Department of Signal Processing and Acoustics

Improving the Metrological Traceability of Online Dry Grammage Measurement Used in the Paper Industry

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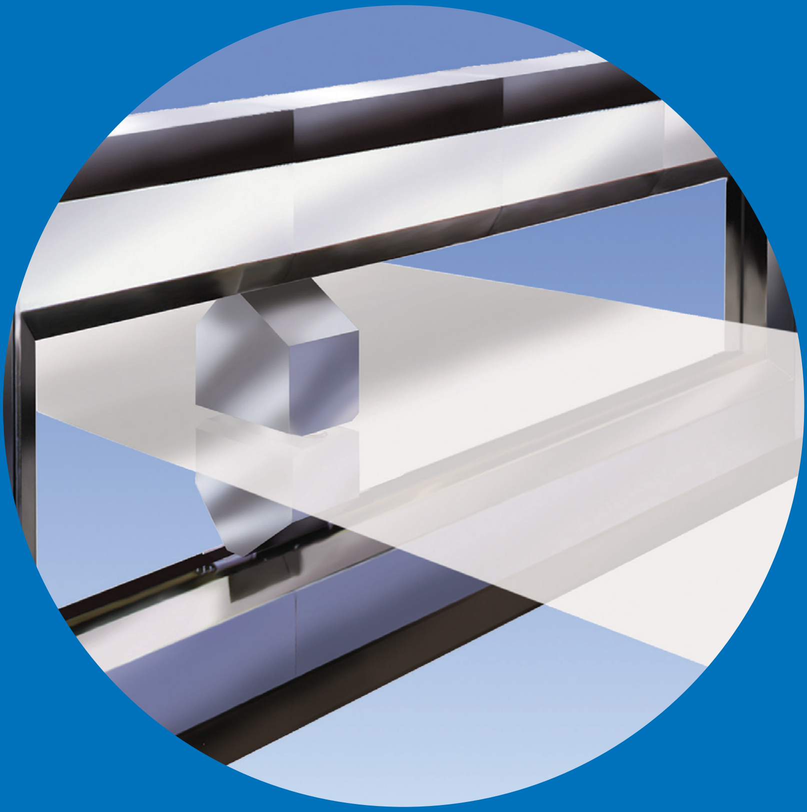
Juha Kangasrääsiö



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Improving the Metrological Traceability of Online Dry Grammage Measurement Used in the Paper Industry

Juha Kangasrääsiö

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Abstract

In the paper industry, the automatic control of raw material consumption and product quality relies upon online grammage (total mass per unit product area) and moisture measurement. The optimisation of raw material consumption is vital to the profitability of production, and the even quality is essential for satisfying customer needs.

It was shown in this work that the basic errors of radioisotope Kr-85 based grammage and IR based moisture measurements, which are commonly used in the paper industry, can be decreased on average approximately 85% with traceable calibration methods. Moreover, a novel method was created for the simultaneous grade specific calibration of these measurements by applying machine reel sampling and traceable sample area measurement. It was demonstrated that a relative uncertainty of 0.5%, at the 95% confidence level, can be achieved in dry grammage calibration. The calibrations were performed according the EN ISO 536 (grammage) and the EN ISO 287 (moisture) standards. The uncertainties of existing measurement systems can be reduced usually over 50% with the created methods.

The decrease of measurement uncertainty offers an opportunity for significant economic savings since one per cent change in the dry raw material consumption of a typical production line affects several hundreds of thousands euros in the annual raw material costs. The presented calibration methods can basically be applied to all paper and board production lines.

Keywords Automatic process control, Online measurement, Grammage, Moisture, Calibration, Traceability

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Tiivistelmä

Paperiteollisuudessa tuotannon raaka-ainekulutuksen ja laadun automaattinen säätö tukeutuu tuotannonaikaiseen neliömassan (massa pinta-alayksikköä kohti) ja kosteuden mittaukseen. Raaka-ainekäytön optimointi on tärkeää tuotannon kannattavuuden kannalta ja asiakastarpeet voidaan täyttää vain tasalaatuisella tuotannolla.

Työssä osoitettiin, että paperiteollisuudessa yleisesti käytettyjen radioisotooppi Kr-85:een pohjautuvien neliömassamittausten ja infrapunasäteilyyn perustuvien kosteusmittausten virheitä voidaan pienentää keskimäärin noin 85 % jäljitettävien kalibrointimenetelmien avulla. Lisäksi kehitettiin menetelmä neliömassa- ja kosteusmittausten samanaikaiseen tuotekohtaiseen kalibrointiin. Menetelmä perustuu konerullista tehtävään näytteenottoon ja näytteiden pinta-alojen jäljitettävään mittaukseen. Tehtyjen kalibrointien avulla osoitettiin, että kuivan neliömassan kalibroinnissa voidaan saavuttaa 0,5 %:n suhteellinen epävarmuus 95 %:n luotettavuustasolla. Kalibroinnit tehtiin soveltaen standardeja EN ISO 536 (neliömassa) ja EN ISO 287 (kosteus). Käytössä olevien mittauslaitteiden mittausepävarmuutta voidaan pienentää yleensä yli 50 % kehitetyillä menetelmillä.

Mittausepävarmuuden pienentäminen avaa mahdollisuuden merkittäviin taloudellisiin säästöihin, koska tyypillisesti yhden prosenttiyksikön muutos tuotantolinjan kuivaraaka-aineen käytössä vaikuttaa satoja tuhansia euroja tehtaan vuotuisiin raaka-ainekustannuksiin. Tässä työssä käytettyjä kalibrointimenetelmiä voidaan periaatteessa hyödyntää kaikilla paperin ja kartongin valmistuslinjoilla.

Avainsanat Automaattinen prosessin säätö, online-mittaus, neliömassa, kosteus, kalibrointi, jäljitettävyys

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Preface

This thesis work is a part of the research and development work which has been performed at JMK Instruments Oy, in order to improve the performance of the existing online quality measurement systems used in the paper, board and pulp industry. The research for this study was performed during the years 2006-2013. However, the calibration database of the company was also utilised in this work, beginning from the year 1998 when the company got a FINAS Accreditation.

I wish to express my sincere respect to my supervisor and thesis advisor Professor Erkki Ikonen, Metrology Research Institute, Aalto University, for encouraging and guiding me patiently through the thesis project. I am also grateful to the official preliminary examiners Professor Steven Keller, College of Engineering and Computing, Miami University, and Professor Kai-Erik Peiponen, Department of Physics and Mathematics, University of Eastern Finland, for carefully reviewing the manuscript and their improvement suggestions.

I am thankful to the people from MIKES (Centre for Metrology and Accreditation) who performed a feasibility study of the flatbed scanner based paper grammage determination method at the beginning of this thesis project: Dr Björn Hemming and Dr Antti Lassila from the Length group, MSc Jussi Hämäläinen from the Electricity group, Dr Kari Riski and MSc Leena Stenlund from the Thermometry and Mass group. Without you it would have been much more difficult to proceed in this project. In particular, I am grateful to Björn for being my co-author in the first article of this thesis work. I also acknowledge TEKES (Finnish Funding Agency for Technology and Innovation) for supporting this preliminary study financially.

The personnel of JMK Instruments Oy deserve my sincere gratitude, especially Mr Ilkka Hujanen and Mr Sami Gråsten. Ilkka for assisting me in the calibrations performed at the production lines, manufacturing the customised tools needed and performing calibrations in our laboratory, and Sami for his skilful programming work and finishing some of the drawings.

I owe gratitude to my son Antti for assisting me in some of the calibrations and proofreading the manuscript. I am also thankful to my daughter Annaliisa for helping me in marketing the calibration services to the paper industry and taking care of my dog during all those long evenings when I was writing the articles and the manuscript.

Most of all, I am grateful to my wife Paula for all her love, support and empathy during our marriage. I am a blessed man having such a worthy wife (Proverbs 31:10-31).

Kuopio, 19 November 2013

Juha Kangasrääsiö

Paul the Apostle (approximately AD 53-54):
”ἐκ μέρους γὰρ γινώσκομεν καὶ ἐκ μέρους προφητεύομεν· ὅταν δὲ ἔλθῃ τὸ τέλειον, τὸ ἐκ μέρους καταργηθήσεται.” ΠΡΟΣ ΚΟΡΙΝΘΙΟΥΣ Α 13:9-10
The 28th edition of the Nestle-Aland Novum Testamentum Graece 2012

Apostoli Paavali (noin 53-54 jKr):
”Sillä tietämisemme on vajavaista ja profetoimisemme on vajavaista, mutta kun täydellinen tulee, vajavainen käy tarpeettomaksi.” 1 Kor. 13:9-10
(kirjoittajan vapaa käännös)

List of Publications

This thesis consists of an overview and the following selection of the author's publications.

I Kangasrääsiö J and Hemming B, Calibration of a flatbed scanner for traceable paper area measurement, *Measurement Science and Technology* 2009 **20** 107003 4 pages

II Kangasrääsiö J, Improving integrity of on-line grammage measurement with traceable basic calibration, *ISA Transactions* 2010 **49** 257-263

III Kangasrääsiö J, Improving the integrity of IR based online moisture measurement used in the paper and board industry, *Measurement* 2011 **44** 1937-1944

IV Kangasrääsiö J, Application of a traceable area measurement in the calibration of online dry grammage measurement used in board production, *Measurement* 2013 **46** 1585-1591

Author's contribution

The development of the calibration methods described in Publication I was a team project, where the author co-developed the geometric calibration procedure, developed the radiometric calibration procedure, the procedure to correct the drift of the systematic geometric errors and the measurement model, calculated the uncertainty budget, and wrote the manuscript.

For Publications II and III, the author performed most of the calibrations, analysed the calibration results, created the measurement models and the suggested adjustment algorithms, calculated the uncertainty budgets, and wrote the manuscripts.

For Publication IV, the author developed the simultaneous grade calibration method of grammage and moisture measurements, the sample edge detection method based on two threshold levels, performed the basic calibrations of the online sensors and part of the sampling and laboratory analysis work, created the measurement model and analysed the calibration results, calculated the uncertainty budgets, and wrote the manuscript.

List of Abbreviations

CD	Cross machine direction
EN	European Standard (Europäische Norm)
FINAS	Finnish Accreditation Service
GUM	Guide to the Expression of Uncertainty in Measurement
IEC	International Electrotechnical Commission
IR	Infrared radiation
ISO	International Organization for Standardization
MIKES	Centre for Metrology and Accreditation (Mittatekniikan keskus)
NIR	Near infrared radiation
NMI	National Measurement Institute
SI	International System of Units (Système International d’Unités)
TEKES	Finnish Funding Agency for Technology and Innovation

List of Symbols

a_i, b_i	Adjustment parameters of grammage or moisture reading for product grade i
A	Sample area
A_R	Calibrated area of the reference
A_{Rs}	Scanned area of the reference
A_s	Scanned area of a sample
c, d	Adjustment parameters of basic grammage reading
c_j, d_j	Adjustment parameters of basic percentage moisture reading for product grade group j
E_G	Error of the basic grammage reading G_s
E_M	Error of the basic percentage moisture reading M_s
G	Grammage of a web
G_i	Grammage reading of a measurement system for product grade i
G_r	Calibrated grammage of the reference
G_s	Basic grammage reading of a measurement system
I	Transmitted β-radiation intensity
I_0	Full β-radiation intensity
k	Coverage factor
m_0	Wet mass of a moisture sample
m_1	Dry mass of a moisture sample
M_i	Percentage moisture reading of a measurement system for grade i
M_r	Calibrated percentage moisture content of the reference
M_s	Basic percentage moisture reading of a measurement system
n	Number of calibrated measurement points
N	Number of calibrated sensors
p	Number of calibrated product grades

T	Calibration temperature of the reference
$u(x_i)$	Standard uncertainty of the input estimate x_i
$u_\mathrm{c}(y)$	Combined standard uncertainty
$w_{\mathrm{H_2O}}$	Percentage moisture content of a sample
x_i	Estimate of input term i in a measurement model
y	Output estimate of a measurement model
α	Thermal expansion coefficient of the reference
δA_d	Correction for the drift of the reference since the previous calibration
δA_h	Correction for sample thickness
δA_i	Correction for illumination
δA_rep	Correction for repeatability
δA_res	Correction for scanner resolution
δA_T	Correction for the temperature drift of the reference at the scanner
δG_a	Correction for the moisture absorption of the reference
δG_b	Correction for the mismatch of the sensor beam size to the calibrated area of the reference
δG_rep	Correction for the repeatability of the grammage measurement
δG_res	Correction for the finite resolution of the grammage reading
δG_rh	Correction for the humidity expansion of the reference
δG_t	Correction for the transmission angle of the sensor beam through the reference
δG_T	Correction for the thermal expansion of the reference
δG_z	Correction for the location of the grammage reference
δM_rep	Correction for the repeatability of the moisture measurement
δM_res	Correction for the finite resolution of the moisture reading
δM_z	Correction for the location of the moisture reference
μ	Absorption coefficient of β-radiation

Contents

1 Introduction

1.1 Background

The paper and board industry have to continuously improve their internal efficiency in order to stay competitive and profitable. One way of adapting to this situation is the automatisation of production processes. The ongoing trend of increasing automation also affects traditional manual laboratory measurements, which are being replaced by automated laboratory and online measurements [1]. The better these measurements perform, the more they can be trusted in the quality assurance and better control results can be expected [2].

The automatic control of total and dry raw material consumption in a production line relies upon online grammage (basis weight, total mass per unit product area) and moisture measurements which are performed with an online quality measurement system (Fig. 1) [3, 4]. Since online quality measurement and computer control systems emerged in the paper industry during the 1950s and 1960s, the motivation for using and developing these systems has been the fact that they assist reducing material and energy consumption, increasing production rates, improving product quality, eliminating product rejects and maximising production efficiency [5-8].

However, because the performance of the process control is limited by the performance of the online measurements, there is a need to create and use reliable calibration and verification methods for these measurements, as well as, to reduce the effects of the factors which disturb measurements at the mill [9, 10]. The aim of this work is to respond to these needs by improving state of the art calibration practices.

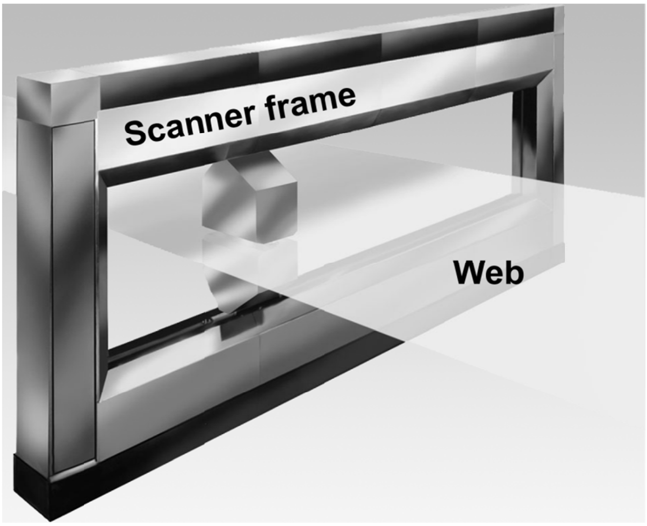

Figure 1. Continuously scanning online measurement system installed in a production line (Publ. II)

The calibration of online grammage and moisture measurement is generally executed in two parts in order to effectively minimise the effects of those factors which cause uncertainty in these measurements [11-14]. At first, the errors of the measurement system are minimised by performing a basic calibration/adjustment with a set of reference samples. After this, the grade parameters of each product grade are defined by comparing the online measurement results, for instance, with the respective laboratory results.

A fundamental problem concerning the current calibration practises is the fact that they are not traceable to SI-units. This problem results from a research gap:

Nobody has so far created a metrological traceability chain to the calibration of the online dry grammage measurement used in the paper industry.

1.2 Metrological traceability

A metrological traceability chain to the International System of Units (SI) is a documented unbroken chain of calibrations ending at the realisation of the definition of the respective SI-unit(s) [15]. An example on such a traceability chain is presented in figure 2. The presented uncertainties are typical for grammages lower than 100 g/m².

A National Measurement Institute (NMI, in Finland MIKES) maintains the realisation of the SI-unit of mass (kilogram) and length (metre) in its facilities. With reference standards and several calibrations the measurement capability of mass and length is transferred and combined in such a way that the accredited calibration laboratory is able to perform grammage determinations and calibrations. In order to calibrate an online grammage measurement system the laboratory traceably calibrates a set of reference standards which are suitable for the basic calibration of the grammage measurement. The laboratory then calibrates the online measurement system with these references and gives a certificate of calibration.

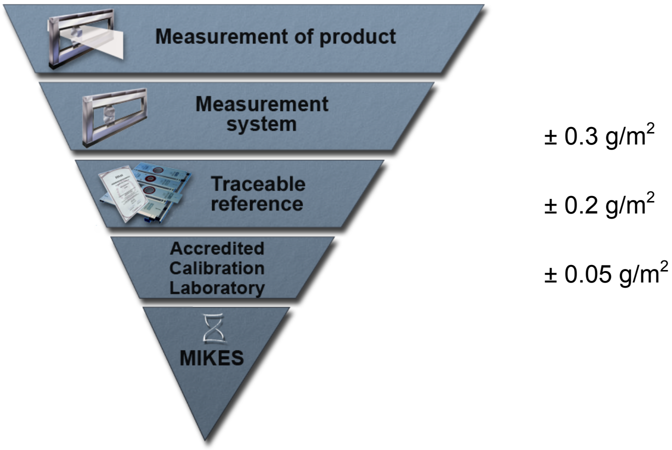

Figure 2. Traceability chain of the basic calibration of online grammage measurement

All these calibrations belonging to the traceability chain need to have credibly evaluated uncertainties which all contribute to the final stated uncertainty [15]. In order to evaluate the uncertainty of a calibration, a measurement model[1] has to be established for the measurement [16]. Each input term estimate x_i in the measurement model has its own uncertainty $u(x_i)$. These uncertainties (standard uncertainties) are evaluated separately by using the principles of the GUM (Guide to the Expression of Uncertainty in Measurement) [16]. For uncorrelated input quantities, the combined standard uncertainty $u_c(y)$ for the output of the measurement model can be obtained by taking the square root from the following equation, which is called the law of propagation of uncertainty,

$$u_c^2(y) = \sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i). \tag{1}$$

The partial derivatives $\partial f / \partial x_i$ in the previous equation are called sensitivity coefficients, because they describe how the output estimate y varies with the changing value of each input estimate x_i. An example of an uncertainty budget can be seen in Publ. I table 1. The combined standard uncertainty is usually multiplied by the coverage factor $k=2$ to express the expanded uncertainty at the 95% confidence level.

Traceability is maintained by repeating calibrations regularly [17]. Thus, the traceability and the measurement capability of the accredited calibration laboratory are maintained by performing all needed calibrations by pre-set time intervals, or whenever needed. The traceability of the references is usually maintained by calibrating them once a year. Similarly, for instance, the traceability of the basis of the online grammage measurement can be maintained by repeating the basic calibration procedure regularly.

1.3 Objectives and scope

This work concentrates on studying the traceability and uncertainty of online dry grammage measurement used in paper and board production.

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[1] For example, equation 1 in Publication I is a measurement model $y=f(x_i)$.

The focus of this thesis work is to find out how a traceability chain could be established for the calibration of online dry grammage measurement. The aim is also to evaluate how much the performance of typical online dry grammage measurement can be improved with such a traceable calibration methods. Thus, the research problem is:

Is it possible to establish a metrological traceability chain to the calibration of the online dry grammage measurement used in the paper industry?

The online dry grammage measurement results are calculated from the results of simultaneously executed grammage and moisture measurements. Both of these separate measurements can be calibrated stepwise in two parts (basic and grade specific calibration). Therefore, the research problem is divided respectively into more limited research questions, which are then studied separately.

Research questions:

1. *How can a paper or board sample area be measured in a traceably way?*
2. *How much can the measurement errors of online grammage measurement be decreased with a traceable basic calibration executed at site?*
3. *How much can the measurement errors of online moisture measurement be decreased with the basic calibration executed at site with custom-made reference standards and by using traceable calibration methods?*
4. *Is it possible to achieve traceability in the grade calibration of online dry grammage measurement executed at site?*

The traceability of the grammage determination of a paper or board product requires that the area of a product sample can be measured in a traceable way. Therefore, the first research question concentrates on this specific issue. This question is studied in Publications I and IV.

The basic calibrations and the adjustments based on these calibrations establish the basis for the grade specific calibration work of grammage and moisture measurements. Nevertheless, sometimes the basic calibrations are ignored in paper and board mills and just grade calibrations are executed. In order to evaluate the justification of the basic calibration work, the improvement potential of grammage and moisture measurements was investigated. Thus, the second and third questions, which are investigated in Publications II and III, deal with reducing the measurement errors by applying traceable methods in the basic calibrations. The analysis of the second and third research questions are limited to the radioisotope Kr-85 based grammage sensors and IR based moisture sensors, which are the most common online quality measurement sensors installed in the paper mills [13].

The final research question concentrates on establishing traceability to the grade specific calibration of grammage and moisture measurement. This research question was researched by executing grade specific calibrations for scanners locating at the end of board production lines, and the results were analysed in Publication IV.

1.4 Research process and dissertation structure

Publl. I

In the determination of paper and board grammage, according to the standard SFS-EN ISO 536, the area of a paper or board sample (a test piece) needs to be determined together with the determination of sample mass, as the grammage of a sample is calculated by dividing the sample mass by the sample area [18]. Naturally, both the area and the mass of a sample need to be determined in a traceable way in order to gain traceability in grammage determination.

Novel geometric and radiometric calibration methods were developed for a flatbed scanner in order for it to be used for traceable area measurements. Also a measurement model was created and it was shown with the model that a relative uncertainty of 0.1%, at the 95% confidence level, can be achieved in area measurements performed with the calibrated scanner.

Publ. II

The performance of Kr-85 based online grammage measurement systems made by different manufacturers was studied by analysing basic calibration results, which were performed with traceably calibrated plastic reference standards at the site. It was demonstrated that the average relative basic errors can be reduced over 80% with the traceable calibration method. Also a simple algorithm, based on the experience from the executed calibrations, was proposed to standardise and ease the adjustment of different grammage measurement systems.

Publ. III

A study, similar to the one presented in Publ. II, was performed for the IR based online moisture measurement systems. The calibrations were executed with references sealed between two glass plates. It was shown that the average basic errors can be decreased approximately 85% by performing the basic calibration at site with traceable methods. As in Publ. II, a similar though slightly modified algorithm was proposed for the adjustment of the moisture measurement systems.

The reduction of the basic errors of the online grammage and moisture measurement systems decreases the need for laborious grade calibration work. This is especially valuable when many different product grades are produced on a production line.

Publ. IV

A new calibration method was developed for the simultaneous grade calibration of online grammage and moisture measurements used in the board production. The method is based on traceable sample area measurement technique and machine reel sampling. The area measurement technique which was created in Publication I was improved with a novel edge detection method, based on two threshold levels, and with a sample thickness correction. It was demonstrated that a relative uncertainty of 0.5% in dry grammage calibration can be achieved at the 95% confidence level by applying the created calibration technique.

The raw material consumption of production cannot be controlled any better than it can be measured during the production, and therefore, a small measurement uncertainty is vital for the performance of process control. However, there are no well documented and published results concerning the uncertainties of dry grammage measurements performed on different production lines, but according to the author's experience they vary usually from 1.5% to 2.5% at the 95% confidence level. The smaller the measurement uncertainty is, the better paper and board producers can optimise their raw material consumption. This optimisation is economically interesting for the paper industry since one per cent change in the dry raw material consumption affects usually several hundreds of thousands euros in the annual raw material costs of a production line.

This thesis is organized as follows: In Chapter 2, a new method is presented by which paper and board sample area measurements can be performed with a flatbed scanner in a traceable way [Publications I and a part of IV]. In Chapter 3, a basic calibration technique is described by which basically all the online grammage measurement systems can be brought on the same traceable basis [Publ. II]. In Chapter 4, an approach similar to the one in Chapter 3, is applied to the basic calibration of online moisture sensors [Publ. III]. The application of traceable sample area measurement in the simultaneous grade calibration of online grammage and moisture measurement is described in Chapter 5 [Publ. IV]. Discussion and Conclusions are presented in Chapters 6 and 7.

2 Traceable sample area measurement

2.1 Dimension measurement with a flatbed scanner

Scanners can be classified in drum and flatbed scanners according their scanning mechanism. The geometric accuracy of flatbed scanners are better than that of drum scanners which are mainly applied in scanning large maps and paper documents [19]. Photogrammetric and desktop publishing scanners are usually flatbed scanners.

Photogrammetric scanners are used to digitise high quality aerial images taken by film-based cameras [19-21]. These expensive professional scanners can, in some cases, be replaced with more common and less expensive desktop publishing scanners [22-25]. However, this is possible only provided that the desktop scanners are calibrated and the errors corrected, as the desktop scanners are designed for much less demanding applications.

Flatbed scanners have also been utilized in various other applications requiring geometrical, grey scale or colour intensity measurements [26-36]. Manual measuring methods have often been replaced with scanner based methods because a scanner provides more objective measurement results, reduced variability in results, higher speed and lower costs. The digital images taken by the scanner also enable the automatisation of measurement and documentation. The tasks performed with a scanner can usually also be performed with a digital camera based machine vision system. Nevertheless, a scanner is usually less dependent on external lighting conditions and less expensive than a camera based system.

It is known that there are several sources of uncertainty in flatbed scanner based measurements [22-25]. In order to execute reliable dimension measurement with a scanner, the geometric measurement errors of the scanner need to be identified by performing a geometric calibration. The edge positions of the inspected object are detected by thresholding technique [37], and therefore, also the differences in the grey scale or colour channel responses need to be identified and corrected.

The geometric calibration of a scanner can be performed, for instance, with a two-dimensional grid or a line-scale that can be traceably calibrated [23-25, 38]. Once the reference is scanned with a scanner, the positions of the calibrated reference points are identified and their relative positions are compared to the calibrated values to find out the errors of the scanner. The errors can usually be decreased by creating a measurement model that describes the found errors and then these errors are corrected mathematically. With this kind of geometric calibration, errors that are systematic and stable can be decreased. However, there are also sources of error which cause the geometric errors to vary randomly or drift [23]. The effect of these errors can be decreased with another reference that is scanned more frequently than the reference by which the actual calibration is performed. This reference may, for instance, be so small that it can be scanned together with the object to be measured, a so called internal reference.

Radiometric calibration is usually performed with a grey scale wedge [23, 39], but it can also be performed with separate grey value references [40]. The local differences in the grey scale responses of the effective pixels need then to be modelled and corrected so that the edge positions will be located correctly.

2.2 Developed flatbed scanner based area measurement system

Paper and board samples of various sizes are needed in the calibration of online grammage and moisture measurement. For instance, the basic calibration of IR based online moisture measurement is based on a set of product sample references whose areas need to be determined. The shapes and sizes of these references vary as they are custom-made for each measurement system. Typically, these sample sizes vary from 50 cm^2 to 250 cm^2. During the machine reel based grade calibration of online grammage measurement several sets of product samples are also cut from the machine reel. In the determination of grammage values of these samples, their areas have to be measured. The sizes of these samples are usually approximately 500 cm^2.

In order to decrease the size measurement uncertainty of these samples, a flatbed scanner based area measurement system was developed. The system consists of a flatbed scanner (Epson Expression 1680) which was used in a greyscale mode and connected to a PC. The user interface was created in custom-made software. Special calibration software was also developed for the geometric and radiometric calibration of the scanner.

The flatbed scanner based area measurement equipment determines the total number of pixels covered by the sample. Therefore, the size and the shape limitations of the sample can be quite flexible. This is especially useful when measuring the areas of hand-cut machine reel samples, the sizes of which would otherwise be very laborious to measure manually with a ruler. Another valuable property of the apparatus is that the calculation of uncertainty budgets can be executed by build-in measurement software, and thus, the uncertainty calculation can be automated.

2.2.1 Geometric and radiometric calibration

A geometric calibration procedure was created to identify local differences in the effective pixel size of the flatbed scanner, and to create correction tables by which these deviations can be corrected. The local differences in the effective pixel size have to be corrected in order to get consistent measurement results which do not depend on the location of the sample on the scanner. The sample areas are so large that high geometric resolution is not critical, and therefore, the effective pixel size was restricted to 600 dpi (42 μm × 42 μm). By limiting the resolution, the image file sizes can be kept manageable and the measurement time reasonably short.

The geometric calibration of the scanner is based on a 200 mm line-scale with 1 mm subdivision. The line-scale is etched on a glass plate (265 mm × 30 mm × 5 mm). The line-scale was calibrated with a line-scale interferometer at MIKES. The expanded uncertainty of calibration *U* was 0.5 µm (coverage factor *k*=2). With this line-scale a 200 mm × 200 mm area was calibrated (Fig. 3) as described in Publ. I.

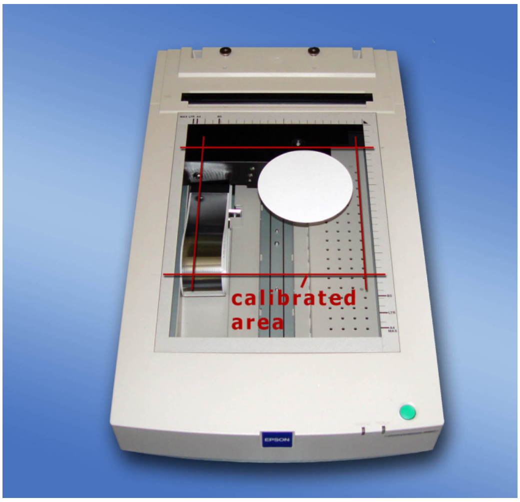

Figure 3. The calibrated area of the scanner

Also a radiometric calibration procedure was created to identify local differences in the grey scale response of the effective pixels, and to create a correction table by which these differences can be corrected. If the grey scale response of the effective pixels varies locally, then also the edge location of the sample appears to vary respectively. Therefore, the correction of the local differences in the grey scale response of the effective pixels decreases the uncertainty of area measurement.

The radiometric calibration was performed as described in Publ. I and was based on a 203 mm × 24 mm grey scale wedge (Fig. 4) which has 20 density levels from 0.05 to 1.95. The density values are nominal values given by the manufacturer of the wedge and they are not traceable.

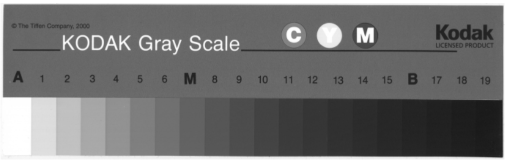

Figure 4. A grey scale wedge (Kodak Gray Scale Q-13) (Publ. I)

2.2.2 Edge detection and area measurement

For area measurement, the sample edge needs to be detected. However, the sample is a 3-dimensional object, whereas the scanner can only measure its 2-dimensional projection, and thus, the uncertainty of edge position increases with increasing sample thickness. The machine reel samples have to be cut manually, and therefore, they are not very precise. For instance, in case the sample is coated, the coating material can crack close to the edge, and thus, parts of the edge area may become more transparent than other areas. For this reason, a special edge detection technique was developed for the detection of the average edge position and the uncertainty estimation. The technique is based on the use of two separate threshold levels. With these levels, the minimum and maximum area of a sample can be estimated.

The principle of detecting the sample edge with two threshold levels is presented in figure 5. A sample is scanned against a black background to get a grey scale picture of the sample. The black background is used, because it usually increases contrast between the sample and the background. A histogram (the small picture in figure 5) is created based on the captured image. The horizontal axis of the histogram presents grey values and the vertical axis the occurrence of effective pixels (in logarithmic scale) with a particular grey value. The peak on the left in the histogram represents the effective pixels with low grey value (mainly the black background) and the peak on the right those pixels which have high grey value (mainly the sample). The valley between these peaks represents all those pixels with a grey value belonging in a transition area between the background and the sample. By setting the lower threshold level (the red line in the small picture) in the valley close to the background peak, the maximum area of a sample can be estimated (red line in figure 5), and by setting the another threshold level close to the sample peak, the minimum area of a sample can be estimated. These threshold levels are manually set during the area measurement procedure.

The red and blue lines which follow the sample edge in figure 5 have been constructed by colouring those pixels whose grey value is equal or larger than the lower (red line) or the higher threshold level (blue line). These rim lines include only the outermost pixels of the sample which pass these threshold levels, and thus, their width is 42 µm. The relative difference between the maximum and minimum area of a sample is typically less than 0.5%. In general, the larger and thinner a sample is, the smaller is the difference.

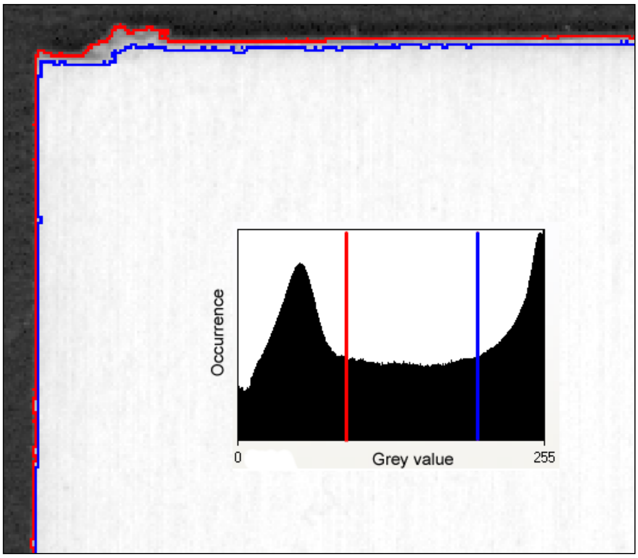

Figure 5. The principle of edge detection with two threshold levels (Publ. IV)

As mentioned in Ch. 2.1, the error sources can be systematic or random. The effect of systematic errors is reduced with geometric and radiometric calibrations. In this thesis, a method was created for the reduction of the influence of the drift of the systematic geometric errors. In this method, the reduction is performed with the aid of a special reference standard which is scanned both before and after the samples are scanned. The scanned area is then scaled by the ratio of the calibrated area of the reference and the scanned area of the reference. The schematic presentation of the calibration and area measurement procedures is shown in figure 6.

The reference standard used in scaling the sample scanning results is a custom made 100 mm diameter circle etched on a 140 mm × 140 mm × 3 mm glass plate. The area of the standard was calibrated using an optical coordinate measuring machine at expanded uncertainty of 0.5 mm^2 (coverage factor k=2) by MIKES.

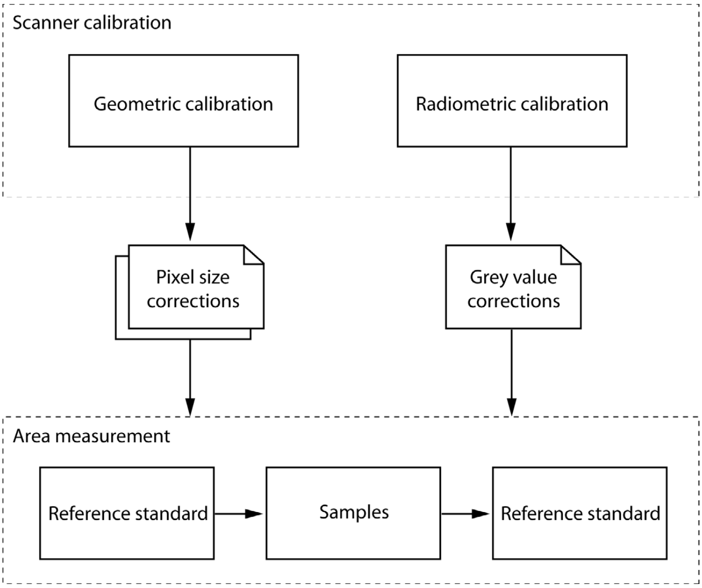

Figure 6. The area measurement principle and calibration corrections

2.3 Traceability and uncertainty

The geometric calibration, which is based on a traceable calibrated line-scale, is used to alleviate the uncertainty caused by varying effective pixel sizes of the scanner using the pixel size correction tables. Likewise, the radiometric calibration, which is based on a grey scale wedge, is used to decrease the uncertainty caused by varying grey scale responses of the effective pixels using the grey value correction table. However, the traceability of the sample area measurement to the SI-unit metre is ultimately based on the traceably calibrated reference, which is scanned before and after the sample scanning, and by area of which the sample area is scaled.

An estimation of measurement uncertainty according to [16] includes a mathematical measurement model, together with a description of each uncertainty component. In order to estimate the uncertainty of sample area measurement, a measurement model was created. It was presented originally in Publ. I for an ideal situation where the paper edge is straight cut and the thickness of a sample can be ignored. Later on, this ideal model was improved by adding a correction for sample thickness (Publ. IV). The final measurement model for sample area A is

$$A = \frac{A_\mathrm{R} + 2A_\mathrm{R}\alpha(296.15\,\mathrm{K} - T) + \delta A_\mathrm{T} + \delta A_\mathrm{d}}{A_\mathrm{Rs}} \times \left(A_\mathrm{s} + \delta A_\mathrm{h} + \delta A_\mathrm{i} + \delta A_\mathrm{res} + \delta A_\mathrm{rep}\right), \tag{2}$$

where A_R is the calibrated area of the reference, α is the thermal expansion coefficient of the reference, T is the calibration temperature of the reference (in Kelvins), δA_T is the correction for the temperature drift of the reference at the scanner, δA_d is the correction for the drift of the reference since the previous calibration, A_Rs is the scanned area of the reference, A_s is the scanned area of a sample, δA_h is the correction for sample thickness, δA_i is the correction for illumination, δA_res is the correction for scanner resolution and δA_rep is the correction for repeatability.

An example of the uncertainty budget is presented in Publ. I table 1. The standard uncertainties were combined in the table according to the principles of GUM [16]. A typical achieved relative uncertainty in the area measurements which were executed during this work was 0.2% at the 95% confidence level.

It has been demonstrated in this thesis work (Publications I and IV) that sample area can be measured in a traceable way with a traceably calibrated flatbed scanner. This is one possible answer to the first research question: *How can a paper or board sample area be measured in a traceably way?*

3 Basic calibration of online grammage measurement

3.1 Grammage measurement

Online grammage measurement exists practically in every paper and board production line. It is usually based on the attenuation of transmitted β-radiation (electrons) through a product web [12, 13, 41, 42]. β-radiation attenuates mainly as high-speed electrons lose their kinetic energy when they collide with web material atoms and scatter. A part of the energy of the electrons energy may also be converted into electromagnetic radiation when they decelerate, or they may escape from the detector by backscattering. The radioisotopes used in the grammage sensors emit electrons with continuous energy spectrum, which makes the attenuation of β-radiation to be a complicated process, as these different attenuation mechanisms depend on the energy of β-radiation electrons. However, from the grammage measurement point of view it is essential that the observed attenuation of β-radiation into a web depends mainly on the grammage of a web without being much sensitive to its material composition [13, 43, 44]. The ratio of the transmitted β-radiation intensity I to the full intensity I_0 can be approximated by an exponential function

$$\frac{I}{I_0} = e^{-\mu G}, \qquad (3)$$

where μ is an absorption coefficient (m²/g) and G is the grammage of a web [43].

The most common radioisotopes of the grammage sensors applied in the paper industry are Promethium (Pm-147), Krypton (Kr-85) and Strontium (Sr-90). Their maximum measurement ranges are approximately 200 g/m^2, 1100 g/m^2 and 5000 g/m^2, respectively [45]. Typical attenuation curves of these radioisotopes are shown in figure 7. Sensors made by different manufacturers have slightly different attenuation curves because the absorption coefficient is affected also by the measurement geometry of the sensor [46, 47]. An ionisation chamber is the most generally used detector in the grammage sensors, but also photomultipliers [48] and semiconductor detectors [49] are used.

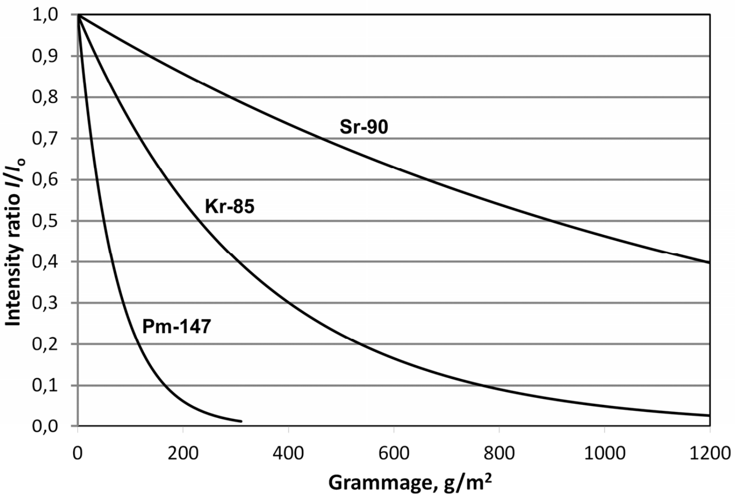

Figure 7. Examples of the β-radiation attenuation curves

A successful online grammage measurement in industrial environment requires overcoming multiple technical challenges. For example, the sensor response is affected by a possible mechanical misalignment of the sensor heads. This problem can be alleviated by sensor measurement geometry design [50-53], shielding the supporting structures of scanner frame against temperature variations [54-56] and measuring and correcting the effects of misalignments to the grammage sensor readings [57, 58]. Also, the air gap between the measurement heads can be kept clean by removing extraneous material from the radiation path with an air wipe system [59], and the effects of drifting sensor response can be reduced by zeroing the sensor periodically

[60]. Additionally, compensations based on the internal temperatures of the sensor heads and the air column temperatures may be performed [61] as well as active dirt compensation [62].

3.2 Basic calibration technique

Each sensor manufacturer applies its own proprietary algorithm to calculate the grammage reading given by the measurement system [63-65]. These models are generally calibrated and adjusted by performing a basic calibration with a set of plastic reference samples [11-14]. Plastic is used as a reference material because its chemical composition is close to that of wood fibres and water without being as vulnerable as true paper. The sensors are usually calibrated and adjusted by their manufacturers before they are installed in the mill, though sometimes the sensors are also calibrated at the mill [61, 65].

It is known that the local conditions in the mill affect the response of the measurement system [54, 58, 66], and therefore, the calibrations performed before the systems are installed at the mill are not completely valid in the mill. In this thesis, in order to investigate how much the errors of the grammage measurements systems can be decreased, sensors installed in the production lines were calibrated with a set of traceably calibrated grammage references (Fig. 8, 9) as described in Publication II.

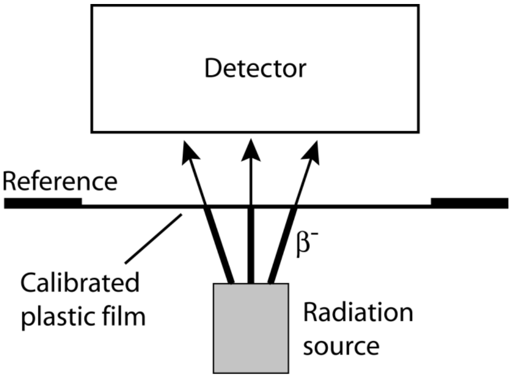

Figure 8. The basic calibration principle of online grammage measurement

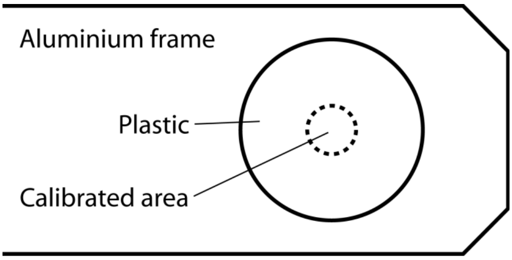

Figure 9. Grammage reference (Publ. II)

3.3 Results of Kr-85 based grammage measurement calibrations

During the calibration the references were measured one by one with the aid of a measurement system specific jig to guarantee the precise positioning of the reference. Examples of the observed basic measurement errors at the first time calibration are shown in Fig. 10. The horizontal axis represents the grammage value of the reference and the vertical axis is the basic grammage reading of the calibrated sensor subtracted by the grammage value of the reference. It is obvious from these examples that there can be quite large deviations between different grammage sensors. However, as can been seen in Fig. 11, these differences can be efficiently decreased with adjustments.

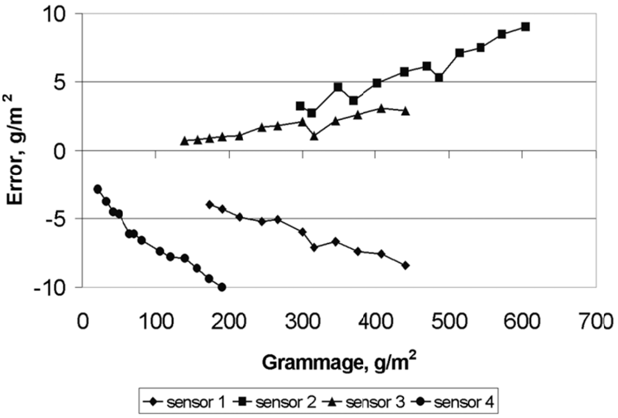

Figure 10. Examples of grammage calibration results before adjustment

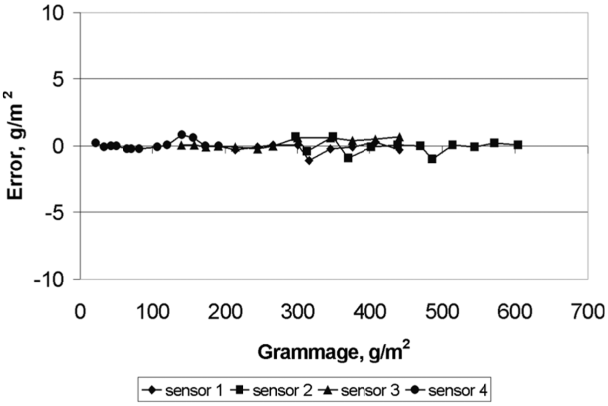

Figure 11. Examples of grammage calibration results after adjustment

The improvement potential of the radioisotope Kr-85 based online grammage measurement systems was evaluated by performing a statistical analysis. The analysed calibrations were executed at Finnish production lines during the years 1998-2010. The data set included 87 calibrations with 957 measurement points. These calibrations concerned 16 measurement systems which were manufactured by several different suppliers.

The first main result from the analysed calibration data was that the average relative basic measurement errors can be decreased approximately 85% with adjustments based on the calibration results. The expanded (k=2) standard deviation of the relative measurement errors being 3.3% at the first time calibrations (Fig. 12) and 0.5% after the adjustments (Fig. 13) (Publ. II). This result answers to the second research question: *How much can the measurement uncertainty of online grammage measurement be decreased with a traceable basic calibration executed at site?*

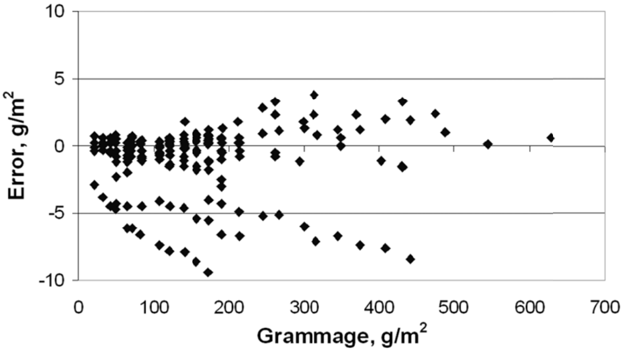

Figure 12. Observed basic errors at the first time calibration. Number of calibrated sensors N=16 with number of measurement points n=174 (Publ. II)

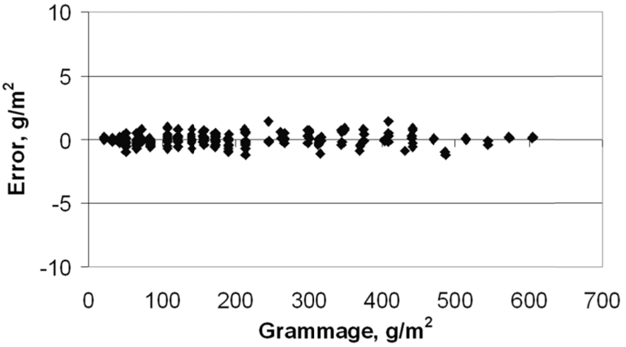

Figure 13. Basic errors after adjustment (N=20 with n=228) (Publ. II)

The second main observation from the results of repeated calibrations was that the basic errors of the grammage measurement systems drifted between calibrations, the expanded (k=2) standard deviation of the relative drift being 2.1% (Fig. 14). It was also demonstrated in Publication II that the calibration method can be used to study the temperature and dirt sensitivity of grammage measurement system.

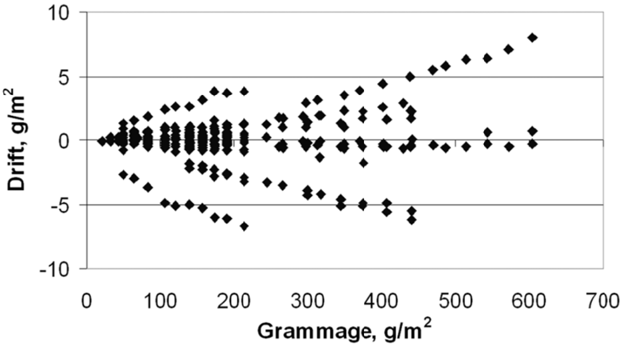

Figure 14. Drift since last calibration (N=28 with n=288) (Publ. II)

3.4 Proposed adjustment algorithm

It was observed during this study that usually a linear adjustment is adequate to correct the basic measurement errors (Publ. II). However, these adjustments were sometimes cumbersome to perform because of the varying adjustment possibilities of different measurement systems. Therefore, a standardised algorithm for the calculation of the grammage reading G_i for each product grade i is suggested to ease the adjustment of the different grammage measurement systems. The algorithm is

$$G_i = a_i \left(cG_s + d \right) + b_i \,, \tag{4}$$

where G_s is the basic grammage reading of the measurement system, a_i and b_i are adjustment parameters, which are optimised based on the grade calibration results for each product grade *i*, and *c* and *d* are adjustment parameters, which are optimised based on the basic calibration results.

From the calibration point of view it is essential that there are accessible separate parameters (*c* and *d*) by which the basic errors can be decreased without a need to change the grade parameters (a_i and b_i), or the parameters of the manufacturer specific proprietary algorithms. This kind of standardised approach could streamline the adjustment of different measurement systems.

3.5 Traceability and uncertainty

The traceability of the calibration is based on traceably calibrated references and a traceable field calibration method. According to this method, the error of the basic grammage reading E_G and its uncertainty were estimated with a simplified measurement model

$$\begin{aligned} E_\mathrm{G} &= G_\mathrm{s} + \delta G_\mathrm{z} + \delta G_\mathrm{rep} + \delta G_\mathrm{res} \\ &\quad - \left(G_\mathrm{r} + \delta G_\mathrm{T} + \delta G_\mathrm{rh} + \delta G_\mathrm{a} + \delta G_\mathrm{b} + \delta G_\mathrm{t} \right), \end{aligned} \tag{5}$$

where G_s is the basic grammage reading of the measurement system, δG_z is the correction for the location of the grammage reference between the source and the detector, δG_{rep} is the correction for the repeatability of the grammage measurement, δG_{res} is the correction for the finite resolution of the grammage reading, G_r is the calibrated grammage of the reference, δG_T is the correction for the thermal expansion of the reference, δG_{rh} is the correction for the humidity expansion of the reference, δG_a is the correction for the moisture absorption of the reference, δG_b is the correction for the mismatch of the sensor beam size to the calibrated area of the reference, and δG_t is the correction for the transmission angle of the sensor beam through the reference.

The uncertainty of the basic error E_G (Eq. 5) was evaluated at each measurement point. The best measurement capability of the calibration method was 0.3 g/m² when the grammage was under 100 g/m², and 0.3%

from the reading when the grammage was greater than 100 g/m^2, at the 95% confidence level.

4 Basic calibration of online moisture measurement

4.1 Moisture measurement

Unlike grammage sensors, which are not molecule sensitive, moisture sensors are designed to respond to the presence of water molecules (H_2O) in a web. Molecules have bonds between their basic elements (atoms), which enable them to absorb energy, making them vibrate in molecule specific modes. Water molecules absorb specifically at certain NIR (near infrared radiation) wavelengths. Usually, one of those wavelengths (for example 1.94 µm) is utilised to detect the presence of water in a web. In order to decrease the disturbances associated with the measurement, the attenuation of at least one other reference wavelength, which is not sensitive to water, is also detected. The intensity ratio of the water sensitive and the non-sensitive wavelengths is then used as an indicator of the amount of water in the web [13, 67].

Usually, an IR moisture sensor measures the relative attenuation of different wavelengths of transmitted radiation through the paper or board web (Fig. 15). The surface moisture of the web can be measured with an IR reflection based sensor.

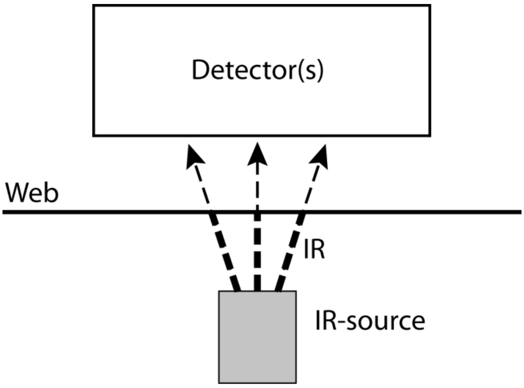

Figure 15. The measurement principle of IR-transmission based online moisture measurement

Water can also be detected with microwave radiation based techniques. These sensors are more penetrating and less sensitive to the optical properties of web material than IR based sensors. They are used in applications which are not suitable for IR sensors, such as the moisture measurement of heavy grammage products and products having high carbon black content.

There are basically two different measurement principles by which the moisture can be detected with microwaves. The most common technique is based on sensing the dielectric constant (the relative permittivity) of a web. The higher the dielectric constant of a material, the easier it can be polarised with an electric field. Since the water molecule has a permanent dipole moment (it is electrically polarised) its dielectric constant (approximately 80) is higher than that of other web materials (typically less than 10), and thus, the presence of water in a web has a substantial effect on its dielectric constant. These sensors consist of two cavities located on the opposite sides of a web, forming a microwave resonator whose resonance frequency shifts as the dielectric constant changes. The other technique relies on microwave absorption on a specific frequency (22.2 GHz) that sets water molecules to rotate around their axes in a rotational resonance. This is similar to the absorption of specific IR wavelength that sets the molecules to vibrate. Sensors applying the microwave absorption technique can be either two-sided (transmission based) or single-sided (reflection based) [13, 68].

The moisture content of a paper or board product is normally expressed as percentage mass fraction, which is the percentage ratio of the water mass of the product to the total mass of the product [69]. A moisture sensor usually measures only the water grammage of a web. The total mass, which is needed in the calculation of percentage mass fraction, is measured with a separate grammage sensor [67, 70]. However, the moisture measurement can also be performed without a grammage sensor [71, 72].

A typical measurement range of an IR based online moisture sensor is from 2% moisture to 15% moisture with grammage range up to 400 g/m^2. Moisture contents as high as 70% with water grammages up to 1500 g/m^2 can be measured with the microwave technique. The measurement ranges of these techniques overlap partly since some microwave sensors are able to measure moisture from newsprint papers (approximately 50 g/m^2) [73].

Moisture sensor specific techniques are applied to minimise the sensitivity of the moisture measurement. For instance, measurement head misalignment, web pass line location changes (flutter), the optical properties of a web, ash content variations and the effect of changing web temperature [13, 74-78]. Most of the methods that alleviate the effects of harsh industrial environment on online grammage measurement (Ch. 3.1) also improve the performance of moisture measurement.

4.2 Basic calibration techniques

Similarly to the basic grammage reading (Ch. 3.2), the basic percentage moisture reading given by the measurement system is also calculated using a manufacturer specific mathematical model. The basic calibration of the sensor is usually performed with reference samples sealed in plastic bags [11, 12, 79-83]. However, some manufacturers also apply reference samples sealed between glass plates in their calibrations. These samples last longer and are more convenient to handle at the production line than the bagged samples, which need to be stored in a desiccator above saturated salt solutions in order to remain valid.

In this thesis, the sensors installed in the production lines were calibrated with a set of calibrated moisture references (Fig. 16) with the procedure described in the Publication III. All the references were custom made for each measurement system out of the paper or board material collected from that specific production line and sealed between two glass plates.

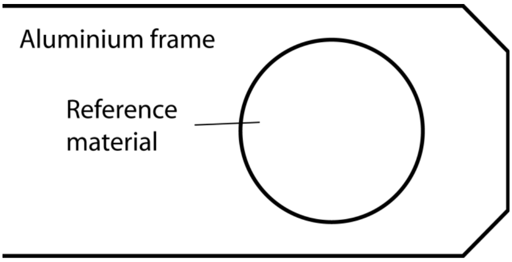

Figure 16. Moisture reference

4.3 Results of IR based online moisture measurement calibrations

The improvement potential of the basic errors of moisture measurements was evaluated in a similar way as for grammage measurements (Ch. 3.3). The analysed data set consisted of basic calibration results of IR based online moisture measurement systems. These calibrations were executed at several Finnish paper and board production lines during the years 1998-2008. A total of 22 moisture measurement systems, made by different manufacturers, were inspected with 141 calibrations, including 205 product grades and 843 measurement points.

The errors in the first time calibrations were remarkable as can be seen in figure 17. The horizontal axis represents the calibrated moisture content of the reference and the vertical axis is the basic moisture reading of the calibrated sensor subtracted by the moisture of the reference. Both axes are in the units of percentage moisture. By performing adjustments, these errors decreased significantly as is demonstrated in figure 18.

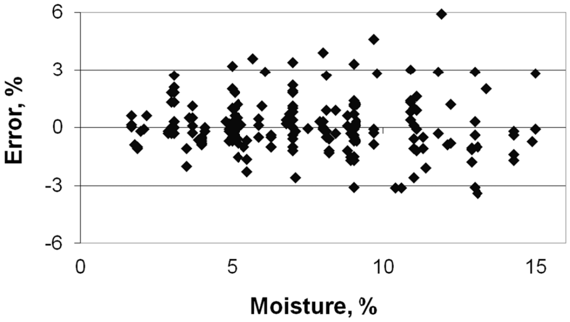

Figure 17. Basic errors at the first time calibration. Number of calibrated sensors *N*=22 with number of calibrated product grades *p*=49 and with number of measurement points *n*=193 (Publ. III)

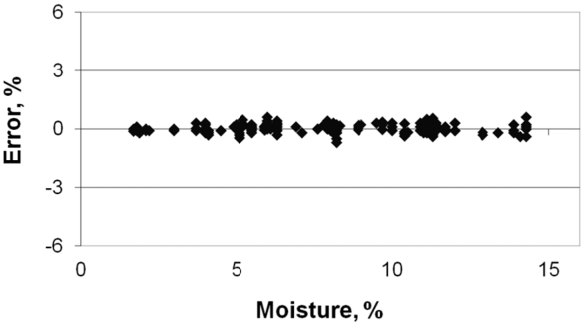

Figure 18. Basic errors after adjustment (*N*=23 with *p*=47 and with *n*=174) (Publ. III)

The first main result from analysed calibrations data was that the average relative basic measurement errors could be decreased over 85% with adjustments based on the calibration results. The expanded (*k*=2) standard deviation of the measurement errors being 2.9% moisture at the first time calibrations and 0.4% moisture after the adjustments (Publ. III).

The above result gives answer to the second research question: *How much can the measurement uncertainty of online moisture measurement be decreased with the basic calibration executed at site with custom-made reference standards and by using traceable calibration methods?*

From the results of repeated calibrations it could be noticed that the basic errors of the moisture measurement systems drifted in the same way as the basic errors of the grammage measurements, the expanded (k=2) standard deviation of the relative drift being 1.9% moisture between the calibrations. These results indicate that both grammage and moisture measurements need to be calibrated and adjusted regularly in order to keep their errors small.

4.4 Proposed adjustment algorithm

It was demonstrated (Publ. III) that a linear adjustment is usually adequate to correct the basic measurement errors, as was the case also with the grammage measurement errors (Ch. 3.4). Therefore, having a similar standardised algorithm for the calculation of the percentage moisture reading M_i for each product grade i available in every measurement system would also ease the adjustment of different moisture measurement systems. The proposed algorithm was

$$M_i = a_i\left(c_j M_\mathrm{s} + d_j\right) + b_i\,, \tag{6}$$

where M_s is the basic percentage moisture reading of the measurement system, a_i and b_i are parameters which are optimised based on the grade calibration results for each product grade i, and c_j and d_j are parameters which are optimised based on the basic calibration results of each product grade group j.

The difference between the proposed adjustment algorithms (Eq. 5 and 6) is, that whereas there is only one plastic reference set for the basic calibration/adjustment of grammage sensor, there can be more than just one set for a moisture sensor, and therefore, there needs to be a possibility to adjust the sensor for each set with separate parameters (c_j and d_j). For example, if both uncoated and coated products are measured with a moisture sensor, there can be one reference set made out of the main uncoated grade and a respective set for coated grades. The basic calibration/adjustment is then performed separately for uncoated and coated grade groups with these reference sets.

4.5 Traceability and uncertainty

The dry mass and moisture content of the reference material was determined by applying the EN ISO 638 standard [84] and the grammage by applying the EN ISO 536 standard [18]. All the weighings were performed with a traceably calibrated and maintained laboratory balance. The area of the reference was measured with a ruler at the beginning of this study, but later on with a traceably calibrated flatbed scanner.

To estimate the calibration uncertainty, a simplified measurement model was created for the basic moisture measurement error E_M:

$$E_\mathrm{M} = M_\mathrm{s} + \delta M_\mathrm{z} + \delta M_\mathrm{rep} + \delta M_\mathrm{res} - M_\mathrm{r}\,, \qquad (7)$$

where M_s is the basic percentage moisture reading of the measurement system, δM_z is the correction for the location of the moisture reference between the source and the detector, δM_rep is the correction for the repeatability of the moisture measurement, δM_res is the correction for the finite resolution of the moisture reading, and M_r is the calibrated moisture content of the reference.

The uncertainty of the basic error E_M was evaluated at each measurement point. The best measurement capability of the calibration method was 0.3% moisture at the 95% confidence level.

A systematic error is introduced to the calibration results since the measurement of a reference through glass distorts the sensor reading. To decrease the effect of this error, the sensor was standardised with plain glass before the calibration. However, the reported uncertainties do not include the possible contribution of this error because no practical method was available to evaluate it at each calibration point.

An oven-drying method was applied for the determination of the dry mass and the moisture content of the reference according to the ISO standard [84].

It has been reported in the previous publications [85, 86] that since the oven is ventilated with laboratory air, which is not completely dry [87], there might still be some residual moisture in the samples after drying. The remaining moisture content varies due product grades and is usually less than 0.2% moisture, but can be for some grades over 1% moisture. Another problem of this standardised method is that the sample material may contain small amounts of materials other than water which evaporate during the oven-drying which may affect the results. These two issues can cause a systematic grade specific error in the moisture determinations of the references. The possible contributions of these errors were not estimated nor included in the reported uncertainties in this thesis work.

5 Grade calibration of online dry grammage measurement

5.1 Grade calibration techniques

Obtaining correct dry grammage results for each product grade requires grade calibration of both grammage and moisture measurements. In this procedure the grade specific parameters of a measurement system, for example, the parameters a_i and b_i in Equations 4 and 6, are optimised so that measurement results that are as correct as possible will be obtained for each product grade.

Traditionally, the grade calibration of the grammage measurement is performed by taking laboratory samples from a machine reel and comparing the online results with the respective laboratory results. A sampling method for the determination of average quality and sample grammage has been standardised [18, 88]. Later on, an average reel grammage method was created for the grade calibration of online grammage measurement. It is based on comparing the average machine reel grammage, measured by the online grammage sensor, with the respective average calculated from the net mass, lengths and widths of the rolls cut from the machine reel [14, 54, 89]. Basically, a whole machine reel is a sample which evens out efficiently the effects of process variations, resulting in small spread in repeated comparison results. For this reason, and because of the possibility to automate the collection of comparison data, this method has replaced the traditional sampling method in many mills.

The grade calibration of the moisture measurement is commonly performed by taking samples from a machine reel and comparing the online results with the respective laboratory results [14, 89]. The average moisture content of a web can be determined according the same ISO standard [88] which is used for the machine reel sampling of grammage. A standardized method [69] based on oven-drying is applied for the determination of the moisture content in which the percentage moisture content of a sample $w_{\mathrm{H_2O}}$ is calculated from the wet mass m_0 and dry mass m_1 of a sample based on the equation

$$w_{\mathrm{H_2O}} = \frac{m_0 - m_1}{m_0} \times 100 \quad . \qquad (9)$$

There have also been attempts to create methods by which a sample could be taken from a moving web instead of a machine reel in order to get samples which are as representative as possible [91, 92]. This would be useful, for instance, for the calibration of a scanner which is located far away from the machine reel. However, these methods are technically challenging and rarely used.

Probably the greatest challenge related to the grade calibration of grammage and moisture measurement is to get representative samples from the product because the moisture content of a web usually changes after the scanner frame when it is exposed to the atmosphere. Another issue is, that the calibration of both grammage and moisture measurement needs to be based on similarly conditioned web material references if one wants to achieve accurate dry grammage results. The environmental conditions vary around the production line in different seasons, and therefore, for example, the combination where grammage is calibrated based on the average reel grammage method and moisture based on machine reel sampling, does not guarantee accurate dry grammage measurement results. At the moment, whatever the applied grade calibration methods are in the mills, the calibration of grammage and moisture is performed separately, resulting in the fact that the calibration of grammage and moisture measurements are based on differently conditioned references.

5.2 Developed sampling based dry grammage calibration method

A novel method was developed for the grade calibration of dry grammage measurement. In this method both the grammage and moisture content of a product web are determined from the same sample set taken from a machine reel. By this way, the fundamental problem of current calibration methods, differently conditioned grammage and moisture references, can be eliminated.

During the sampling the samples are manually cut from a machine reel. The sizes of these samples are measured with the traceable sample area measurement technique (Ch. 2.2.) which was also created during this thesis work. The application of this technique decreases the uncertainty of area determination and brings along traceability to SI-unit metre.

In the created calibration procedure, the last grammage and moisture CD-profiles (cross-machine direction) before the reel turn-up are recorded to document the online sensor readings at the end of a machine reel. The samples are then taken from the machine reel at evenly spaced positions in cross-machine direction (Fig. 19) immediately after a reel turn-up by applying the standard EN ISO 186 [69], as described in Publ. IV. The samples are then transported to the laboratory for the determination of their grammage and moisture content.

One of the main uncertainty sources in the grade calibration procedure is caused by the fact that the samples cannot be taken exactly from the web area measured by the scanning sensors. The effect of this mismatch can be reduced with the presented method by taking samples from several machine reels and calculating grade parameters based on the average results.

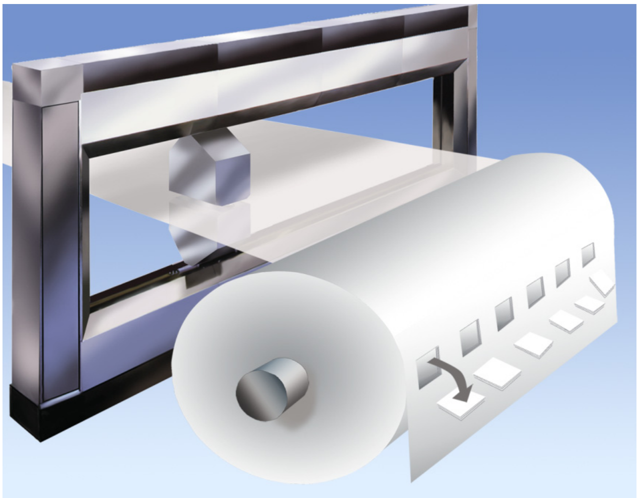

Figure 19. Sampling from a machine reel

5.3 Dry grammage calibration results

Before the new method was applied to the grade calibration of grammage and moisture measurements in the board mills, the basic errors of the grammage and moisture measurements were reduced with basic calibration and adjustment, as described in Ch. 3 and 4. This was performed in order to provide a good starting point for the grade specific calibration work.

For the estimation of the smallest achievable uncertainty in grade calibration, the best data sets from the performed calibrations were collected and analysed statistically. It was demonstrated in Publication IV that by applying the developed new method the grade calibration of online dry grammage measurement can be performed with a relative uncertainty of 0.5% at the 95% confidence level, the relative uncertainty of the grammage calibration being 0.4% and the uncertainty of moisture calibration being 0.3% moisture.

Examples of sampling profiles are shown in figures 20 - 22. The dry grammage profiles (Fig. 22) were calculated from the respective grammage and moisture results. These examples demonstrate how the correctness of the online measurement profiles can be evaluated with the sampling results.

To ease the profile shape comparison the online measurement results were adjusted with their grade parameters so that the profile averages were identical with the respective laboratory result averages.

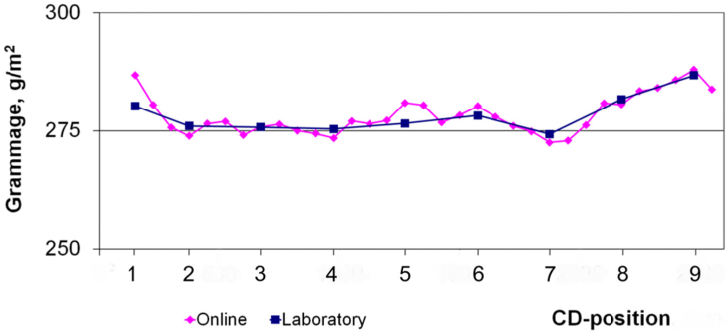

Figure 20. Example of a grammage sampling profile and the respective online profile

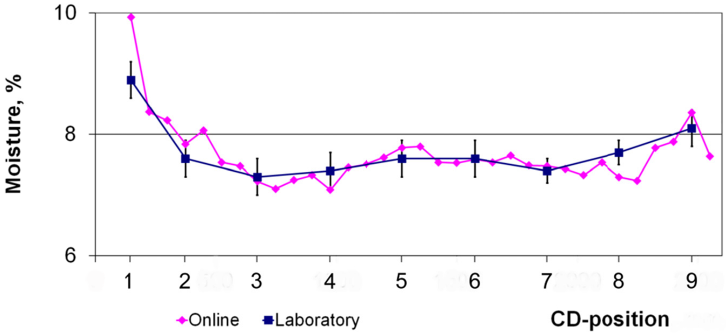

Figure 21. Example of a moisture sampling profile and the respective online profile

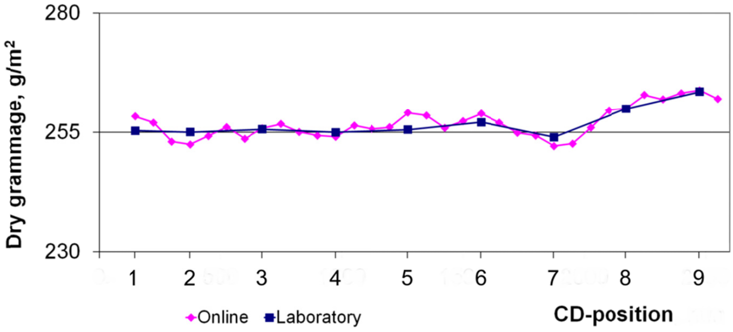

Figure 22. Example of a dry grammage sampling profile and the respective online profile

5.4 Traceability and uncertainty

The determination of the sample grammage was performed by applying the EN ISO 536 standard [18]. The traceability of the grammage determination was based on a traceably calibrated laboratory balance and the traceable sample area measurement. The uncertainty of the grammage was determined for each sample, the relative uncertainty of the grammage being typically below 0.2% at the 95% confidence level.

The determination of the sample moisture content was performed by applying the EN ISO 287 standard [69]. The weighing of a sample, both before and after drying, was executed with a traceably calibrated balance. The temperature measurement of the oven was also calibrated, as well as the equipment which was used to monitor the atmosphere temperature and relative moisture of the laboratory. Generally, the uncertainty of the moisture content determination was below 0.2% moisture at the 95% confidence level.

The answer to the fourth research question (*Is it possible to achieve traceability in the grade calibration of online dry grammage measurement executed at site?*) is not fully satisfying. For the grammage, an unbroken traceability chain to the SI-units can be established. The moisture content can be determined with oven-drying according to the EN ISO 287 standard [69], but the result is not the true moisture content of the material because the residual moisture is ignored from the result and the effect of possible other volatiles than water is included in the result. Thus, the moisture determination is not traceable to SI-units when obeying the ISO standard [69]. As the dry grammage is determined with the aid of the same oven-drying procedure, the dry grammage determination is also not fully traceable to SI-units.

As discussed at the end of Ch. 4.5, the residual moisture is usually less than 0.2% moisture when applying the standard EN ISO 638 [83] for the determination of dry matter content. The same oven-drying procedure was applied when the moisture content was determined from machine reel samples according the EN ISO 287 standard [69]. These standards are intended to be used providing that the sample material does not contain any substances, other than water, that are volatile at the oven-drying temperature. Nevertheless, because the presence of these materials could not be tested there may have been small amounts of them present. Both the residual water and the possible presence of other volatile materials than water cause a grade specific, systematic error to the moisture determination.

The amount of residual water could be reduced by ventilating the oven with dry air instead of laboratory air, but this approach would violate against the ISO standards [69, 84]. The presence of other volatile materials than water could be tested if there were a practical test method available.

6 Discussion

6.1 Application of results

The developed calibration methods can basically be applied in all paper and board production lines to improve the performance of existing online quality measurement systems. However, these methods require quite a lot of background work, maintained equipment, facilities and routines in order to guarantee small uncertainties and traceability.

When the uncertainties of online grammage and moisture measurements are reduced it will also indirectly improve the performance of those other online measurements which need grammage or moisture content information for their operation. For instance, the X-ray absorption based filler (total ash) measurement [93-95] requires support from both grammage and moisture measurements.

Once the online measurement scanner located at the dry end of a production line is calibrated, as presented in this thesis, the average moisture content of rolls in the packaging area can be calculated. By supposing that the dry grammage of the product remains constant between the scanner frame and the packaging area, the average moisture content of the rolls can be obtained from the average reel dry grammage, measured by the scanner, and the average reel grammage in the packaging area, which is calculated from the net weights, lengths and widths of the rolls cut from the machine reel. In many mills, the production personnel monitor that the average grammage of the product stays within specifications in the packaging area. By this approach, they will be able to monitor also the average moisture content of rolls in the packaging area.

Furthermore, this will also enable the control of this moisture content, for instance, by changing the set points of the grammage and moisture content of the product at the scanner. By this way, all moisture content changes between the scanner frame and the packaging area can be compensated, and the moisture and dry material content of the produced rolls can be kept constant at different seasons. This will result in reduced product quality variations and decreased annual dry raw material consumption, since currently just the product grammage is kept constant in the packaging area.

6.2 Reliability and limitations

The EN ISO/IEC 17025 standard [17], which is the requirement for accredited calibration laboratories, was applied in the measurements and calibrations performed during this thesis work. The most essential procedures are FINAS-accredited, for instance, the traceable calibration method of grammage references and the traceable field calibration (calibration at site) of grammage sensors have been accredited since 1998 and 2003, respectively. The traceability of the performed measurements has been discussed more detailed in chapters 2.3, 3.5, 4.5 and 5.4.

Data sets which contained results from a total of 87 grammage and 141 moisture calibrations were used as a basis of the analysis when the improvement potential of the basic errors of grammage and moisture measurements was evaluated. These data sets are believed to contain realistic and representative information on the measurement systems used in Finnish paper and board production lines as they include results from various age measurement systems made by different manufacturers. According to the author's knowledge, the analyses which were performed in Publications II and III are based on the largest published data set concerning basic calibration results of online quality measurements.

For the estimation of the smallest achievable uncertainty in the grade calibration of online dry grammage measurement, a total of 15 calibrations were performed which each included online and sampling results from three machine reels. Out of the most successful calibrations a representative data set was selected for the analysis. The data set includes calibration results from different board grades (6 grades) with various grammage values

(190 g/m^2 – 450 g/m^2). This data set is also large enough so that the results of the executed statistical analysis can be relied on (Publ. IV, table 1).

The research problem concerning the possibility of establishing a metrological traceability chain to the dry grammage calibration was studied by splitting the problem into several research questions and by analysing each of them separately. It was demonstrated how a traceability chain can be created for both the basic and grade calibration of grammage measurement. However, it seems that metrological traceability to the SI-units for the determination of the true moisture content of the paper or board product cannot be fully established as long as the EN ISO 287 standard [69] (and the EN ISO 638 standard [84]) is applied in moisture determination.

6.3 Recommendations for future research

Establishing a true traceability chain to the SI-units for the determination of moisture content of a paper of board sample requires a method to estimate the amount of the sample residual moisture after oven drying. It will also require a method to estimate the uncertainty caused by the possible evaporation of other volatile materials than water during the moisture determination. A development of practical methods, to overcome these obstacles, would increase the reliability of the moisture and dry grammage calibration further.

Beside grammage and moisture measurements, there are also other online quality measurements used in the paper industry, such as ash content (inorganic substances) and coat grammage, which do not yet have traceability chains to the SI-units. These inorganic additives are an essential part of modern paper and board products, and therefore, it may be justified to develop traceability chains also for their measurements in the future.

This thesis work concentrated on the currently dominating grammage and moisture measurement techniques. However, there are emerging new multi-measurement sensors which are based on the utilisation of near-infrared spectrum [96-98]. There may also be a need to build traceability chains for the calibration of these new sensors.

7 Conclusions

It was shown that the uncertainties of the existing online grammage and moisture measurement systems can be significantly reduced with traceable calibration methods. For instance, it was demonstrated that the basic measurement errors of these systems can be decreased on average approximately 85% with the traceable methods.

A novel method was developed for the simultaneous grade calibration of grammage and moisture measurements based on machine reel sampling. The method applies the traceable sample area measurement technique, which was also created during this work. It was demonstrated that the grade calibration of online dry grammage measurement can be performed with the relative uncertainty of 0.5% at the 95% confidence level with the created method. This means that the uncertainty of a typical measurement system can be decreased over 50% with the new calibration method. The calibrations were performed by applying the standards EN ISO 536 [18] and EN ISO 287 [69].

Reliable measurements are based on a metrological traceability chain. It was shown in this thesis how the traceability chain can be established to the calibration of online grammage and moisture measurement. The calibration of grammage measurement can be fully traceable to the SI-units. The moisture measurement can be calibrated by applying the EN ISO 287 standard, which is commonly used in the paper industry, but this prevents the calibration to be SI-traceable because this standard accepts a small amount of water (residual moisture after oven-drying) to be excluded from the determined moisture content result. It is known from the previous publications [85, 86] that the amount of residual moisture is product grade specific and it is usually less than 0.2% moisture. This causes a systematic error to moisture calibration which then transfers further on to online dry grammage measurements.

The calibrations performed during this thesis work concerned mainly the radioisotope Kr-85 based grammage sensors and IR transmission based moisture sensors, but the presented calibration methods can basically be applied in all paper and board production lines. In those production lines where the average grammage of rolls can be calculated based on their weighing results in the packaging area of a mill, it is possible to calculate also their average moisture content based on the calculated average roll grammages and the online dry grammage measurement results, once the online scanner calibrated as presented in this work. This will open up a possibility to monitor the moisture content of the rolls and keep both the grammage and moisture content of rolls constant during different seasons.

Based on the calibration experience of different online measurement system, it is also demonstrated how the adjustment of both grammage and moisture measurement algorithms could be standardised. By this way, the maintenance of measurement accuracy could be more straightforward than it is today.

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The optimisation of raw material consumption and even product quality are vital to the profitability of the paper and board industry. In the automatic control of production, the total grammage and moisture content of produced material are key parameters which are measured during production. It was shown how these online measurements can be made traceable to the SI-units. It was also demonstrated that the application of traceable calibration methods significantly decreases the uncertainties of these measurements.

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