ERROR SUSCEPTIBILITY AND IMPERFECTION OF RESEARCH PROCEDURES - IMPLEMENTATION OF SIX SIGMA METHODOLOGY IN LEIBNIZ CENTRE FOR AGRICULTURAL LANDSCAPE RESEARCH (ZALF)

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Abstract. Currently one of the most widely used method to estimate greenhouse gas emissions and their impact on agricultural landscape is used e.g. by the Institute for Biogeochemistry at ZALF – „closed chamber method". A lack of adequate training of staff and inappropriate standardized procedures for various stages of the analysis of environmental data directly affect the final results. A specific experiment, corresponding to the different stages of the field measurement was performed, referring to the subjective assessment of reliability and accuracy of obtained flux rates. The paper presents observed results and try to provide economical as well as practical solutions to reduce the impact of a "human factor".

Key words: management, quality, six sigma, optimization.

1. Introduction

For many thousands of years, man has influenced the natural environment in which he lives. Interactions between the human society and the surrounding environment are highly complex and dependent on many different variables. Increasingly, man uses the natural environment in unconventional ways, leading to over-exploitation of natural resources, pollution of the environment by various types of waste, destruction of forests, emissions of multiple harmful substances into the atmosphere, which in turn leads to the impoverishment of the flora and fauna of the world. Since the turn of the last decades, humanity has been trying to fix all the errors of his conduct towards the environment. Through the introduction of modern production technologies and a broader environmental education of children and young people, new forms of nature protection have arisen in order to preserve the environment in the least modified form.

Probably the most important part of the environment is the climate as it affects the development of all living beings on Earth. For growth plants need water, minerals and air. Similarly, animals need water, food and oxygen. However, all organisms require a suitable temperature environment for their proper development. Therefore, even the smallest disturbance of this delicate balance is reflected in every element of the ecosystem, which also has an influence on human life. For a long time, people did not realize the scale of the changes in the natural environment made by them. Garbage and industrial waste, contaminated and poisoned rivers, lakes and seas and polluted air in big cities and industrial districts are today the most visible impact of man. Environmental pollution threatens our health and even our lives.

Nowadays, reflections on greenhouse gas emissions are a major subject of scientific debate. There are two groups of scientists arguing about the truth of the thesis of human impact on global warming through increased emissions of greenhouse gases caused by human activity. Rationalists mobilize public opinion to make changes in the manufactory energy sector. However, the second group of people defends the status quo concerning the impact of GHG emissions on global warming caused by human activity.

According to the IPCC report of 2007 [1], the global mean temperature is gradually increasing; a phenomenon which is observed around the world and is correlated with an increase in anthropogenic GHG emissions. Furthermore comparing the emission covered by the Kyoto Protocol, GHGs increased about 70% from 1970 to 2004. According to research data the biggest source is CO2, which grown about 80%. Regarding to all GHGs emissions growth: energy supply - 26%, industry - 19%, the gases came from land use change and forestry - 17%, agriculture - 14%, transport - 13% residential and service sectors - 8%, waste - 3% (IPCC Report, 2007).

Currently, there is not enough information about the functioning of the biosphere to certainly assess the anthropogenic impact. On the other hand, in situ measurements of agriculturally used areas allow us to estimate GHG emissions and to improve knowledge on the impact of agricultural land on the overall balance of GHG in the atmosphere. Since this subject directly embodies on the environmental strategy of European Union, there are many different research units investigating the issue related to this topic.

One of the scientific institutions dealing with various environmental issues is the Leibniz Centre for Agricultural Research (ZALF) e.V., in particular its Institute of Landscape Biogeochemistry - where the author worked during a student internship – which is investigating greenhouse gas emissions from various agricultural areas. To learn more about the complex functioning of the environment concerning trace gas emissions and to quantify the impact of different ecosystems on global warming, advanced environmental analysis need to be to carried out. For more than 10 years, periodic trace gas measurements using the well-established closed chamber method have been continuously conducted by the "trace gas research group" at the Institute for Landscape Biogeochemistry of the Leibniz-Centre for Agricultural Research Müncheberg e.V. (ZALF).

2. Materials and methods

2.1. Environmental analysis and gas flux measurements in general

For conducting trace gas measurements and to quantify the net ecosystem exchange (NEE) and its components ecosystem respiration (R_{eco}) and gross primary productivity (GPP), Drösler [2] developed a "non-flow-through non-steady-state closed chamber measurement system", which is used not only by the trace gas research at ZALF, but also by other research groups throughout Germany. The closed chamber method in general is a widely used method for determining trace gas fluxes (CO_2, CH_4 and N_2O) of agricultural areas as well as wetlands, and described by several researchers all over the world. The measurement system is primary related to relatively low costs and a simple field operation (even in logistically difficult areas) [3].

Dynamic-closed-chamber measurements are based on the changing concentration gradient of CO_2 inside the chamber during each measurement. The measurement system consists of a portable infrared gas analyzer (IRGA) and two types of rectangular PVC chambers: non-transparent and transparent. Each chamber has a volume of 0,296 m^2 and is placed on a permanently installed PVC frame (0,75 x 0,75 cm). Additionally, non-transparent and transparent PVC extensions are used to accommodate plant growth, e.g.,

in the case of maize[4]. Properly carried out, a measurement takes 5 minutes. Following each measurement, the chamber must be ventilated to prevent gas accumulation from the previous measurement, and to prepare for the next measurement plot. It also must be noted that when plants are really high, it is necessary to use an additional fan within the chamber to ensure adequate gas distribution inside the chamber.
However, the closed dynamic chamber method is associated with certain risks and potential error sources. Drösler [2], Livingston [5] and Hutchinson [6] mentioned as potential error sources:

- inaccurate determination of the headspace volume which can have a big influence on final results [7],
- direct leakage at the chamber components or through the soil pore space, which can cause inaccurate flux measurements [8],
- temperature changes of the soil and the atmosphere within the chamber, which can cause change of the environmental conditions inside the chamber [9],
- artificial water vapor accumulation within the chamber, which may affect the stomata regulation of plants and thus plant gas exchange [10],
- modification of atmospheric air pressure due to chamber placement [11],
- huge $CO2$ concentration changes within the chamber headspace, which can disturb the underlying concentration gradients and thus affect flux rates [12].

2.2. DMAIC methodology

The DMAIC method (an acronym of the first letters: Define - Measure - Analyze - Improve - Control) is a quality improvement cycle, a method of supporting quality improvement. The main focus is set on implementing methodical improvements and on enhancing the cost-benefit-relation. It provides a systematic and rigorous five step scheme on how to improve imbedded processes. Each of these phases (steps) is logically linked with the preceding and following phases [13]. DMAIC as a method of improving the quality is one of the tools used in Six Sigma techniques.

3. Author's methodology – DEFINE

To be able to perform environmental analyses, it is necessary to conduct field research to collect field data on gas fluxes and environmental parameters. During the field measurements campaigns, which represent important stage of research, the author seized the opportunity to examine the quality of the field data acquisition. Incorrect measurements which occur during normal measurement campaigns suggested that it makes sense to conduct experimental field measurements to quantify the relative importance of various measurement errors on the final outcome of the whole analysis. Following the DMAIC methodology, the characteristics of the tests and procedures with a flow chart was analyzed. The following section of this chapter explains how the field experiment was planned and executed. Measurements were carried out in a specially designed and constructed plan for the purposes of this study.

The first stage of the DMAIC cycle – DEFINE, has been implemented by defining the operation of the Institute, illustrating the algorithm of conduct in the different stages of the research.

The Algorithm of actions is presented below. Additionally, the steps of DMAIC cycle are presented in Figure 1.

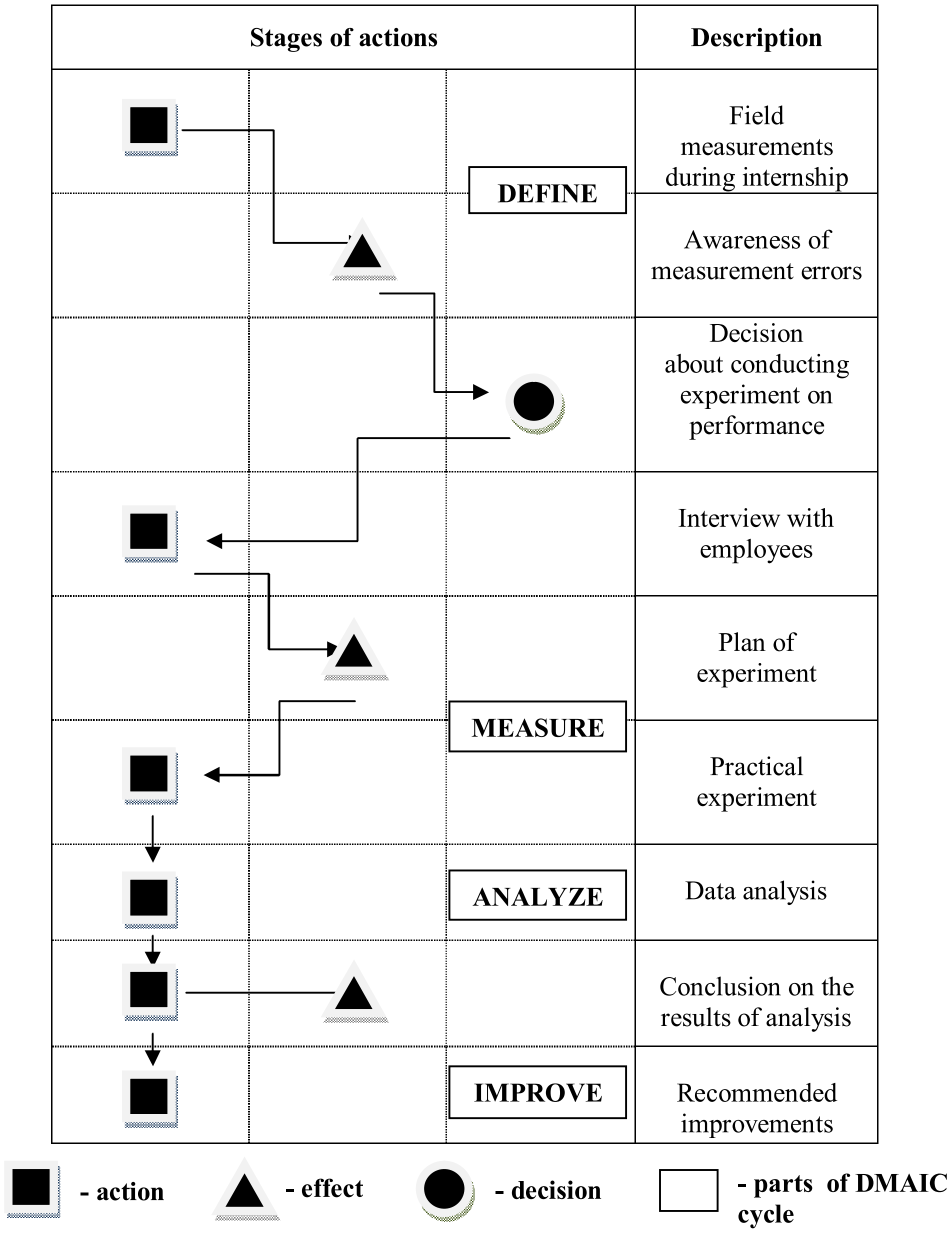

Figure 1. The algorithm of actions taken by the author during the experiment including parts of DMAIC cycle.

3.1. Analysis of the current use of the closed chamber method

Analysis of gas emissions from different land use types is always carried out by a specific algorithm. Therefore this paper mainly focuses on the practical element of work, especially the data collection in the field. The flow chart in Figure 2 describes the sequence of the process which is carried out in order to obtain the final results. According to this, further consideration was made to describe every single process in details.

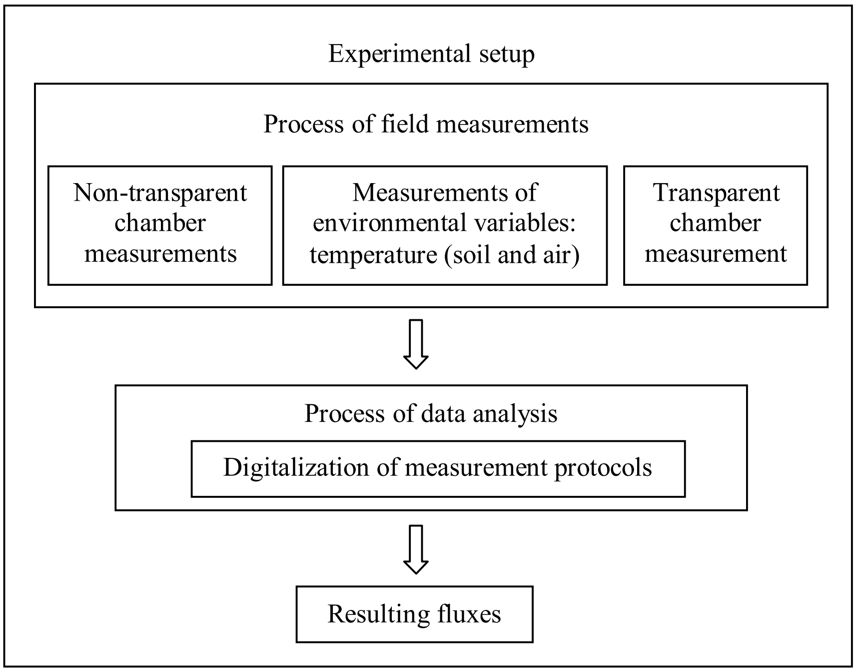

Figure 2. Flow chart of processes

The process of field measurements is divided into three main parts:

- Non transparent chamber measurements exclude the impact of radiation on gas emissions inside the chamber. The chambers are made of a special non-transparent PVC material. For this type of measurement, the incoming photo synthetically active radiation (PAR) is negligible, but the current temperature of the soil and air inside the chamber is very important, because it has an impact of gas emission changes.
- Transparent measurements allow to study the combined effects of solar radiation and temperature on greenhouse gas emissions over time. The chamber used for this kind of measurements is made of special PVC material with a light transmission of approx. 76%. For this type of measurement both the photo synthetically active radiation and the current temperatures of the soil and air inside the chamber are very important.
- Measurements of environmental variables: temperature (soil and air) and PAR are important elements of the field measurements as they are the driving factors of the underlying gas exchange processes. Soil temperature measurements are carried out by three electronic thermometers placed at different depths in the soil (2 cm, 5 cm and 10 cm). Temperature measurements inside and outside the chamber are made using temperature sensors attached to the chambers and connected to the data logger.

Measurements are performed with both chamber types in order to obtain measurements for a wide range of temperature and PAR values during the day. The next step is related to the use of the collected field data on CO_2 concentration changes and environmental parameters to subsequently calculate CO_2 flux rates. All data analyses and calculation of CO_2 fluxes are done with Microsoft Excel 2010, where after data digitalization flux rates are calculated by means of programmed VBA macros.

3.2. Field experiment: error measurements – MEASURE

3.2.1. Experimental concept

The experimental error measurement campaign was conducted at an experimental site with bio energy maize in Müncheberg. Although during standard field measurement campaigns situations arise that may affect the accuracy of the measurements, to data there has not been any systematic error classification with subsequent recommendations for improvement. In order to better understand contribution of individual measurement errors to the overall measurement error an experimental error measurement campaign was thus conducted.

In order to identify errors with a tangible impact on the final measurement results, narrative interviews with experienced field staff were performed to collect information about the most common errors occurring during measurements campaigns. Based on interviews with field-experienced members of the working group, 8 common error sources were identified. In addition, the experience gained during field work also helped the author a personal assessment of the types of potential errors and ways of dealing with them.

Based on the results of the interview, a measurement plan was developed (Table 1), which included replicated measurements for each identified measurement error. The choice of experimental error measurements was also made based on the available methodological documentation which showed measurements with various imperfections. The descriptions of errors have been made with more or less detail on what may often have an influence on the appropriate analytical approach

Furthermore, in order to compare the results, correct measurements were also scheduled at three different times of the day (8.00 am, 11.00 am and 2.00 pm.) to allow for the comparison of the relative flux rate error of deliberate error and correct measurements. In both cases, a distinction by chamber type has been considered.

In this section and its subsections the second part of the DMAIC cycle - MEASURE has been implemented by the presented plan and the determination of the general characteristics of the measurements.

Table 1. Plan of experiment

Type of measurements	Kind of measurements	Number of measurements by type		Sum of measurements
		Non - transparent	Transparent	
Error measurements	Measurement without external fan (ventilator)	3	3	6

	Measurement without internal fan	6	6	12
	Measurement with wrong internal fan direction	6	6	12
	Shadowed measurement (changing PAR)	0	3	3
	Insufficient airtight closure of chamber	3	3	6
	Measurement with steamy/dirty chamber and extension	0	3	3
	Badly ventilated chamber right before measurement (high initial CO2 conc.)	3	3	6
	20 min measurements	3	3	6
Correct measurements	Correct measurements 8.00 AM	6	6	12
	Correct measurements 11.00 AM	6	6	12
	Correct measurements 2.00 PM	6	6	12
		$\sum = 81$		

It was planned that for each error measurements are repeated three times. Although more measurements, of course, would be desirable, due to the combination of this experiment with regular field campaigns it was necessary to make a compromise between the possible and the recommended number of measurements. Each of the three error measurements was made at another plot to capture a wide range of flux magnitudes.

3.2.2. Types of error measurements

The most common errors which are made during field work are presented below. According to this, experimental error measurements were performed in order to verify how much these error types may affect the final results. Descriptions of error measurements are grouped according to the expected impact on the final outcome, starting with the potentially largest impact. In addition, measurements are also grouped according to the type of error: I - Insufficient airtight closure, II - Insufficient headspace mixing , III – Non-atmospheric conditions. Individual descriptions of error measurements are presented below in Table 2.

Table 2. Descriptions of error measurements

Id.	Kind of error measurement	Description	Expected effect
Insufficient airtight closure			
1	*Insufficient airtight closure of chamber and extension*	Incorrectly placed extensions or chambers may result in the exchange of within-chamber air and ambient atmospherically air. As a result, the observed flux rates are highly unreliable. During this error measurements, one extension and the chamber were not properly placed on the frame.	Inappropriate air exchange inside and outside the chamber
Insufficient headspace mixing			
2	*Measurements without external ventilator*	The external ventilator is used to provide appropriate air circulation inside the chamber when more than one extension is used. Error measurements were conducted without an external ventilator.	Inappropriate air circulation inside the chamber.
3	*Measurements without internal ventilator switched on*	The internal ventilator is one of the most important elements in the chamber providing appropriate air circulation inside the chamber and therefore ensuring an even distribution of the CO2 concentration.	Inappropriate air circulation inside the chamber
4	*Measurements with wrong direction of internal ventilator*	The internal ventilator is supposed to circulate the air around the sides of the chamber. A wrong fan direction may not provide enough ventilation.	Inappropriate air circulation at the top of the chamber.
Non atmospheric conditions			
5	*Insufficiently ventilated chamber prior to measurement*	After every measurement, the chamber should be ventilated to prevent accumulation of gases from the previous measurement. Experimental measurements were conducted with a chamber placed on the ground for 5 min without ventilation to induce an artificial buildup of CO2 within the chamber.	High CO2 concentration at the beginning of measurement
6	*20 min measurements*	Time related reduction of the CO2 concentration gradient between chamber and soil	Accumulation/ deficiency of emitted CO2
7	*Shaded transparent measurement*	The top of the chamber tower was covered with a towel to simulate a shadow which a field worker sometimes unconsciously makes	Modification of PAR within the chamber and thus reduced NEE flux rates

		during transparent measurements.	
8	*Measurement with steamy chamber and extension*	Very often chambers steam up during measurements, particularly early in the morning. For the experimental error measurements steamy chambers were not cleaned. Steam inside or outside the chamber may reduce the input of PAR and therefore influencing the measured NEE flux rates through modification of GPP.	Modification of PAR within the chamber and thus reduced NEE flux rates

3.2.3. Results

In this section, a summarized analysis of the results of all correct and incorrect errors (81 measurements) is presented in Table 3. The average values for all relative slope errors by error type, chamber type and plant (n = 3) presence or absence were calculated and are also presented in Table 3.

Table 3. Average relative slope errors and standard deviation by error type, chamber type and plant presence.

Error type	**Average of relative slope error ± standard deviation [%]**			
	Transparent chamber		**Non-transparent chambers**	
	With plants	**Without plants**	**With plants**	**Without plants**
Measurements without external fan (ventilator)	1,072 ± 0,854	-	0,901 ± 0,426	-
Measurements without internal fan	1,058 ± 0,790	15,191 ± 15,734	0,889 ± 0,838	3,719 ± 6,662
Measurements with wrong internal fan direction	2,200 ± 2,462	4,776 ± 1,915	0,679 ± 0,612	2,454 ± 3,334
Shadowed measurements	1,301 ± 0,266	-	-	-
Insufficient airtight closure of chamber	2,026 ± 1,189	-	-	4,530 ± 2,049
Measurement with steamy/dirty chamber/extension	11,450 ± 7,646	-	-	-
Badly ventilated chamber right before measurement	4,068 ± 1,510	-	1,375 ± 0,346	-
20-minute measurements	-	0	-	1,725 ± 1,052
Correct measurement	0,784 ± 0,693	1,613 ± 0,792	0,679 ± 0,179	1,844 ± 1,307

As it is shown in the Table 3. results of particular errors measurement are significantly differ from each other. This difference is also evident in the individual measurement pairs are grouped according to the presence or absence of "the measurement without internal fan switched on", “wrong internal fan direction” or "insufficient airtight closure of chamber". In the first case, a significant difference is the amount of built structures caused by measuring with height plant, despite the lack of fan off, inside there was an additional fan which caused air circulation inside the chamber. However, the measurement without the presence of plants, despite the use of only the chamber was made without any fan. In this case, we can see that an additional fan during measurements with a high plants altitude plays the most important role. Similar explanation refers to the measurement with the wrong set of the internal fan, which affect on inappropriate air circulation which is particularly evident in the measurement with the absence of plants where there is no other fan, opposed to measurements made with the presence of high plants. In the third case so big difference is mainly caused by the volume of air contained in the chamber. Presence of plants impose higher construction which affect of the volume of air inside the measurement construction. Because this error is caused by the leaky closure of the elements of the measurement constructing, lost of air has an influence on change of gas concentration, which is more apparent in the measurement without plants where is much less air.

Generally, there are also differences in the results of measurement error depending on the presence of plants. Results of measurements made with the presence of plants, except for "steamy" and "bad ventilated" are more or less similar to the correctly performed measurements. Steamy error measurement is definitely a fatal error which must be repeated due to the large differences compare to the correct measurements. It is due to inappropriate exposure to sunlight. "Bad ventilated" measurements without adequate ventilation has influence in an initial accumulation of gases, and therefore internal environment does not suit into the real conditions.

Some error measurements do not significantly differ from the correct measurements, therefore, it is important to decide whether such measurements should be repeated or can be treated as correct. As you can see above, these errors are much less important than those listed above.

3.3. Proposition of solutions – IMPROVE

The next stage of DMAIC analysis is IMPROVE, that applies patches used to implement based on the results of measurements. One of the proposed solutions for the development of detailed procedures and instructions for the various analyzes is a guide for new employees. It should contain detailed descriptions of each step, and instructions for preparing and carrying out the experiment. The rationale for this guide would be the continuous development based on new insights and comments.

Preparation of detailed procedure containing all the necessary information and the recommendation for the proper conduct of the experiment is next proposition of improvement. Information on the possible occurrence of certain errors and how to deal with them should be obligatory placed there as well.

In addition, from time to time specific checks should be carried out concerning knowledge of the procedures and dealing with error it any will occur. This would ensure adequate quality of staff training which would have an influence into the quality of obtained data.

3.4. CONTROL

The final stage of DMAIC cycle - CONTROL includes checks carried out on the specified implemented improvements. Checks if the new proposed solutions works in practice and whether are they properly used by employees. Therefore the specification of CONTROL part of DMAIC cycle has not been defined yet.

4. Conclusions

The DMAIC methodology allowed implementing the various stages of work during internship into certain parts of the DMAIC cycle. The implementation of these principles allowed conducting an appropriate classification and the enforcement activities in a logical sequence. Therefore, only the last stage of that cycle - CONTROL - was not executed. However, application of the DMAIC methodology allowed showing the possibility of using well-known methods of quality management to improve the functioning of a research institute associated with environmental studies. This demonstrates also the versatility of this method and the possibility to use it in many areas of science.

Another element of the quality management used in this study is the created algorithm of conduction. It represents the most important tasks performed during the experiment implemented by taking into account the practical elements of the DMAIC cycle.

The development of measurement experiment containing a detailed description as well as the expected impact of measurement errors on the measurement results has also been presented. Moreover, at this stage, this experiment can also be used as a source of further improvements concerning creating a stringent measurement procedure.

The observed significant differences between the results of some error measurements: "The measurement without internal fan switched on", "Wrong internal fan direction", "Insufficient alright closure of chamber" illustrate that some errors can be characterized as "critical". These kinds of erroneous measurement must be discarded and repeated. However, result of some error measurements are much similar to normal measurements and there is the possibility to use such measurements by taking appropriate analysis depending on the type of error. Applying these considerations should be used to construct a procedure containing appropriate instructions. Taking measurements and analyses following these recommendations would significantly improve measurement quality and save the time associated with repeated measurements.

The analysis shows how inaccurate measurements can be, and how errors can affect the final results. Depending on an employee's competence, errors can occur with a certain frequency. Therefore, it is really important to conduct continuous improvement of the competence of staff and procedures. Only by such action reliable results can be achieved.

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