Quantifying sources of variation in process analytical measurements

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Chapter 1

General introduction

In production process environments, many sources of variation have an influence on the quality of the final product. To comply with product specifications, the production process has to be monitored and controlled. For process control, reliable analytical, preferably on-line, chemical measurements are needed. A problem arises when the total variation apparent in the measurements approaches, or even exceeds the allowed variation as specified in the product specifications. Analysis results are used for both process control and qualification of products. In case of qualification, a product can be accepted, downgraded or rejected based upon analysis results obtained from samples taken from the product. Because of the variation in the analysis results, errors in these decisions are inevitable. Two types of errors can be recognized:

1. Acceptance of a product while it does not meet the product specifications.

2. Rejection of a product while it meets the product specifications.

In the first case a product that does not meet the requirements is delivered to the customer. The second case results in downgrading or even total rejection of a product that is well suited for the application it is produced for. Both errors cause economic damage and should be prevented. To prevent both false rejection and false acceptance of the product, the variation in the analysis results should be small compared to the allowed product variation. If this prerequisite is not met, variation reduction is needed to comply with the demands from clients.

Another situation can occur as well. When clients request for products with improved product specifications, the process needs to be optimized in such a
way that variations in the product are reduced. To reduce the process variation and thereby improve the product quality, the process needs to be controlled more strictly. This is possible only when the variation in the measurements for process control are reduced. Hence, before optimizing the production process, both sampling and analysis have to be improved to be able to measure the consequences of adjustments made to the production process.

At the Dutch steel company Hoogovens Staal BV research on the topic of variation reduction in the production, sampling and analysis of steel was initiated to ensure that concentrations of the element in the final products comply with demands from clients, now and in the future. Others have recognized as well that improvements in the process analytical measurements are needed and that variation reduction is an important issue in the control of the steel making process. Although current demands give no rise to concern, effort has to be put in variation reduction in the analysis results because demands will become more and more stringent in the future.

The production of steel products such as soda cans and car parts is a complex process. Chapter 2 describes this process in some detail to put the research presented in this thesis into perspective. The research concentrates on that part of the production process where liquid iron has been transformed into steel and is ready to be cast into moulds to form slabs of steel.

One method of improving the analyses is the development of more accurate and precise methods of analysis. Many researchers have searched for methods to improve process control in steel production. Several investigations involved the use of a laser as both a sampling and an excitation source. Others focussed their research on in-situ analyses. In Chapter 3, an overview is presented of studies that may give more insight in possibilities of improving process control in steel production.

Currently, spark optical emission spectroscopy (spark OES) is still the most favourite analysis method for analysing steel samples. The method is fast and both accuracy and precision meet the required conditions. With spark OES, solid steel samples are analysed and analysis results for the elements C, Mn, P, S, Si, Al, Cu, Sn, Cr, Ni, Mo, Nb, V, B, Ti and Ca are reported to the process engineer. A detailed description of the spark OES and the used samples is presented in Chapter 4.

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*a* The name *Hoogovens* translates to blast-furnaces in English.

*b* Hoogovens Staal BV merged with *British Steel plc* in 1999 forming a new company called *Corus Group plc*. The research described in this thesis was performed before the two companies merged and therefore, only the Dutch steel company is mentioned.
The analysis results of the spark OES are used to monitor and control the chemical composition of the steel bath. As stated before, the customer demands are expected to become more and more stringent in the future and therefore, improvement of the analyses is needed. Replacing the spark OES with laser based analysis methods is one solution but these methods still need to prove their applicability. Another method is improving the currently used methods of sampling and analysis. Such improvements can be performed by means of trial and error but this is not preferable because the model according to which the variation is build-up will be unknown. A more strategic approach in which possible sources of variation are taken into account will give a better insight in the structure of the total variation apparent in the final analysis result. Chapter 5 presents such a strategic method consisting of six steps, each to be performed consecutively. The strategy has been applied to the sampling and analysis procedure as used at the Hoogovens Staal BV Laboratory for Process Control.

Two chapters are devoted to the subject of identification and quantification of variation in the production, sampling and analysis of steel. Chapter 6 describes the setup of the experiments and reports the results for one type of steel. To test whether results for one type of steel holds for other types of steel as well. Chapter 7 presents the results obtained for three different types of steel.

In Chapter 4, a description is given of the calibration method used for the spark optical emission spectrometers (spark OES) at the Hoogovens laboratories for process control. This method has been developed and changed time after time over the years based on new insights and the introduction of better instruments. The calculation is a rather complex combination of different procedures. Although this method has been developed and improved over the years, alternative methods may be used to improve the accuracy and precision of the analytical results. However, before implementation, such methods need to be verified to certify the continuation of the steel making process. Implementation of an alternative method might result in further deviations thereby influencing the process control. Experiments presented in Chapter 8 have been performed to test whether changes to the current calibration method can reduce the variations in the analysis results. The experiments concerned simulations in which alternative methods to calculate the concentrations from intensities were tested. Results for the elements carbon (C), manganese (Mn) and phosphorus (P) are shown in this last chapter.

"Steel can be produced in many different compositions. Steel with a certain composition, for instance composition 1, is called type 1 steel in this thesis."