr>
<td>4</td>
<td>AREA 9 HYDROX PROCESS FLOW</td>
<td>18</td>
</tr>
<tr>
<td>5</td>
<td>SIX SIGMA METHODOLOGY</td>
<td>25</td>
</tr>
<tr>
<td>6</td>
<td>NITRITE PROCESS MAP</td>
<td>30</td>
</tr>
<tr>
<td>7</td>
<td>NITRITE GAUGE STUDY</td>
<td>32</td>
</tr>
<tr>
<td>8</td>
<td>FMEA RISK PRIORITY RANKING (RPN) SCALE</td>
<td>36</td>
</tr>
<tr>
<td>9</td>
<td>FISHBONE DIAGRAM FOR NITRITE TOWER</td>
<td>36</td>
</tr>
<tr>
<td>10</td>
<td>FMEA FOR NITRITE TOWER</td>
<td>37</td>
</tr>
<tr>
<td>11</td>
<td>MULTI-VARIABLE ANALYSIS RAW DATA</td>
<td>39</td>
</tr>
<tr>
<td>12</td>
<td>MULTI-VARIABLE ANALYSIS (BEST SUBSETS REGRESSION)</td>
<td>40</td>
</tr>
<tr>
<td>13</td>
<td>MULTI-VARIABLE ANALYSIS (BASIC REGRESSION)</td>
<td>41</td>
</tr>
<tr>
<td>14</td>
<td>REGRESSION RESIDUAL DIAGNOSTIC PLOTS</td>
<td>42</td>
</tr>
<tr>
<td>15</td>
<td>RATIONAL SUBGROUPING: CONVERSION INDEX VS. BICARBONATE LEVEL</td>
<td>43</td>
</tr>
<tr>
<td>16</td>
<td>BICARBONATE EXPERIMENTAL PLAN</td>
<td>46</td>
</tr>
<tr>
<td>17</td>
<td>BICARBONATE EXPERIMENT #1 RESULTS</td>
<td>48</td>
</tr>
<tr>
<td>18</td>
<td>BICARBONATE EXPERIMENT #2 RESULTS</td>
<td>50</td>
</tr>
<tr>
<td>19</td>
<td>CARBONATE BASELINE PROCESS CAPABILITY (MARCH 1997)</td>
<td>54</td>
</tr>
<tr>
<td>20</td>
<td>CARBONATE CAUSAL LOOP DIAGRAM</td>
<td>55</td>
</tr>
<tr>
<td>21</td>
<td>CARBONATE PROCESS CAPABILITY - AFTER PROCEDURE CHANGES (SEPT. 1997)</td>
<td>56</td>
</tr>
<tr>
<td>22</td>
<td>CARBONATE PROCESS CAPABILITY - AFTER CO2 SETPOINT CHANGES (OCT. 1997)</td>
<td>56</td>
</tr>
<tr>
<td>23</td>
<td>NITRITE TEAM BENEFITS</td>
<td>59</td>
</tr>
</tbody>
</table>
1. Thesis Overview

1.1 Company Background

AlliedSignal Inc. is a large, diversified Fortune 100 manufacturing company comprised of eleven business groups supplying products for the aerospace, automotive and engineered materials markets. This thesis involves the Polymers business group. AlliedSignal's 1996 revenue was $14 Billion with Polymers contributing $1.7 Billion of that number. The majority of the revenue in Polymers comes from the sale of carpet fibers, plastics and industrial fibers.

Figure 1 - AlliedSignal Business Units

![Diagram showing AlliedSignal Inc.'s business units]

- **AlliedSignal Inc.**
  - Aerospace Engines
  - Commercial Avionics Systems
  - Electronic Systems
  - Aerospace Equipment Systems
  - Aerospace Marketing Sales & Service
  - Automotive Products Group
  - Truck Brake Systems
  - Turbocharging Systems
  - Polymers
  - Specialty Chemicals
  - Electronic Materials
The Polymers business unit can be split into two general product lines: polyester and nylon-6. The raw material for nylon-6 is a chemical called caprolactam, which is produced internally at the Hopewell, Virginia plant, part of the Chemical Intermediates group.

**Figure 2 - Nylon Value Chain**

![Nylon Value Chain Diagram]

**1.2 Company Culture**

AlliedSignal’s culture is results-driven. One of the three key corporate goals each year from CEO Larry Bossidy is “Make the Numbers”. This means each business is expected to meet their revenue, income and cash flow targets. One tangible benefit from this results-driven culture has been the favorable view by Wall Street investors as seen in the 429% increase in AlliedSignal market value since Larry Bossidy joined the corporation in 1991. One disadvantage of this results-driven culture is the tendency for employees to look for quick-fixes or short-term solutions when confronting problems. This tendency to focus on short-term solutions leads to treating symptoms without addressing root causes. Some of the
problems identified during the Area 9 Hydrox project had been recurring problems in the production area and could be traced to this short-term focus. When addressing production problems, changing the culture is often more challenging than changing the production process. The key methods used to overcome some of the cultural issues were communication and education.

1.3 Thesis Objectives

The internship project covers the use of Six Sigma methodology to increase Hydrox capacity in Area 9 of the AlliedSignal Hopewell plant. In 1994, AlliedSignal launched its Six Sigma program to improve manufacturing operations by training engineers and scientists in a statistical-based problem-solving method. These trained employees became known as Black Belts.

At the Hopewell plant, the Hydrox production area has been the bottleneck for the past few years. To achieve the business goals, the Production Manager and Process Engineering Leader formed a task force led by Black Belts with the goal of dechoking the Hydrox area by 50 million pounds per year.

This thesis has three objectives:

1. Provide an overview of AlliedSignal’s Six Sigma methodology.

2. Explain how the Six Sigma methodology was used to increase the capacity of a continuous chemical process.

3. Identify opportunities for future improvements

1.4 Terms

This thesis will be using a number of terms related to Six Sigma and to AlliedSignal. Since different companies may use these terms differently, the following definitions are provided.
Six Sigma Terminology

- Six Sigma - A statistical-based problem solving method used to improve manufacturing processes. (A process with six sigma capability generates only 3.4 defects per million units and is considered world-class).

- Unit – A task or physical entity such as: number of rail cars shipped, pounds of lactam produced, or number of customer phone calls received.

- Defect – Non-compliance to a standard such as a product parameter outside of customer specifications, spillage, emission losses, etc.

- Capacity-Productivity (C-P) - A measure of first-quality capacity of a manufacturing operation.

- Rolled Throughput Yield - Measures the first-quality yield for a process by multiplying together first-quality yield of each major process step.

- Cost of Poor Quality - The costs of failing to produce and deliver 100% quality products to the customer. This metric includes the cost of waste, scrap, rework, and returns.

- First-quality - Product from a unit operation that meets all customer specifications.

AlliedSignal/Chemical Terminology

- Caprolactam (lactam) - The main product of the Hopewell Plant. It is the raw material used to make Nylon-6 polymer.

- Hydroxylamine Sulfate (hydrox) - One of the three key intermediate chemicals used to produce caprolactam. Hydrox is produced in Area 9 of the Hopewell plant by reacting carbon dioxide, ammonia, sulfur and water in a series of unit operations.

- Area 9 – The hydrox production area.

- Ammonium carbonate (carbonate) - The first unit operation in hydrox production. Carbonate is formed by absorbing carbon dioxide and ammonia in water.

- Ammonium nitrite (nitrite) - The second unit operation in hydrox production. Nitrite is formed by reacting ammonia and air to form nitrous oxide and reacting it with carbonate.
• Hydroxylamine disulfonate (disulfonate) - The third unit operation in hydrox production. Disulfonate is formed by reacting sulfur and air to form sulfur dioxide and reacting sulfur dioxide with nitrite.

• Train - A series of unit operations that make up a single production line. Area 9 has five hydrox trains.

• Unit Operation – Each sub-process (piece of major process equipment) within a train

• Mppy – abbreviation for million pounds per year

1.5 Thesis Outline

Chapter 2 provides background material on AlliedSignal’s nylon value chain, the Hopewell plant, and a review of the literature on hydrox production. Chapter 3 provides an overview of AlliedSignal’s Six Sigma methodology which includes the three key metrics (capacity-productivity, rolled throughput yield, cost of poor quality) and the four step methodology (Measure, Analyze, Improve, Control)1. Chapters 4 through 7 discuss how the methodology was applied in Area 9 to increase hydrox capacity. Chapter 4 provides a discussion of the Measure phase and the tools used to provide baseline information on the hydrox process. Chapter 5 covers the Analyze phase and provides insight into the statistical methods used to analyze baseline process data. Chapter 6 describes the Improve phase and the experiments that were conducted to identify improvements in operating conditions. Finally, Chapter 7 outlines the Control phase and the steps taken to institutionalize the process changes. Chapter 8 provides conclusions, recommendations for future improvement efforts and closing comments.

1 Note: The four phases of Six Sigma process improvement (Measure, Analyze, Improve, Control) will be capitalized when used to identify a process improvement phase and not capitalized when used as a verb.
2. Caprolactam and the Nylon System

This chapter provides a brief overview of AlliedSignal's nylon value chain and discusses the market conditions facing management.

2.1 Plant Background

AlliedSignal's Polymer division is vertically integrated with two chemical intermediates plants feeding two downstream nylon plants which supply material to six business areas. The Hopewell plant, one of the chemical plants, converts phenol, natural gas, and sulfur to caprolactam, ammonium sulfate and other by-products through a series of complex unit operations as shown below. The main product from the Hopewell plant is caprolactam, which is shipped to downstream plants for polymerization and conversion into nylon fibers, films and plastic resins.

Figure 3 - Hopewell Plant Process Flow
2.2 Hydrox Process Background

Within the Hopewell Plant, Area 9 produces hydroxylamine sulfate, which is referred to as hydrox. The hydrox is produced in a series of four unit operations: Carbonate, Nitrite, Disulfonate and Hydrolysis. The hydrox product is reacted with cyclohexanone and ammonia in Area 8 to produce cyclohexanone oxime (a precursor to caprolactam). A small portion of the hydrox stream is used on-site in the Specialty Chemicals plant to produce a family of oxime products.

Figure 4 - Area 9 Hydrox Process Flow
Hydrox is produced in the following steps:

1. Ammonium carbonate and bicarbonate are produced in the carbonate section from ammonia, carbon dioxide and water.
   
   \[ 3 \text{NH}_3 + 2\text{CO}_2 + 2\text{H}_2\text{O} \rightarrow (\text{NH}_4)_2\text{CO}_3 + \text{NH}_4\text{HCO}_3 \]

2. Nitric oxide is produced in the ammonia oxidation section by reacting ammonia and air over a cobalt catalyst bed. Ammonium nitrite is produced in the nitrite section by reacting nitric oxide with ammonium carbonate/bicarbonate.
   
   \[ 4\text{NH}_3 + 5\text{O}_2 \rightarrow 4\text{NO} + 6\text{H}_2\text{O} \quad (\text{very exothermic}) \]
   
   \[ \text{NO} + \frac{1}{2}\text{O}_2 \rightarrow \text{NO}_2 \]
   
   \[ \text{NO} + \text{NO}_2 + 2(\text{NH}_4)_2\text{CO}_3 + \text{H}_2\text{O} \rightarrow 2\text{NH}_4\text{NO}_2 + 2\text{CO}_2 \]

3. Sulfur dioxide is produced in the sulfur burning section from sulfur and air.

Unhydrolyzed hydrox (and byproduct ammonium sulfate) is produced in the disulfonate section by reacting sulfur dioxide, ammonium nitrite, and ammonia.

   \[ \text{S} + \text{O}_2 \rightarrow \text{SO}_2 \quad (\text{very exothermic}) \]
   
   \[ 3\text{SO}_2 + \text{NH}_4\text{NO}_2 + 3\text{NH}_3 \rightarrow (\text{NH}_4\text{SO}_3)_2\text{NOH} + \text{H}_2\text{O} + (\text{NH}_4)_2\text{SO}_4 \]

4. Hydrox, the final product, is produced in the hydrolysis section from unhydrolyzed hydrox and water.

   \[ 2(\text{NH}_4\text{SO}_3)_2\text{NOH} + \text{H}_2\text{O} \rightarrow \text{NHOH}(\text{NH}_4\text{SO}_3) + \text{H}_2\text{SO}_4 + (\text{NH}_4)_2\text{SO}_4 \]
2.3 Improvement Goals

A number of improvement opportunities were identified in Area 9. These opportunities were not necessarily new, but with the increasing competitiveness of the market, they were becoming more important to “harvest”. Therefore, aggressive improvement goals had been established.

2.3.1 Carbonate system

Since the carbonate system has excess capacity beyond the hydrox production goals set by the team champions, the main goal for the carbonate system was to improve process capability of carbon dioxide and ammonia in the carbonate product.

2.3.2 Nitrite System

The nitrite system is the bottleneck operation in the hydrox production area for the majority of the year. During the warmer months of the year, ammonia burning capacity is limited due to ambient air temperature restricting air blower capacity. As the ambient temperature increases, air density decreases, which reduces blower throughput. The majority of this thesis focuses on increasing nitrite capacity by improving process yields and increasing capacity-productivity.

2.3.3 Disulfonate System

The disulfonate system can become the bottleneck operation during the colder months due to sulfur burning limitations. The main goal for the disulfonate area was to increase sulfur burning capacity to support the projected nitrite rate increases.

2.3.4 Refrigeration

To improve process yields, heat is removed from the nitrite and disulfonate processes by circulating the liquid through refrigerated chillers and ammonia vaporizers. The main goal for the refrigeration system was to improve refrigeration operations to lower tower temperature and increase nitrite and disulfonate yields.
2.4 Review of the Literature

Before using the Six Sigma methodology to improve the hydrox process, a review of the technical literature on the process was undertaken. Since the hydrox production process is relatively uncommon and since AlliedSignal is one of the world’s largest producers, internal technical reports yielded most of the information from the literature search. A technical information summary published in 1992 provided a comprehensive overview of internal research, plant trials and process data for each unit operation in the Area 9 hydrox process. The findings from the literature search were used as input for the failure modes and effects analysis.

Key findings from the literature search were as follows:

- Nitrite tower yield increases as the ratio of bicarbonate to carbonate in the feed stream increases. Operating with a higher bicarbonate ratio reduces the partial pressure of ammonia; decreasing side reactions that result in ammonia yield loss.

- Nitrite tower yield increases as tower temperature decreases. Reducing tower temperature decreases side reactions that result in ammonia yield loss.

- Increasing nitrite tower pressure can reduce NOX emissions out of the vent stack, which reduces ammonia yield loss and increases tower yield. A trade-off is that increasing tower pressure increases the formation of nitrate, which is another form of yield loss.
3. Six Sigma

This chapter provides a brief overview of AlliedSignal's Six Sigma program. It also provides a framework of how the Hopewell Plant planned to use Six Sigma to dechoke the plant bottleneck (Area 9 hydrox) and meet the business goals.

3.1 Six Sigma: Business Goals

AlliedSignal launched Six Sigma in early 1995 to reduce waste and improve capacity and yields in their manufacturing operations. The mission of the Six Sigma program is to reduce defects, waste and variability in the manufacturing process to exploit the 'hidden factory'.

The hidden factory is the lost capacity and waste that keep a manufacturing process from fully utilizing its assets. Through the Six Sigma method, AlliedSignal planned to capture a portion of this 'hidden factory' to reduce costs, increase profits and improve cash flow. Historical data shows that average companies lose approximately 25 percent of their total sales in this hidden factory; world-class companies lose less than ten percent [Harry, 1993].

Prior to launching Six Sigma, AlliedSignal's 300+ manufacturing sites each had their own measures for capacity utilization, first pass yield, and cost improvements. One of the key benefits from launching the corporate-wide Six Sigma program was providing a standard nomenclature, methodology and metrics for each manufacturing site to use. This common language facilitated sharing results and best practices and ensured each site was measuring the true impact of variation in their processes. The three measures used to understand the impact of variation are capacity productivity (C-P), rolled throughput yield (RTY) and cost of poor quality (COPQ).

3.1.1 Capacity Productivity

*Capacity productivity (C-P)* measures the output of each process over a specified time period. *Entitlement* is the theoretical capacity of a process and measures output based on 100% up-time, operating at maximum capacity and at theoretical yields. Dividing the capacity-productivity of a process by its entitlement, gives *capacity utilization*; an indication of asset utilization.
3.1.2 Rolled Throughput Yield

Rolled throughput yield (RTY) provides an indication of process quality. Most manufacturing processes have multiple steps or sub-processes. By analyzing one step or sub-process and examining its first pass yield (first quality, first time through), we can capture the effect of any loss or variation in the downstream processes by calculating the rolled throughput yield. RTY is calculated using the product of all the first pass yields as follows:

\[(\text{Process 1 Yield}) \times (\text{Process 2 Yield}) \times (\text{Process 3 Yield}) \ldots = \text{Rolled Throughput Yield}\]

3.1.3 Cost of Poor Quality

The cost associated with defects and with detecting and eliminating defects is called the cost of poor quality (COPQ). COPQ quantifies the cost of internal and external failures. Internal failures include scrap, rework, blending, recycle streams, quality inspection and testing and special packaging or handling. External failure is the cost to correct products or services after delivery to the customer and includes claims, returns, and price adjustments. COPQ quantifies the financial impact of operating a process at less than 100 percent rolled throughput yield and 100 percent capacity utilization.

3.2 Six Sigma Methodology

In addition to defining the three productivity metrics (C-P, RTY, COPQ), AlliedSignal's Six Sigma program trained employees how to use a standardized process improvement methodology: Measure, Analyze, Improve, Control.
3.2.1 Measure

The Measure phase includes:

- Defining project objectives, metrics and resources requirements; usually documented in a team charter.

- Creating a detailed process map which includes key process input variables (KPIV) and key process output variables (KPOV) for each step in the process.

- Identifying and implementing gauge studies to determine the capability of the measurement system.

- Performing a short-term capability study to determine the process baseline.

3.2.2 Analyze

Once the Measure phase has been completed, the Analyze phase begins by:

- Conducting a failure modes and effects analysis to identify potential root causes of low yields, lost capacity, and high COPQ.

- Conducting a multi-variable study using historical process data to identify key process input variables for further study.
• Using the results from the FMEA and multi-variable study to develop a project roadmap for the Improvement and Control phases.

3.2.3 Improve
Using the results from the Analyze phase, the Improvement phase includes:

• Planning and conducting design of experiments (DOE) to determine the impact of KPIVs on KPOVs and establishing an optimum operating window

• Using analysis of variance techniques and other statistical tools to analyze the results from the DOE

3.2.4 Control
The Control phase is the final process improvement step and involves:

• Developing a detailed process control plan which includes training materials, updated operating procedures and contingency plans

• Conducting a long-term capability study to validate the new operating conditions

• Documenting the results and lessons learned in the project closure report.

3.3 Six Sigma History
The Six Sigma program is based on the methods developed by Motorola. Much of the training material and experience to date was based on using statistical techniques to improve discrete manufacturing operations. The majority of AlliedSignal’s 300+ sites utilize primarily discrete manufacturing operations. In applying the Six Sigma methodology to a continuous process, a number of modifications had to be incorporated into the methodology.

When conducting gauge R&R studies on a chemical analysis, sampling methods and timing are critical. If the sample port is not properly purged, the sample can be non-representative of the process. Also, if the sample is not promptly brought to the lab and analyzed, it continues to react and its composition can change over time introducing additional error into the gauge study. For a chemical analysis device, the measurement error can vary over the
sample range. To address this problem, a gauge study conducted on a chemical analysis method encompasses three dimensions. Multiple samples are taken that represent the expected process range and a gauge study looks at operator to operator repeatability and reproducibility over the sample range.
4. Six Sigma: Measure

This chapter first discusses the purpose of the Six Sigma Measure phase. Second, the use of tools such as the Team Charter, Process Map and Gauge Study are discussed. Finally, the key findings from the Measure phase are presented.

4.1 Purpose

The purpose of the Measure phase is to:

- define project objectives, metrics and resource requirements using a team charter,
- map the process and identify key process input and output variables,
- evaluate measurement system error using gauge R&R studies,
- establish baseline process performance through a short-term capability study.

4.2 Tools

4.2.1 Team Charter

The team charter identifies the strategic goals, specific objectives and metrics, process boundaries, stakeholders, potential benefits, resources required and team members for a specific project. The first step in a Six Sigma project is preparing the charter with the champion(s) to ensure the team objectives are aligned with strategic business goals and that measures of success are identified up-front. By identifying clear objectives and process boundaries, the charter can help prevent ‘scope creep’ where the team’s objectives keep expanding to incorporate the needs of additional stakeholders. The team charter also identifies key milestones and provides the team with a schedule to ensure steady progress toward the goal. The Nitrite team charter identified the strategic goal of 800 Mppy of hydrox production and the specific team objectives to increase capacity of the nitrite system to support 800 Mppy hydrox by increasing the ammonia burning rate and the nitrite conversion index.
4.2.2 Process Map

After the team charter is completed, the next step is to generate a process map detailing the key process input variables (KPIVs) and key process output variables (KPOVs) for each unit operation. The process map provides two benefits. First, it identifies the KPIVs and KPOVs to be used in the failure modes and effects analysis. Second, creating the process map serves as a knowledge transfer mechanism by allowing team members to gain greater insight by discussing the process step-by-step as a group. The process map indicated that nitrite capacity could be increased through two approaches: decreasing pressure drop to increase throughput or optimizing KPIVs such as temperature and feed composition to increase yield.

Figure 6 - Nitrite Process Map

<table>
<thead>
<tr>
<th>KPIV</th>
<th>KPOV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature - Ambient Air</td>
<td>Flow - Outlet Air</td>
</tr>
<tr>
<td>Humidity - Ambient Air</td>
<td>Discharge Pressure</td>
</tr>
<tr>
<td>Blower Motor AMPS</td>
<td>Temperature - Outlet Air</td>
</tr>
<tr>
<td>Suction Pressure</td>
<td>%Water - Outlet Air</td>
</tr>
<tr>
<td>Flow - Inlet Air</td>
<td>Temperature - Outlet Air</td>
</tr>
<tr>
<td>Flow - Cooling Water</td>
<td></td>
</tr>
<tr>
<td>Temperature - Inlet Air</td>
<td>Flow - Outlet gas</td>
</tr>
<tr>
<td>Temperature - Cooling Water</td>
<td>Temperature - Outlet gas</td>
</tr>
<tr>
<td>Pressure Drop - Air side</td>
<td></td>
</tr>
<tr>
<td>Flow - Inlet Air</td>
<td></td>
</tr>
<tr>
<td>Flow - Ammonia</td>
<td>Flow - Nitrite product</td>
</tr>
<tr>
<td>Temperature - Inlet Air</td>
<td>%Nitrite strength</td>
</tr>
<tr>
<td>%Water - Inlet Air</td>
<td>Nitrite Conversion Index</td>
</tr>
<tr>
<td>Catalyst Efficiency</td>
<td></td>
</tr>
<tr>
<td>Pressure Drop</td>
<td></td>
</tr>
<tr>
<td>Temperature - Bottoms</td>
<td></td>
</tr>
<tr>
<td>%Bicarbonate in Feed</td>
<td></td>
</tr>
<tr>
<td>Pressure Drop</td>
<td></td>
</tr>
</tbody>
</table>
4.2.3 Gauge Studies

In a typical gauge study, a single sample is analyzed multiple times by different operators to determine the measurement system error. A single sample is used since measurement error is assumed to be constant over the sample range. For a chemical analysis lab method, this assumption is not valid since the measurement error may vary over the sample range. To address this issue, when a gauge study is conducted on a chemical analysis method, multiple samples are used which are representative of the normal process range. The repeatability and reproducibility variances are calculated and added together to get the measurement system variance as shown in Figure 7. The measurement system error (%R&R) indicates how much of the total observed variance can be attributed to the measurement system. The %R&R is the ratio of measurement system sigma to total sigma.

To evaluate the nitrite measurement system, a gauge study was conducted on the lab method for analyzing nitrite product strength. Two gauge studies were conducted on the nitrite analysis. During the first gauge study, five different nitrite samples were obtained and analyzed by two different operators in triplicate for a total of 30 sample results. The reproducibility and repeatability (%R&R) of the first gauge study was 49% which meant that 49% of the total process variation was attributed to the measurement system. This result was quite poor as target %R&R is below 10%. Upon investigation, the spectrophotometer was found to have a defective bulb, so the instrument was repaired and calibrated and a second gauge study was conducted.

The second gauge study was conducted using five different samples, each analyzed twice by three different operators for a total of 30 samples as shown below. The results were much better with a %R&R of 5.7% (below the target of 10%).
4.2.4 Short Term Capability Studies

Using the key process input variables identified from the nitrite process map, baseline data was gathered for each of the five nitrite trains for a one month period. Variables included: ammonia burning rate (lbs/hr), nitrite conversion index (%), oxidizer efficiency (%), oxidizer temperature (°C), nitrite product flowrate (gal/min), nitrite product strength (%), tower temperature (°C), tower pressure (psig), and carbonate feed composition.
(%carbon dioxide, %ammonia). Customer specifications are not provided for the nitrite product since it is consumed internally. However, standard operating ranges are used to control some of the process variables in nitrite production.

4.3 Summary of Measure

The key tools used during the Measure phase are the team charter, process map, gauge studies and short-term capability study. The team charter outlines the project objectives and goals and identifies potential risks and benefits. The process map provides a graphical means for identifying key process input and output variables. Evaluating measurement system error is accomplished using the gauge study. Finally, a short-term capability study is used to evaluate initial process capability and establish baseline performance.

The key insights from the Measure phase were:

- The key output variable for measuring project success is the nitrite conversion index.
- The lab analysis for nitrite product strength is robust with low variability.
- The baseline nitrite conversion index was well below the theoretical level.
5. Six Sigma: Analyze

This chapter first discusses the purpose of the Six Sigma Analyze phase. Second, the use of tools such as the failure modes and effects analysis (FMEA) and multi-variable analysis are discussed. Finally, the key findings from the Analyze phase are presented.

5.1 Purpose

The purpose of the Analyze phase is to:

- determine high risk input variables based on the risk priority number (RPN) generated by the FMEA,
- use multi-variable analysis to get an initial look at the relationships between KPIVs and KPOVs,
- use the results from the FMEA and multi-variable analysis to outline an experimental design for the process improvement phase.

5.2 Tools

5.2.1 Failure Modes and Effects Analysis (FMEA)

The FMEA is used to identify high risk input variables based on a risk priority number. Before conducting the FMEA on the nitrite process, a ranking system was first defined. The risk priority number puts a quantitative score on the failure mode based on three factors: the estimated frequency of occurrence, the ability to detect the occurrence and the severity of the occurrence. The RPN is calculated by multiplying the three factors as follows:

\[ RPN = \text{Frequency} \times \text{Detection} \times \text{Severity} \]

To ensure consistent ranking during the FMEA, the team developed a 1 to 5 scale for each of the three factors as shown below. The RPN scale ranges from 1 (no risk to production) to a 125 (major risk to production).
Using this RPN scale, a failure modes and effects analysis was conducted for each step in the nitrite production process. First, a fishbone diagram was generated for each piece of equipment by identifying all the failure modes that could occur. The failure modes were grouped into six generic categories: People, Inputs, Instrumentation, Procedures, Equipment and Environment.

**Figure 9 - Fishbone Diagram for Nitrite Tower**
Once the fishbone diagram was completed, these failure modes were transcribed into a FMEA table and root causes were identified, a risk priority numbers were assigned and recommended actions were recorded.

**Figure 10 - FMEA for Nitrite Tower**

<table>
<thead>
<tr>
<th>Process:</th>
<th>Nitrite Tower</th>
</tr>
</thead>
<tbody>
<tr>
<td>Responsible:</td>
<td>AB Nitrite</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Cause</th>
<th>O C T</th>
<th>Failure Mode</th>
<th>Current Controls</th>
<th>D E T</th>
<th>Effects</th>
<th>S E V</th>
<th>R P N</th>
<th>Recommended Actions</th>
<th>Resp.</th>
</tr>
</thead>
<tbody>
<tr>
<td>High tower bime. temp.</td>
<td>8</td>
<td>Reduced yield</td>
<td>Temperature indicator</td>
<td>4.5</td>
<td>Reduced yield</td>
<td>5</td>
<td>115</td>
<td>REFRIGERATION TEAM</td>
<td>B1</td>
</tr>
<tr>
<td>Low bicarbonate in feed</td>
<td>5</td>
<td>Reduced yield with increased NO3 vapor press</td>
<td>Lab analysis - CO2 (every 5 hrs) / On-line CO2 analyzer</td>
<td>3</td>
<td>Reduced yield</td>
<td>4</td>
<td>60</td>
<td>Conduct test to determine optimum bicarbonate level</td>
<td>C3</td>
</tr>
<tr>
<td>Entrainment spray from tower</td>
<td>5</td>
<td>Nettie yield loss</td>
<td>Damper in line to towers</td>
<td>6</td>
<td>Reduced yield</td>
<td>2</td>
<td>60</td>
<td>Vari losses are reduced with NO3 reduction controls (being installed on each train)</td>
<td>GW</td>
</tr>
<tr>
<td>NH3 burning rate change</td>
<td>6</td>
<td>Carbonate not on feedforward control - instability</td>
<td>(none)</td>
<td>6</td>
<td>Reduced yield</td>
<td>1.5</td>
<td>38</td>
<td>Evaluate control strategy for carbonate feed</td>
<td>WCI</td>
</tr>
<tr>
<td>High NH3 in carbonate feed</td>
<td>3</td>
<td>High NO3/Nettie ratio</td>
<td>Lab analysis - 4PS (every 2 hours)</td>
<td>4</td>
<td>Reduced yield</td>
<td>3</td>
<td>38</td>
<td>CARBONATE TEAM (On-line NO3 analyzer being investigated)</td>
<td>BK</td>
</tr>
<tr>
<td>Low nitrite strength</td>
<td>3</td>
<td>High NO3/Nettie ratio</td>
<td>Lab analysis (every 8 hours)</td>
<td>4</td>
<td>Reduced capacity</td>
<td>3</td>
<td>38</td>
<td>(none)</td>
<td>BK</td>
</tr>
<tr>
<td>Low tower RH</td>
<td>2</td>
<td>Higher bime. temp</td>
<td>Flowmeter indication</td>
<td>3</td>
<td>Reduced yield</td>
<td>2</td>
<td>32</td>
<td>(none)</td>
<td>(none)</td>
</tr>
<tr>
<td>High tower pressure</td>
<td>4</td>
<td>Lower air feed flow</td>
<td>Pressure control loop on tower</td>
<td>1</td>
<td>Reduced capacity</td>
<td>2</td>
<td>32</td>
<td>(none)</td>
<td>(none)</td>
</tr>
<tr>
<td>Low tower pH</td>
<td>1</td>
<td>Exchanger run blown rupture disc</td>
<td>pH indicator &amp; alarm</td>
<td>1.5</td>
<td>Increased downtime</td>
<td>5</td>
<td>8</td>
<td>(none)</td>
<td>(none)</td>
</tr>
<tr>
<td>UV analyzer broken</td>
<td>1.5</td>
<td>Variable nitrite strength</td>
<td>Measurement off-scale / Nettie strength lab analysis (every 4 hrs)</td>
<td>3</td>
<td>Reduced capacity</td>
<td>1.5</td>
<td>7</td>
<td>(none)</td>
<td>(none)</td>
</tr>
<tr>
<td>Carborate feed pumps fail</td>
<td>1</td>
<td>Loss carbonate feed</td>
<td>pH indicator &amp; alarm</td>
<td>3</td>
<td>Increased downtime</td>
<td>2</td>
<td>8</td>
<td>(none)</td>
<td>(none)</td>
</tr>
<tr>
<td>High bicarbonate in feed</td>
<td>2</td>
<td>Plug chill tubes - lower recirc flow</td>
<td>Lab analysis - CO2 (every 2 hrs) / On-line CO2 analyzer</td>
<td>2</td>
<td>Increased downtime</td>
<td>1.5</td>
<td>8</td>
<td>(none)</td>
<td>(none)</td>
</tr>
</tbody>
</table>

Over 100 failure modes were identified and 60 action items were assigned. To prioritize the action items, the failure modes were sorted by RPN with the top quartile being recorded on a separate action item tracking sheet. Many of the action items captured ‘low hanging fruit’ which resulted in significant benefits but required minimal labor and capital. Some of these included: replacing undersized static mixers and check valves to increase air flow, upgrading undersized spray nozzles in the quench coolers to reduce
byproduct formation and adding temperature and pressure compensation to process control software to improve ammonia flowmeter accuracy.

### 5.2.2 Multi-variable Study

Using some of the insight provided by the FMEA, a multi-variable study was then conducted to understand how the KPIVs vary in the production process and to get a initial look at the relationship between KPIVs and KPOVs. During the multi-variable study, the Hopewell plant was in the process of converting each of its nitrite production lines from pneumatic instruments to electronic instruments with a distributed control system (DCS). Two key benefits of converting to DCS are a reduction in process variability (improved Cpk) and the ability to track process information on a continuous basis. Process data such as temperature, pressure and flow are electronically recorded for the production trains that have been converted over to DCS. The production trains that use older, pneumatic instruments require operators to manually record process parameters once an hour. Process data from the pneumatic instruments is subject to a greater degree of measurement error than that from DCS since operators visually estimate process values from paper strip charts and key these estimated values into notebook computers. To ensure data accuracy, data from C-train nitrite was used for the multi-variable study since this train had been converted to DCS.

The multi-variable study involved the following steps:

1. Identify the KPOVs and KPIVs to be analyzed.
2. Obtain historical data for each of these variables and screen the data to remove outliers due to non-routine conditions. Non-routine conditions include time periods when equipment is shutdown or started up or when instruments are calibrated.
3. Using best subsets regression, identify the model with the highest adjusted R-squared.
4. Run a basic regression using the best model identified in step three.
5. Check the residual diagnostic plots to verify model assumptions.
For the multi-variable study, the nitrite conversion index (ConvIndx) was chosen as the output variable while the initial input variables included: calculated bicarbonate strength (Bicarb), carbonate carbon dioxide (CO2), carbonate ammonia (NH3), nitrite tower temperature (BtmTemp), ammonia burning rate (FeedRate) and oxidizer efficiency (OxidEff). Daily averages were obtained for each variable for a three week period.

<table>
<thead>
<tr>
<th>Day</th>
<th>ConvIndx</th>
<th>BicLev</th>
<th>Bicarb</th>
<th>CO2</th>
<th>NH3</th>
<th>BtmTemp</th>
<th>FeedRate</th>
<th>OxidEff</th>
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<tbody>
<tr>
<td>1</td>
<td>85.89</td>
<td>1</td>
<td>3.4</td>
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<td>95</td>
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<td>4</td>
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<td>3599</td>
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<td>3142</td>
<td>77.9</td>
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<td>13</td>
<td>9</td>
<td>13.5</td>
<td>3753</td>
<td>97</td>
</tr>
</tbody>
</table>

Using Minitab statistical software, best subsets regression was conducted. The highest adjusted R-squared (59.2%) and lowest C-P (1.8) was obtained for the model using carbonate carbon dioxide (CO2), tower bottoms temperature (BtmTemp), and oxidizer efficiency (OxidEff).
A basic regression model was run using the three input variables from the best subsets model. Based on the model output, each of the three factors was significant at greater than 93% confidence. Of the three variables, the carbon dioxide in the carbonate feed stream is the only variable that can be directly controlled by the operators. The tower temperature is operated as low as possible by running the refrigeration system at maximum capacity. The desired oxidizer efficiency is to be as close to 100% as possible. However, the efficiency decreases as the catalyst activity in the oxidizer declines over time. When the catalyst efficiency reaches a lower specification limit, the maintenance coordinator shuts down the oxidizer for catalyst replacement.
Figure 13 - Multi-variable Analysis (Basic Regression)

Regression Analysis
The regression equation is
\[ \text{ConvInedx} = -152.09 + 16.0 \text{CO2} - 1.78 \text{BtmTemp} + 0.536 \text{OxidEff} \]

19 cases used 4 cases contain missing values

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coef</th>
<th>Stdev</th>
<th>t-ratio</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-152.09</td>
<td>45.77</td>
<td>-3.32</td>
<td>0.005</td>
</tr>
<tr>
<td>CO2</td>
<td>15.956</td>
<td>3.204</td>
<td>4.98</td>
<td>0.000</td>
</tr>
<tr>
<td>BtmTemp</td>
<td>-1.7845</td>
<td>0.9127</td>
<td>-1.96</td>
<td>0.069</td>
</tr>
<tr>
<td>OxidEff</td>
<td>0.5359</td>
<td>0.2716</td>
<td>1.97</td>
<td>0.067</td>
</tr>
</tbody>
</table>

\[ s = 4.926 \quad R-sq = 66.0\% \quad R-sq(adj) = 59.2\% \]

Analysis of Variance

<table>
<thead>
<tr>
<th>SOURCE</th>
<th>DF</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>p</th>
</tr>
</thead>
<tbody>
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<td>Regression</td>
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<td>707.36</td>
<td>235.79</td>
<td>9.72</td>
<td>0.001</td>
</tr>
<tr>
<td>Error</td>
<td>15</td>
<td>363.96</td>
<td>24.26</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>18</td>
<td>1071.32</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>SOURCE</th>
<th>DF</th>
<th>SEQ SS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO2</td>
<td>1</td>
<td>557.58</td>
</tr>
<tr>
<td>BtmTemp</td>
<td>1</td>
<td>55.29</td>
</tr>
<tr>
<td>OxidEff</td>
<td>1</td>
<td>94.48</td>
</tr>
</tbody>
</table>

Unusual Observations

<table>
<thead>
<tr>
<th>Obs.</th>
<th>CO2</th>
<th>ConvInedx</th>
<th>Fit</th>
<th>Stdev.Fit</th>
<th>Residual</th>
<th>St.Resid</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>12.6</td>
<td>62.59</td>
<td>67.50</td>
<td>4.40</td>
<td>-4.91</td>
<td>-2.22RX</td>
</tr>
</tbody>
</table>

R denotes an obs. with a large st. resid.
X denotes an obs. whose X value gives it large influence.

Residual diagnostic plots were used to check the regression model assumptions. On the normal probability plot, the residuals fell roughly into a straight line so the assumption of a normal distribution is valid. In the time sequence plot, no repeating pattern is apparent. Finally, in the plot of residuals versus fits, no funnel-shape is seen which would indicate a relationship between the residual and the value of the response.
To get a better indication of the relationship between the carbonate CO₂ level and the nitrite conversion index, rational subgrouping was used. First, two of the input variables (carbonate carbon dioxide and ammonia) were transformed into a third variable (bicarbonate strength) using the stoichiometric equation:

\[
\% \text{ Bicarbonate} = 3.6 \times [\% \text{carbon dioxide} - (1.3 \times \% \text{ammonia})]
\]

Second, using the historical data set, three periods were selected where the bicarbonate level was significantly different. Since the three data sets were not equal in size, a general linear model was used for the analysis. The null hypothesis is that the carbonate/bicarbonate ratio has no significant effect on the conversion index. The GLM analysis shows that the null hypothesis can be rejected (p-factor < 0.05) and the ratio does impact conversion index. The multi-variable study indicated that increasing the %bicarbonate in the nitrite feed (raising the %carbon dioxide level while holding the %ammonia level constant) could increase nitrite conversion index.
Figure 15 - Rational Subgrouping: Conversion Index vs. Bicarbonate Level

### General Linear Model
**Factor Levels Values**
- C-BICL3: 3 1 2 3

#### Analysis of Variance for C-CI3
<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-BICL3</td>
<td>2</td>
<td>312.65</td>
<td>312.05</td>
<td>156.02</td>
<td>3.49</td>
<td>0.054</td>
</tr>
<tr>
<td>Error</td>
<td>17</td>
<td>759.47</td>
<td>759.47</td>
<td>44.67</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>1071.52</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

At least one of the yields is different.

### General Linear Model
**Factor Levels Values**
- C-BICL3: 3 1 2 3

#### Analysis of Variance for C-BIC3
<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-BICL3</td>
<td>2</td>
<td>23.649</td>
<td>23.649</td>
<td>11.824</td>
<td>23.82</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>17</td>
<td>8.441</td>
<td>8.441</td>
<td>0.497</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>32.090</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

At least one of the bicarb. levels is different.

C-train Conversion Index vs. Bicarbonate

Conversion Index (%)

3.5% (low) 4.75% (current) 6.0% (high)

Bicarbonate (%)

82
77
72
5.3 Summary of Analyze

The key tools used during the Analyze phase are the failure modes and effects analysis (FMEA) and the multi-variable study. The FMEA identifies high risk input variables by ranking potential failure modes. The FMEA can help identify key process input variables to be evaluated in multi-variable study. The multi-variable study provides insight into potential relationships between input variables and output variables and provides key information needed for effective experimental design.

The key insights from the Analyze phase were:

- Many ‘low hanging fruit’ opportunities were identified and captured using the results from the FMEA.

- The FMEA indicated two approaches to increase nitrite production capacity: improve process yields by optimizing carbonate composition and by increasing ammonia burning capacity by reducing system pressure drop.

- The multi-variable analysis indicated that increasing the %bicarbonate in the nitrite feed stream could increase nitrite conversion index.
6. Six Sigma: Improve

This chapter first discusses the purpose of the Six Sigma Improve phase. Second, the use of tools such as the design of experiments and analysis of variance are discussed. Finally, the key findings from the Improve phase are presented.

6.1 Purpose

The purpose of the Improve phase is to:

- use design of experiments (DOE) to verify the relationship between key process input variables and output variables and to determine sensitivity of the output variables to changes in the input variables,

- use DOE methods to identify the optimum operating window for the key process input variables.

6.2 Tools

6.2.1 Design of Experiments I

Using the results from the multi-variable study, an experimental planning sheet is completed prior to running experiments on the production line. The experimental planning sheet provides information such as the objective of the experiment, start and end dates, output variable(s) measured, potential noise variables, input variables and their specified levels as shown below.
Figure 16 - Bicarbonate Experimental Plan

Date: 08/21

Product: Ammonium Nitrite

Team Leader: C. Mastro           Process(es): Nitrite

Expected Start: 8/28           Expected Completion: 9/11

Problem Statement: To increase nitrite production to support the Hydrox800 goals.

Objective: To evaluate the impact of bicarbonate feed on nitrite conversion index.

List of Experiment Parameters:
Response Nitrite Conversion Index        Type: Quantitative
Unit of Measure 0 to 100%
Specification Baseline= 78%, Goal= +81%

Input Variables Levels Specifications
1. %CO2 in carbonate 3 12.2%, 12.8%, 13.4%
2. %NH3 in carbonate 1 8.8%

Noise Variables Units
1. NH3 feed rate pph
2. Nitrite temperature °C
3. CV efficiency %

Brief Outline of the Experimental Design to be used:
Narrow inference study to evaluate impact of three different bicarbonate feed levels on A & B Nitrite Conversion Index. Trial will be conducted with 2 days at each level and repeated during a second week. Refer to trial plan for schedule.

A trial was conducted on A-train nitrite using the carbonate/bicarbonate ratio (%carbon dioxide) as the input factor, at three different levels, and using the A-train nitrite conversion index as the output variable. The experiment was conducted over a two week period and the three levels were run twice in randomized order to reduce the impact of noise variables. The results of the experiment were not as clean as originally expected.
Due to poor process capability in the upstream carbonate process, the operators had a difficult time maintaining the carbon dioxide levels at the intended targets during the two week experiment. As a result, the two weeks worth of data was screened and periods of relative stability were selected for the subsequent data analysis. Data was analyzed using a 35 hour period at the high carbon dioxide level and a 28 hour period at the low carbon dioxide level.

The two sample populations were analyzed using two sample t-test. The null hypothesis was that the carbon dioxide level has no significant impact on nitrite conversion index. The t-test yielded a T-value of 3.58 (p-factor=0.0007) which meant the null hypothesis could be rejected at greater than a 99% confidence level. As seen in the interval plot, the conversion index increases as the carbon dioxide level is increased from the low level to the high level.
**Figure 17 - Bicarbonate Experiment #1 Results**

Two Sample T-Test and Confidence Interval

**Null Hypothesis:** The conversion index at the two different CO2 levels is the same.

**Twosample T for ACI_LAB**

<table>
<thead>
<tr>
<th>CO2 Level</th>
<th>N</th>
<th>Mean</th>
<th>StDev</th>
<th>SE Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>13.4</td>
<td>36</td>
<td>77.90</td>
<td>2.93</td>
<td>0.49</td>
</tr>
<tr>
<td>12.4</td>
<td>29</td>
<td>72.07</td>
<td>4.07</td>
<td>0.76</td>
</tr>
</tbody>
</table>

95% C.I. for mu 13.4 - mu 12.4: ( 4.02, 7.64)

T-Test mu 13.4 = mu 12.4 (vs not =): T= 6.48  P=0.0000  DF= 49

**Reject Null Hypothesis:** The two conversion indexes are different (p < 0.05)

Three key recommendations from the trial were:

- Conduct a second trial by running the higher carbon dioxide level on one train to evaluate the long-term effects and verify the benefits.

- Reduce variability (improve Cpk) in carbon dioxide composition in the carbonate tower using the on-line carbon dioxide analyzers.
- Investigate methods to obtain higher carbon dioxide concentrations in the carbonate towers such as improved tower packing, improved circulation cooling and increased carbon dioxide header pressure.

6.2.2 Design of Experiments II

Based on the results for the initial bicarbonate experiment, a second experiment was run on another train to verify the benefits and to identify any potential long-term risks. Prior to conducting the second experiment, two of the recommendations from the first experiment had to be completed. First, to support operating the carbonate towers at a higher carbon dioxide concentration, the carbonate tower packing was replaced to reduce pressure drop and improve carbon dioxide absorption. Second, to reduce carbon dioxide variability in the carbonate product, on-line CO₂ analyzers were installed on each of the carbonate towers. Once these two action items had been completed, a second experiment was conducted by operating one nitrite tower at a higher carbon dioxide feed concentration. As seen in the first experiment, maintaining the carbon dioxide feed within the target range proved difficult, and carbon dioxide Cpk was quite poor. However, during the periods of consistently higher carbon dioxide concentration, the nitrite conversion index increased. As shown in the box plot, at the higher carbon dioxide concentration, the conversion index increased by 2-3% as expected.
During the period of higher carbon dioxide levels, no ill effects such as carbonate or nitrite tower plugging were observed. The key observation from the second experiment is that raising the carbon dioxide level can result in a 2-3% increase in conversion index with no major negative impacts. Two key recommendations from the trial were:

- Modify the standard operating procedures and conduct operator training classes on raising the carbon dioxide targets for the carbonate towers.
• Address the sources of variability in carbon dioxide control to improve process capability of the nitrite feed stream.

6.3 Summary of Improve

The key tools used during the Improve phase are the design of experiments (DOE) and data analysis techniques (t-tests, ANOVA). Experiments are conducted to identify the relationship between key process input variables and output variables and to determine the optimum operating window. The experimental data is analyzed using techniques such as ANOVA to verify if the observed effects are statistically significant. Analyzing experimental data can also identify the key variables that should be controlled.

The key insights from the Improve phase were:

• Increasing the carbon dioxide concentration in the carbonate feed to the nitrite towers can improve nitrite conversion index.

• To fully capture these benefits, process capability for carbon dioxide in the carbonate towers must be improved.
7. Six Sigma: Control

This chapter first discusses the purpose of the Six Sigma Control phase. Second, the use of tools such as the operator training, instrumentation upgrades and long-term capability studies are discussed. Finally, the key findings from the Control phase are presented.

7.1 Purpose

The purpose of the Control phase is to:

- develop, document and implement process support systems (training, operating procedures, control system improvements) to institutionalize the proposed changes,
- verify the process improvement benefits through a long-term capability study,
- document the results in a final report to record the process improvement effort and facilitate knowledge transfer.

7.2 Tools

7.2.1 Process Support Systems

The Hopewell plant has a standard Management of Change (MOC) process in order to comply with federal Process Safety Management regulations. This MOC process was followed in implementing the process changes. Training classes were held for each operating shift in order to explain the process change, the reasons for the change and to address operator concerns. Standard operating procedures were updated to reflect the higher carbon dioxide targets.

To facilitate tracking the nitrite conversion index over time, special process data tags were programmed into the process information management system which then calculated the nitrite conversion index and displayed the results to the operators on a continuous basis.
Once the feed valve problem was addressed, the second step was to reduce the variability introduced by frequent setpoint changes. The carbon dioxide feed flowrate to each carbonate tower is controlled directly by on-line CO₂ analyzers. The operators also pull carbonate product samples for lab analysis every two hours. Some operators, trusting the lab results over the analyzer, will change the carbon dioxide setpoint every two hours based on the lab results.

To quantify the variance between the lab analysis and the analyzer reading for each train, a short-term capability study was conducted for two days on A, B and C-trains where the analyzer setpoints were held constant. During this period, the process capability improved on A and B-trains.
To reduce variability in the carbonate system, these two problems were addressed separately. When the carbon dioxide feed valves are greater than 90% open, the operators effectively lose one of their key control variables. Operating with the carbon dioxide valve in the fully open position, the carbon dioxide feed rate fluctuates significantly and results in increased process variability. To reduce this variability, the area supervisor issued a production memo to the operators. This memo set a maximum limit of 90% open for the carbon dioxide feed valve. Due to the valve design, above the 90% open position the control valve can no longer effectively control the carbon dioxide gas flow. Operating the valves below 90% open improved process capability as shown below.
7.2.2 Long-term capability study

After the higher carbon dioxide targets were implemented on each nitrite train, a long-term capability study was conducted to verify the benefits and re-evaluate process capability. As seen in the table, the process capability for carbon dioxide control in the carbonate towers remains quite poor with Cpk between 0.1 and 0.4.

**Figure 19 - Carbonate Baseline Process Capability (March 1997)**

<table>
<thead>
<tr>
<th>Description</th>
<th>A-train</th>
<th>B-train</th>
<th>C-train</th>
<th>D-train</th>
<th>E-train</th>
</tr>
</thead>
<tbody>
<tr>
<td>%CO₂ (analyzer) Specification</td>
<td></td>
<td></td>
<td>13.1 to 13.7%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>13.5</td>
<td>13.3</td>
<td>13.4</td>
<td>13.3</td>
<td>13.6</td>
</tr>
<tr>
<td>Standard Dev.</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>Cpk</td>
<td>0.26</td>
<td>0.24</td>
<td>0.42</td>
<td>0.21</td>
<td>0.13</td>
</tr>
<tr>
<td>% of time control valve fully open</td>
<td>13%</td>
<td>26%</td>
<td>12%</td>
<td>31%</td>
<td>19%</td>
</tr>
</tbody>
</table>

To address the sources of the carbon dioxide variability, a causal loop diagram was generated based on input from production engineers, process engineers and operators. As seen in the diagram below, carbon dioxide variability is caused by two main factors: using the carbonate towers to absorb excess ammonia which leads to operating the carbon dioxide feed valve wide open and by frequent setpoint changes based on fluctuating lab results.
During this stable trial period, the process capability on C-train was unacceptably low (Cpk=-0.99) due to a low carbon dioxide level. This had been a recurring problem on C-train and was due to the lab analysis reading consistently higher than the on-line analyzer. The hypothesis was that the problem was caused by sample contamination from the long sample tubing on C-train. If the sample tubing is not purged prior to pulling each sample, some precipitated material in the tubing may lead to a sample with higher than expected carbon dioxide levels. To address this problem, the sample tubing will be modified on C-train to reduce sample contamination.

7.3 Summary of Control

The Control phase is one of the most important process improvement steps and is often overlooked. Key tools used during the Control phase are the control plan and the long-term capability study. The control plan ensures that the process support systems (training, procedures, and instrumentation) are in place to support the proposed process changes. The long-term capability study verifies the benefits of the process change and identifies areas for further improvement.

The key insights from the Control phase were:

- Operating training and constant communication were crucial for successful implementation of the proposed process changes.

- Failure to address process capability problems in upstream operations prevented obtaining the full benefits from the nitrite process changes.
8. Conclusion/Recommendations

This chapter first provides a brief summary of the results of the internship project along with some of the key insights from using the Six Sigma methodology. Second, a number of recommendations are presented for future improvement efforts (in keeping with the spirit of continuous improvement).

8.1 Conclusions

The benefits from the increasing the carbon dioxide levels as well as from action items generated from the FMEA are summarized below. Using the Six Sigma methodology, the team increased Area 9 capacity by over 8 Mppy of equivalent lactam, worth approximately $3.1 million per year.

![Figure 23- Nitrite Team Benefits](image)

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
<th>Lactam Gain</th>
<th>Benefit</th>
<th>Capital Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Replace A train static mixer &amp; check valve</td>
<td>1.3 M ppy</td>
<td>$550K/yr</td>
<td>$24K</td>
</tr>
<tr>
<td>2</td>
<td>Replace B train static mixer &amp; check valve</td>
<td>1.3 M ppy</td>
<td>$550K/yr</td>
<td>$24K</td>
</tr>
<tr>
<td>3</td>
<td>Replace A train carbon</td>
<td>1.5 M ppy</td>
<td>$660K/yr</td>
<td>($45K exp)</td>
</tr>
<tr>
<td>4</td>
<td>Replace C train carbon</td>
<td>0.9 M ppy</td>
<td>$380 K/yr</td>
<td>($45K exp)</td>
</tr>
<tr>
<td>5</td>
<td>Replace E train axiblower air cooler</td>
<td>0.2 M ppy</td>
<td>$100 K/yr</td>
<td>$100K</td>
</tr>
<tr>
<td>6</td>
<td>Modify quench cooler spray on C-train</td>
<td>0.07 M ppy</td>
<td>$30 K/yr</td>
<td>$15K</td>
</tr>
<tr>
<td>7</td>
<td>Modify quench cooler spray on D-train</td>
<td>0.06 M ppy</td>
<td>$25 K/yr</td>
<td>$15K</td>
</tr>
<tr>
<td>8</td>
<td>Increase %CO2 in carbonate (13.4%)</td>
<td>3.1 M ppy</td>
<td>$1300 K/yr</td>
<td>$0</td>
</tr>
<tr>
<td></td>
<td>TOTAL</td>
<td>8.3 M ppy</td>
<td>$3.1 M/yr</td>
<td>$320K</td>
</tr>
</tbody>
</table>

This internship project provided many learning opportunities. Some of these include:

- Ensuring the upstream process is capable before optimizing downstream processes. Experimentation and data analysis on process output variables becomes difficult if there is a large variability in the process input variables.

- The failure modes and effects analysis (FMEA) is a powerful tool for identifying and prioritizing process improvement opportunities. Using the structured FMEA
approach allows a more thorough analysis of a process than just brainstorming improvement opportunities.

- Learning to integrate tools such as causal loop modeling with the Six Sigma method help promote a systems view and reduce local optimization. Using a causal loop diagram to show undesirable effects caused by certain operating conditions helped change those operating conditions.

- Although the gauge study provides an indication of the variance contributed by the measurement system, it can overlook a significant source of variance; sampling error [Barrentine, 1991]. In the carbonate process, we found that although the lab analysis had adequate repeatability and reproducibility, the error between the on-line analysis and the lab analysis was still significant due to poorly designed sample ports.

- During the data analysis phase, more data is not always better. With day to day variability in the plant operations often caused by special-cause variation such as equipment downtime, analysis of large data sets often become meaningless due to the noise factors. Using smaller data sets over periods where special-cause variation could be identified provided greater insight for process improvements.

- Consider the Theory of Constraints when claiming capacity benefits. First, increased capacity in a specific production area can be valued as additional production only during the period that the area is a bottleneck. If the area is a bottleneck operation six months out of the year, benefits can only be claimed for six months. Second, the excess capacity between the bottleneck and the next constraint must be sufficient to handle the capacity increase or the bottleneck is shifted.

8.2 Recommendations

In keeping with the spirit of continuous improvement, a number of observations and recommendations are made for future improvements. These are:

- Disulfonate Optimization. A process capability analysis conducted on the downstream disulfonate process identified a low Cpk on excess nitrite. Due to limitations on the
existing control strategy, the disulfonate towers are operated using excess nitrite feed. Operating the towers with excess nitrite feed reduces process yields and ultimately, lactam production. Shifting from a single loop control strategy to a multi-loop control strategy could reduce standard deviation for excess nitrite control by over 50%, allowing a lower excess nitrite target. Standard benefits calculations show this optimization could provide an additional $2.5 million/year of lactam.

- **Education and Communication.** One of the key factors in institutionalizing process change is effective communication and education. By incorporating process changes into the existing area training programs, operators can learn the reasons for the process change as well as what the change encompasses. Without an understanding for the reasons driving the change, operators often return to previous operating conditions if problems arise.

### 8.3 Closing Comments

I would like to thank AlliedSignal, and in particular the Hopewell plant for giving me this internship opportunity. This success of this process improvement project can be attributed to the dedicated efforts of many of the team members. I would like to extend my gratitude for all the support provided during this project by the Hopewell plant.
9. References


Box, Hunter, Hunter. Practical Statistics for Experimenters.


