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# Lab-data Based Process Control- Comparing SPC and Automatic Control

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<i>Title and subtitle</i> Lab-data Based Process Control-Comparing SPC and Automatic Control. (Reglering med hjälp av labdata-jämförelse av SPC och regler teknik.			
<i>Abstract</i> <p>In many process industries samples are taken and analyzed in laboratories to monitor and ensure proper production conditions. If the analysis result is not within specification limits measures are taken to correct errors. It has proved to be hard to take the appropriate measures. The problem description fits into the confines of control theory and the aim is to make a fairly simple solution to a very complex problem. For example, a standard proportional-integral-controller block in the control system can not be used. As a case study, this project tries to stabilize and control the content of residual formaldehyde in a Neo production plant situated in Perstorp. The content is affecting the yield of the two main reactants in the reaction and therefore important in an economical aspect. The thesis answers the following questions:</p> <ul style="list-style-type: none"> <li>• What is statistical process control and is it the solution to the case study?</li> <li>• What are the similarities and parallels between classic automatic control and statistical process control?</li> </ul> <p>The theory of statistical process control is based on defining processes to be in statistical control or out of statistical control. The founder of the theory, Dr Shewhart, puts it into words like this: <i>While every process displays variation, some processes display controlled variation, while others display uncontrolled variation.</i> Many tools are developed to signal when a process changes from displaying in-control properties to out-of-control properties. Quite a few of those tools are described in the statistical process control chapter. A chapter recalling some useful parts of the automatic control theory is followed by a chapter describing tools and theories in the borderland between statistical process control and classic automatic control. The two theories contradict each other in some aspects. While statistical process control always try to identify the actual cause of production malfunction, automatic control can in some cases hide the actual cause by manipulating the control parameter. Tools coping with situations like that have been developed and are described in chapter 7: Comparing and Combining SPC and Automatic Control. Analyzing the whole process in depth is too complicated and had to be limited into focusing on the parameters dominating the variations in the residual formaldehyde content. Extensive data analysis pointed out the formaldehyde loading not to be sufficiently accurate. A control system consisting of both feed-forward and feedback terms is designed. Only the feed forward term could be tested in reality. The semi-manual Excel based solution showed good results in time periods when used correctly.</p> <p>The conclusion is that statistical process control is not the solution to the case study problem but nevertheless interesting, especially for its diagnostic tools. The theory should not be considered an alternative to automatic control but as a good complement. The project shows that it is definitely possible to stabilize and control the residual formaldehyde content. In order to do this an automatic implementation is needed.</p>			
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### **3 Problem Description**

The purpose of this master thesis is to improve the yield of reactants in a semi batch reaction process by developing methods for systematic and automatic correction of operating parameters based on lab data.

Some lab results are used for adjustments in the process, especially for changes in set points and recipe. In many cases, the adjustments were previously made in an ad-hoc manner, and were not based on any form of calculations. In this project we study which improvements that are possible to achieve by using systematic adjustments, and also suggest how improvements should be implemented on a real process within Perstorp AB.

Several methods exist for analysing lab data, including 6-sigma methods and statistical process control. In the thesis, methods from statistical process control are compared with methods from the theory of automatic control.



## 4 Working Procedure

The purpose of this chapter is to introduce a systematic way of identifying unwanted and unknown sources of process variation. There are many potential application areas, not only reactor processes in this thesis. The procedure for minimizing variations in a process is suggested below step by step.

### 1. Data collection

The first step is to collect existing data and evaluate if any more information is needed. Make sure that all necessary data will be logged for future work. AspenTech Information Plus 21 is a good tool for data logging and analyzing even though not all data are automatically available here. An example of data that is not always available in IP21 is results from manual lab analyses.

### 2. Investigate the variance of lab analysis method

When the data is available it is important to validate the correctness of the information. The lab method should be examined in at least two aspects. First of all, it needs to be investigated if the analysis method is appropriate. This could be done by analyzing the same sample with different methods and comparing the results. Another important aspect is the standard deviation of the analysis method. This parameter can be estimated by doing many analyses on the same sample. Lab methods usually have upper and lower detection limits and the measured results should not be too close to these limits. Offsets and time drifts should also be studied.

### 3. Investigate the variance of sample taking

The procedure for taking the samples to the laboratory also needs to be examined in order to be able to explain process variations and deviations rather than operator errors.

### 4. Perform extended sampling and analyses

The aim of the working procedure is to minimize all variations and when trying to do that as much data as possible is needed. Therefore a testing period with extra samples is suggested. It needs to be studied if the process varies in between normal sampling times. The temporary sampling rate differs from application to application but the number of new data samples needs to be big, maybe 50 or more. From the data obtained obvious time constants and dynamics should be looked for.

### 5. Model the process based on mass balance and molar ratio

A process model based on mass balance should be developed. The model could be validated with real data but bear in mind that the mass balance model does not take into account dynamics such as time delays and feed back terms of the real process.

### 6. Make a sensitivity analysis

The mass-balance based process model should be used to perform a sensitivity analysis on the process. This means to add equal theoretical disturbances on all input parameters separately and study how much they affect the theoretical process value.

**7. Investigate the variance of input parameters**

From collected data input variances should be examined. Combining the knowledge of input variations and the result of the sensitivity analysis one has a good base for making priorities on where to start improving.

**8. Investigate reasons for variation**

It is time to study the real process and especially the reasons for variations in input parameters. Examples are potential uncertainties in weighing procedures, illogically placed sensors and mistuned controllers.

**9. Define measures**

Problems can be solved in different ways and with different degrees of success. One needs to consider what measures that could be taken and compare different solutions with aspect to costs and potentials. Reconstructions, controller tuning or reprogramming etc could be considered.

**10. Use automatic control**

When appropriate actions have been taken, the remaining sources of variances should be examined again. The actions could or should have changed the properties of the system and new priorities can be made. As a next step the opportunities of control theory should be considered. Measured input deviations can be compensated for by feed forward terms and there could also be situations present where new automatic controllers could be implemented. Of course, existing controllers should be properly tuned.

**11. Use statistical process control**

After performing the 10 steps above successfully, the process hopefully displays an process value pattern that indicates in-statistical-control properties. Different SPC tools could then be used to signal whether the process remains in control or an assignable cause becomes present. The concept of statistical process control including the notions *statistical control* and *assignable causes* is explained in the SPC chapter.

## 5 Statistical Process Control

The purpose of this chapter is to give an overview of the theory of Statistical Process Control. Only a few parts will actually be used in practice in this master thesis. One aspect of the thesis was to investigate whether SPC was something that was directly or indirectly applicable in a process industry subject to automatic control. For this reason, the SPC chapter is as extensive as it is.

### 5.1 Introduction

Ever since the industrial revolution people have tried to manufacture two or more identical objects. Since the same time people have been coming to the same conclusion: It is very hard, if not impossible, to manufacture series of identical objects. One of the engineers working at Bell Laboratories, Dr Shewhart, put a lot of effort into thinking about this conclusion. He accepted it and started to analyse different production lines and processes. From this study *The Shewhart Concept of Variation* was formed [1]. In words:

*While every process displays variation,  
some processes display controlled variation,  
while others display uncontrolled variation.*

Based on these wise words Dr Shewhart founded the theory of *Statistical Process Control*.

### 5.2 Assignable and Chance Causes

According to Shewhart, a controlled variation is characterized by a stable and consistent pattern of variation over time. The cause of such a variation is called a *chance cause*. Errors occurring when a control chart signals an assignable cause only due to chance causes are called *Type I* errors. An uncontrolled variation is on the other hand characterized by a pattern of variation that changes over time [1]. A *Type II* error comes from a test concluding a process to be in control when it is really out of control. The mentioned test could be called hypothesis testing or control charting. The tested hypothesis  $H_0$  is that  $y(t)$  does not belong to the same distribution as the observations  $\{y(1), y(2), \dots, y(N)\}$  made from an in-control process. Table 5.1 shows how the two types of errors and the process properties are related to hypothesis testing.

Process Property Hypothesis	In statistical control	Out of statistical control
Accept $H_0$	-	Type II error
Reject $H_0$	Type I error	-

Table 5.1: Relations between process properties, type of faults and hypothesis testing.

The variations due to chance causes are those who depend on variations in a properly working machine, for example a small variation in incoming raw material. On the other hand, a variation caused by an assignable cause indicates that something is wrong with the process or machine, for example a worn out drill. An assignable cause can have devastating effects on the process but on the good side, if it is identified it can be dealt with. One of statistical process control's major subjects is to detect assignable causes. To be able to do this there is a need for developing tools that separate chance causes from the assignable ones.

### **5.3 Motivation for Detecting Assignable Causes and Using Control Charts**

A process that is not producing any defective products due to assignable causes is said to be in statistical control. A process that displays lack of control due to assignable causes is said to be out of control. These two states for a process are fairly obvious but note that a process can be dominated by assignable causes but still produce products within specification and control limits. In [1] a process is said to be in one of four possible states, described below.

#### **5.3.1 Process States**

##### **The Ideal State**

When a process is in the ideal state it will produce 100 % conforming units within control and specification limits. It will keep doing this as long as it is in control. It is found empirically that it is hard to get a process to this state and very easy to get it out of it.

##### **The Threshold State**

A process in the threshold state shows signs of being in statistical control but will still be producing defective products. This is because the specification limits are too narrow compared to the process ability to produce conforming products. There are two ways of getting the process to the ideal state: Changing the specification limits or modifying and improve the process. The first option will hardly be accepted by many customers and the latter will demand investments. Even though it will probably cost money to improve the process it is usually the best solution.

##### **The brink of chaos**

The first two states are fairly easy to imagine in a real situation. The definition of a process in the state of *brink of chaos* is when a state is out of control but is still producing 100 % acceptable units. This can be hard to imagine but the explanation is logical. If the specification limits are generous the process can still seem to be in control if it is not examined thoroughly. This calls for some other way to detect when the process is out of control. Control Charts would detect that it is out of control. But, if the process is still producing conforming products, why is there a need for a change? A process on the brink of chaos is controlled by the assignable causes and is not predictable. It can suddenly start making defect units and at that moment the process can have several assignable causes. This leads to a situation where process engineers need to detect and repair lots of damages. A long involuntarily stop in the production line can be very costly.

## The State of Chaos

The state of chaos is as the name implies out of statistical control. It will be producing nonconforming units and the percentage of nonconforming units will vary over time. The process is not predictable. In this state improvements are usually short lived because other assignable causes change the process over time. All the assignable causes need to be removed before the process will be in control. A control chart might come in handy here.

### 5.3.2 The Cycle of Despair

Imagine a process in the state of chaos. A common case is to let an engineer remove some assignable causes and push the process to brink of chaos. The engineer starts to work on another project and simultaneously the remaining assignable causes will be a force back towards the state of chaos. This negative cycle is called *the cycle of despair* and will keep on turning until a more systematic approach is done. The good and systematic approach will be helped by properly designed control charts.

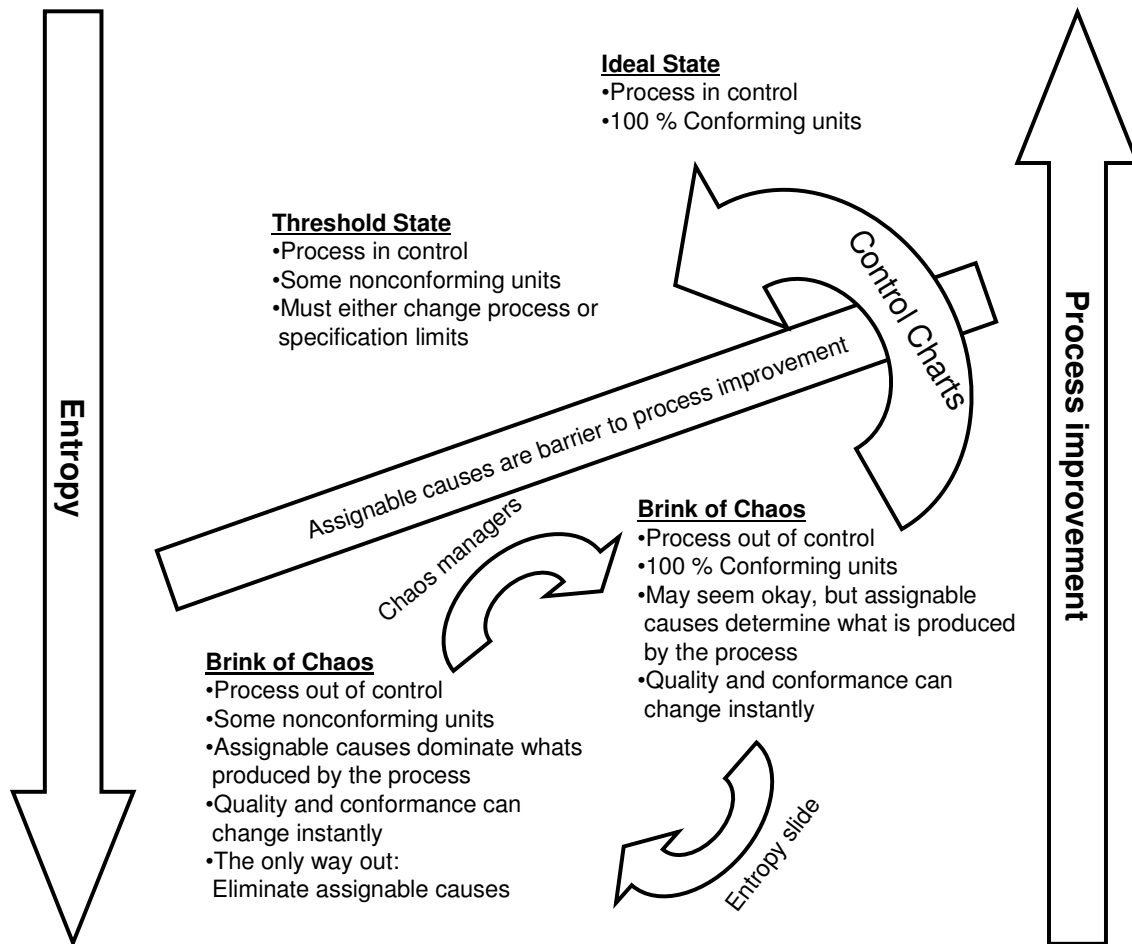


Figure 5.1: A graphical chart showing the cycle of despair.

## 5.4 Control Charts

A control chart is a tool for detecting assignable causes. There are different types for detecting different types of assignable causes. In general, when applying control charts on a process in statistical control it could be looked upon as continuous hypothesis testing.

### 5.4.1 Shewhart Chart

The Shewhart chart is named after its developer, Dr Shewhart. Another name for the same chart(s) is the  $\bar{X}$  and  $\bar{R}$  chart. The Shewhart chart consists of two sub charts, one for monitoring mean value of groups of data and one for monitoring the range within the groups of data. The charts also contain centrelines and control limits.

The groups of data mentioned above are collected from measurements of finished products. An example could be measuring ten products of an all day running process each morning and form a subgroup of these measurements.

#### The $\bar{X}$ Chart

Every point in the  $\bar{X}$  chart corresponds to the mean value of data collected from the process

$$\bar{x} = \frac{x_1 + x_2 + \dots + x_n}{n} \quad (5.1)$$

The size of the group,  $n$ , is a design parameter for the chart constructor and varies heavily from application to application. In the example below the size of the groups are  $n=3$ .

A common assignable cause is a shift in the process mean and therefore it is logical to draw a line in the chart where the process has its mean value. An unbiased estimation of the process mean is

$$\bar{\bar{x}} = \frac{\bar{x}_1 + \bar{x}_2 + \dots + \bar{x}_m}{m} \quad (5.2)$$

It is important to point out that the process mean must be calculated on data collected from a process in statistical control.

In order to make the control chart effective it is crucial to have limits indicating when there is reason to believe the process is out of control. For this reason the upper and lower control limits are introduced. The equations defining these limits are

$$UCL = \mu + Z_{\alpha/2} \sigma_{\bar{x}} \quad (5.3)$$

$$LCL = \mu - Z_{\alpha/2} \sigma_{\bar{x}} \quad (5.4)$$

$\mu$  is the process mean value and can therefore be estimated by  $\bar{\bar{x}}$  and  $\sigma_{\bar{x}}$  is the estimated standard deviation of the subgroup which can be estimated from the process standard deviation  $\sigma$ .

$$\sigma_x = \frac{\sigma}{\sqrt{n}} \quad (5.5)$$

$Z_{\alpha/2}$  in (5.3) and (5.4) comes originally from the theory statistics and especially the normally distributed density function. It is defined by being the  $Z$  that fulfils

$$\int_{-\infty}^{Z_{\alpha/2}} f(x)dx = 1 - \alpha \quad (5.6)$$

where  $f(x)$  is the density function of a normally distributed variable, defined by

$$f(x) = \frac{1}{\sqrt{2\pi}\sigma} e^{-\frac{1}{2}\left(\frac{x}{\sigma}\right)^2} \quad (5.7)$$

and  $q$  is a constant.

It is common to use  $Z_{\alpha/2} = 3\sigma$  and from this the well known *three sigma* discussion is formed.

There is one remaining unknown parameter and that is the process standard deviation  $\sigma$ . A common way to estimate this parameter is to use the range  $R$  of the subgroups  $\bar{x}$

$$R = x_{\max} - x_{\min} \quad (5.8)$$

$$\bar{R} = \frac{R_1 + R_2 + \dots + R_m}{m} \quad (5.9)$$

$$\hat{\sigma} = \frac{\bar{R}}{d_2} \quad (5.10)$$

If the data is normally distributed  $E[R]$  will be proportional to  $\sigma$ . According to [2] this proportional constant is  $d_2$  which is tabulated in all SPC books. From this fact the unbiased  $\hat{\sigma}$  is obtained by dividing  $\bar{R}$  with  $d_2$ .

Based on this it is possible to develop the equations for the control limits as functions of process mean value  $\bar{\bar{x}}$  and the range of  $\bar{x}$ ,  $R$ .

$$UCL = \bar{\bar{x}} + \frac{3}{d_2 \cdot \sqrt{n}} \cdot \bar{R} = \bar{\bar{x}} + A_2 \bar{R} \quad (5.11)$$

$$LCL = \bar{\bar{x}} - \frac{3}{d_2 \cdot \sqrt{n}} \cdot \bar{R} = \bar{\bar{x}} - A_2 \bar{R} \quad (5.12)$$

$A_2$  is often tabulated for different values of  $n$ .

For clarification, control limits are calculated based on the process ability to produce conforming units. This has nothing to do with specification limits that are based on accepted variance in process value. A quote including the two concepts is called process capability and is described in chapter 5.7.

It is now time for looking at a  $\bar{X}$  chart.

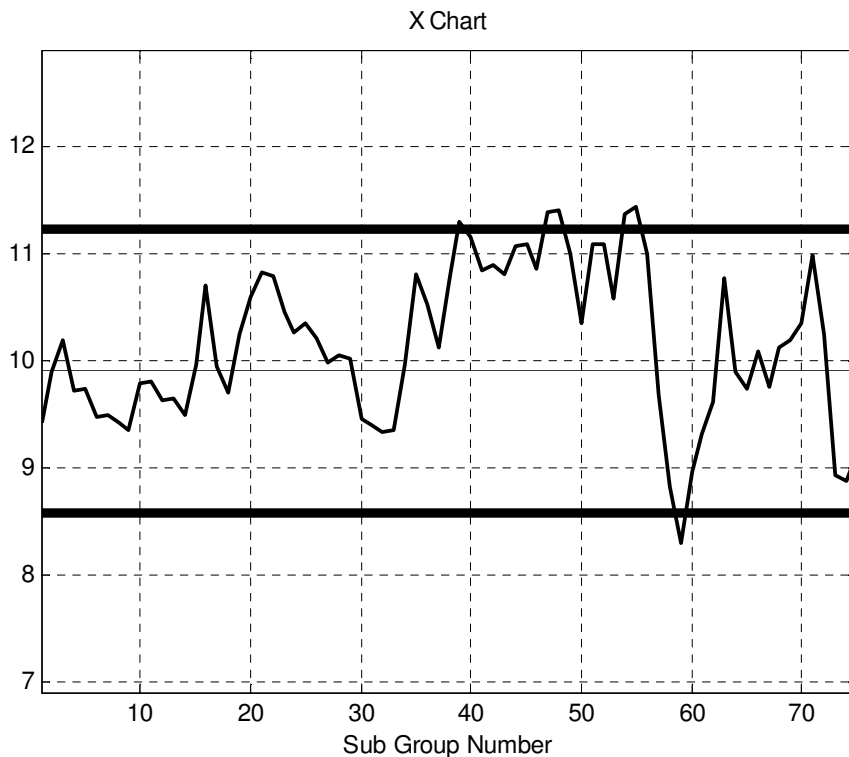


Figure 5.2: The Shewhart  $\bar{X}$  Chart. The two bold lines are the upper and lower control limits while the straight thinner straight line is the mean value.

The dataset was created in Matlab with the following syntax

```
x = [10+randn(1,100) 11+randn(1,50) 10+3*randn(1,80)];
shewhart(x,3)
```

The elements of vector  $x$  are simulated values of a process observations. The first 100 elements are generated from a normally distributed randomized number table with a mean value of ten and standard deviation of one. The next two sub sequences simulates two assignable causes, first a shift of process mean and then a shift in standard deviation. The last



command sends the vector  $x$  into a function called Shewhart that returns a control chart containing the elements of  $x$ . The 3 defines the size of the sub groups.

The red lines are the upper and lower control limits. When trying to detect an assignable cause, sensitizing rules are used. The first rule fires if an observation falls outside the control limits. In figure 5.2 an assignable cause is detected in group 39. More sensitizing rules are discussed in section 5.5.

The discussion about control limits in the  $\bar{X}$  chart can be extended into dividing the area between the control limits in zones. Zone 1 is the area between one standard deviation above the centreline and one standard deviation below. Zone two actually consists of two zones, the ones between one and two standard deviations above and below the centreline. The last zone, three, is the zones between zone two and the upper respectively lower control limit.

### The $\bar{R}$ Chart

The range of a group was defined above as subtracting the minimum value from the maximum value. In the R chart the ranges of each group of data is plotted. Similar to the  $\bar{X}$  chart it also has centre lines and control limits. The centre line is logically defined as the mean value of all ranges. The control limits are defined as

$$UCL = D_4 \bar{R} \quad (5.13)$$

$$LCL = D_3 \bar{R} \quad (5.14)$$

$D_3$  and  $D_4$  are usually tabulated and are functions of the group size  $n$ .

In most cases the lower control limit is set to 0. This is due to the fact that it is almost never a bad thing to have a low range within subgroups of data.

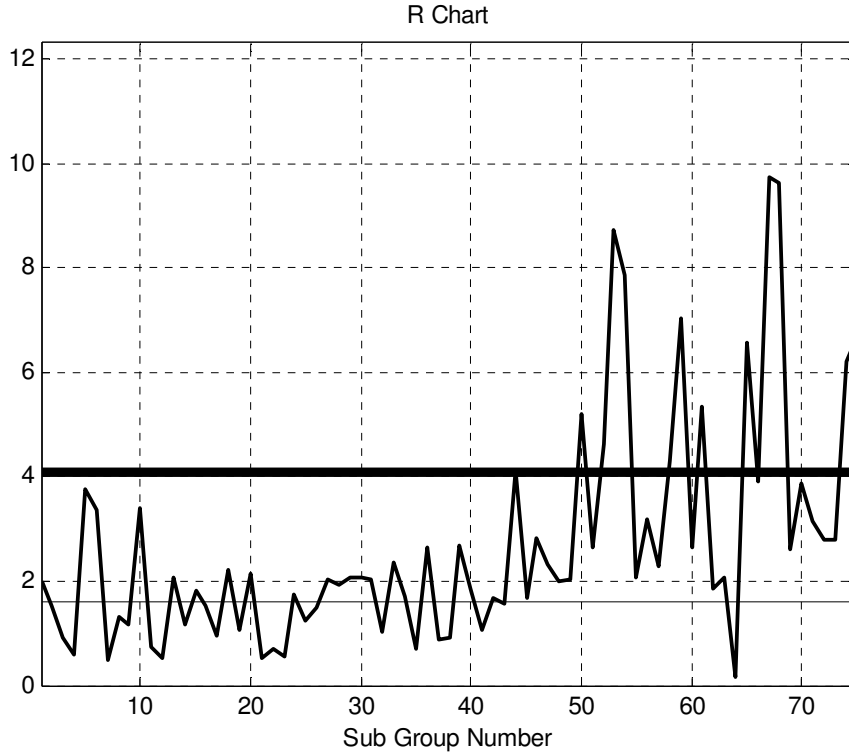


Figure 5.3: The Shewhart R Chart. The chart is created with the same code as in figure 5.2. The bold line is the upper control limit while the thin straight line is the mean of all sub groups from the in-control process.

### Detecting assignable causes in Shewhart charts

The purpose of the chart is to obtain an instrument for detecting when a process is out of statistic control. It is time to show how it is done. There are several rules for detecting assignable causes in the  $\bar{X}$  and  $\bar{R}$  charts that soon will be presented but first some general guidelines for working with the charts. It is important to remember that the  $\bar{X}$  chart should be looked upon as a chart that monitors the mean value of the process while the  $\bar{R}$  chart monitors the standard deviation of the process. The constant control limits in the  $\bar{X}$  chart are calculated based on previous  $\bar{R}$  values. Therefore it is essential to never try to examine the  $\bar{X}$  chart when the  $\bar{R}$  chart displays out of control behaviour.

#### 5.4.2 The Moving Average Control Chart

The Moving Average (MA) Control Chart is an extension to the Shewhart  $\bar{X}$  control chart. Instead of collecting fixed sized groups of data one tries to gather observations periodically and form groups of these data. The big difference from Shewhart's Chart is that the number of points in the MA control chart equals the number of observations instead of number of observations divided by the group size.

If the moving group size is  $w$ , the moving average at time  $i$  is calculated as

$$M_i = \frac{x_i + x_{i-1} + \dots + x_{i-w+1}}{w} \quad (5.15)$$

In words, every  $M$  is calculated from current and the last  $w$  observations. Every control chart needs at least one control limit and suitable upper and lower control limits are calculated as in the Shewhart  $X$  chart, but with the exception that the standard deviation for  $M$  is

$$\sigma_m = \frac{\sigma}{\sqrt{w}} \quad (5.16)$$

hence

$$UCL = \mu_0 + \frac{3\hat{\sigma}}{\sqrt{w}} \quad (5.17)$$

$$LCL = \mu_0 - \frac{3\hat{\sigma}}{\sqrt{w}} \quad (5.18)$$

Both Wheeler [1] and Montgomery [2] state that the MA control chart is suitable for processes with slow changes compared to the sampling frequency.

### 5.4.3 The Exponentially Weighted Moving Average Control Chart

The Exponentially Weighted Moving Average Control Chart is good for detecting small shifts in the process mean. It was first described in Roberts (1959). The difference from the MA control chart is that the EWMA does not have a group size; instead it weights the historical data. Really old data will be weighted almost to zero while new data will count more. See equation below

$$z_i = \lambda \sum_{j=0}^i (1-\lambda)^j \cdot x_{i-j} + (1-\lambda)^i \cdot z_0 = z_{i-1} + \lambda \cdot (x_i - z_{i-1}) \quad (5.19)$$

Sometimes this algorithm is called Geometric Moving Average (GMA).

The EWMA has lots of advantages and one is its insensitivity to the normality assumption of the process variation. [2, page 412]

The control limits and centre line are calculated according to

$$UCL = \mu_0 + L\hat{\sigma} \sqrt{\frac{\lambda}{2-\lambda} \cdot [1 - (1-\lambda)^{2i}]} \quad (5.20)$$

Centre line =  $\mu_0$

$$LCL = \mu_0 - L\hat{\sigma} \sqrt{\frac{\lambda}{2-\lambda} \cdot [1 - (1-\lambda)^{2i}]} \quad (5.21)$$

In the equation for the control limits there are two design parameters,  $\lambda$  and  $L$ .  $L$  controls how many standard deviations from the centre line the control limits are placed.  $\lambda$  is a parameter

that should be chosen carefully and it decides how much new observations will influence the EWMA. When comparing the expression for the EWMA with similar ones in theory of signal processing one should bare in mind that  $\lambda$  is not the same as  $\theta$ , but  $\theta = 1-\lambda$ .

#### 5.4.4 The Cumulated Sum Chart

The Shewhart Chart has a big disadvantage; it is not good at detecting small shifts in either the process mean or the process standard deviation. If only the first sensitizing rule (points outside the three-sigma-limits) is applied there is a high probability that for example a process mean shift of  $0.5 \sigma$  will run undetected for many subgroups. One of the later described sensitizing rules fires when many observations are on the same side of centre line. This can help detecting small shifts but it is definitely not a fail safe procedure.

The idea behind the CUmulative SUM Control Chart is to summarise all the deviations from a target value,  $\mu_0$ . If the process is in control and the deviations are normally distributed the sum will alternate around zero. Even a slightest shift in the process mean will cause a drift in the sums of deviations. The CuSum test quantity is described by the following formula

$$C_i = \sum_{j=1}^i (\bar{x}_j - \mu_0) = (x_i - \mu_0) + C_{i-1} \quad (5.22)$$

As a parallel to the Shewhart Chart the CuSum is most effective with group size  $n=1$ .

The cusum tool could be looked upon as the integral part of an proportional integral controller.

#### Tabular CuSum

The tabular CuSum is a version of a CuSum chart with two series accumulating the values above or below target value, respectively. This is a double side control chart. The variables are called  $C_i^+$  and  $C_i^-$  are calculated according to

$$C_i^+ = \max[0, x_i - (\mu_0 + K) + C_{i-1}^+] \quad (5.23)$$

$$C_i^- = \max[0, (\mu_0 + K) - x_i + C_{i-1}^-] \quad (5.24)$$

with starting values  $C_i^+ = C_i^- = 0$

The variable  $K$ , called allowance, should be chosen as

$$K = \frac{\delta}{2} \cdot \sigma = \frac{|\mu_1 - \mu_0|}{2} \quad (5.25)$$

which makes  $K$  a design parameter that corresponds half the shift size one wants to detect.

Montgomery [2] also brings up another variable  $H$ , called Decision Interval. This is the allowed interval for the  $C_i^+$  and  $C_i^-$ . It is usually defined in numbers of standard deviations and a rule of thumb is to use  $H = 5$ .

When a situation arises where an adjustment is called for, it is a good idea to estimate the shift size. This can be done with

$$\hat{\mu} = \begin{cases} \mu_0 + K + \frac{C_i^+}{N^+} \\ \mu_0 - K - \frac{C_i^-}{N^-} \end{cases} \quad (5.26)$$

and should be taken into account when adjusting the manipulatable variable.

In (5.26) the first case should be used if  $C_i^+ > H$  and the second if  $C_i^- < H$ .

$N^+$  and  $N^-$  are counters counting number of observations since the process shift occurred. It is straightforward to find  $N$  in the CuSum Chart.

### Standardized CuSum Control Chart

If there is reason to compare many cusum charts it could be a good help to standardize the variable  $x_i$

$$y_i = \frac{x_i - \mu_0}{\hat{\sigma}} \quad (5.27)$$

This makes all control charts independent of their different standard deviations. The algorithm for plotting  $y_i$  now becomes

$$C_i^+ = \max[0, y_i - k + C_{i-1}^+] \quad (5.28)$$

$$C_i^- = \max[0, -y_i - k + C_{i-1}^-] \quad (5.29)$$

### Headstart Feature for CuSum Control Charts

The tabular and standardized cusum control charts both have the disadvantage that they are bad at detecting process shifts during start up phases. It takes a couple of observations for the shift to accumulate. This disadvantage can be suppressed by giving the control chart a head start.

The headstart is typically implemented by defining

$$C_0^+ = \frac{H}{2} \quad (5.30)$$

$$C_0^- = -\frac{H}{2} \quad (5.31)$$

By implementing this, two scenarios can occur. If no assignable cause (shift in process mean) is present  $C_i^+$  and  $C_i^-$  will drift towards zero after just a couple of observations. If, on the other hand, there is a shift in the process mean it will be detected faster than without the headstart feature. The initial condition on  $C_i^+$  and  $C_i^-$  is a design parameter and it is possible to place it closer to  $H$ , but this will increase the false alarm rate. Natural process variations can push the cusum line over  $H$  if the initial condition is too large (even though it will go back towards zero after a while).

### **CuSum for monitoring process variability**

So far the cusum control chart has been used to detect shifts in the process mean. It would be practical to have a control chart able to detect shifts in the standard deviation. According to Montgomery this feature was invented by Hawkins (1981, 1993a). Hawkins uses a

standardized cusum line  $y_i = \frac{(x_i - \mu_0)}{\sigma}$ . From this, a new standardized quantity can be formed

$$v_i = \frac{\sqrt{|y_i|} - 0.822}{0.349} \quad (5.32)$$

While Hawkins states that this variable  $v_i$  will be sensitive to changes in process variance rather than in process mean, Montgomery says it is sensitive to both mean and variance changes. The control chart is called Scale Cusum Control Chart. Two one-sided standardized scale cusums are constructed with this formula

$$S_i^+ = \max[0, v_i - k + S_{i-1}^+] \quad (5.33)$$

$$S_i^- = \max[0, -v_i - k + S_{i-1}^-] \quad (5.34)$$

The headstart feature works well on scale cusum control charts.

Hawkins also suggests that the scale cusum should be accompanied by a standardized cusum. If the scale cusum exceeds  $H$ , then it should be a signal that a shift in process variance, but if both control charts signals it should be interpreted as a shift in process mean.

### **5.4.5 Control Charts for Correlated Data**

The control charts above are very sensitive to even so slightly correlated data. The false alarm rate increases strongly with the amount of correlation between observations. In correlated data a high value will probably be followed by another high value, this means that for example one observation above a warning limit will be followed by another one even if no assignable cause is present. One way to overcome effects of correlation is to sample more seldom. This is not recommended! There are other, better ways of dealing with the problem.

### **Measure or estimate the autocorrelation**

The autocorrelation function is defined as

$$\rho_k = \frac{\text{cov}(x_t, x_{t-k})}{V(x_t)} \quad (5.35)$$

where  $\text{cov}(x_t, x_{t-k})$  is the covariance of observations that are  $k$  time periods apart.  $V(x_t)$  is the variance of  $x$ .

In many practical situations there is a need for estimating the autocorrelation. It is done by using the sample autocorrelation function:

$$r_k = \frac{\sum_{t=1}^{n-k} (x_t - \bar{x}) \cdot (x_{t-k} - \bar{x})}{\sum_{t=1}^n (x_t - \bar{x})^2}, k = 0, 1, \dots, K \quad (5.36)$$

### Model based approach

In this approach the SPC engineer will construct a model of the process, subtract modelled data from the observations and use control charts on the residuals. In mathematical terms, the model will be described by

$$x_t = \zeta + f(x_{t-1}, x_{t-2}, \dots, x_{t-n}) + f(\varepsilon_t, \varepsilon_{t-1}, \dots, \varepsilon_{t-k}) \quad (5.37)$$

where  $\zeta$  is an offset and  $\varepsilon$  is the deviation from the process model. The residuals are defined by

$$e_t = x_t - \hat{x}_t \quad (5.38)$$

If the model  $\hat{x}_t$  is correct the residual  $e_t$  will be independent observations from a normally distributed variable.

The process value can be looked upon as white noise filtered through a filter that describes the process. From the theory of signal processing and especially [3] following filter (process) models are borrowed.  $z$  is the complex variable used in the Z transform which is a discrete version of a La Place transform.

Process Name	Filter Equation
Moving Average (MA)	$H(z) = \sum_{k=0}^q \theta_q(k) \cdot z^{-k} \quad (5.39)$
AutoRegressive Moving Average (ARMA)	$H(z) = \frac{\sum_{k=0}^q \theta_q(k) \cdot z^{-k}}{1 + \sum_{k=1}^p \phi_p(k) \cdot z^{-k}} \quad (5.40)$

Table 5.2: Time series models

The values of  $p$  and  $q$  define the model order.  $\theta$  and  $\Phi$  defines the filter coefficients.

The two general models in Table 5.2 are members of the family AutoRegressive Integrated Moving Average models.

The process model can either be derived by setting up theoretical equations of the system or be identified by theory of system identification [4]. Montgomery and Mastrangelo [5] suggest an approximation procedure based on EWMA.

Suppose a process that can be modelled by

$$x_t = \xi + \phi x_{t-1} + \varepsilon_t - \theta \varepsilon_{t-1} \quad (5.41)$$

then an EWMA with  $\lambda = 1 - \theta$  is the best one-step-predictor, according to

$$z_t = \lambda x_t + (1 - \lambda) \cdot z_{t-1} \quad (5.42)$$

The best predictor will, in some sense, be when the squares of  $e_t$  is minimized. By testing different values on  $\lambda$  and choosing the one minimizing  $e_t$  a good approximate is achieved. As stated above, the residuals can be plotted in a control chart. The Shewhart  $\bar{X}$  Chart with group size  $n = 1$  is a good choice here.

An interesting aspect is to think about how the residuals are calculated. If they are normally distributed with a standard deviation of one, then

$$P[-3\sigma \leq e_t \leq 3\sigma] = P[-3\sigma \leq x_t - \hat{x}_t(t-1) \leq 3\sigma] = 0.9973 \quad (5.43)$$

Since the best predictor of  $\hat{x}_t = z_{t-1}$



$$P[-3\sigma \leq x_t - z_{t-1} \leq 3\sigma] = P[z_{t-1} - 3\sigma \leq x_t \leq z_{t-1} + 3\sigma] = 0.9973 \quad (5.44)$$

A new Shewhart chart can now be applied on the observations  $x_t$  but this time with the dynamic control limits

$$UCL = z_{t-1} + 3\sigma \quad (5.45)$$

$$LCL = z_{t-1} - 3\sigma \quad (5.46)$$

### Moving Centreline EWMA

The EWMA algorithm for monitoring correlated data can be extended to a Moving-Centreline EWMA Control Chart. In this case the centre line is defined as

$$CL = Z_t \pm 3\sigma_e \quad (5.47)$$

with  $Z_t$  as above and  $\sigma_e$  is the standard deviation of the single-period forecast error.

The forecast error is then the difference between an observation and the forecasted centreline. If the forecasting system is working as according to specifications, the forecast errors are expected to be close to zero and unbiased. A disadvantage of the moving centreline EWMA is that it tends to track data and may lead to biased forecast errors.

### 5.4.6 Control Charts with Tracking Signals

In [7] two versions of tracking signals for monitoring the forecast errors are introduced. One is called cumulative-sum tracking signal and the other smoothed-error tracking signal.

#### Cumulative-Sum Tracking Signal

There is a way of monitoring the mean average deviation. The formula reminds of a EWMA but there is a main difference, the absolute value of the forecast error is used to feed the algorithm. The Mean Average Deviation (MAD) is defined as

$$\hat{\Delta}(t) = \alpha \cdot |e_t| + (1 - \alpha) \cdot \hat{\Delta}(t-1) \quad (5.48)$$

where  $\alpha$  is typically chosen to be between 0.05 and 0.15. The MAD is an increasing value where the increasing rate is defined by  $|e_t|$  and  $\alpha$ .

By just summing up all the forecast errors

$$Y(t) = \sum_{j=1}^t e_j \quad (5.49)$$

a quote between  $|Y(t)|$  and  $\hat{\Delta}(t)$  can be created according to

$$T_C(t) = \frac{|Y(t)|}{|\hat{\Delta}(t)|}. \quad (5.50)$$

This is the Cumulative-Sum tracking signal.

As long as the MAD is growing faster than  $Y(t)$  the  $T_C(t)$  will be close to zero. This implies that the EWMA corresponds well to the actual system. With a bad EWMA, the forecast errors will not be normally distributed white noise, and the  $Y(t)$  will steadily increase (or decrease). If the tracking signal is started on a in-control process with an optimal EWMA, the parameter  $T_C(t)$  will only increase when the system is no longer in statistical control.  $T_C(t)$  is usually compared with a critical value,  $K_C$ . According to Montgomery, [2], this value is in practice chosen to be  $4 \leq K_C \leq 6$ .

### Smoothed Error Tracking Signal

The smoothed error tracking signal is an extension of the tracking signal described above. In this case, the nominator is a smoothed value of forecasted errors

$Q(t) = \beta \cdot e_t + (1 - \beta) \cdot Q(t - 1)$  and  $0.05 \leq \beta \leq 0.15$ . Therefore, the smoothed error tracking signal is defined as

$$T_S(t) = \frac{|Q(t)|}{|\hat{\Delta}(t)|}. \quad (5.51)$$

Of course, there is a need for a critical value  $K_S$  as well. This time, usual values falls between  $0.4 \leq K_S \leq 0.6$ .

A common way of using these tracking signals is to use a three-in-a-row rule to minimize potential false alarms, while maintaining an acceptable ARL.

In the article [7], an extensive case study is shown and the conclusion indicates that the smoothed-error signal has a better false-alarm (FA) rate and ARL. The  $\alpha$  and  $\beta$ -parameters are designed with aspect to the process-shifts magnitude that wants to be detected.

## 5.4.7 Common Fault Patterns in Control Charts

### Cyclic patterns

The cyclic patterns are common in many processes. It can be due to different reason and here are some examples: Temperature and regular rotation of operators.

### Mixture

The phenomenon called mixture is indicated when there are many points close to the upper and lower control limits but few points close to the process mean value. Overcompensation and when the products come from different parallel machines can be the reasons for this.

### Shift in process level

This is a permanent shift of the process mean value, can occur from different factors as shift in raw materials or skills of operators.

## Trend

A is a drift from the process mean value and can be blamed on for instance worn out equipment or leakage of a pipe.

## Stratification

In this case the points seem to cluster artificially around the centreline with very small variance. There are several reasons for this and one of them is miscalculated control limits.

## 5.5 Cuscore Statistics

In a process there are disturbances known to happen, maybe not regularly, but at least a couple of times during a long time period. In some cases these phenomena can be devastating for the process performance if not dealt with in an appropriate way. One example could be clogging in a main pipe of a chemical plant. A common way to deal with this is to clean the pipe after equal time intervals to make sure the clogging does not influence the process performance. The time interval is usually short in comparison to the time it usually takes to plug the pipe. This maintenance approach ensures no malfunction but will probably tell the operators to clean the pipe a couple of times when it is not necessary. What if there is a way of detecting when it is time to remedy the disturbance?

In the example above, the clogging will probably affect the flow through the pipe in a similar way each time it happens. The flow signal could be monitored by some of the charts mentioned above, but they will not be selective to the known-to-happen clogging. The Cuscore statistics is used to detect known patterns in a signal with noise. The general formula for Cuscore is

$$Q = \sum_{t=0}^n e_{t0} r_t \quad (5.52)$$

where  $e_{t0}$  is the residual from the process when it is in statistical control and  $r_t$  is a detector that measures the rate of change.

Another way of writing the Cuscore is

$$Q_t = \max [Q_{t-1} + (x_t - \mu + k_f) \cdot f(t, \delta, \tau); 0] \quad (5.53)$$

where

$x_t$  is an observation of the process

$\mu$  is the process target value

$k_f$  is a reference value or handicap

$\delta$  is a parameter that reflects the nature and magnitude of the process shift

$f(t, \delta, \tau)$  is the function of the signal that should be detected. The known assignable cause occurs a time  $\tau$ .

**Example: Designing a Cuscore for detecting a shift in process drift**

In a simple process model with a parameter drift,  $\beta t$ , the observation  $x_t$  can be described

$$x_t = \mu + \beta t + \varepsilon_t \quad (5.54)$$

The residuals will be written as

$$e_{t0} = x_t - \mu - \beta t \quad (5.55)$$

and with an assignable cause,  $\delta t$ , present

$$e_t = x_t - \mu - \beta t - \delta t \quad (5.56)$$

The model of (5.55) representing an in-control process is called the null model.

In (5.52) a detector,  $r_t$ , is introduced. In this case the detector will be calculated as

$$r_t = \frac{e_{t0} - e_t}{\delta} = \frac{x_t - \mu - \beta t - (x_t - \mu - \beta t - \delta t)}{\delta} = t \quad (5.57)$$

The Cuscore will then be

$$Q = \sum e_{t0} r_t = \sum (x_t - \mu - \beta t) \cdot t \quad (5.58)$$

A Cuscore for detecting a drift during time will have a factor  $t$  in its mathematical expression. This will explain the  $f(t, \delta, \tau)$ -term in the somewhat, maybe before, cryptic (5.53). For monitoring the process, the Cuscore,  $Q$ , is plotted against  $t$ . A decision interval,  $H$ , is used as a control limit for detecting the presence of the searched assignable cause.

Montgomery [2] brings up more examples with different types of assignable causes. He also states that it is possible to design a Cuscore to detect almost any kinds of signals. The disadvantage of the cuscore statistics is that it performs at its best when  $\tau$  is known!

**Example: Looking for sinusoidal wave in noise with cuscore**

In figure 5.4 b) a signal containing white noise and a sinusoidal wave after  $t=200$  is showed. A cuscore for detecting this sinusoid imagined to be caused by an assignable cause would look like (5.58) but with a sinusoid  $\sin t$  instead of  $t$ .

The white noise disturbing the process value (Figure 1.3) is normally distributed with a standard deviation that is twice as big as the amplitude of the sinusoid. By just looking at the signal it is hard to detect the disturbance but the cuscore has no trouble detecting it (figure 5.4c). Worth mentioning is that even though the cuscore managed to signal the sinusoid most of the times simulated, a couple of runs passed undetected. By adjusting the designing parameters  $H$  and  $k_f$  the numbers of misses are minimized. The disadvantage of this procedure is that the frequency of the sinusoid needs to be known.

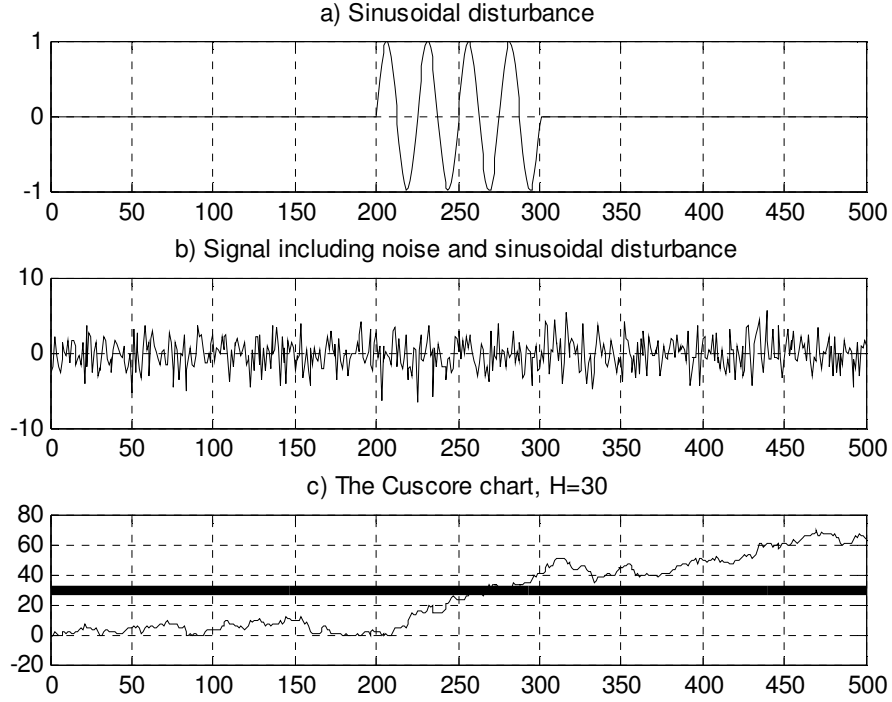


Figure 5.4: a) A sinusoidal wave with amplitude  $I$ , caused by an assignable cause, occurring at time  $t=200$  and is present during for  $100$  samples. b) The process value disturbed by white noise with  $\sigma=2$  and the assignable cause in a). c) The cuscore for detecting a sinusoidal wave.  $\mu$  and  $k_f=0$ ,  $H=30$ .

Box and Luceño [9] has built up the following table for detecting a couple of standard patterns

Noise Signal	White noise ( $e_t$ ) Step Change	White noise ( $e_t$ ) Sine wave with known frequency	White noise ( $e_t$ ) Slope change
Null model	$e_{t0} = x_t - \mu$	$e_{t0} = x_t - \mu$	$e_{t0} = x_t - \beta t$
Discrepancy model	$e_{t0} = x_t - \mu - \delta$	$e_{t0} = x_t - \mu - \delta \cdot \sin(t)$	$e_{t0} = x_t - \mu - \delta t$
Detector	$I$	$\sin(t)$	$t$
Cuscore	$\Sigma(x_t - \mu)$	$\Sigma(x_t - \mu) \cdot \sin(t)$	$\Sigma(x_t - \mu) \cdot t$
Value of Q when discrepancy model is true	$\Sigma e_t + [\delta n]$	$\Sigma e_t \cdot \sin(t) + [\delta \cdot \Sigma \sin^2 t]$	$\Sigma e_t t + [\delta \cdot \Sigma t^2]$

Table 5.3: Noise, signals, models, detectors, and cuscore statistics for a step change, intermittent sine wave and a change of slope in the mean.

The observant reader notices that the cuscore for detecting a step change is the same as a cusum chart. When detecting a spike with cuscore you get the Shewhart chart and for an exponentially increasing signal one ends up with the EWMA chart.

## 5.6 Sensitizing Rules

To be able to detect different phenomena in control charts there has evolved a lot of different rules. The ten most common rules are stated in table 1. [2]

### Standard action signal:

1. One or more points outside the control limits
2. Two of three consecutive points outside the two-sigma warning limits but still inside the control limits
3. Four of five consecutive points beyond the one-sigma limits
4. A run of eight consecutive points on one side of the centre line
5. Six points in a row steadily increasing or decreasing
6. Fifteen points in a row in zone C (both above and below the centre line)
7. Fourteen points in a row alternating up and down
8. Eight points in a row on both sides of the centre line with none in zone C
9. An unusual or non-random pattern in the data
10. One or more points near a warning or control limit

Table 5.4: Sensitizing rules for detecting an assignable cause

Rule one to four are called *The Western Electric Rules*.

Not all of the rules directly imply assignable causes. Some of them should instead be interpreted as detecting potential assignable causes. For example another common rule, “if one or two points are outside the  $2\sigma$  limit but inside the control limit” it does not say that an assignable cause is present, but it is indicating that special care should be taken. One action could be increased sampling frequency.

The different sensitizing rules can in some sense be compared to strategies in automatic control. The first rule can be looked upon as an on-off controller, the third and fourth as integral controller while number five reminds of a derivative controller.

Care should be taken when using these rules and especially when using them simultaneously. The more rules used the higher probability for false alarm. If the probability for rule  $i$  is  $\alpha_i$  then the overall false alarm probability will be

$$\alpha = 1 - \prod_{i=1}^k (1 - \alpha_i) \quad (5.59)$$

if all the  $k$  rules are independent. This is not always the case with the rules above but the formula still makes its point.

## 5.7 Process Capability

The control limits of a control charts are calculated based on the natural variations of the process. Specification limits are limits that decide whether a unit is acceptable or not. In one sentence, the process decides the control limits while the market and management decides the specification limits. Even though the two sets of limits not are correlated in any way it is useful to measure them according to each other. The easiest and a common way to do this is to compare the difference between the Upper Specification Limit (USL) and the Lower Specification Limit (LSL) with the difference between the two control limits. From this the Process Capability Ratio (PCR) is formed

$$C_p = \frac{USL - LSL}{UCL - LCL} = \frac{USL - LSL}{6\sigma} \quad (5.60)$$

If the standard deviation is unknown an estimate  $\hat{\sigma}$  is used to form an estimate  $\hat{C}_p$ .

One sided process capability ratios are defined as

$$C_{pu} = \frac{USL - \mu}{3\sigma} \quad (5.61)$$

$$C_{pl} = \frac{\mu - LSL}{3\sigma} \quad (5.62)$$

A newer and a bit more advanced way to calculate the PCR is discussed in [2]. It is called the third generation of CPR and follows as

$$C_{pkm} = \frac{C_{pk}}{\sqrt{1 + \left(\frac{\mu - T}{\sigma}\right)^2}} = \frac{C_{pk}}{\sqrt{1 + \xi^2}} \quad (5.63)$$

$\mu$  is the process mean value and  $T$  is the target value.

The process capability ratio could be used in many ways but the obvious use is to predict how many defect units a process will produce (while in statistical process control).

The PCR has no meaningful interpretation if the process is out of statistical process control. The reason for this is that an estimated PCR from an out-of-control process does not necessarily describe the rate of defect units in the future. [2]

## **5.8 Applying SPC**

### **5.8.1 Phase I and Phase II**

There are situations where SPC is applied from scratch on an existing process. A reason for implementing SPC is that the process does not perform as wished. The management perhaps gives an engineer the task of improving the process using control charts. A good assumption is that the process is out of statistical control. If it is not, the management probably were satisfied with the process and the need for improvement would not arise. When SPC is applied for the first time on a process that is (probably) out of control it is said to be in phase I. The first thing to do is to gather information about the process and observations from the process value. The observations are divided in to subgroups. At least 20 to 25 subgroups are usually needed. These facts are used to construct *trial control limits*. Since the process was not working in the first place the most dominating assignable causes are removed by the engineer. Subgroups of data are collected and plotted. New trial control limits are calculated. The procedure of collecting data, forming subgroups, plotting and calculating limits will together with removing assignable causes result in an in-control process. The trial control limits will finally converge into proper control limits. During phase I the Shewhart chart is a good control chart. It is easy to use and construct and has a clear physical interpretation.

A process that is said to be in phase II has been in statistical control but it does not necessary have to be in control all the time. In phase II the engineer is using control charts to monitor the process. If the control charts detects an assignable cause it should be dealt with. This is why a process not have to be in control all the time, but can still considered being in phase II. While the Shewhart chart is a good option for phase I the cusum or EWMA chart is probably a better solution in phase two. A process in phase II is more likely to be affected by a small assignable cause and since the cusum and ewma charts are better at detecting small shifts (compared to the Shewhart chart) these charts should be used here.

### **5.8.2 Data Collection and Subgroups**

In order to have a properly working control chart it is important to choose appropriate sampling sizes and sampling frequencies. Good values of these parameters are heavily dependent of the nature of the individual process. A first approach is to be certain to gather as much information as necessary. In a practical situation, this does not imply sampling of everything all the time. Let us say that there is a cost on sampling like discarding the observed unit or a salary for an operator, then too many observations should not be gathered. Another parameter to consider is the size and frequency of the shifts caused by the assignable causes.

For the Shewhart chart there are two possible ways of creating subgroups. One strategy is to maximize the probability of shifts occurring between subgroups and minimizing the probability of shifts occurring between the samples within the group. This calls for observing a few observations in a row when the sampling interval begins. In doing this the engineer could obtain a good estimate of the standard deviation but the observations might not be typical values for the whole sampling interval.

If a representative value of the whole sampling interval is desired the spaces of the samples should be constant. Constant sampling period and rather sparse sampling has the advantage of suppressing the effects of weakly correlated data. On the other hand, both Wheeler and Montgomery states with emphasis that Shewhart charts should not be used on correlated data.



Due to technological developments it is becoming easier and easier to obtain observations of the process value. If the sampling is cheap and automatic there is no need for discarding information. Instead the control charts should be designed in a way that optimizes the use of all information. If correlated data is present the SPC engineer ought to use control charts designed for correlated data, either by a model based approach or model free approach. The complexity of the system decides whether it is possible to obtain a process model.

### 5.8.3 Average Run Length

To design a good control chart it might be a good practice to think about the control chart's average run length (ARL). The ARL of a control chart is a number on for how long the chart will run before detecting a Type I error. If the probability of signalling an assignable cause even if such a thing is not present, is  $p = 0.0027$ . Then the average run length is defined as

$$ARL = \frac{1}{p} = \frac{1}{0.0027} = 370. \quad (5.64)$$

The calculation is based on normally distributed data. ARL is measured in subgroups. A more intuitively parameter might be the ATS (Average Time to Signal) which is measured in second, hours or days.

$$ATS = ARL \cdot h \quad (5.65)$$

where  $h$  is the sampling interval. One should not trust the ARL or ATS blindly since its standard deviation is about the same figure as the ARL itself. Another disadvantage is that a skew distribution might give an ARL that is not representative for actual run length.

## 6 Automatic Control

The reader is assumed to be familiar with the basics of continuous and discrete automatic control. This chapter will review the parts that are used in this thesis when it comes to control and analyzing the actual system.

### 6.1 Discretization

There are numerous ways of discretizing continuous time systems. One, and maybe the most common used, is to sample the continuous system with a zero-order-hold circuit. Euler introduces a backward and forward approximation method and the methods are extended by Tustin's version.

#### 6.1.1 Euler Backward Method

Using Euler's backward differentiation method a stable continuous time system will become a stable discrete system.

$$\frac{dx(t)}{dt} \approx \frac{x(t) - x(t-h)}{h} \quad (6.1)$$

hence

$$s' = \frac{z-1}{zh} \quad (6.2)$$

### 6.2 PI Controllers

A regular PI controller consists logically of two terms, one Proportional and one Integral term. A basic control schedule is shown in figure 6.1.

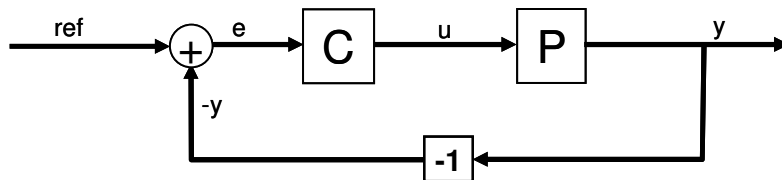


Figure 6.1: Basic Control Scheme

It is straight forward to realize that the proportional term is reacting on the error at the moment of the observation. The integral part is reacting on the sum of all faults since start-up. If the process combined with the controller is stable the fault will go towards zero as long as the reference value is constant. This assumes that nothing disturbs the process. Disturbances will be treated in another section. It is time to look at the formulas for implementing a PI controller. In the time domain the control signal  $u(t)$  will look as

$$u(t) = K_C \cdot \left[ e(t) + \frac{1}{T_I} \int_0^t e(\tau) d\tau \right] \quad (6.3)$$

while in the continuous frequency domain

$$U(s) = K_C \cdot \left[ E(s) + \frac{1}{T_I s} \cdot E(s) \right] \quad (6.4)$$

The parameter  $K_C$  is the gain of the controller and  $T_I$  is the integration time and is, as the name implies, given in the dimension seconds. Since the real process in this master thesis is, at least partly, discrete it is reasonable to have a discrete regulator. By using Euler Backward Approximation and a sampling time  $h=1$  batch

$$s' = \frac{z}{z-1} \quad (6.5)$$

the discrete control signal  $u(k+1)$  will be calculated as

$$u(k+1) = u(k) + \left( 1 + \frac{K_C}{T_I} \right) \cdot e(k+1) - K_C e(k) \quad (6.6)$$

### 6.2.1 Minimum Mean Square Error Controller

In many references like [9], [11] and [12] the authors introduce controllers that minimize the variance of process value, Minimum Mean Square Error (MMSE) Controller. Equations for the controller are derived in cases where a good process model is available. All references on the subject are also quick to emphasize the fact that even though the controller minimizes the mean square error it will yield a heavily varying control signal. As the controller reacts very strong to small process value deviations the system is sensitive to disturbances and model uncertainties. With these facts at hand, another approach is often made.

### 6.2.2 Constrained Adjustment Controller

The MMSE controller minimizes the variance of the process value deviations from its target value  $\text{var}(e_t) = \sigma_e^2$ . A constrained controller takes into account the benefits of having a slower and calmer control signal. If a function of control signal variance,  $\text{var}(u_t) = \sigma_u^2$  and  $\sigma_e^2$ ,

$$\sigma_e^2 + \beta \sigma_u^2$$

is minimized, instead of just the process value error variance, a proportional integral controller can be derived. Box and Luceño [12] say that the calculations needed to do this are long and complicated; hence they are not presented here. Instead tables for tuning discrete PI controllers into performing approximately as the optimal constrained versions are introduced.

Many examples show that a constrained controller, theoretically optimal or not, is able to reduce  $\sigma_u^2$  by up to 95 % when only increasing  $\sigma_e^2$  with typically 10 %.

### 6.3 Stability Analysis

Given the system in figure 6.1, it is well known that if the closed loop system

$$\frac{Y_c(s)}{R_c(s)} = \frac{C_c(s) \cdot P_c(s)}{1 + C_c(s) \cdot P_c(s)} \quad (6.7)$$

has its poles in the left half plane of the frequency domain, the system is stable. The *characteristic polynomial* is another name for  $1 + C_c(s) \cdot P_c(s)$  and since it is the denominator of the closed loop transfer function the zeros have to be situated in the left half plane.

The subscript  $c$  indicates that the transfer functions are continuous.

The discrete version of (6.7) will be looking like

$$Y(z) = \frac{C(z) \cdot P(z)}{1 + C(z) \cdot P(z)} \cdot R(z) \quad (6.8)$$

and poles need to be inside the unity circle for stability.

### 6.4 Final Value Theorem

#### Final Value Theorem

The  $z$ -transform has the following properties [10]

*If  $(1 - z^{-1}) \cdot F(z)$  does not have any poles on or outside the unit circle, then for a signal  $f$  for which  $\lim f$  is defined*

$$\lim_{k \rightarrow \infty} f(kh) = \lim_{z \rightarrow 1} (1 - z^{-1}) \cdot F(z) \quad (6.9)$$

### 6.5 Random Processes

#### 6.5.1 Autocorrelation Function

An autocorrelation function is a function that describes how much a signal at time  $k$  matches the same signal at time  $k-l$ . It is frequently used in signal processing theory, like the one described in Hayes [3]. The autocorrelation function is defined as

$$r_x(k, l) = E\{x(k) \cdot x^*(l)\} \quad (6.10)$$

where  $x(k)$  often is a stochastic discrete variable.

The  $*$  in  $x^*(l)$  is referring to the conjugate of  $x(l)$ .

A white noise sequence will have an autocorrelation function that has only one value that is not zero. E.g. if  $x(k)$  is a vector containing a white noise sequence, the corresponding autocorrelation function will be

$$r_x = \left[ \dots \quad 0 \quad 0 \quad \left| \sigma_x^2 \quad 0 \quad 0 \quad \dots \right. \right] \quad (6.11)$$

where the | symbol indicates where  $k=0$ .

### 6.5.2 Filtering Random Processes

Consider a signal,  $x(k)$ , used to excite a time discrete filter,  $h(k)$ . The filter output will be called  $y(k)$ .  $x(k)$  is described by an autocorrelation function,  $r_x$ . The autocorrelation function  $r_y(k)$  will then be calculated according to

$$r_r(k) = r_x(k) \cdot h(k) \cdot h^*(-k) \quad (6.12)$$

The equation above can be transformed into the frequency domain and will then look like

$$P_y(z) = P_x(z) \cdot H(z) \cdot H^*(z^{-1}) \quad (6.13)$$

If  $x(k)$  fits the description of (6.11)  $P_x(z)$  will be equal to  $\sigma_x^2$ , then

$$P_y(z) = \sigma_x^2 H(z) \cdot H^*(z^{-1}) \quad (6.14)$$

### 6.6 Sample Interval

A controller is suggested to have a sample time that gives the controller at least four samples per rise time [10]. A higher sampling rate is often a good choice but not always possible.

## 7 Comparing and Combining SPC and Automatic Control

### 7.1 SPC and Feedback Adjustment

The original theory of statistical process control stresses that adjustments should only be made when there is an assignable cause present. This should be proven statistically to rule out the possibility of making adjustments due to a chance cause. The process should be tuned into almost perfection and afterwards only be adjusted occasionally. This is an appealing approach, but in many real life situations this is not enough. Many manufacturing processes tend to drift away from their set point value and some of them will by their nature drift away (think of a level in a tank for example). The drifts can maybe be remedied by repeatedly reconstructing the factory, but this will sooner or later probably wind up in costly reconstructions for small improvements. If the process has one or more manipulatable process variables that affects the process in a known way, why not make adjustments here instead? If it is cheaper to adjust some parameters of the process than rebuilding the whole thing, why not do that if it yields the same result? These are some philosophical questions that the SPC practitioners had to cope with. Luckily there was a solution to the problem. Statistical process control can be used also in situations where regular adjustments are called for.

An integrated system is created by letting controllers based on automatic control theory handle the adjustments and letting the SPC theory be the foundation of monitoring the process.

Feedback adjustments sabotages the common use of control charts described in the SPC chapter. The automatic controller will compensate for the effects of assignable causes and in a way conceal them. Even though feedback adjustments make assignable causes harder to detect, there are tools for monitoring process values from controlled systems.

### 7.2 Tools for Monitoring Controlled Systems

#### 7.2.1 Monitoring Feedback Systems Subject to Integral Control

The following example is borrowed from [9].

Consider a system controlled by an incremental integral controller

$$\Delta u_t = -\frac{G}{g} e_t \quad (7.1)$$

with  $e_t$  as the deviation from target value,  $G$  damping factor [9] and process gain  $g$ .

A disturbance is affecting the system on  $e_t$  and is modelled

$$z_t - z_{t-1} = a_t - \theta a_{t-1} \quad (7.2)$$

with  $a_t$  being normally distributed white noise with  $\sigma_a$  representing the standard deviation of  $a_t$ .

In this example a spike needs to be detected. A spike affecting a system controlled by an integral controller will make the process value look much like a spike affecting a EWMA chart (spikes with exponentially decreasing magnitude). The cuscore for detecting an EWMA signal in noise will look as

$$Q_s = D \cdot e_t + (1 - D) \cdot (e_t - \tilde{e}_{t-1}) \quad (7.3)$$

Where  $D = \frac{G}{\lambda}$  and  $\tilde{e}_{t-1}$  is an EWMA of the output errors with smoothing constant  $\theta$ .  $\lambda$  is a tuning parameter where  $\lambda = G$  will produce a minimum mean square error controller.

If the system in previous example is in perfect statistical control, no controller like the one in (7.1) is needed. According to the foundations of SPC no adjustments should be made, which contradicts the nature of the controller. If a controller is used, it can increase the standard deviation on the process value. An integral controller like the one in (7.1) will produce a process value standard deviation,

$$\sigma_y = \sqrt{\frac{2}{2 - G}} \cdot \sigma \quad (7.4)$$

where  $\sigma_y$  is the process value standard deviation from the controlled system, while  $\sigma$  is the equivalent for the uncontrolled system.

### 7.3 Overcompensation

Overcompensation is a phenomenon that arises when an assignable cause is removed from a system controlled by a regulator. For example, when there is a drift in the process mean present the regulator will start compensating for it. The control signal will increase (or decrease) and the process value will again be close to the nominal value. As long as the drift continues the control signal will continue to rise, but all products will be acceptable. The compensating controller will of course try to hide the assignable cause but eventually it will show or be noticed by an operator. In practice there is always a delay between the occurrence of the assignable cause and the detection of the same. Due to the delay, the control signal increases as mentioned above. When the assignable cause is removed and the system restarted the control signal will be inappropriate for the new conditions. The phenomenon is in [8] called *overcompensation*. Depending how fast the regulator adapts to the new environment, it will take some time and perhaps a couple of nonconforming units before the system runs according to specifications.

In automatic control theory *overcompensation* can symbol other phenomena which are totally separated by the one described here.

### 7.4 Recognition of Disturbance Types

Recognizing the disturbance type is crucial in providing the operators guidance to seek the present assignable cause. In some fields of applications the SPC theory has the disadvantages to detect assignable causes but not indicate where to or how to look for them. SPC applied, without extra care, on correlated data is one of those fields.

### 7.4.1 Process drifts and shifts

Huang and Lin [8] introduce a way to distinguish process drifts from process shifts.

A process controlled by a properly tuned controller will have an process value that is varying around a target value. Shifts and trends will be, if not fully hidden, at least hard to detect.

Given a system

$$y_i = qx_{i-1} + d_i \quad (7.5)$$

with

$$d_i = d_{i-1} + a_{i-1} - \theta a_{i-2} \quad (7.6)$$

$a$  is normally distributed white noise and  $\theta$  is the smoothing constant.

$y_i$  is process value deviation from target value at time  $i$ .

$x_i$  is the difference between control variable and nominal variable at time  $i$ .

$q$  is the system gain.

Huang and Lin refer to Shao and Lin when they are saying that the process value will be described by following equations.

A process shift,  $D$ , would in the process value look like

$$y_i = D\theta^i + a_{i-1} \quad (7.7)$$

while a drift,  $S$ , will be modelled as

$$y_{i-1} = S \cdot \frac{1 - \theta^{i+1}}{1 - \theta} + a_{i-1} \quad (7.8)$$

A process shift is only changing at the moment when it occurs and remains thereafter constant. It is then easy to comprehend that (7.5) will converge to the process value before the shift

$$y_i = a_{i-1} \quad (7.9)$$

A process drift continues to increase forever and the process value will not converge to (7.8) since there will always be a deviation present. Instead, a process drift, converges to

$$y_{i-1} = \frac{S}{1 - \theta} + a_{i-1} \quad (7.10)$$



The systems above could be monitored with the aid of a Shewhart Chart and a Cusum Chart. Properly designed, the Shewhart chart, will detect a process shift since it produces a sudden (but decreasing) deviation in the process value. Even more intuitively is the detection of a process drift. Since a Cusum chart summarizes all process value deviations it is easy to understand that the chart will detect the drift by looking at (7.10). Huang and Lin extend the discussion to a more systematic way of detecting and recognizing the two assignable causes.

1. Monitor the process value with a Shewhart and a Cusum Control Chart.
2. If the Shewhart chart signals, stop the update of the Cusum chart for a time period  $T_w$ , then restart the updating again.
  - a) If the Cusum chart signals within a time  $T_{det}$ , the assignable cause should be identified as a process drift.
  - b) If the Cusum chart does not signal within  $T_{det}$ , the assignable cause should be looked upon as a process shift.
3. If, instead of the Shewhart chart, the Cusum chart signals, the assignable cause should be regarded as a process drift.

$T_w$  and  $T_{det}$  are design parameters for the engineer. The time  $T_w$  is used for letting the effects of a shift decrease enough to not make the Cusum chart signal when started again. A too large  $T_w$  will increase the detection time.  $T_{det}$  should also be carefully chosen since a too large time will increase detection time and a too short will increase the misdetection rate.

### 7.4.2 General Approach

In chapter 5.5 Cuscore Statistics a general approach is defined. A detector,  $r_t$ , is introduced and the Cuscore is developed from

$$Q = \sum_{i=0}^n e_{i0} r_i \quad (5.52)$$

to

$$Q_t = \max[Q_{t-1} + (x_t - \mu + k_f) \cdot f(t, \delta, \tau); 0]. \quad (7.11)$$

This approach has the disadvantage of needing to know what signal to look for and when to look for it.

#### Method for obtaining the detector when looking for signal in noise

There are practical situations when there is a need for estimating the detector,  $r_t$ . This could be when an assignable cause introduces a non-trivial pattern in the process value, probably disturbed by some kind of noise. Box and Luceño, [9], introduces a way of achieving the appropriate detector from the process value.

Consider a system where the process value,  $\{z_t\}$ , is noise modelled by

$$z_t - z_{t-1} = e_t - \theta e_{t-1} \quad (7.12)$$

where  $e_t$ , as usual, is white noise.

If an assignable cause occurs and introduces a unknown signal,  $\{x_t\}$ , the process value will be the sum of  $\{z_t\}$  and some multiple,  $-\delta$ , of  $\{x_t\}$  and will be a new series like  $\{z_t - \delta x_t\}$ .

Equation (7.12) can easily be transformed into producing white noise

$$e_t = z_t - z_{t-1} + \theta e_{t-1} \quad (7.13)$$

In Appendix 10 B of [9], a proof shows that the detector,  $r_t$ , will parallel (7.13) exactly and therefore the detector can be obtained from the series  $\{x_t\}$  according to

$$r_t = x_t - x_{t-1} + \theta r_{t-1} \quad (7.14)$$

from which the Q statistic

$$Q = \sum_{t=0}^n e_{t0} r_t \quad (7.15)$$

can be obtained.

## 8 Case Study at Perstorp AB

In this chapter the following glossary is needed:

**Formaldehyde:** Chemical substance with the formula  $CH_2O$ . Used to index the total mass of formaldehyde in a solution.

**Strong Formaldehyde:** A solution of water and formaldehyde. In this text the actual percentage is censored. The maximum possible concentration is 52 %.

**Weak Formaldehyde:** A mix of process condensate and strong formaldehyde. The formaldehyde concentration is varying somewhat but the actual percentage is censored.

### 8.1 The Task in Details

The reaction process is semi-batch wise will be described in the section below. The most expensive reactant is the isobutyric aldehyde, and therefore it is important that all isobutyric aldehyde is reacted into neopentyl glycol. One way to ensure this is to have an excessive amount of the other reactants in the reactor. By measuring the remaining formaldehyde after the reaction is stopped, it is possible to monitor and make sure that all isobutyric aldehyde has reacted with formaldehyde. Research and simulations [16] et. al. show that an amount of 0.25 – 0.35 % in the synthesis solution is minimizing the costs of raw materials. Almost all of the formaldehyde that has not reacted with any isobutyric aldehyde will be separated from the product in the evaporator. From the evaporator the formaldehyde will be pumped into the condensate tank. This condensate is then mixed into the synthesis solution; hence hardly any formaldehyde is lost even though not all of it reacts into neopentyl glycol in the reactor.

The amount of residual formaldehyde has turned out to be hard to control. The main task of this master thesis is to control the residual formaldehyde content in the reactor solution after the batch is finished. This problem should be investigated and hopefully solved without increasing the numbers of analysed samples every shift.

### 8.2 History of Perstorp AB

This section is based on the text *125 years of winning formulas*. [6]

Perstorp AB was founded, under the name of Stensmölla Tekniska Fabrik AB, 1881 by Wilhelm Wendt. He used beech wood from his fathers' forests to produce simple chemical products. The two biggest products were acetic acid and charcoal. The success of especially the acetic acid results in a name change to Skånska Ättikfabriken (Acetic acid is in swedish called ättika). The year of 1907 was a milestone when the company learned how to produce formaldehyde from methanol. A product the company still manufactures.

A great deal of money is made on formaldehyde product and is used for researching on new products. This renders in building Scandinavia's first plastic industry in 1918. Year goes by and laminate is added to the product family. Especially the decorative laminate is a big success and in the years after World War II the sales numbers of *Perstorp-plattan* shoots through the roof. The design called *virrvarr* is developed by royal designer Sigvard Bernadotte. A second milestone is reached 1955 when the first factory abroad is constructed. In Sao Paulo, Brazil a factory is manufacturing laminate.

The company starts focusing on chemistry and builds its first factory for producing the polyol pentaerythritol. Together with another polyol, trimethylolpropane, Perstorp founds the company's product base for many years to come. Parallel with the chemical industry the company starts to develop more plastic products and in a couple of years it becomes Scandinavia's biggest plastic producer with over 10 000 different products in the catalogue.

In the 1960's the times are changing and the company needs funding for big investments. As a result, the Wendt family changes the name to Perstorp AB and introduces it on Stockholm stock market in the year of 1970.

Perstorp expands a great deal in the 1980's. Many companies are bought and the growing company is introduced on the big stock markets of Paris and London. When the laminate floor Pergo sees the daylight for the first time it does not take many years to become a world renowned product.

At this moment Perstorp is a heavily diversified company with many different business areas. This tends to weaken the company and a process is started with the aim of specializing the company on speciality chemicals. Pergo and the plastic division are sold and at more or less the same time the Wendt family sells Perstorp AB to the investment company Industri Kapital in the year of 2001. Industri Kapital already owns Neste Oxo in Stenungsund and the company's focus on specialty chemicals is clear. Today, 2006, Perstorp AB is owned by the french company PAI and celebrates that 125 years have passed since Wilhelm Wendt started producing formic acid.

### **8.3 Neopentyl Glycol**

Neopentyl Glycol is sold under the name of Neo and is described on [www.perstorp.com](http://www.perstorp.com) as:

"Neo has two primary hydroxyl groups. It is a white material in flakes. Neo is used in saturated polyesters for powder coatings, coil coatings and other stoving enamels, unsaturated polyesters for gel coats and reinforced plastics and in esters for synthetic lubricants."

When used in coatings Neo improve properties as crystallinity, heat resistance, chemical resistance and outdoor durability.

The structure formula looks like this

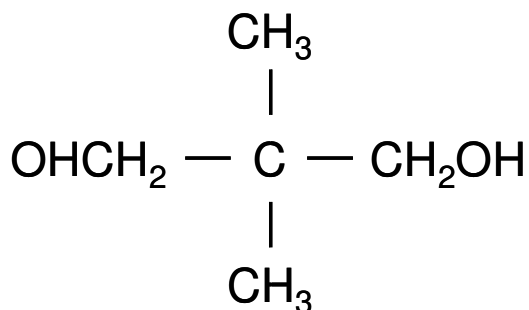


Figure 8.1: The structure formula of Neopentyl Glycol

### **8.4 The Neopentyl Glycol Production Plant**

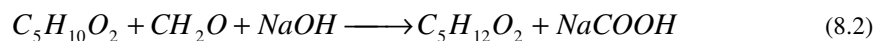
The production plant consists mainly of many different processes but they will not be described in this censored version.

### 8.4.1 Reaction

The chemical reaction is divided into two parts. The first part is to let formaldehyde react with isobutyric aldehyde. The reaction is called *aldol condensation* and is performed with an  $\text{OH}^-$  ion as a catalyst.



The product of this reaction is called HPAL and is used as a reactant in the second part of the reaction. This part is called a *Crossed Cannizzaro Reaction*.



As the observant reader realizes not only neopentyl glycol is created, but also sodium formate. All reactions are temperature dependent but this property is not covered in this thesis.

## 8.5 The Uncontrolled Residual Formaldehyde Content

When this master thesis was started the surplus of formaldehyde was measured three times daily. The specification limits for the residual content was 0.10 – 0.30 %. There were no official control law but the operators were adjusting recipe in order to force the process value into the specification limits. The adjusting parameter is the amount of formaldehyde added into the reactor each batch. The concentration of the formaldehyde varies and is measured, but was not taken into account when adjusting. The recipe used to have a mean of about 8200 kg strong formaldehyde and adjustments of 25 to 50 kg were considered to be a good choice. If there were an observation outside the specification limits adjustments were made and sometimes the result was as hoped, but normally not.

In figure 8.2 the results of the residual formaldehyde analysis is shown. The plot shows values from 2006-09-27 to 2006-10-23. During this period of time the specification limits (black lines) were 0.1 – 0.3 %. It is seen that the majority of the observations are inside the specification interval, which is good. But on the other hand it is also easy to see that the value at sample 54 (0.38 %) is about five times as high as sample 79 (0.08 %).

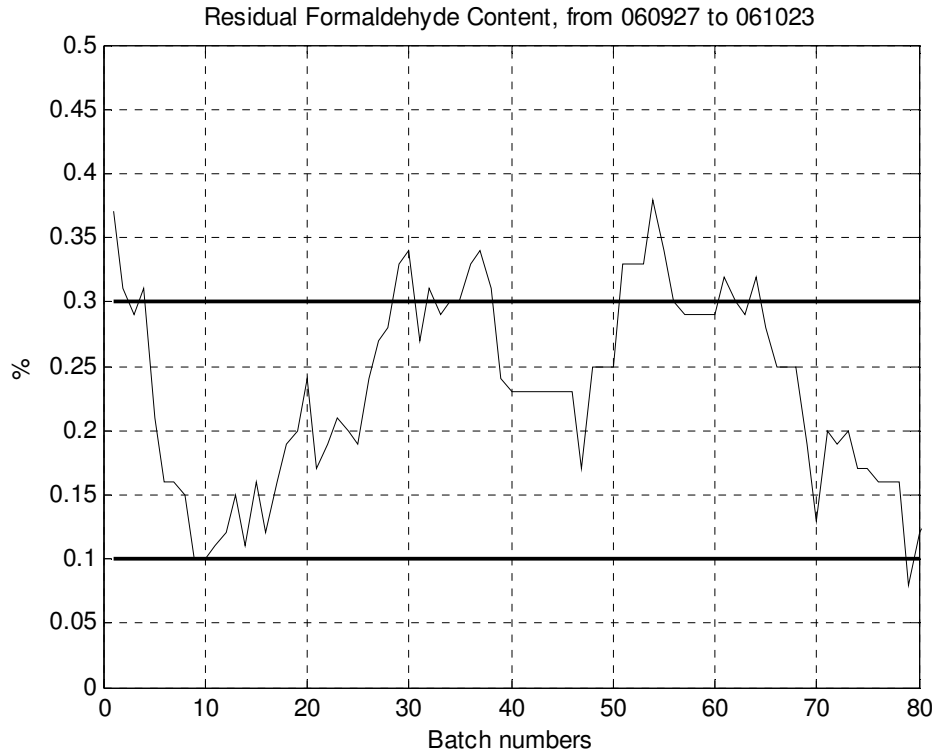


Figure 8.2: Residual formaldehyde content, three observations daily

### 8.6 Analysis of Disadvantages and Faults of Current System

The operators used to make adjustments on the set point for strong formaldehyde with respect to past residual formaldehyde contents. A big deviation from target value used to, but not always, result in a big adjustment and vice versa. No calculations were made and no model of the process was identified. If one should describe the past somewhat imprecise control strategy in mathematical terms, it would look something like in the text below.

In some sense the process could be thought of to be a linear model from strong formaldehyde to residual formaldehyde content. An attempt to identify this obviously wrong model, is made by approximating existing data to (8.3).

$$ExFa = k \cdot Fa - m \quad (8.3)$$

System identification on this model gives, not surprisingly, a poor result that does not match the process values of residual formaldehyde content.

On the data from the time period above, the identification gives the result

$$ExFa = -0.0004 \cdot Fa - 3.6660 \quad (8.4)$$

If this model were describing the real process in a realistic way, it should be possible to calculate the residual formaldehyde content and the result should match the actual values. In figure 8.3 the calculated and real values are shown. It is clear that the model does not describe

the real process in a good way. This discussion motivates this master thesis: The current model does not correspond to the real system, and there is a need for another approach.

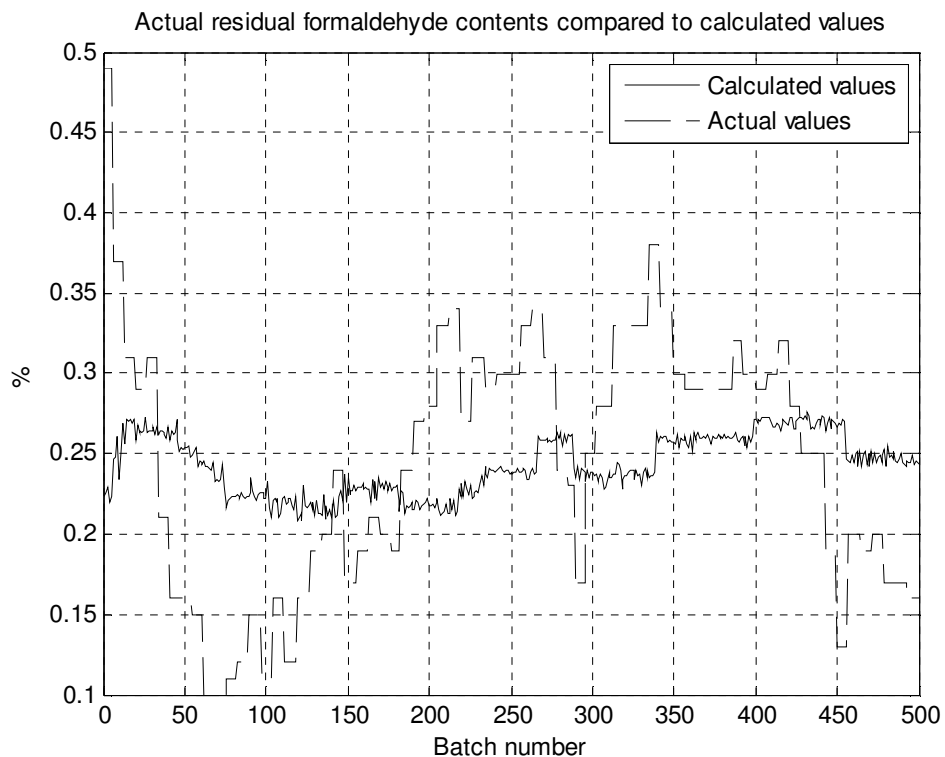


Figure 8.3: Calculated and actual values of the residual formaldehyde analysis

The amounts of chemicals loaded into the reactor are stated in a recipe in the control system. All the amounts of the different chemicals are constant values over long time intervals in this recipe, except one: The total amount of strong formaldehyde is a variable adjusted due to inappropriate values of the residual formaldehyde content.

As with all processes there is a variation in the loading procedure. In Table 8.1 the reactants and their target values are shown with the actual mean value and standard deviations. The set point values are figures from the recipe.

	Set point [kg]	Mean value [kg]	Standard deviation [kg]
Strong formaldehyde			7.5
Isobutyric aldehyde	4800	4816	2.0
Sodium hydroxide	5670	5667	33.8

Table 8.1: Loading accuracy

The strong formaldehyde amount shifted several times during the measurement series and therefore it is not possible to give a target value, neither an accurate mean value. By subtracting the target values from actual values it was possible to create data for calculating the standard deviation.

The biggest disadvantages is that the model or operators does not take into account if the formaldehyde concentration changes or not. A process model from formaldehyde concentration to residual formaldehyde content is linear and is describing the real process in good way.

## **8.7 Theoretical Process Model Based on Mass Balance**

The main object of the process is to turn all of the isobutyric aldehyde into neopentyl glycol; this due to the fact that this chemical is the most expensive of the raw materials. The recipe states that 4800 kg isobutyric aldehyde should be loaded into the reactor. The reaction formula shows that for every mol isobutyric aldehyde there should be two moles of formaldehyde. The molar mass of isobutyric aldehyde is 72.11 [g/mol] and of formaldehyde 30.03 [g/mol]. To calculate the amount of formaldehyde needed to turn all isobutyric aldehyde into neopentyl glycol, following calculation should be done

$$F_a = \frac{I_{bal} \cdot 2 \cdot 30.03}{72.11} = 0.833 \cdot I_{bal} \quad (8.5)$$

The amount of strong formaldehyde is then calculated by dividing the amount of formaldehyde with the concentration of incoming strong formaldehyde. One part of the formaldehyde is fed to the reactor from the incoming raw material and another part is reused from the condensate. The residual formaldehyde from previous batches winds up in the process condensate after the evaporation process (see section about evaporation).

The residual formaldehyde content is then calculated as amount of remaining formaldehyde in the reactor divided by total mass in the reactor. These sentences explain the following figure, Figure 8.4. In the figure the isobutyric aldehyde concentration is assumed to be constant, for this reason there is no input parameter called *isobutyric aldehyde concentration*.



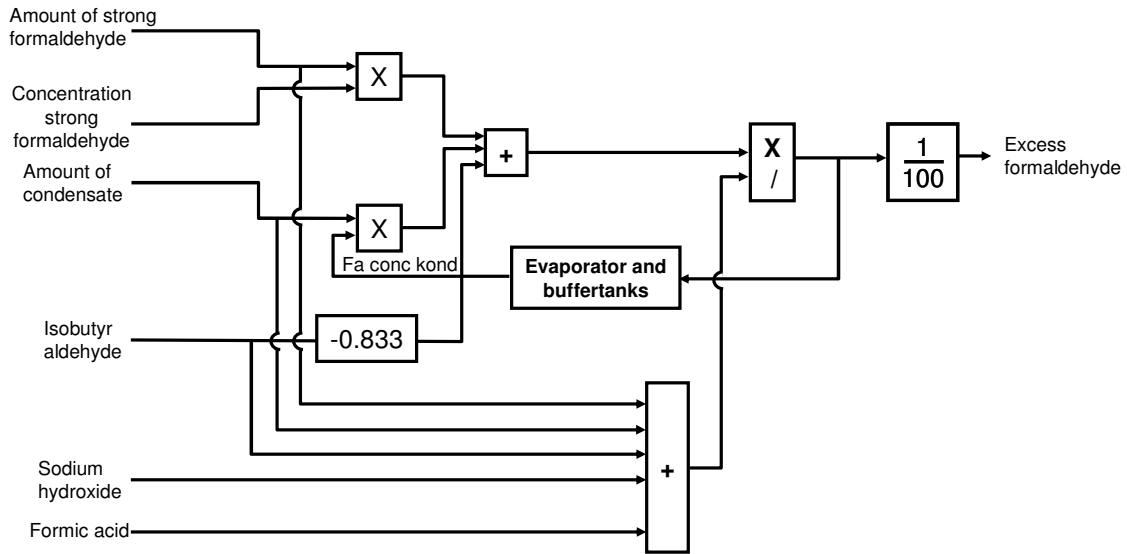


Figure 8.4: Process model, with feedback

In Figure 8.4, there is a feedback term symbolizing reused formaldehyde. The block named *Evaporator and buffer tanks* will in practise act as a low-pass filter with a low cut-off frequency. Intuitively, this phenomenon can be imagined as batch wise adding of formaldehyde through tanks and columns with continuous output. Any sharp differences in the batch wise adding will be mixed with the content of the tank; hence a low-pass filtering will take place.

Matlab and Simulink were used to simulate the system above. The following model (Figure 8.5) rendered a result that was approximately equivalent of real values observed on the real process.

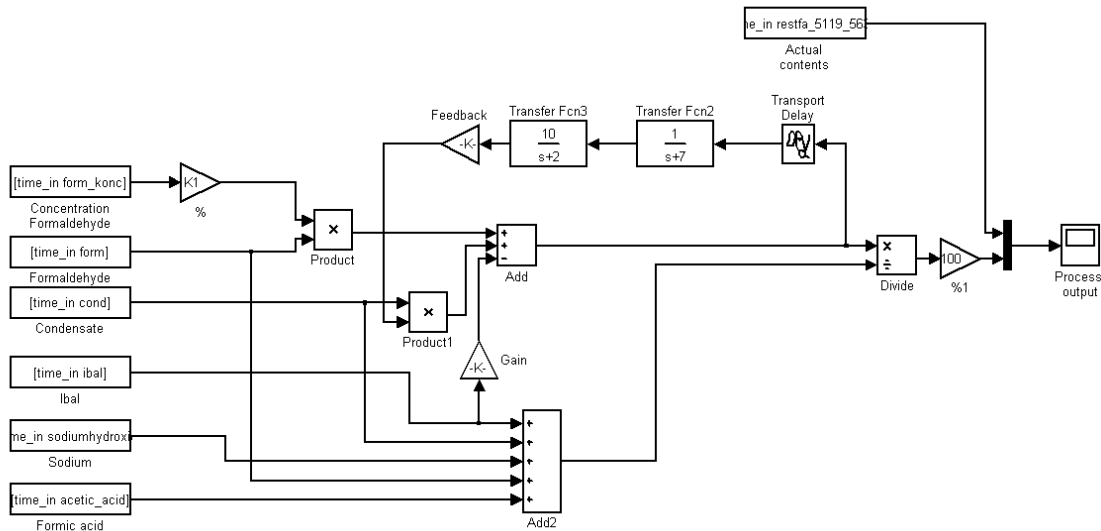


Figure 8.5: A simulation captured from Simulink.

The system of Figure 8.5 was able to produce the following result.

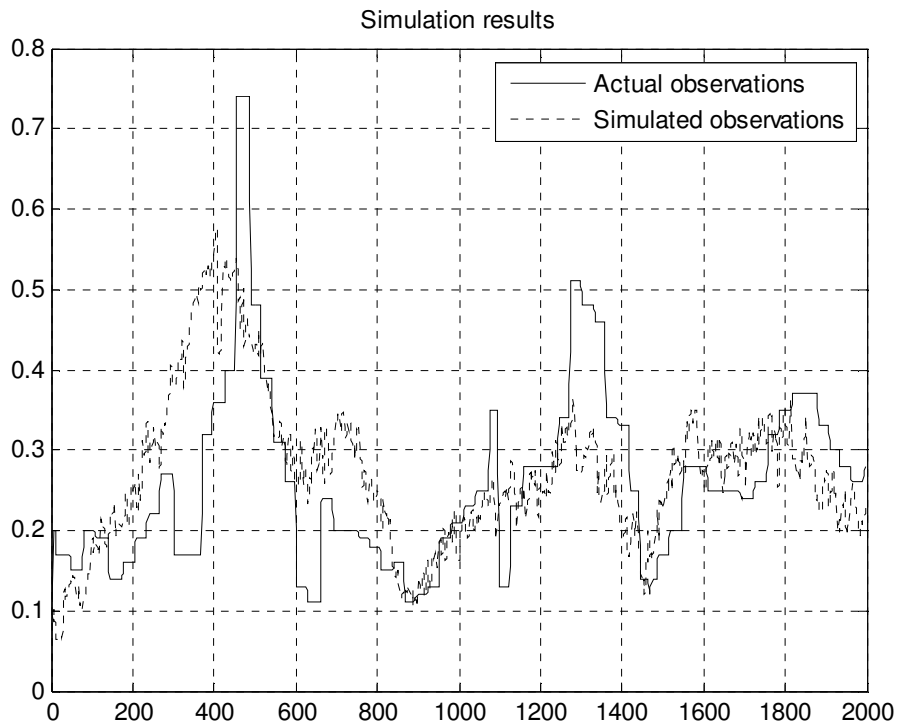


Figure 8.6: Simulated and real values. It is clear that the model is fairly good for values in the interval 0.15 – 0.30. The simulated model has troubles in reproducing the sharp spikes at sample ~1100 and ~1300.

A linearized discrete transfer function from excess formaldehyde (amount off formaldehyde not reacted with isobutyric aldehyde) to residual formaldehyde in the simulation in Figure 8.5 can be derived.

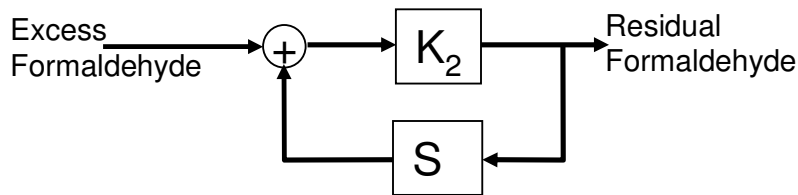


Figure 8.7: Simplified part of process model

In Figure 8.7  $K_2$  is representing the calculation from total amount of formaldehyde left in the synthesis solution to residual formaldehyde. In this derivation  $K_2$  is said to be constant

$K_2 = \frac{1}{54900}$ .  $S$  is representing the formaldehyde in process condensate and corresponds to the time delay, two low pass filters and a constant situated in the upper part of Figure 8.5.  $S(s)$  will look like

$$S(s) = D(2h) \cdot K_1 \cdot \frac{K_3}{(s + P_1) \cdot (s + P_2)} \quad (8.6)$$

where  $K_1$  is the inverse of  $K_2$  and  $D(2h)$  is a time delay corresponding to two sample periods. By discretizing with Euler's backward method, the discrete version will look like

$$S(z) = z^{-2} \cdot \frac{K_1 K_3}{\left(\frac{z-1}{z} + P_1\right) \cdot \left(\frac{z-1}{z} + P_2\right)} = \frac{K_3 K_1}{(z-1+P_1 z) \cdot (z-1+P_2 z)} \quad (8.7)$$

The equation above can be transformed into the following

$$S(z) = \frac{K_3 K_1}{\left(\frac{z}{p_1} - 1\right) \cdot \left(\frac{z}{p_2} - 1\right)} \quad (8.8)$$

The constants  $p_1$  and  $p_2$  are the poles for process part  $S(z)$ .

The discrete transfer function from *Excess Formaldehyde*  $U(z)$  to *Residual Formaldehyde*  $Y(z)$  will then look like

$$P(z) = \frac{K_2}{1 - K_2 S(z)} = \frac{K_2 \cdot \left(\frac{z}{p_1} - 1\right) \cdot \left(\frac{z}{p_2} - 1\right)}{\left(\frac{z}{p_1} - 1\right) \cdot \left(\frac{z}{p_2} - 1\right) - K_3} \quad (8.9)$$

### 8.7.1 Sensitivity Analysis

The process model is only linear from input to output for one parameter, the formaldehyde concentration. For all other variables there is nonlinearity from input to output. It is feasible to linearize the system around an equilibrium point and examine how the theoretical system corresponds to perturbations in input variables.

Perturbation from equilibrium point:	Residual formaldehyde content	Change in residual formaldehyde content due to perturbation		Normalized parameters
According to recipe	0.0900			
1% higher formaldehyde concentration	0.1585	$k_1$	0.0685	1
1% higher mass of strong formaldehyde	0.1583	$k_2$	0.0683	0.9966
1% higher formaldehyde concentration in condensate	0.0926	$k_3$	0.0025	0.0369
1% higher mass of condensate	0.0920	$k_4$	0.0019	0.0280
1% less isobutyric aldehyde	0.1602	$k_5$	-0.0701	-1.0238
1% higher mass isobutyric aldehyde	0.0199	$k_6$	-0.0701	-1.0240
1% higher mass sodium hydroxide	0.0900	$k_7$	-0.0001	-0.0013

Table 8.2: Shows the effect of input perturbations in a linearized model on the output residual formaldehyde content.

Table 8.2 shows the result of making a perturbation of 1 % on the different input parameters, respectively. It is clear from the table and intuitive from chemical reaction formulas that the perturbations on the formaldehyde and isobutyric aldehyde parameters are the ones that affect the amount of residual formaldehyde content the most. Isobutyric aldehyde is a chemical that is bought from Perstorp's Stenungsund site and the purity of this reactant is high (~99.7%) and stable, it has a standard deviation of 0.16 percentage units. The procedure of adding isobutyric aldehyde to the reactor shows very little variation from batch to batch, the standard deviation is estimated to 2 kg.

Since the procedure of adding strong formaldehyde into the reactor, via a mixing tank, shows a large variation in mass and concentration it is natural to focus on the formaldehyde loading of the reactor as a first step.

As mentioned in chapter 8.4.1, the temperature dependency is neglected, but generally a too high reaction temperature will produce low residual formaldehyde contents, compared to lower reaction temperatures.

## **8.8 Controlling the Theoretical Model**

The first approach on stabilizing the residual formaldehyde content is to stabilize the amounts of chemicals fed into the reactor. The interesting number here is the number of moles, not the absolute number of kilograms of the reactant mixed with water, or in other words: kilograms pure product. Since all reactants have different concentrations, the amount of pure product is of course the number of kilograms solution multiplied with its concentration. As mentioned before, both the amount of isobutyric aldehyde fed into the reactor and its concentration are relatively stable. Also mentioned before is that the amount of strong formaldehyde is subject to a rather large variation from batch to batch and that the formaldehyde concentration also varies in a way that can not be neglected.

Hence, while the isobutyric aldehyde can be considered well enough something has to be done about the formaldehyde loading. The formaldehyde concentration is measured continuously and therefore the target value for the strong formaldehyde loading should be a function of the formaldehyde concentration and the residual formaldehyde content from the previous observation, and not only the latter.

When the concentration issue is taken care of the biggest variations should have been suppressed, but it would be a bit too naive to think that the residual formaldehyde content would become stable for a long time after the improvement. Even though it has not been thoroughly examined there are strong reasons to believe that the formaldehyde concentration in the condensate varied a lot. Sporadic tests show that values between 0.22 to 0.55 % has been present. From the sensitivity analysis it is clear that the formaldehyde concentration in the process condensate affects the residual formaldehyde content in small amounts (compared to the formaldehyde concentration) for a one percent deviation from equilibrium point. A change from 0.22 % to 0.55 % is in itself a change of more than 100 %. Therefore these variations should not be neglected. Stabilization in the input of formaldehyde should have the positive side effect of stabilizing the formaldehyde concentration in the process condensate or at least reducing its variance.

Another factor that affects the residual formaldehyde content is the self cannizzaro reaction. The reaction speed is heavily dependent on the temperature and hence should the temperatures in the incoming formaldehyde and the process condensate be as stable as possible. The temperature of the incoming raw material can not be adjusted without reconstruction. The condensate temperature is depending on a large number of condensate flows, but the dominant one is the one from the second step of the evaporator. Stabilisation of the temperature here should not just aid the stabilization of the residual formaldehyde content but also help a smooth operation of the evaporator.

The loading of sodium hydroxide shows a large variation from time to time and should be improved, but since a couple of kilograms extra sodium hydroxide does not influence the residual formaldehyde content this improvement has low priority.

The purpose of a future controller should be to compensate for the deviations that could not be remedied by other measures.

### **8.8.1 Suggested Improvements**

Due to restrictions in time and resources of this project there is just time to test and improve the formaldehyde loading. A PI-controller is developed and tested in theory but will not be tested on the real process. The procedure of weighing the strong formaldehyde is done in two steps. First an amount of 6500 kg is dropped into the mixing tank, second a mass of approximately 2000 kg (this is the manipulatable variable) is added into the same tank.

A feed forward term should be added into the target value for the second weighing. This term should be a function of the formaldehyde concentration and will ideally cancel the effects of varying formaldehyde concentration. A second feed forward term is added to the same place and will compensate for variance due to the first weighing.

A detailed plan (pseudo code) for how the formaldehyde loading should be programmed is stated below.

### **Pseudo code for suggested improvement**

#### **Variable declaration:**

- w1:** Variable that shows the value of the weight cell before loading the first sub batch strong formaldehyde into the mixing tank
- w2:** Weight cell value after loading the first sub batch into mixing tank
- w3:** Target value for the second sub batch
- w4:** Value of weight cell before loading the second sub batch
- w5:** Weight cell after loading the second sub batch
- cfa:** Strong formaldehyde concentration
- fa:** Target value for the total amount of strong formaldehyde given in a nominal concentration
- nomcfa:** Nominal strong formaldehyde concentration
- fatot:** Amount of loaded strong formaldehyde given in nominal concentration
- k:** Adjustable value that defines when the valve starts to close

**Valves, switches and weight cells (See Appendix Fel! Hittar inte referenskölla. for tank numbers and tag addresses)**

- AUV-2105:** Valve controlling the flow to tank V-6210
- WV-2101:** Valve controlling the flow from V-6210 to V-6216
- WC-2101:** Weight cell that gives the weight of V-6210's content
- AY-2084:** Density sensor that calculates the formaldehyde concentration

**Program (instruction list):**

Only the first time the program runs:

**Read formaldehyde concentration from AY-2084 and save in nomcfa**

---

Every time the program runs:

**Open AUV-2105**

**When WC-2101 show 5000 kg, read AY-2084, save in cfa**

**When WC-2101 show 6500 - k, close AUV-2105**

**Wait 10 seconds**

**Read WC-2101, save in w1**

**Open WV-2101**

**When WC-2101 have shown less than 50 kg for 25 seconds, close WV-2101**

**Read WC-2101, save in w2**

**Calculate  $w3 = fa * nomcfa/cfa - (w1 - w2)$**

**If  $w3 > 1900$ , set  $w3 = 1900$ .**

**If  $w3 < 1500$ , set  $w3 = 1500$ .**

**Open AUV-2105**

**When WC-2101 show  $w3 - k$ , close AUV-2105**

**Wait 15 seconds**

**Read WC-2101, save in w4**

**Open WV-2101**

**When WC-2101 have shown less than 50 kg for 25 seconds, close WV-2101**

**Read WC-2101, save in w5**

**Tare the weight cell**

**Calculate  $fatot = ((w1-w2) + (w4-w5))*cfa/nomcfa$**

**Write fatot in batch report**

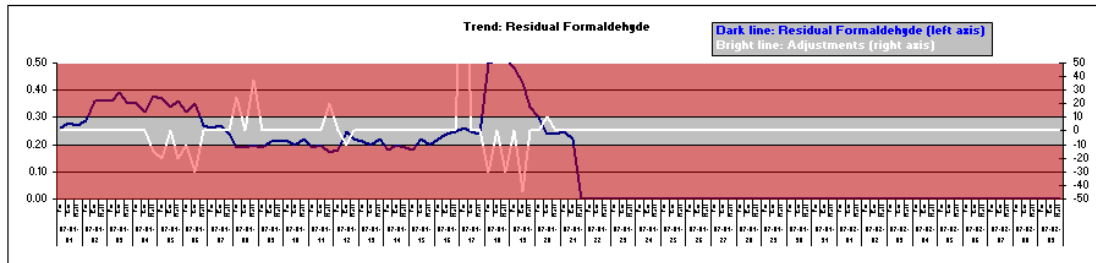
**Start over**

The signal from AY-2084 ought to be filtered before using in program above.

A future controller should give its control signal to the previous feed forward implementation. The variable  $fa$  could be used for this purpose. The controller should also be possible to run in a manual mode, including bump less mode transfer when switching between the modes.

### 8.8.2 Implementing the Feed Forward Term

Due to lack of resources there was no time to implement the program described above in the control system. Instead a bit simpler MS Excel based semi manual feed forward term was implemented. The implementation included sampling the formaldehyde concentration every four hour instead of every batch. When knowing the concentration the target value for strong formaldehyde loading could be calculated. The concentration was read from the control system by the operators and fed into the Excel sheet. The new calculated strong formaldehyde amount was then manually written into the control system. When unsatisfying residual formaldehyde content was obtained, an adjustment parameter was adjusted.



Residual Formaldehyde Adjustment

Date	Shift	Time	Form. Conc.	Adjustment parameter	Calculated amount	Residual Formaldehyde
2007-01-08	Morning	06:00	47.6	25	2181	0.19
		10:00	47.6		2181	
	Afternoon	14:00	47.6	2181	0.19	
		18:00	47.7	2163		
	Night	22:00	47.7	37	2200	0.20
02:00	47.7	2200				
2007-01-09	Morning	06:00	47.5		2236	0.19
		10:00	47.6		2218	
	Afternoon	14:00	47.6		2218	0.21
		18:00	47.7		2200	
	Night	22:00	47.7		2200	0.21
02:00	47.7		2200			

Figure 8.8: A screen shot from the Excel based feed forward implementation. In the trend the light grey area is inside the specification limits while the darker area is outside. The dark line shows the residual formaldehyde contents in percentage and belongs to the left axis. The white line shows made adjustments in kilograms and belong to right axis.

The results from this implementation are described in the result section.

### 8.8.3 Stability Analysis and Final Value Theorem

Before implementing a controller on a system it is necessary to investigate if there will be a stationary error in a step response. In this chapter two controllers are investigated, the proportional controller and the proportional integral controller. The controllers are meant to be implemented according to the basic control scheme in figure 6.1.

The final value theorem and a stability analysis are explained in chapter two.



### Final Value Theorem applied on system with P and PI Controller

The Final value theorem applied on the system simulated in Figure 8.5 controlled by a proportional controller,  $C(z) = K_C$ , will look like

$$\begin{aligned} \lim_{k \cdot h \rightarrow \infty} f(kh) &= \lim_{z \rightarrow 1} (1 - z^{-1}) \cdot F(z) = \lim_{z \rightarrow 1} \left( \frac{z-1}{z} \right) \cdot \frac{P(z) \cdot C(z)}{1 + P(z) \cdot C(z)} \cdot \frac{z}{(z-1)} = \\ &= \lim_{z \rightarrow 1} \frac{K_C \cdot P(z)}{1 + K_C \cdot C(z)} = \lim_{z \rightarrow 1} \frac{K_C K_2 \cdot (3z-1)(8z-1)}{24z^2 - 11z + 11} = \frac{7K_C K_2}{12 + 7K_C K_2} \end{aligned} \quad (8.10)$$

Since the final value does not equal zero, there will be a gain dependent stationary error.

$F(kh)$  is any general signal and in this case it is defined as the reaction in  $E(z)$  on a step change in  $R(z)$ .

The Final value theorem applied on the system simulated in Figure 8.5 controlled by a PI controller will look as

$$\begin{aligned} \lim_{k \cdot h \rightarrow \infty} f(kh) &= \lim_{z \rightarrow 1} (1 - z^{-1}) \cdot F(z) = \lim_{z \rightarrow 1} \left( \frac{z-1}{z} \right) \cdot \frac{1}{1 + P(z) \cdot C(z)} \cdot \left( \frac{z}{z-1} \right) = \\ &= \lim_{z \rightarrow 1} \frac{1}{24z^2 - 11z + 11 + K \cdot K_2 \left( 1 + \frac{1}{T_i} \cdot \frac{z}{z-1} \right) \cdot (3z-1) \cdot (8z-1)} = 0 \end{aligned} \quad (8.11)$$

### 8.8.4 Simulated Controller

The system is simulated with a feed forward term compensating for deviations in formaldehyde concentration and with a PI controller compensating for process value errors. The model in Simulink is shown in Figure 8.9.

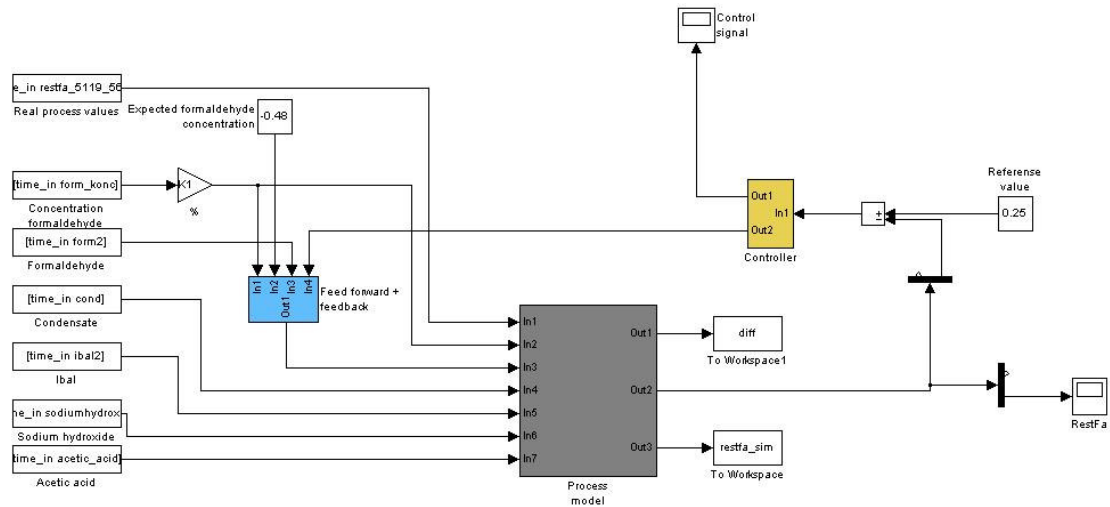


Figure 8.9: Simulink model used to simulate a system including feed forward term and a PI-controller

The strong formaldehyde input signal is contains normally distributed randomized values with a mean of 8450 kg and standard deviation of 15 kg for the first 350 batches, and 8500 kg respectively 30 kg for batch number 351 to 500. A discrete PI controller is used with a gain of 300 and integral time of 20 working shifts. In the plot below, the simulated residual formaldehyde contents are shown batch wise while the control signal is showed shift wise. This is due to the fact that the controller has a sample time of 7 batches, corresponding to sampling once every working shift.

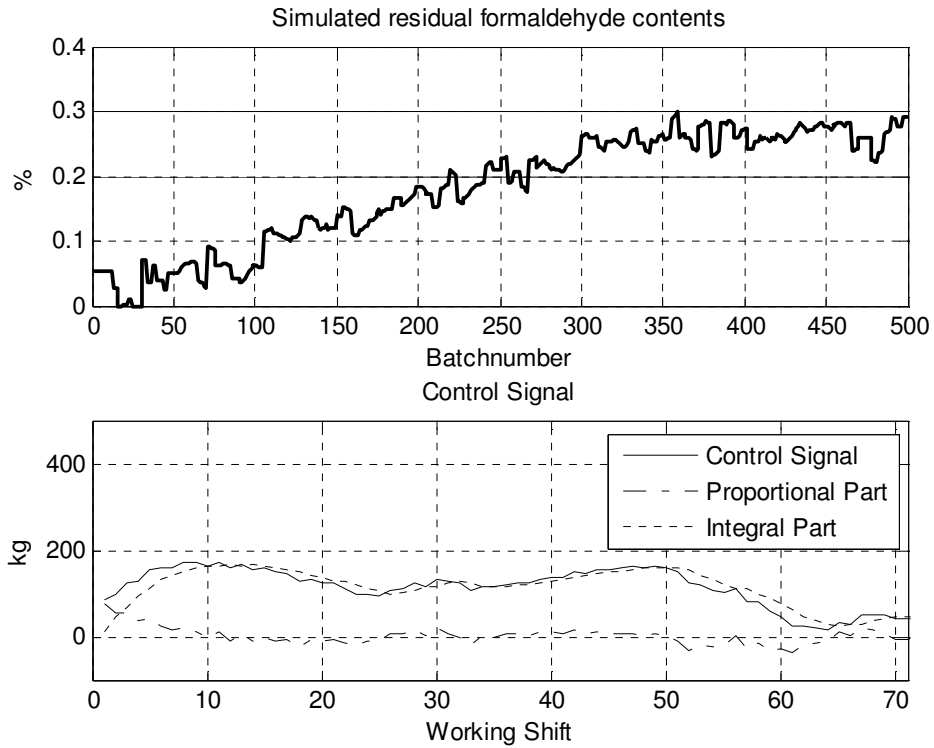


Figure 8.10: The process value from a controlled process.

One can see in the plot above that the model output is fairly stable from batch 250 to 500. Even though it is not realistic to start a system from 0 that has been done here (the real process condensate will always contain some formaldehyde) it is interesting to see that the system reacts slowly to step changes.

## 9 Results

### 9.1 Data Available From the Beginning

The first step of the master thesis was to analyse available data of residual formaldehyde content. In figure 9.1 the results from one month's analyses are shown together with the set point for strong formaldehyde loaded into the reactor.

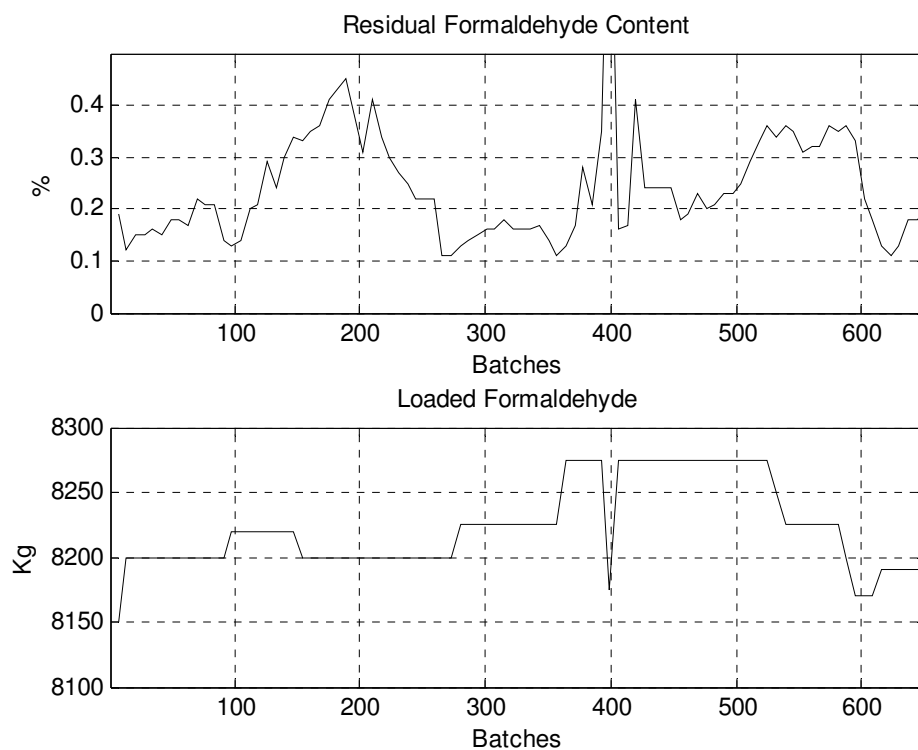


Figure 9.1: Available data on residual formaldehyde content from July 7 to August 6. During this period a sample was taken and analyzed once every working shifts (approximately every seventh batch). The spike at sample number 395 is an outlier due to a poorly calibrated pH sensor. The lower plot shows the target value of loaded strong formaldehyde. No data on actual values on formaldehyde concentration are available for this period of time.

One can see that there is no perfect correlation between the amount of strong formaldehyde solution loaded in the reactor and the residual formaldehyde content.

Figure 8.3 shows the predicted residual formaldehyde by a linear combination between the two variables and it is clear that the process should not be described in that way. Something else needs to be included in the model. The two main participants in the reaction is formaldehyde and isobutyric aldehyde. From available batch reports it is shown that the isobutyric aldehyde loading procedure is very exact and repeatable with a standard deviation of about just two kilograms, in comparison with a total amount of approximately 5000 kg.

## 9.2 High Frequency sampling

To gain more information about the system and especially to get to know if there was high frequent variation that was invisible when sampling just every seventh batch. During one weekend 52 consecutive samples were analysed. The result is shown in figure 9.2 along with the adjustments that were made on the target value for loading strong formaldehyde into the mixing tank.

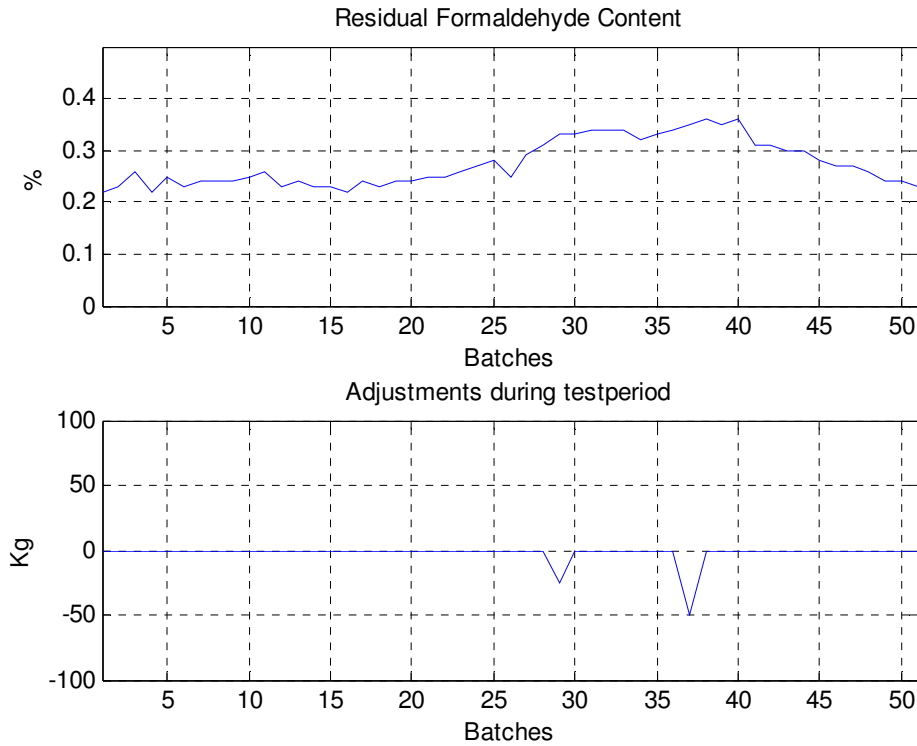


Figure 9.2: Residual formaldehyde content from the test series performed from August 25 to August 27.

During the test series the residual formaldehyde content showed little variation but the values from batch ~25-45 are a bit higher than the others. By looking at figure 9.2 one can imagine some effects from the adjustments, especially after the second one. What can not be seen is a reason for the rise in formaldehyde content after 20 batches. Of course, the amount of loaded strong formaldehyde will have affect on the residual formaldehyde content, but it seems that something else is also affecting the result.

The RS3 control system has a trend over the incoming-formaldehyde concentration but with a poor resolution. The Anyhow, from the printed and scanned trend in figure 6.3 a variation in the concentration can be seen. For this reason it is impossible to plot the actual amount of loaded formaldehyde with good-enough precision during this period of time.

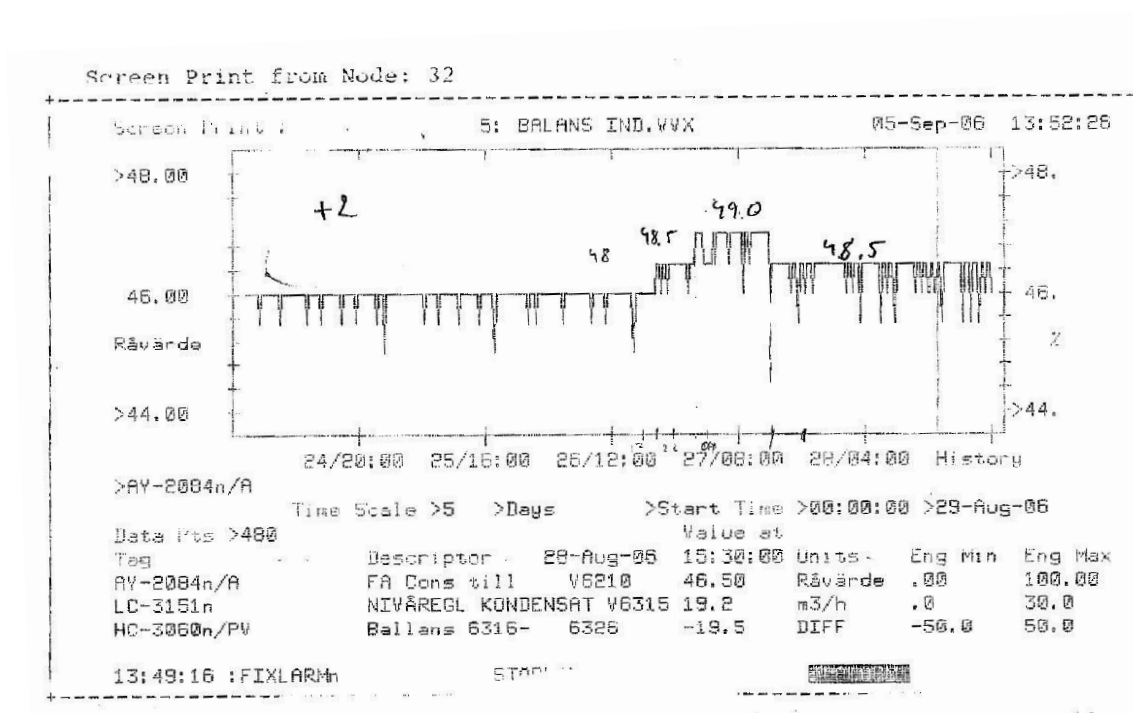


Figure 9.3. The strong formaldehyde concentration from August 24 to August 29. The line is subjected to an offset of 2 percentage units and therefore all values of the scales should be increased by two units. The author apologises for the poor quality of the figure. At the time AspenTech was not installed and there was no other way of extracting the data but making a print on an antique matrix printer.

Without too much imagination and taken into account the poor resolution of figure 9.3 one can see a correlation between the formaldehyde concentration and the residual formaldehyde content.

### 9.3 Analysis Method Verification

The samples taken from the synthesis solution are analysed in a small laboratory in the factory. To be able to establish the accuracy of the method a test series were made on reference solutions and a series where 16 samples were analysed in the factory lab and also with two different methods in a professional lab, later in the text called PA Lab. The two methods used in PA Lab are called the sulphite method and the Hantsch method. The sulphite method is used by the operators in the small factory lab. The latter one is a bit more complicated and takes longer time to perform but has the advantage of being selective to formaldehyde while the sulphite method is selective only to aldehydes in general.

As a start the factory laboratory was tested with three different reference solutions of formaldehyde and water. The solutions contained 0.05, 0.1 and 0.5 % formaldehyde. The results from the sulphite method were

Reference sample concentration	Result of analysis in factory laboratory
0.05 %	0.06 %
0.10 %	0.16 %
0.5 %	0.54 %

Table 9.1: Results from analysing reference solutions of formaldehyde in factory laboratory

The results from the 16 samples are shown in Table 9.2 and figure 9.4. Eight samples were taken from the synthesis solution and eight from the process condensate. Two samples were not analyzed in the factory lab.

Analysis	Sulphite method	Hantsch method	Sulphite method
Location of analysis	Factory laboratory	PA laboratory	PA laboratory
Sample name /Unit	%m/m	%m/m	%m/m
Synteslösn 061115 1000	0.12	0.11	0.10
Synteslösn 061115 1110	0.12	0.12	0.12
Synteslösn 061115 1210	0.15	0.15	0.15
Synteslösn 061115 1335	0.16	0.15	0.15
Synteslösn 061115 1545	0.19	0.18	0.16
Synteslösn 061115 1645		0.11	0.12
Synteslösn 061116 0900	0.17	0.15	0.16
Synteslösn 061116 1010	0.14	0.14	0.14
Process kond 061115 1000	0.24	0.24	0.24
Process kond 061115 1110	0.23	0.24	0.23
Process kond 061115 1210	0.22	0.22	0.23
Process kond 061115 1335	0.20	0.24	0.21
Process kond 061115 1545	0.21	0.20	0.21
Process kond 061115 1645		0.21	0.21
Process kond 061116 0900	0.19	0.17	0.18
Process kond 061116 1010	0.17	0.17	0.18

Table 9.2: Data for verification of factory situated analyses with laboratory analyses.

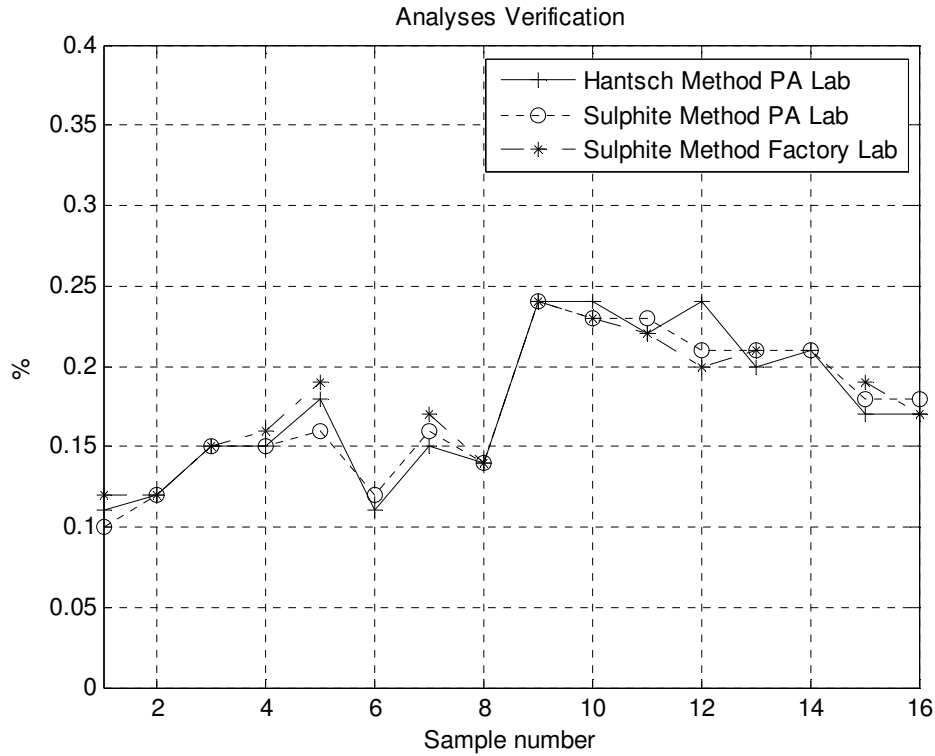


Figure 9.4: The results from Table 9.2 presented in the same plot.

In figure 9.4 it is shown that the three tests (one in the factory lab and two on PA lab) give a good and similar result. One interesting thing is to plot the differences between the three data series to look for offsets or other easily noticeable patterns. In figure 9.5 the results show that no obvious offset or other patterns can be seen in the plots. Therefore, the analyses from the factory lab are considered to be reliable enough for its application.



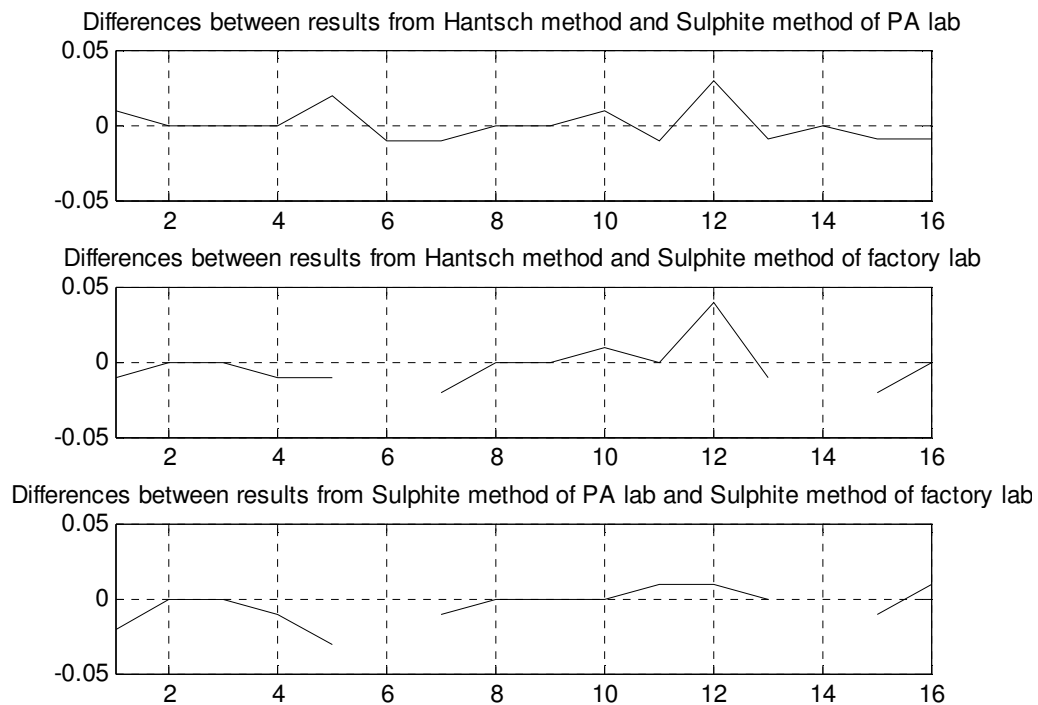


Figure 9.5: Differences between the analysis methods performed on the same samples.

## 9.4 Process Models

### 9.4.1 A Linear Process Model

It was about time to investigate the influence of the chemicals concentration. The sensitivity analysis showed that the concentration of formaldehyde and isobutyric aldehyde affects the system more or less in the same way. Data from the isobutyric aldehyde plant in Stenungsund was obtained while the incoming-formaldehyde concentration was logged once every batch.

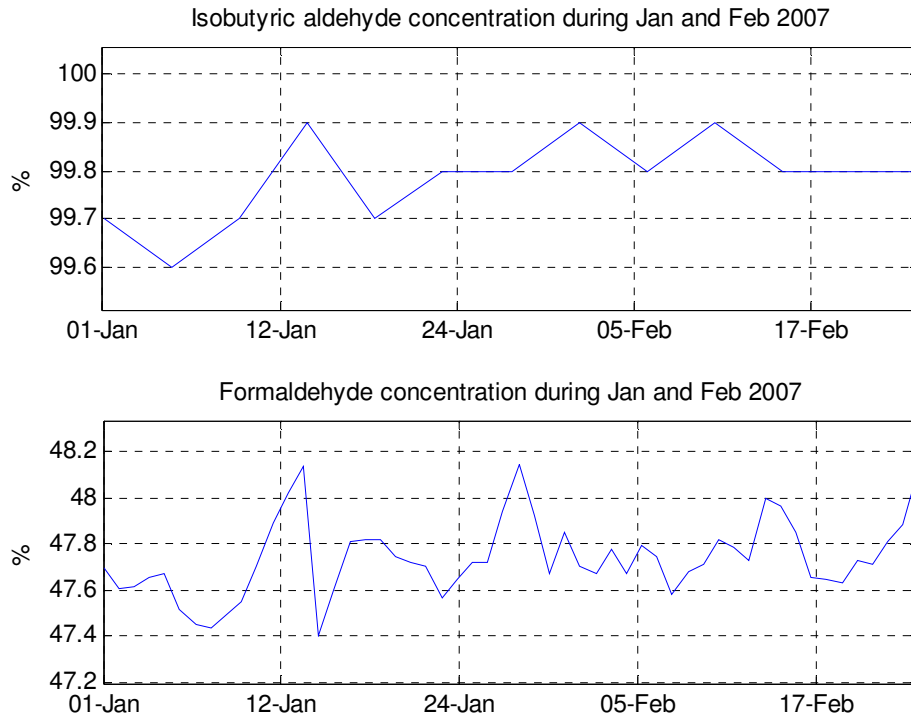


Figure 9.6: Shows the variation in concentration for incoming formaldehyde and isobutyric aldehyde.

The concentration in isobutyric aldehyde is quite stable in comparison with the formaldehyde concentration and can therefore be considered constant. Together with the fact that the loading of isobutyric aldehyde is stable, the focus of this work came to working on formaldehyde loading for quite a while. A new linear process model was developed, this from the amount of formaldehyde to the residual formaldehyde content. The first candidate to a linear model is described in (8.6).

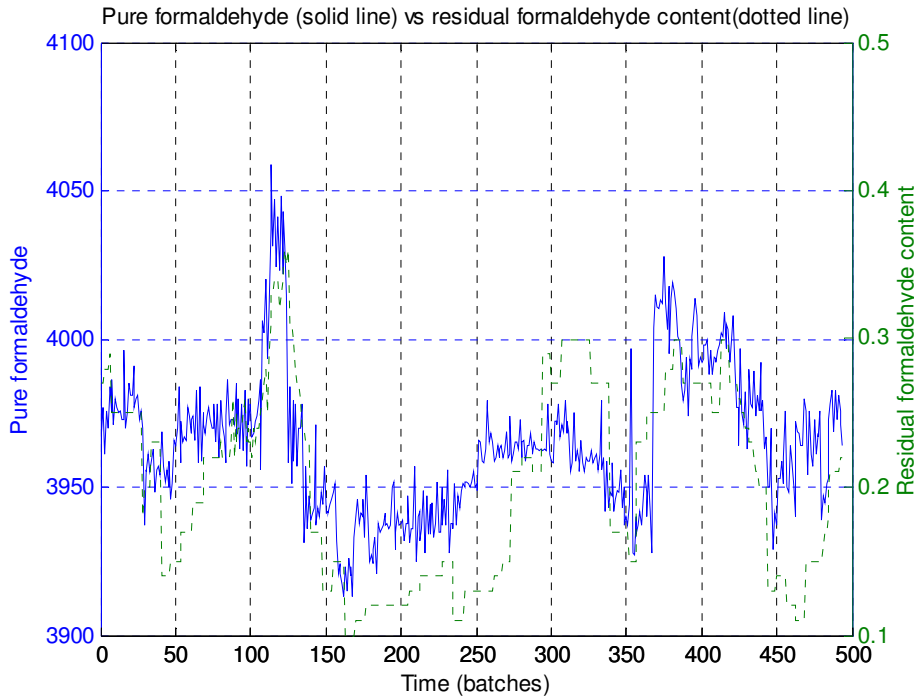


Figure 9.7: Formaldehyde (solid line) and residual formaldehyde content (dotted) in the same figure but with different axes.

The linear model was thought of as

$$Ax = b \tag{9.1}$$

and the residuals

$$\varepsilon = Ax - b \tag{9.2}$$

was minimized in a least square sense.

This is a fairly simple identification process that produces ok result for these data, but by trying to validate the result on another data set will sometimes produce a poor result. Therefore it is incorrect to say that the model can be described by one linear model for a long time period, for example two months or more.

### 9.4.2 Dynamic Process Model

The results of the dynamic process model are shown in figure 8.6

## 9.5 Offset Tuning of Formaldehyde Loading Process

In the beginning of the project the formaldehyde loading was subjected to an offset of about 30 kg (varying a bit over time) and after two adjustments it was tuned into having a close-to-zero offset. Figure 9.8 shows that the loading's offset is close to zero but varying somewhat.

An offset shift can be imagined around batch 300 in the plot. The reason for this shift has been searched but is unknown at the writing moment.

The strong formaldehyde loading is stopped by closing a valve. The stop signal is given when the right amount of strong formaldehyde is loaded. To compensate for extra unwanted formaldehyde loading due to time delays in the valve closing, the stop signal is given at a preset value before the target value is reached. The preset value was adjusted from 10 to 20 kg and thereafter to 27 kg.

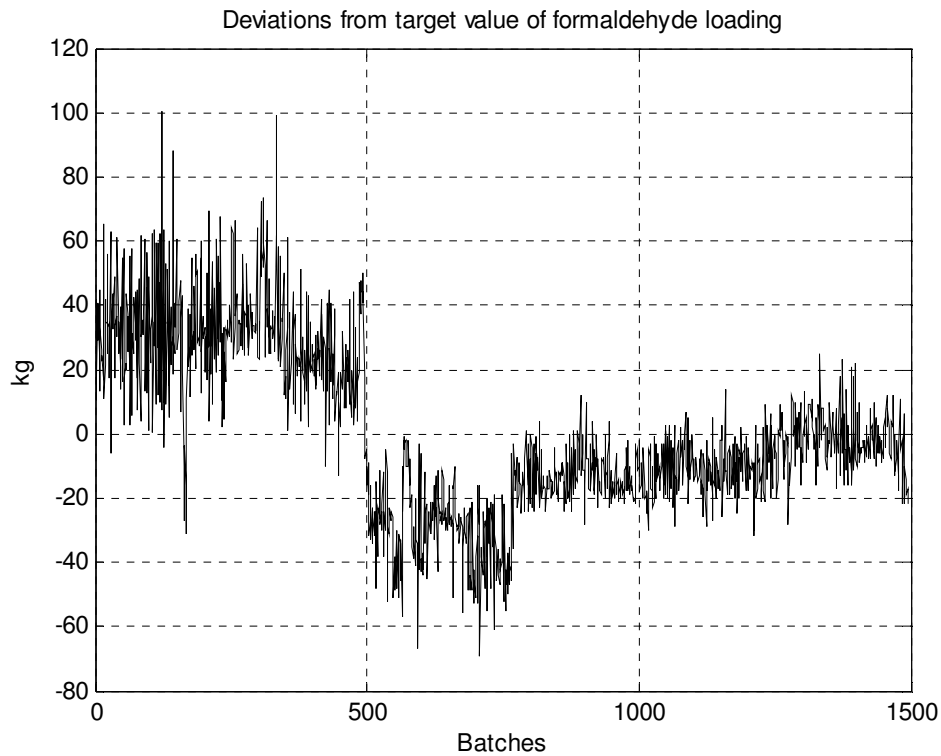


Figure 9.8: Deviations from target value of strong formaldehyde loading. The data are collected from the time period of August 21 to November 10 2006.

## 9.6 Evaluation of Implementing Feed Forward Term

The Excel based implementation of the feed forward term had a good start. After a couple of days stabilizing the process with many samples and extra monitoring, good results were a fact in the beginning.

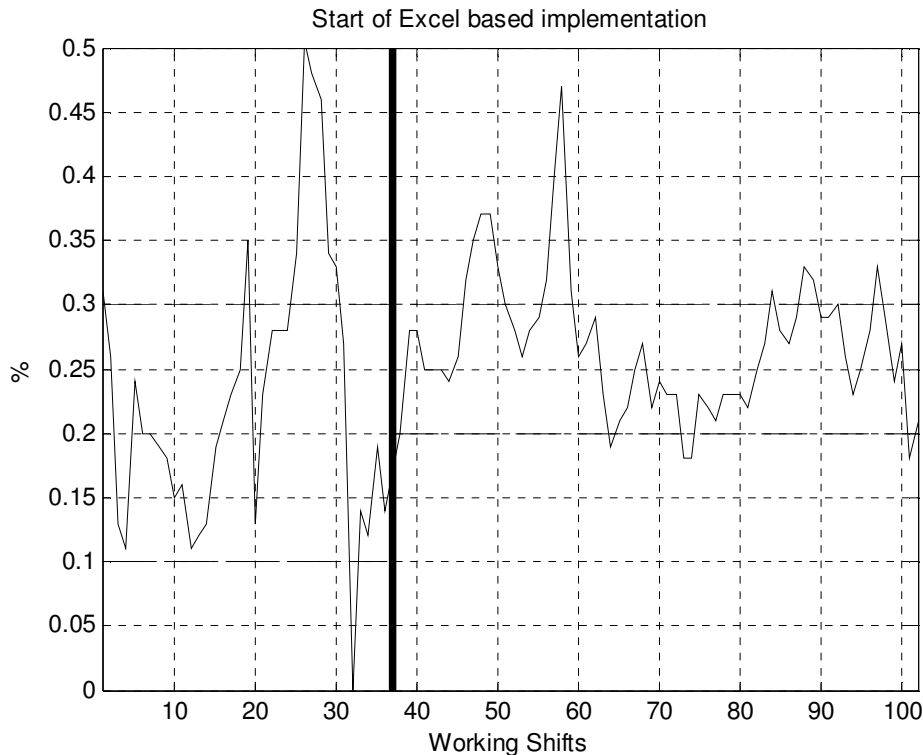


Figure 9.9: The Excel based feed forward term was implemented November 16 which corresponds to sample number 37 and the thick black line in the figure. The specification limits are shown with dotted lines and were before the implementation a bit more generous (0.10-0.30 %) than afterwards (0.20-0.30 %). Sample 32 is an outlier.

As the last sentence implies the results were not as good all the time, this will be presented later but first a more deep presentation of the results for the time period from November 23 to December 7. During this time period the specification limits were changed from 0.10-0.30 % to 0.20-0.30 %. Figure 9.10 shows the residual formaldehyde contents during this time complemented with adjustments from the operator and a plot showing the amount of formaldehyde loaded into the reactor. As can be seen when a stable and even amount of formaldehyde is loaded a stable and even result is attained. In this time period the process could be looked upon as being in statistical control with a fairly good process capability, compared to before. It would be good to estimate standard deviation and process capability but for achieving good results more data is needed. The recipe changes were made during the time period and extra samples were taken in order to have a continuous stable process.

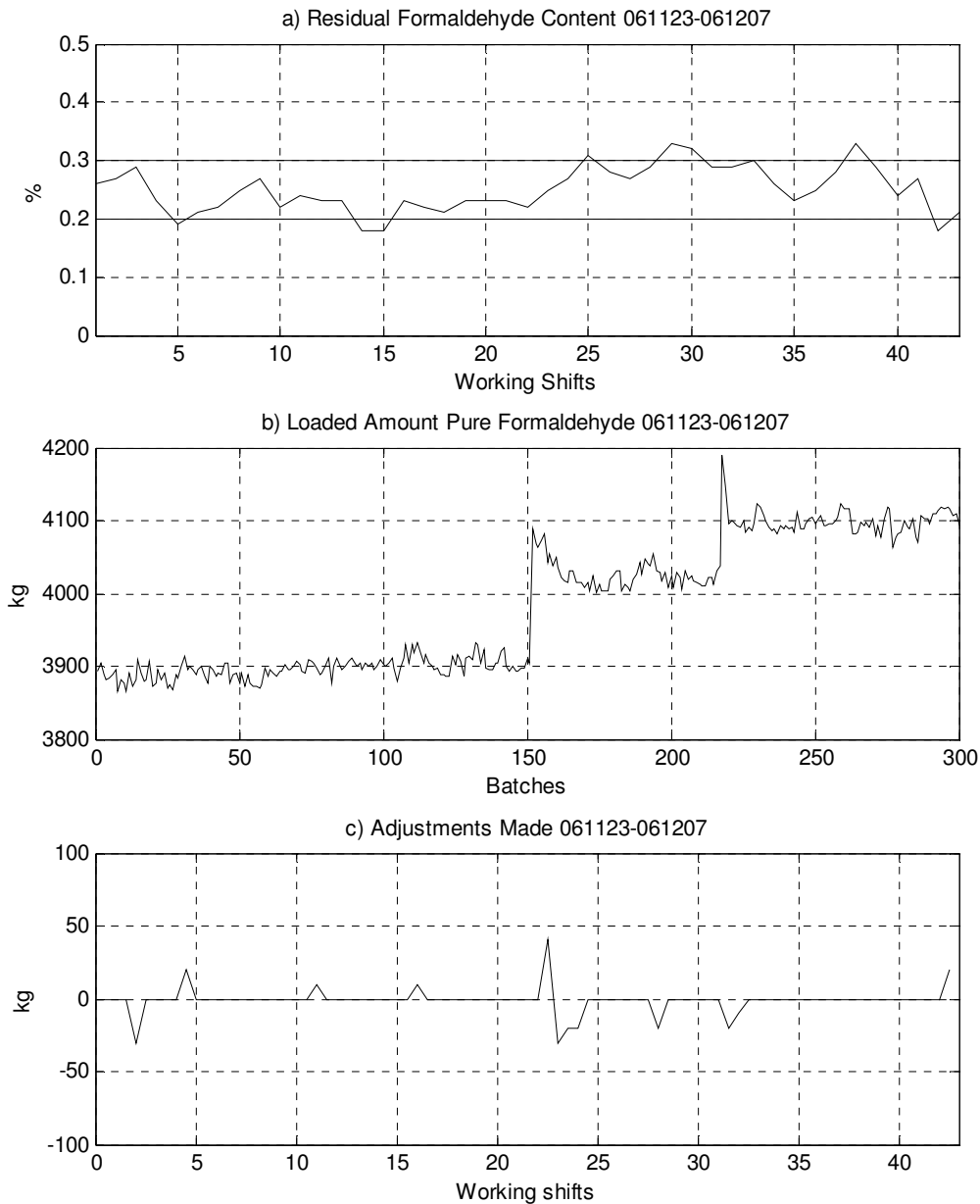


Figure 9.10: a) Results for the two first weeks after the Excel based feed forward implementation. During this period the upper specification limit was 0.3 % and lower specification limit were 0.1 %. b) Formaldehyde during the same time period as plot a). Two recipe changes were made during this period which can be seen in the lower plot after 150 and 220 batches. c) Adjustments made by the operators.

In the time period between December 8 and December 29 the results are not as good as before. In figure 9.11 the results are shown in the same way as figure 9.10. Some of the rises and falls in figure 9.11a) are easy to explain while others are not that easy.

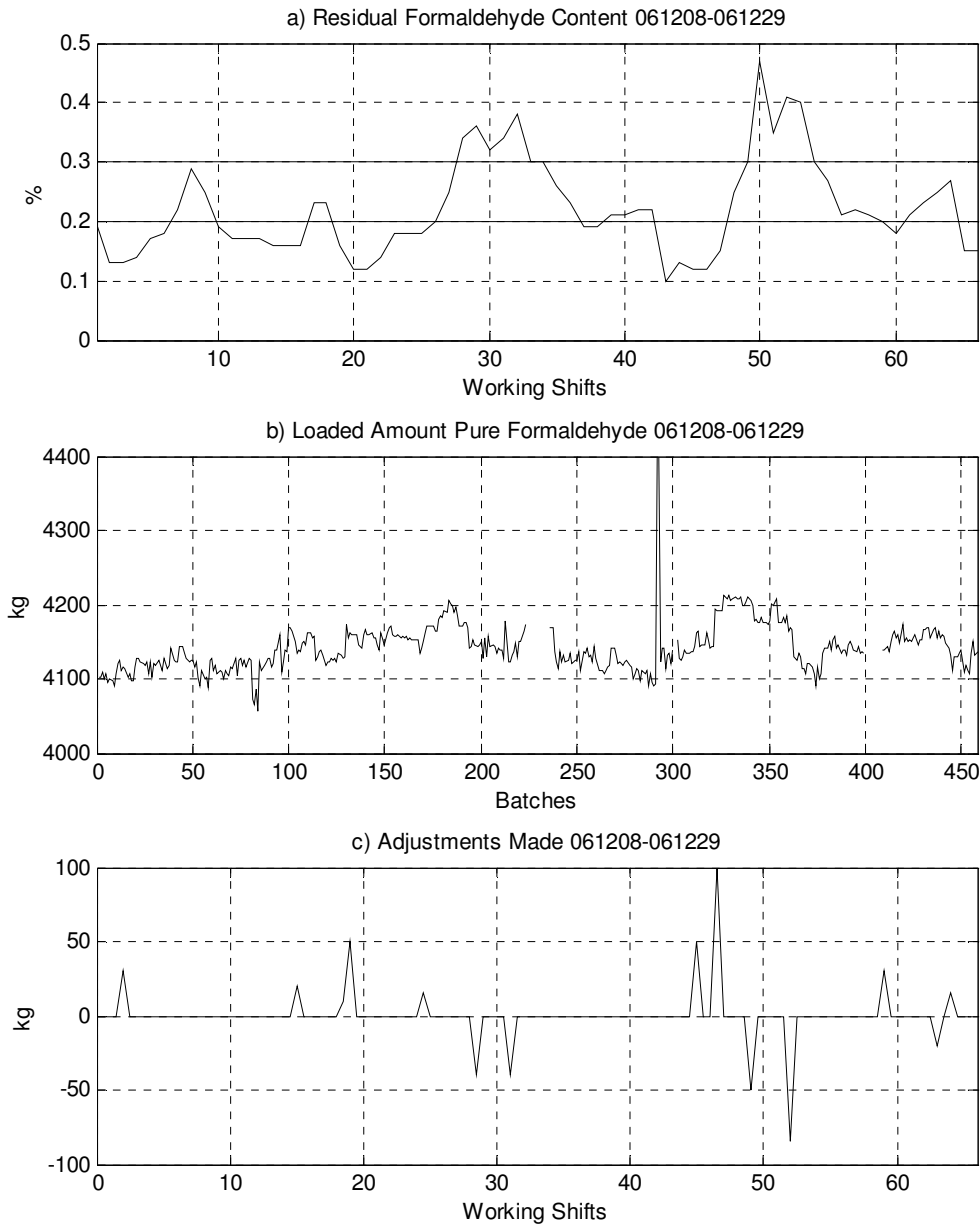


Figure 9.11: a) Residual Formaldehyde during 061208-061229. Sample number 33 is an outlier. b) Loaded amount of formaldehyde. c) Adjustments made by the operator.

Some comments on the variations in figure 9.11.

- Rise and fall between December 10 and 11 (sample 6-10).** The Excel chart was not updated for two working shifts and as a consequence the feed forward term did not compensate for a concentration change of 0.5 % units. A small shift upwards can be noticed in loaded strong formaldehyde due to an operator adjustment. By looking at the reactor temperature, with the help of AspenTech, one can see that there were a couple of degrees warmer during this time period. The uncompensated concentration shift is most likely to explain the rise.

- **Fall between December 13 and 14 (sample 17-21).** As in the last case there is also a change in reactor temperatures during these 48 hours. The outdoor temperature rose 4° C in a couple of hours which caused the temperature of the cooling water to rise. As a consequence many devices where the temperature is measured showed a positive temperature shift. It is known that all the reactions are temperature dependent which possibly could explain the fall in formaldehyde content due to a faster self-cannizzaro reaction.
- **Rise and fall between December 16 and 18 (sample 46-55).** During one day two too big positive adjustments were made. Even though the guidelines for the operators to follow stated that appropriate adjustments were in the range of 20-30 kg, the first adjustment was of 50 and the second of 100 kg. This explains the rise on the residual formaldehyde content, and just as logical is the fall after two too big negative adjustments the following days. On other series of residual formaldehyde content it can be shown that illogical operator adjustment are causing big variations.

### Residual Formaldehyde Content January 2007

The results during this time period are shown in figure 9.12. Unfortunately the exact formaldehyde amounts are not available when the thesis is written.

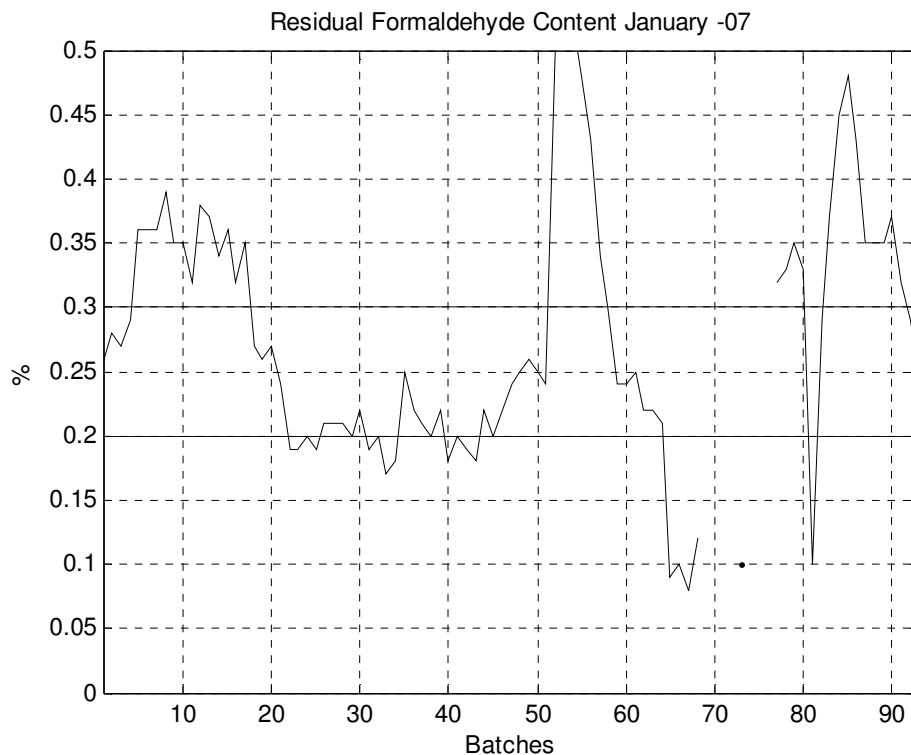


Figure 9.12: Residual Formaldehyde Content during January 2007. The plant was not operating from January 24 to January 26 which is seen in the plot from batch 68 to 77.

One can see that not many observations are below the lower specification limit even though the formaldehyde content is varying quite a bit.



## 9.7 Reactant Yields

One of the goals of the project was to increase the yields of the main reactants formaldehyde and isobutyric aldehyde. In figure 9.13 the yields are shown from January 2004 to January 2007. The Excel based implementation made in this project was introduced in mid November 2006 which corresponds to the bold vertical line in the figure.

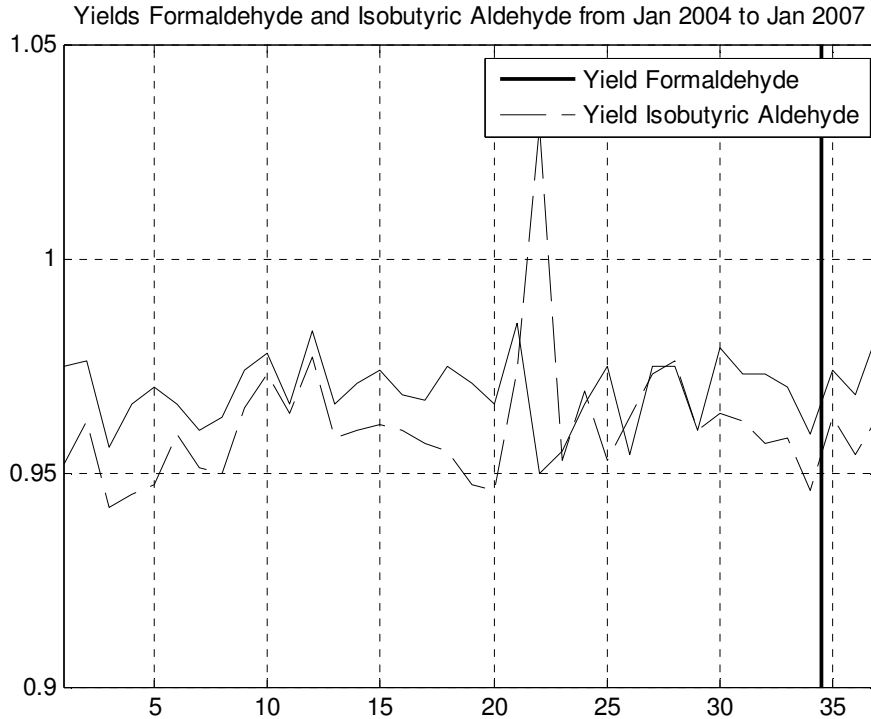


Figure 9.13: Yields of the reactants formaldehyde and isobutyric aldehyde. The Excel based feed forward term was introduced between sample 34 and 35. There is one obvious outlier in the formaldehyde yield.

It is hard to draw any major conclusions from figure 9.13 but it is noticeable that in January 2007 the second highest yield during three years was achieved. During this month there were few residual formaldehyde contents below the lower specification limit. This is probably not a coincidence.

## 10 Comparison, Discussion and Analysis

### 10.1 Reflections on Statistical Process Control

#### 10.1.1 In General

The theory of statistical process control was developed from or for the parts manufacturing industry. Therefore it takes a while and some reading to find parts that are directly applicable for the process industry. Still, the foundations of the subject are interesting in a philosophical sense. The discussion about common and assignable causes should be interesting for almost any worker in any manufacturing industry. By defining phenomena in the factory as something to accept and compensate for, or not, is a good way to intuitively get a good sense for a problem. The theory seems to have its strength in the early phases of handling with a problem like detecting and defining it, whilst the actions taken from conclusions based on SPC theory are heavily application dependent and not covered in the literature. When a project comes to its implementation phase there are some tools for detecting improvements and decide whether a process is in statistical control. The phase I and II concept is used in this area but seems to be rather ad hoc, this because the theory is used for deciding whether a process is said to be in statistical control or not, but on start up after improvements the properties of the in-control process is unknown.

By reading articles and books in the subject it is clear that the areas of SPC and automatic process control are getting closer and closer and many experiments shows that a combination of the two is better than one alone. Automatic control schemes, such as proportional integral controllers, are treated in many SPC books. These schemes contradict in some way the foundations of SPC in the matter of not making adjustments if an assignable cause is not present. Some friendly rivalry between the theories is shown here and there in sentences like “*despite good intentions the best result was reached when the automatic controller was turned off*”. An automatic control practitioner would probably say that the controller was inappropriate or mistuned.

If a controller with a dead zone interval corresponding to the control limits of a control chart was used, there would be no unnecessary adjustments. A condition for this is that all possible (or at least most of them) assignable causes could be remedied by adjusting the manipulated variable. The writer of this thesis has not found any research in the area of SPC combined with nonlinear control such as the dead zone.

The sub theory of cuscore statistics is somewhat more mathematically advanced but it also opens up more areas where SPC can be applied. Especially the ability to recognize known-to-happen faults in noisy signals is of great interest. This has a potential for signalling when maintenance is needed instead of performing maintenance at, sometimes, too short time intervals. One disadvantage with many cuscore statistics is that the time, when a disturbance is starting to affect the system, needs to be known for best performance.

A theory that covers the same area in some sense as cuscore is change detection. Many books are written in this subject, but the theory is not covered or handled in this thesis. The interested reader should consider reading *Adaptive filtering and change detection* [17] by Fredrik Gustafsson.

### **10.1.2 Applying SPC on the Case Study**

The case study of trying to control the residual formaldehyde content has primarily focused on design faults, such as the lack of feed forward term on the formaldehyde concentration. With some good will this could be thought of an assignable cause occurring in the design phase of the project but this is far fetched.

When starting this project the process could be said to be in some sense of statistical control, deciding this is up to the practitioner. This could be investigated with some control chart for correlated data like the ewma chart. Since the system clearly did not display the wanted properties little time was invested on this. After implementing the excel sheet based feed forward term some temporary control limits could have been established, but since the system was troubled by many operational errors it would be hard to use a control chart in a systematic way.

In some cases assignable causes have occurred which were afterwards detected by control charts. For example, a pH-sensor in the plant laboratory displayed a shift in its measurements. The assignable cause was taken care of by the maintenance department when they performed one of their regular pH-sensor calibrations. Stated before was that automatic control and SPC share some areas of work and the example with the pH-sensor indicates that also the area of maintenance share common interests with SPC.

With a complete and good process model there are reasons to believe that the process could have been tuned into almost perfection and the monitored by an appropriate control chart. The process model is fairly accurate but not good enough. From a period with stable values the standard deviation could have been estimated and used to design control charts. This is not a good idea in this phase since the process has produced unpredicted changes without any standard assignable causes present; the changes were probably due to unknown properties of the process instead of occurring errors in some part of the process. Fluctuations in temperature and other unknown disturbances would signal the control chart, probably soon after its start. By following the standards of the SPC theory the process should then be stopped and the assignable cause should be identified and taken care of. Clearly this is not the way to go. For two obvious reasons, no product would come out in the other end of the factory and the improvements of this master thesis should not include massive reconstructions.

Instead there is need for an automatic controller that compensates for the fluctuations mentioned above. In this master thesis, there has not been time or resources to test an automatic controller on the physical process but there are reasons to believe that it would decrease the variance in the residual formaldehyde content.

## **10.2 Complexity of the System**

It is always desirable to have a complete and perfect process model, but this is seldom possible. In this master thesis the process model is based on the reactions stoichiometry. A more skilled person in chemistry might be able to include more parameters. Below is a list over variables that affect the residual formaldehyde content. There is no guarantee that the list is complete but it will still make its point when it comes to show the complexity of the system. The variables are stated but it remains on deciding how they will affect and interact with each other. Some system identification skills would be a good start but it would be too costly to make sufficient tests on all of the parameters in the process plant.

1. Amount of isobutyric aldehyde
2. Amount of formaldehyde
3. Concentration of isobutyric aldehyde
4. Concentration of formaldehyde
5. Amount of condensate
6. Formaldehyde content in condensate
7. Amount of sodium hydroxide
8. Amount of formic acid
9. Starting temperature of the reaction
10. Maximum temperature of the
11. Evaporator performance
12. Batch length
13. Reaction time

Table 10.1: Variables that affects the residual formaldehyde content

On top of all variables in Table 10.1, it remains to decide how correct the sensors are and how much a deviation in some value will propagate through the system. The sensitivity analysis in chapter 8.7.1 takes up the dominant parameters. Other variations and uncertainties affecting the process are stated below.

### **Loading procedure**

All loading procedures in the process show some variation. It is mentioned before, but the object of this section is to show that also the mean value of the loading procedure displays variations that are not predictable. Figure 9.8 shows the difference between actual value and target value for strong formaldehyde. The first 150 batches displays a irregular behaviour while the other batches have a more stable mean value, even though there is a mean value shift around batch 300. Noticeable are the extreme values that have occurred, that in some cases are unexplained but still have happened. Others are due to problems in the factory and the loading procedure.

The same pattern as in figure 9.8 is recognised in the loading of sodium hydroxide but not in isobutyric aldehyde. The difference in loading procedures between formaldehyde and isobutyric aldehyde is when emptying the tanks. Stopping the emptying procedure at a predefined value, as in the isobutyric aldehyde loading shows little variation and it should be investigated if this procedure would reduce the variance of formaldehyde and sodium hydroxide loading.

### **Model**

Model uncertainties have been covered in section 10.2.

## Sensors

Since the sensors are responsible of converting nature into number it is crucial to have a properly working sensor.

## Operators

A process that is not controlled by a controller will depend on adjustments made by the operators. There are general guidelines available for the operator to follow but the guidelines are not very precise and are not always followed by the different operators. This has rendered many sequences of illogical adjustments; some of the sequences are shown in Table 10.2.

	<b>Shift(<i>i</i>)</b>	<b>Shift(<i>i+1</i>)</b>	<b>Shift(<i>i+2</i>)</b>	<b>Adjustments</b>
Example 1	0.17	0.14	0.11	50
	0.18	0.13	0.11	0
Example 2	0.24	0.24	0.24	0
	0.24	0.24	0.25	-50
Example 3	0.23	0.23	0.25	0
	0.24	0.24	0.25	-50
Example 4	0.12	0.12	0.15	100
	0.11	0.12	0.13	0

Table 10.2: The examples in the table show two sequences of residual formaldehyde content values that have resulted in very different adjustments.

This uncertainty could be minimized by automatic control but the operator will always have the opportunity to manually adjust the process. Another uncertainty with operators and the plant itself is that on some working shift there might not be time to perform the residual formaldehyde analysis.

## Analyses

The semi manual procedure of analysing the residual formaldehyde content shows only small variations in the analysis detection area. Results from laboratory verification are stated in the result section. One uncertainty is that the analysis procedure might not be able to detect concentrations below 0.1 %. It is still uncertain what really happens with the result if the concentration is below the one just mentioned. Since the lower detection bound is in more or less the same size as the previous lower specification limit, this is something that should be investigated by experts in the area.

## **The Closed Loop System**

The closed loop system including process model, feed forward term and controller needs to be stable and as robust as possible. There are uncertainties in a controller like mistuning and switching between manual and automatic mode. A bump-less mode change feature can be implemented but the design phase faces the, this far into the report, familiar problem with incomplete process model and restricted possibilities to extensive testing on real process. The robustness of the system is critical and will be deciding how well the system can handle all uncertainties and variances mentioned above.

Based on these complications a simplified model is used.

## **10.3 Suggested Tasks and Improvements for the Future**

### **10.3.1 Statistical Process Control**

The SPC theory has its biggest advantages when it comes to monitoring in-control processes and signalling when it is falling out of control. The residual formaldehyde process needs to show signs of being in statistical process control and have a bigger process capability before it should be monitored by SPC tools.

There are other situations in a production plant that could benefit from clever SPC algorithms. Here is an example of the neopentyl glycol plant.

#### **Example: Malfunction of separation box**

After the evaporator the synthesis solution is pumped into a separation box. When it is properly working it is separating the solution into a salt phase and an organic phase. Each phase is led to a buffer tank. The process is simple but can still malfunction. In this example the pipe for salt phase to its buffer tank gets plugged and therefore stops the flow. As a consequence both phases wind up in the organics phase's buffer tank. This is clearly shown in figure 10.1 where the flow of salt phase is stopped at the first yellow marker and the problem is solved at the second marker. What is shown in the picture is not the flow itself but the levels of corresponding buffer tanks. From the buffer tanks the crystallisation and distillation processes are fed. During this time period the feeding rate for those processes were approximately constant.

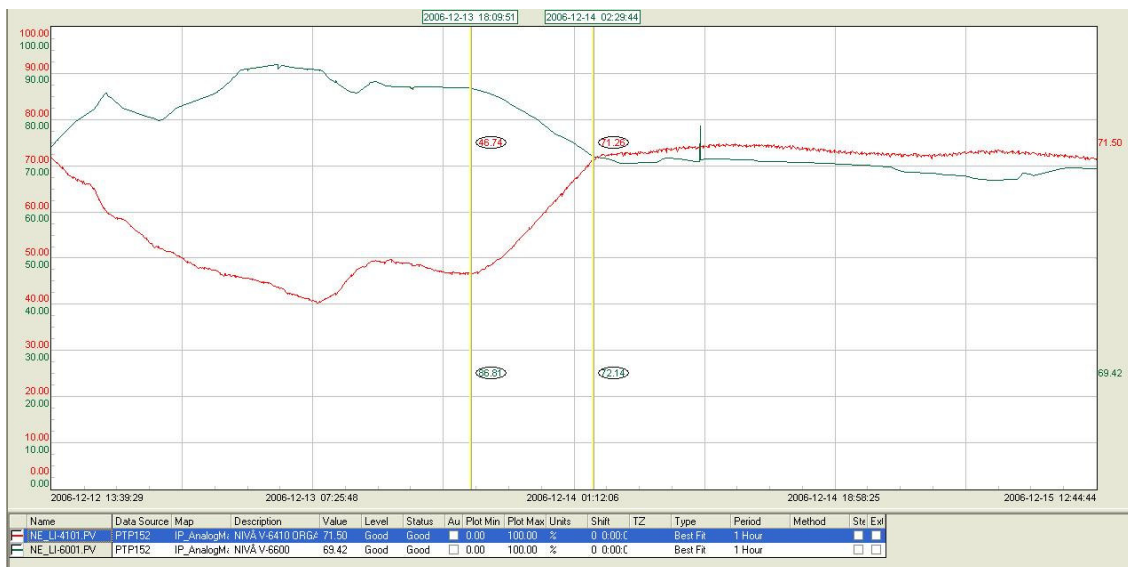


Figure 10.1: An example of when the flow of salt phase from the separation box stops. The red line (the lower one in the beginning of the plot) is the buffer tank for the organic phase while the green (the upper one in the beginning of the plot) is the same for the salt phase. The figure is generated with the AspenTech Process Explorer.

An SPC approach to this problem is to have a control chart, or maybe just a mathematical algorithm, that signals when both lines in figure 10.1 changes its derivative considerably at the same time. This is a clear example of an assignable cause.

This is just one example and there are probably many more of these in Perstorp's factories over the world. In the example the assignable cause affects the system for more or less eight consecutive hours before it is detected and remedied by the operators. In this case, the assignable cause is easy removed by removing the plug with water or steam. The benefit of an automatic signalling of this problem is obvious.

### 10.3.2 The Process and the Process Model

There are definitely possibilities to improve the process model and all knowledge about the process is valuable. The reaction in the reactor and the by-products could, if they are predictable, be included. The self-cannizzaro reaction should also be thoroughly investigated and understood. Without investigating it very deep it is clear that it is heavily temperature dependent. Since the temperature of the condensate and reaction solution is measured continuously and the fact that temperature measurements often are reliable, the including of self-cannizzaro reaction in the process model would probably make it better.

The Excel based feed forward term should be implemented in the process computer and then be evaluated once more. The new batch program should be prepared for taking in a control signal for a future controller. The controller should be able to run in automatic and manual mode with bump less mode transfer. When the controller is evaluated there is need for investigating whether the controller's gain should be a function of variables like temperature or just a constant. As a last approach a more advanced controller should be investigated, but only if considerable benefits are available. There is probably no use of investing time and money if an advanced controller does not perform much better than the simpler one.

There are also some aspects of the process plant that could use some investigation. One is to evaluate if the loading procedure of chemicals into the mixing tank and the reactor could be improved in a sense of variance minimization. The temperature of the condensate depends

heavily on the temperature of the last step of the evaporator. From a brief look at the process values and control signals of the temperature controller, it seems to need a new tuning. This could help the temperature of the condensate to stabilize. An extra approach that might be considered is if the condensate tank could be temperature controlled, by cooling water and a heat exchanger. If cooling the condensate is a good alternative this calls for investments and reconstruction. In the summers there is a problem with the cooling water not being cold enough, during this condition an extra heat exchanger on the condensate tank would not help, but during the rest of the year it will probably make a difference.

### 10.3.3 Control Strategies

A controller has been suggested in many places in this report but there has been no discussion about what kind of controller that should be used. The easiest and most intuitive one is the proportional controller. This will probably not be the best alternative since it does not take into account the past errors and has a stationary fault. A proportional integral controller is in many situations the best alternative and will probably be the one used in a future implementation. It has the advantages of being simple and performs well, if not best, in comparison to other more advanced kinds.

The feed forward term mentioned before is also some kind of controller but will be implemented before the actual controller and the set point value for the latter will be described in *kilograms of pure formaldehyde*.

Another approach could be to analyse the formaldehyde concentration of the condensate at least once a day and take this into account. By doing this a more robust system could be reached, but it would also imply an increased number of analyses and might therefore not be the best solution, but it could be looked into anyway. If it makes the system considerable more stable it might be possible decrease the total number of analyses by decreasing the sampling interval, to for example once a day instead of once a shift.

The analyses frequency is defining the sampling frequency of the controller. In the instructions for the operators, it is stated that one analysis should be performed every working shift of eight hours. Depending on productions speed of the plant, one batch takes in mean about 65 minutes, corresponding to more or less one analysis every seventh batch. Depending on the workload of the operators the analysis is performed at different times, or sometimes not at all, from working shift to working shift. A standard discrete PI controller should ideally be updated directly after every analysis, but this will lead to a controller with irregular sampling time. In the effort of making a, as simple as possible, controller it should be updated after equal time intervals and would in this case be every eight hours. A more advanced solution to this problem could be to implement a Kalman filter that has varying sampling time. This approach is used in for example the paper mass industry.

A more advanced approach could be done to the controller problem. The control system is producing a report from every batch that includes the numbers of kilograms added in the reactor for every reactant. With the use of this information it should be possible to predict the residual formaldehyde content in a fairly good way. Since the process model is not complete the predicted value probably will display a drift from the actual process value. The drift could be dealt with by doing compensation with respect to the actual value obtained once a shift. This can be thought of as a soft sensor, which is an imaginary mathematical sensor that estimates the value that a physical sensor would produce under the same conditions.



## **10.4 Conclusions**

The theory of statistical process control consists of many appealing approaches to different problems. There are benefits in emphasizing on reducing variations, rather than trying to cover them with for example automatic control. On the downside it might be hard to achieve an in-control process when it comes to a continuous process industry. For this reason the different tools of statistical process control are probably not as easy to use effectively in practice as it seems to be in the literature. With this in thought together with the fact that it was not applicable on the case study I still believe that there are situations where the tools, and especially the way of thinking, is useful for the Perstorp group. When comparing the names of statistical process control and automatic control, it is quite easy to believe that they are different alternatives to approach the same problem. This is wrong. Statistical process control is not an alternative to automatic control, but could definitely be a useful complement.

The case study shows that it is definitely possible to considerably reduce the variations in the residual formaldehyde content. I do believe that an automatic controller needs to be implemented in the control system to obtain a predictable process. It is known that the residual formaldehyde content affects the isobutyric aldehyde yield and therefore production costs will decrease with a fully functioning controller. The savings should be compared with costs of installing an automatic analyzer. Even though it is a bit expensive I believe that installing an analyzer would be beneficial, not just for a fully automatic system but also in reducing the work load of the operators.

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