IMPLEMENTATION AND MEASUREMENT UNCERTAINTY OF A HANDHELD MOISTURE METER IN PAPERBOARD MOISTURE MEASUREMENT

Lappeenranta–Lahti University of Technology LUT

Master’s Programme in Chemical Engineering, Master's thesis

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Examiners: Associate Professor Arto Pihlajamäki

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ABSTRACT

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School of Engineering Science

Chemical Engineering

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90 (+7) pages, 33 (+9) figures, 2 tables, and 1 appendix

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Keywords: board, moisture, measurement uncertainty, measurement error, deviation

This thesis aimed to study different methods for moisture measurement and determine their strengths and weaknesses. The suitability of these methods for polymer-coated paperboard was assessed. Measurement error and uncertainty, and different methods for their determination were familiarized. In the experimental part, a capacitive moisture meter was implemented on an extrusion coating line producing polymer-coated liquid packaging board and solid bleached board. A measuring procedure for the meter was created. Measurement error and uncertainty were determined from the experimental data at a 95 % confidence level and Gage Repeatability and Reproducibility measurement system analysis was performed for the meter to determine the main source of variation in the data. The performance of the new meter was compared to the old microwave-based meter. The results from both meters were compared to ISO 287 standardized oven-drying method to determine the absolute error.

The new meter performed well, and the results were generally accurate. A statistically significant difference between the results of the new meter and oven-drying was proven for two of the four product grades studied, but the meter still managed to meet the accuracy requirements that have been created for the old microwave-based meter. The Gage R&R analysis proved that variation in the data is mostly from the different measured reels and not the meter itself or the measuring operator.

Despite the uncertainty caused by an occasional small number of samples, the statistical analysis provided evident results that the new meter performs at least equally well as the older meter, with average absolute errors ranging from 0.04 % to 0.34 % for certain types of products. For the greater absolute errors, the standard deviation was also higher.

TIIVISTELMÄ

Lappeenrannan–Lahden teknillinen yliopisto LUT

LUT Teknis-luonnontieteellinen

Kemiantekniikka

Antti Tolvanen

KÄSIKÄYTTÖISEN KOSTEUSMITTARIN KÄYTTÖÖNOTTO JA MITTAUSEPÄVARMUUS KARTONGIN KOSTEUSMITTAUKSESSA

Kemiantekniikan diplomityö

2023

90 (+7) sivua, 33 (+9) kuvaa, 2 taulukkoa ja 1 liite

Tarkastaja(t): Associate Professor Arto Pihlajamäki ja DI Kati Syväoja

Avainsanat: kartonki, kosteus, mittausepävarmuus, mittausvirhe, hajonta

Tämän diplomityön tarkoituksena oli tutkia erilaisia kosteusmittausmenetelmiä ja määrittää niiden vahvuudet ja heikkoudet. Työssä arvioitiin näiden menetelmien soveltuvuutta polymeeripäällysteiselle kartongille. Yhdessä kappaleessa perehdyttiin mittausvirheeseen ja -epävarmuuteen, sekä niiden määrittämisessä käytettäviin menetelmiin.

Kokeellisessa osassa ekstruusiopäällystyslinjalle otettiin käyttöön kapasitiivinen kosteusmittari. Laitteen mittausvirhe ja -epävarmuus määritettiin kokeellisen datan perusteella 95 %:n luottamusvälillä ja laitteelle suoritettiin Gage Repeatability & Reproducibility -mittausjärjestelmäanalyysi pääasiallisen vaihtelulähteen määrittämiseksi. Uuden mittarin suorituskykyä verrattiin vanhaan mittariin, jonka mittaukset perustuivat mikroaaltoenergian absorptioon. Mittareiden tuloksia verrattiin ISO 287 -standardoituun uunikosteusmenetelmään, jonka avulla määritettiin absoluuttinen virhe.

Uusi mittari toimi hyvin, ja tulokset olivat yleisesti ottaen tarkkoja. Tilastollisesti merkittävä ero uuden mittarin ja uunikuivauksen tulosten välillä todettiin kahdelle neljästä tutkitusta tuoteryhmästä, mutta mittari onnistui silti täyttämään vanhalle mikroaaltomittarille asetetut tarkkuusvaatimukset. Gage R&R-mittausjärjestelmäanalyysi osoitti, että vaihtelu tuloksissa johtuu pääasiassa eri mitattujen rullien välisistä eroista, eikä itse mittarista tai mittauksen suorittajasta.

Ajoittaisesta pienestä näytemäärästä johtuvasta epävarmuudesta huolimatta tilastollinen analyysi antoi selkeät tulokset siitä, että uusi kapasitanssipohjainen mittari suoriutuu vähintään yhtä hyvin tai paremmin kuin vanha mikroaaltopohjainen mittari. Keskimääräiset absoluuttiset virheet verrattuna uunikosteuteen vaihtelivat 0,04 %:sta 0,34 %:iin eri tuotteiden välillä. Virheen ollessa suurempi, myös keskimääräinen hajonta tuloksissa oli myös suurempaa.

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Antti Tolvanen

List of abbreviations and symbols

ABBREVIATIONS

ANOVA	Analysis of Variance
CTMP	Chemi-thermomechanical pulp
EVOH	Ethylene vinyl alcohol
FIR	Far infrared
H_0	Null hypothesis
H_1	Alternate hypothesis
I-MR	Individual - Moving Range
IR	Infrared
ISO	International Organization for Standardization
LCL	Lower Control Limit
LPB	Liquid packaging board
MIR	Mid-infrared
MSA	Measurement System Analysis
NIR	Near-infrared
PDF	Probability density function
PE	Polyethylene
PET	Polyethylene terephthalate
PHA	Polyhydroxyalkanoate
PLA	Polylactic acid
R&R	Repeatability and Reproducibility
SBS	Solid Bleached Board

SD	Standard deviation
UCL	Upper Control Limit
VIM	The International Vocabulary of Basic and General Terms in Metrology

SYMBOLS

A^2	Anderson-Darling test statistic	-
$\overline{X}$	mean value of a sample	-
ε'_r	relative permittivity, dielectric constant	-
ε_0	the permittivity of vacuum	$8.854 \cdot 10^{-12}$ F/m
ε_m	permittivity of the measured material	F/m
μ	expected or mean value	-
A	surface area	m^2
C	capacitance	F
d	distance	m
e_i	error of type i	-
gsm	grams per square meter	g/m^2
n	number of samples	-
x_i	measured value for measurement i	-
θ	true value of a measurement	-
σ	variance	-

Table of Contents

1 Introduction

Paperboard is widely used as a packaging material for various products. Board mills of Kaukopää at Stora Enso's Imatra mills produce different kinds of packaging boards, of which liquid packaging board and cup paperboard for the food industry are the primary products. Moisture greatly affects the physical properties of board, decreasing the tensile strength and causing deformation (Niskanen 2008). Since the legislation in the food industry is very strict, appropriate quality control is especially important. Defective products delivered to the customer always lead to reclamations, where expenses may increase to hundreds of thousands of euros.

Excess moisture causes changes in the fibrous structure of paper and board. The fibers swell and shrink along with changing moisture content, which causes irreversible changes in the structure of the board. If these deformations for some reason manage to get past the quality monitoring system, they may cause problems during post-processing like printing (Seppälä et al. 2000; Paulapuro 2000).

Moisture can be continuously measured online by an automatic quality control system or manually from finished reels with a moisture meter specifically designed for paper and board. The ability to measure moisture manually post-production is important for traceability purposes, and it acts as a protective document for liability when the moisture is proven to be within acceptable limits at the production facility. This is especially important due to European Parliament and of the Council Directive 1935 (2004), which states that the traceability of all materials must be ensured at all stages.

This thesis aims to justify the need for a manual moisture meter, as well as to implement a new meter to the coating lines, and to determine the meter's capabilities and accuracy. This thesis consists of two parts: a literature review and an experimental part. The work was carried out from December 2022 to May 2023.

In the literature review, the goal is to study the effects of water on the structure and physical properties of paperboard. Different paperboard moisture measurement methods are explored and their suitability for measurement in the extrusion coating process is evaluated. Lastly, measurement error and uncertainty are discussed and methods to minimize error during

sampling and measuring in general are examined. The research questions are presented below in Figure 1.

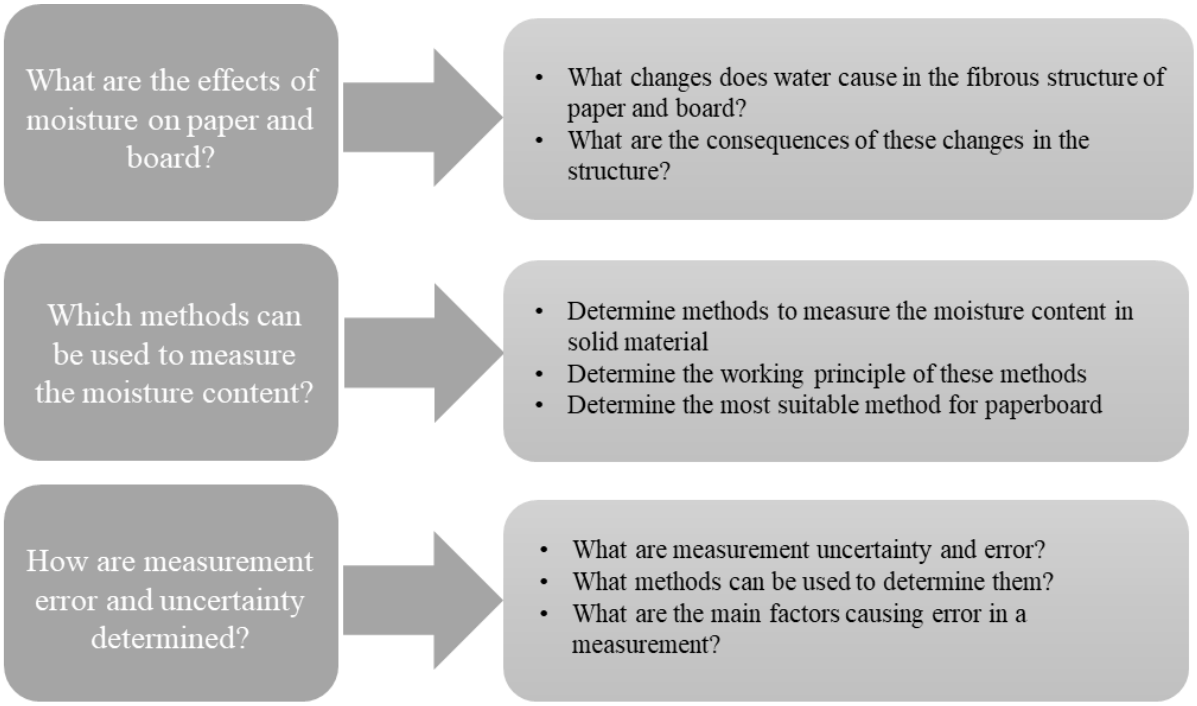

Figure 1. The research questions and objectives of the theoretical part.

In the experimental part, a certain capacitive moisture meter is implemented and configured for coating mills at Stora Enso. The goal is to determine settings that provide the most accurate results for all board grammages, polymer coating types, and their amounts while using oven-drying as a comparable standard. Then, the measurement uncertainty and confidence intervals for the results are calculated. The last goal of the experimental part is to determine the reasons for measurement variance from the collected moisture content data by using Minitab statistical software to perform Gage Repeatability & Reproducibility study for measurement system analysis.

1.1 Structure of the thesis

The first part of the work is the literature review. First, the paperboard structure and extrusion coating process are studied to gain an understanding of the background of this work. Alongside that, the effects of moisture on paperboard, the coating process, and the post-production process such as printing are studied. Then, different moisture measurement

methods for solid materials and more specifically for paper and board are presented. These methods are then compared among themselves to determine which method is possibly most suitable for paperboard. Lastly in the literature section, measurement error and uncertainty and their sources are studied, as well as methods for determining them.

In the experimental part, the new handheld moisture meter and the currently used measurement equipment are presented and after that, the methods for comparing the measurement methods are demonstrated. The performance and accuracy of the new meter and old existing methods are compared against the standardized oven-drying moisture measurement method. The measurement uncertainty is determined by utilizing Minitab data analysis software. Sources of error and their magnitude are estimated in the later chapters, and finally, conclusions from the thesis are summarized at the end.

2 Paperboard

Paper and board consist of fibers that are distributed non-uniformly on a flat plane. The fibers consist mainly of cellulose, hemicellulose, and lignin. Despite non-uniformity, the fibers are more aligned in the machine direction, the long side of the reel, which causes the board to have a higher tensile strength in the machine direction than in the cross-machine direction. Fiber orientation also affects how the board swells and shrinks due to moisture. Fibers are significantly longer than they are thick, but the three-dimensional porous structure influences how fluids travel throughout the material. (Niskanen 2008). This is especially important when examining moisture gradients and their effect on the board. A microscope image of the structure of the paperboard is shown below in Figure 2.

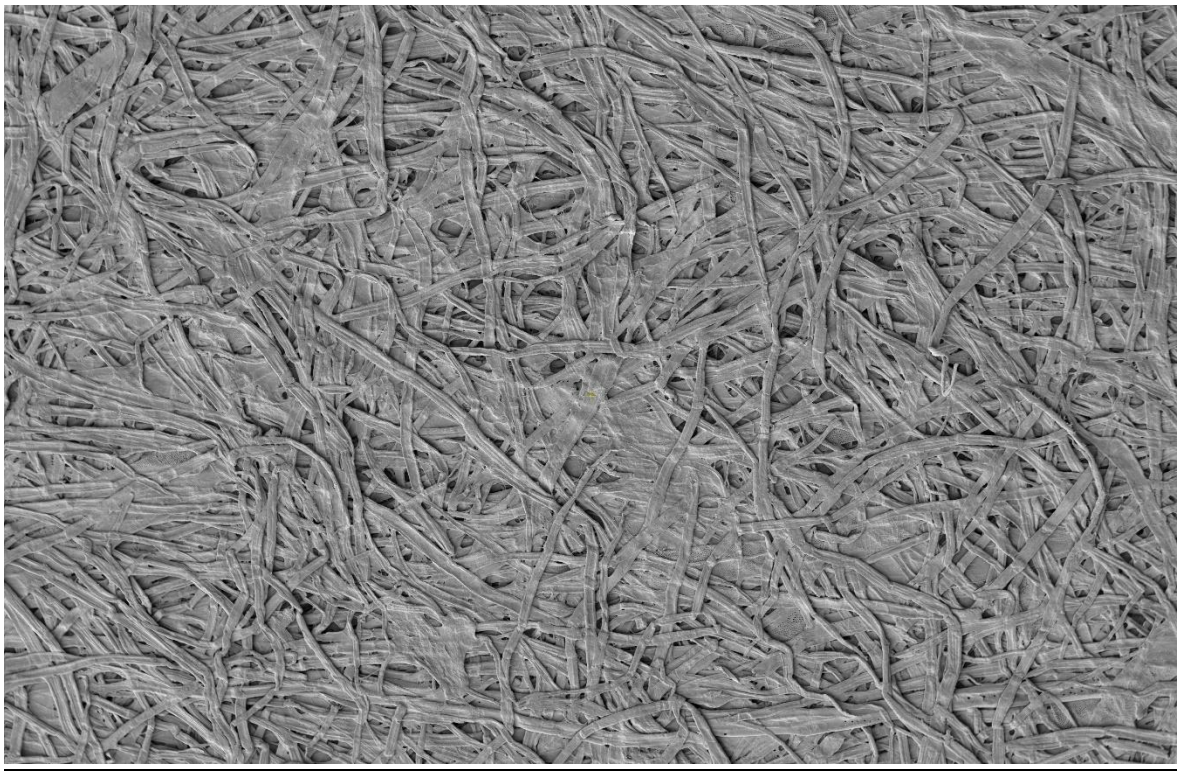

Figure 2. Scanning electron microscope image of paperboard produced at Stora Enso mills. Raw material board from the coating mills was scanned at Stora Enso Research Center Imatra on request.

Paperboard grades are divided into three categories: cartonboards for lighter packaging, containerboards, which are often corrugated board, for more heavyweight packaging and

transportation, and special boards for special applications such as plasterboard or bookbinding board. (Paulapuro 2000). All these board grades have their own structure and most suitable area of use. This thesis will focus on lighter cartonboard packaging grades, especially solid bleached (sulfate) board (SBS), used for example in cardboard cups, and liquid packaging board (LPB).

The raw material composition and structure of the board varies depending on the intended end use. SBS is usually a single-ply product (Figure 3), but it is possible to also produce multi-ply SBS with certain techniques, which allows the composition to be further optimized for desired properties, such as bulk or printability. (Paulapuro 2000).

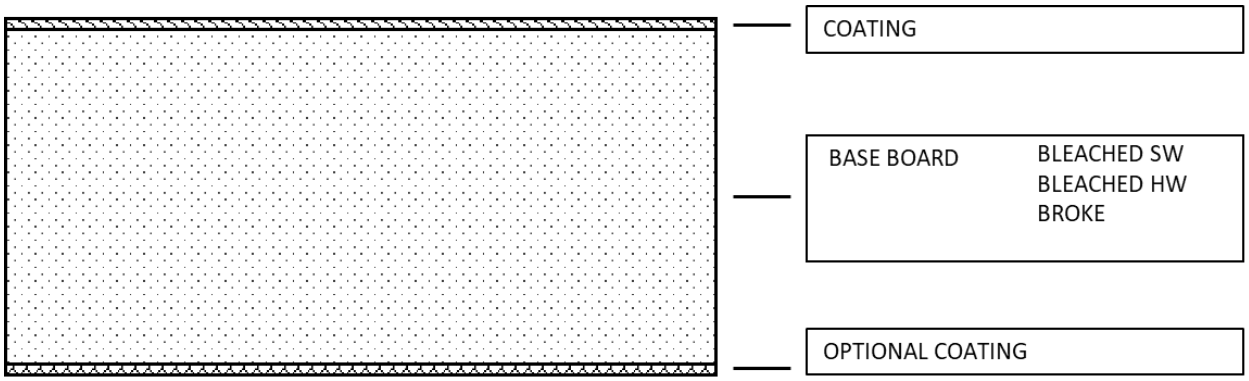

Figure 3. Single-ply structure of the solid bleached board (SBS) (adapted from Paulapuro 2000)

The main component of SBS is usually bleached hardwood (HW) pulp. Softwood (SW) pulp may be used as well, but hardwood is generally considered superior due to its better formation and printing properties. Broke, which refers to repulped paper or board that is discarded from processes can also be utilized in some cases. SBS is very often coated to further increase the surface properties. (Paulapuro 2000).

Liquid packaging board usually has a minimum of three plies along with one or two layers of coating. The back and top plies are usually made from material with high elastic modulus and the middle ply is made of pulp with maximum bulk to provide sufficient stiffness. An example of the structure of LPB is presented in Figure 4 below. (Paulapuro 2000).

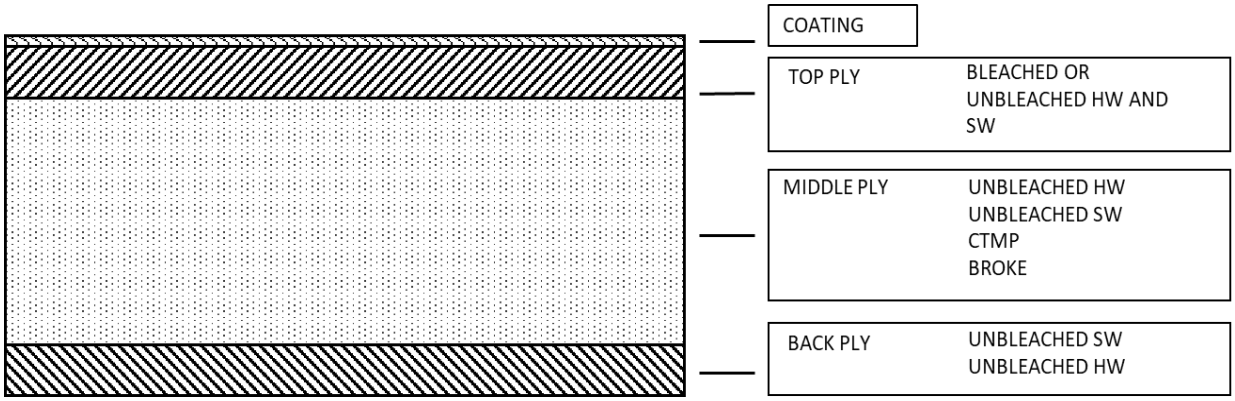

Figure 4. Example of a three-ply composition of liquid packaging board (adapted from Paulapuro 2000)

In LPBs, chemi-thermomechanical pulp (CTMP) is often used in the middle layer as it has greater bulk than traditional chemical pulp. This enables using a lower basis weight for a certain stiffness. However, unbleached hardwood or softwood pulp may be used as well, and often the product is a mixture of these three. Broke is less often used for LPBs, because of the rule of only using virgin fiber. Outer plies are made from pulp with higher elastic modulus, usually from hardwood or softwood pulps as described earlier for the SBS products. Together these plies create a product that is robust enough to stand straight but is also elastic and durable, while the package weight remains low. (Paulapuro 2000).

SBS is often used for packing products such as chocolate, cosmetics, cigarettes, or even food since it usually does not cause issues with odor or tainting. Strength, runnability, attractive appearance, and this previously mentioned tendency to be odor free are all important properties of the solid bleached board. LPB is purely used for packing liquids, as per its name. It is used for brick-type and gable-top packages, such as the common one-liter milk cartons. For LPB, it is extremely important that the material is of high purity and cleanliness. This is ensured by using only virgin fibers, as opposed to utilizing recycled materials even in small amounts. LPB must be strong and stiff, which is ensured by a proper middle ply as demonstrated in the chapter above. Liquid packaging is usually coated from both sides with a high-barrier coating, such as polyethylene. (Paulapuro 2000).

Most cartonboard grades are often coated with pigments for improved printing properties. Multiple pigments are usually applied to the surface of the board. These pigments are usually minerals such as kaolin clay or calcium carbonate. Boards are usually pigment-coated only on one side, whereas paper may be coated on both sides depending on its intended end use.

The first layer of coating is used to fill pores on the surface as well as to improve the opacity of the board. The top layers improve the surface smoothness, gloss and adsorption properties for improved printability, which is the main reason coating is done. The base board or paper must have high and uniform strength and structure, as well as optimal surface smoothness and pore structure to achieve good results during the pigment coating. (Paltakari 2009).

2.1 Extrusion coating

Most of the board produced at Kaukopää mills is shipped to the customer as it comes from the board mill. Roughly one-third of the board is coated at one of the four coating mills (Stora Enso Oyj 2023). These products from paper or board mills are coated to improve their properties to be different from those of uncoated paper or board. Coatings are usually polymers, which enhance barrier properties such as gas and water vapor permeability for food packaging, or water and grease resistance for liquid packaging. These properties increase the shelf life of packed products and protect them from external damage. Polymer coating also affects sealability, printability, and resistance properties to wear. (Kuusipalo 2008).

2.1.1 Coating process

The extrusion coating is the process where molten polymer coating is introduced to a substrate, like paper or board, and immediately nipped between a nip roll and a cooling drum. Tandem extrusion is used when multiple extruders are used for products coated multiple times or on both sides, like liquid packaging. The first extrusion coating was done in a laboratory in the 1940s, from which grew the first commercial extrusion coating line developed by DuPont, St. Regis Paper Company, and Egan Machinery Corporation. (Durling 2017). An example of an extrusion coating line is presented below in Figure 5.

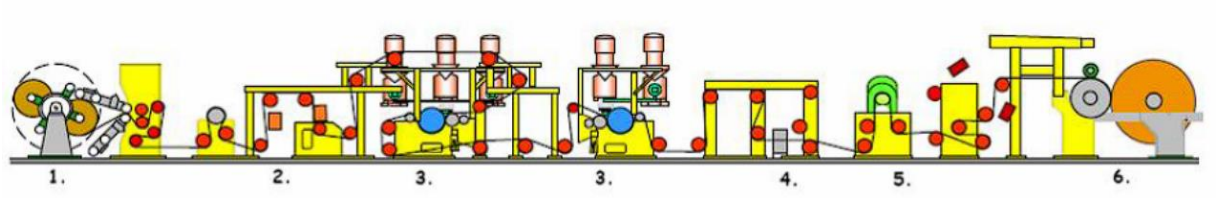

Figure 5. Example of an extrusion coating line. 1. unwinder, 2. pre-treatment, 3. laminator (extruder), 4. online quality measurement, 5. post-treatment, 6. rewinder (Pulkkinen 2009).

The extrusion coating process starts at the unwinding, where the raw material board reels are attached to the moving web. The surface of the board is pretreated to guarantee sufficient adhesion. Several surface treatment options are available: flame treatment, corona treatment, ozone treatment, and even plasma treatment. Of these, flame and corona pre-treatment are the most common options. (Durling 2017).

Corona pre-treatment is more often used for flexible thin paper or aluminum foil webs. The treating system consists of a power supply and a treatment station, where high-frequency power is applied on the surface of the material. Top part of the treater are the electrodes emitting the power, and the bottom part is a grounded roll. The dielectric part between the components is made up of the air gap and the substrate, the material to be treated. The high potential difference of the electrodes is used to produce plasma in the air gap. This forms highly reactive radicals, ions, and ozone, which interact with the polymer surface and break down the chemical bonds to create polar groups, that increase the surface energy. (Das et al. 2021). The treater also removes organic or inorganic contaminants and hence improves adhesion. (Durling 2017). If using too high power, corona treatment will cause etching and roughness on the material surface (Das et al. 2021).

For paperboard pre-treatment, the most commonly utilized method is flame-treatment. It is used to burn off fibers which can cause pinholes in the polymer film and lead to poor adhesion. The flame torch also removes dust particles and other impurities from the surface of the board (Durling 2017). Flame treatment decreases the moisture content of the board significantly. Each flame treatment burner decreases the moisture content by roughly 1 % at full power. If both sides of the board are coated and treated this means that the moisture decreases by about 2 % during the pre-treatment. As long as the web speed is fast enough, the structure of the board is not damaged. (Lankinen 2022).

The extruder is the one unique component of an extrusion line, that melts polymer, glue, and dye pellets to form a melt curtain of plastic coating. In simplicity, the extruder is a single- or multiple-screw conveyor which melts the fed pellets around the midsection of the screws and disperses the materials into a homogenized mixture at the end. After the screw comes the feedblock, which arranges the polymer streams into the desired sequence, which comes out of the die. The die lowers the polymer film on top of the moving board web. After contact between the polymer and board, the web is directed to a nip between the chill roll and a nip roll, and pressed the layers are pressed tightly. The pressing in the nip ensures tight contact between the polymer and the board. (Durling 2017).

The temperature of the molten polymer film is usually very high, from 200 degrees of bioplastics to up to 300 degrees Celsius for more heat-resistant polymers (Seppälä et al. 2000). Heat from the extruder and plastic film transfer to the surrounding air, which causes the ambient temperature to also increase. Especially during hot seasons, the humidity of the hall can be high, which causes water to condense on the chill roll. (Lankinen 2022). This creates the risk for local wet and weak points in the board web, which may in the worst-case cause issues like web tearing leading to shutting the process down.

After the extruder and lamination, the coated board can be post-treated for better surface properties. Corona treatment explained earlier is the most common post-treatment method, as it increases wetting and adhesion capabilities, but most importantly it enables the surface to be printed with better results than the non-treated equivalent (Durling 2017).

Online measurements are located right after the post-treatment section of the process. The amount of polymer coating and the moisture content are measured by an online measurement sensor. This is also where quality sensors are used to detect flaws like scratches or molten polymer droplets, which should not end up in the final product. (Seppälä et al. 2000).

At the end of the coating line, the web is reeled at a winder to a reel of certain tightness. The machine reels are large and heavy since it is the most effective way to store webs. Samples from the product reels are taken at the winder after the reel is done. Samples are usually cut through the cross-direction of the reel by hand and then die-cut into smaller samples if needed. After taking the samples and other required measurements, the reel is forwarded to the slitter, where it is cut into smaller product rolls that are packed and shipped to the customer. (Durling 2017; Seppälä et al. 2000)

2.1.2 Coating polymers

Different polymers can be used to give the product specific desired properties. Polyethylene (PE) is the most used polymer, due to its cheap price, high sealability, and moderate moisture barrier. Polyethylene terephthalate (PET) is a polymer that is highly thermoformable and easy to heat-seal. Over polyolefins, an advantage of PET is the low sorption of flavor and aroma from foods. When a strong barrier is needed, barrier polymers such as ethylene vinyl alcohol (EVOH) can be used. These are often used with PE and adhesives to create strong oxygen and aroma barriers. (Kuusipalo 2008). A disadvantage of the previously mentioned coatings is their non-biodegradable and fossil-based nature.

Biodegradable and renewable polymers used today include starch mixtures, polyhydroxyalkanoate (PHA), and polylactic acid (PLA). These polymers have good resistance and barrier properties against oxygen or grease, but nearly completely lack water resistance. Thus, their use on a larger universal scale is inhibited. (Kuusipalo 2008). Bio-based versions of PE and PET polymers exist and can be used, but similarly to their fossil-based equivalents, they are not biodegradable as they are practically the same molecules as their non-bio counterparts. (Siracusa & Blanco 2020). Bio-based plastics are also more expensive; Bajpai (2019) claimed that the price of bio-PE is roughly 50 % higher than traditional fossil-based PE, which slows down the otherwise increasing demand. However, it was mentioned that the price of bio-PE would go down as the production volumes increase in the future.

Polymers or other coating materials used in extrusion may cause difficulties with moisture measurement. High conductivity from salts or barrier properties that obstruct electromagnetic waves from penetrating beneath the surface, may cause the measurement to give incorrect results (Skaar 1988). All coatings, whether they are polymers or clays, also cause changes in the total product density, which influences the measurement result of many kinds of meters (Bobrov et al. 2019).

2.2 The effects of moisture

The effects of moisture on paper and board have been studied since the 1900s (Byrd 1972; Mark et al. 1983). Many theories for the mechanics of moisture-induced change of properties have been suggested (Panek et al. 2004), but no universal standard consensus exists. Nonetheless, Seppälä et al. (2000) consider the most important property of paper or board to be its moisture content, since most of all other properties directly or indirectly depend on it.

Board or paper moisture content is the ratio of the mass of the absorbed water divided by the mass of the board. The moisture content depends on the relative humidity in the ambient air. The relative humidity is dependent on the amount of water in saturation, which again is heavily but not alone influenced by temperature. Higher temperature equals lower relative humidity and lower moisture content in the board in equilibrium. (Levlin & Söderhjelm 1999).

Since the fibers in board and paper are hygroscopic, they absorb moisture from the surrounding air. The fibers react to moisture by swelling and stretching. This phenomenon is called hygroexpansion. It was found by Lyne et al. (1996) determined that using inorganic fillers such as clay or chalk decreases hygroexpansivity. Paper sheets with 40 % clay filling had roughly 20 % lower hygroexpansivity than sheets without filling. Hygroexpansion is typically up to 20 % larger in lateral cross-machine direction than in longitudinal machine direction due to fiber orientation (Lindner 2018; Ketoja 2008).

Water can exist in fibers in two forms: as free water or as bound water. Free water is absorbed between the pores or inside lumen cavities. Free water in the paper can act as a solvent and thus it can contain salts that inhabit electrolytic properties. Bound water is absorbed inside the pores or chemically bonded to carboxylic acid and hydroxylic groups of the fibers in the paper. (Niskanen 2008).

The moisture content of the board compared to surrounding relative humidity is different when starting from humid conditions and approaching dry conditions or vice versa. This effect is called hysteresis. This effect is caused by irreversible physical changes in the fibers. The effect of this moisture hysteresis is demonstrated in Figure 6 below. The moisture content can be anywhere in between the curves of the hysteresis diagram depending on the humidity history of the product. (Niskanen 2008).

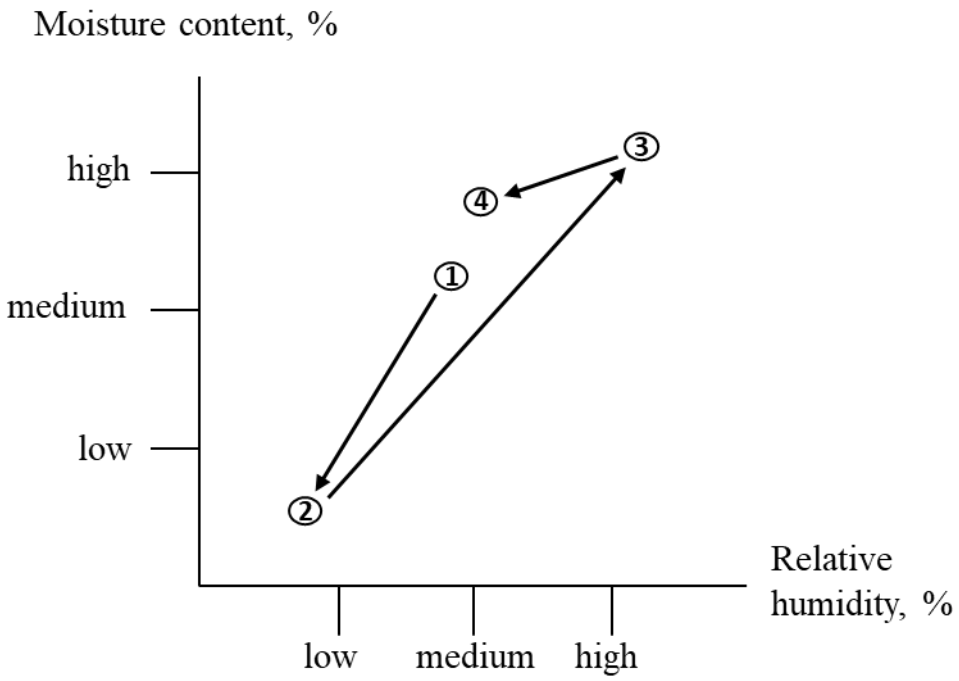

Figure 6. The moisture content of the board vs. relative humidity. Starting from medium moisture at point 1 the fibers of the board suffer permanent deformations when relative humidity changes through points 2 and 3. When returning to medium relative humidity at point 4, the moisture content of the board has increased significantly. (Adapted from Kirwan 2005)

In particularly humid conditions, such as in heavy rain during transportation, the importance of sufficient packaging and protection is emphasized. The board reels are wrapped with a mixture of kraft liner and polyethylene, with inner and outer discs made of board and possible insulative films.

The tightness of reel packages was examined in a thesis by Leskelä (2008). In the thesis, it was determined that the insulation is nowhere near perfect and that a moisture gradient from the outer layers towards the middle of the reel occurs, when the reels are stored in increased humidity (>50%). In the same thesis, it was concluded that the packaging significantly slows down the diffusion of moisture but does not completely prevent it. The majority of the air and moisture leak in through the seams since the wrapping material blocks moisture well. During sealing, excess moisture alongside oxygen can cause film degradation in the wrapper and thus worsen the sealing conditions (Kuusipalo 2008). The conditions during transportation determine the moisture content of the reel, which also highlights the need for a convenient and quick handheld moisture meter.

The excessive moisture content of the board greatly affects the adhesion in the extrusion coating process. Sufficient adhesion between the board and coating plastic may not be achieved if the moisture content is more than 10 %. The surface of the board is usually treated with hydrocarbon flame prior to extrusion to improve the adhesion by oxidating the surface with oxygen from the air. (Lankinen 2022).

2.2.1 Viscoelastic properties

Viscoelastic properties of the board change along with changing moisture content due to the changing dimensions of the fibers. Higher moisture content leads to decreased tensile strength and increased elongation. Wet paper has considerably lower tear strength when compared to dry equivalent. (Höke & Schabel 2010). Salmén & Olsson (2016) also concluded in their experiments that the length changes in the paper are directly linked to the change in its moisture content. If the board is reeled in dry air and stored in increased moisture, these changes in length and increased fiber elongation together may cause the reel to come loose. This may eventually lead to the reel not staying together when handled with forklifts or cranes, creating a safety hazard.

During drying, fibers in the paper shrink anisotropically, which causes shear stresses in the bonding area of the fibers. When laid open to moisture, the stresses release and cause permanent deformation such as fiber shrinkage, which again causes paper curling or wavy edges (Niskanen 2008; Panek et al. 2004). When operating large paper webs at a high speed, seemingly small relative changes may cause severe problems visually and quality-wise. Curl causes most problems in the printing of the final product. The result of curled sheets is not satisfactory during printing, and dramatically diagonally curled sheets may not be printed on at all. (Ketoja 2008)

Paper tends to conform to its surroundings under continuous stress. A simple example of this phenomenon are corrugated boxes, which when fully loaded, over time bulge and eventually break (Coffin & Fellers 2001). In cyclic humidity, where the relative humidity increases and then decreases back multiple times, this so-called *creep* is greatly accelerated, which will result in a shorter lifetime of the paper or board (Panek et al. 2004).

3 Moisture measurement methods

The water content of the board may not be measured directly, so instead all meters for this purpose utilize indirect measurement. Instead of the water content, a measurable characteristic that correlates with the amount of water present is determined. The actual moisture content can then be resolved via calculation. (Jensen et al. 2006).

There are numerous different methods for measuring moisture in different materials. The most commonly known method is measuring electric resistance or conductance. An electric current is passed from the meter's pins through the material, and the moisture content can be then determined from the resistance curve. This method is often used for determining moisture content in building materials such as concrete or wood. However, resistance-type meters are excluded from this review due to their unsuitable moisture content range starting at a minimum of 8 % (Forsén & Tarvainen 2000). Resistance-type meters were also reported to be slower (Forsén & Tarvainen 2000) and in one study even less accurate than capacitance-based meters (Davis et al. 2017). The measurement methods studied in this thesis are capacitive, microwave radiation, and infrared radiation methods.

A common factor for all the studied methods, excluding traditional oven-drying, is their utilization of electromagnetic waves at different wavelengths. Each spectrum of wavelengths has its own strengths and weaknesses in terms of penetration and measurement depth, accuracy, tolerance of interference, and cost of equipment. All electromagnetic methods are quick, and the results are available immediately (Forsén & Tarvainen 2000; Paaso 2007; Kraszewski 2001).

In modern times, online measurements are considered easier in the industry since they do not require constant manual labor and provide real-time results. Online measurements cost more to implement compared to manual measurements but do save a considerable amount of working time since there is no need for taking samples, transporting the samples or meters, or manual measuring. However, online measurements are more novel, and new problems are associated with them. Most difficulties arise with the uncertainty and calibration of online systems. The act of calibration is not traceable, since usually after a basic calibration with reference samples, the fine-tuning part of the procedure is done by taking laboratory samples

and comparing them to respective laboratory results (Kangasrääsiö 2011; Kangasrääsiö 2010). Proper accurate calibration also consumes a lot of time, since every product grade group has to be individually fine-tuned. For processes where multiple parameters change depending on the product, this may lead up to thousands of required samples (Siisiäinen 2023). Online meters are also vulnerable to environmental factors such as temperature, humidity, and fouling due to dirt or dust (Kangasrääsiö 2011).

3.1 Oven drying

For paper and board, the conventional method of determining moisture content is the oven-drying method. Many analysis and determination methods are standardized according to the International Organization for Standardization (ISO). Results from these standardized analyses are usually comparable with each other generally and can be expected to be accurate and reliable. (International Organization for Standardization 2022).

3.1.1 Procedure

Samples are taken according to ISO 186:2002 for paper and board: damaged layers are removed from the reel plus 1-3 layers depending on the paper grammage. For paperboard with grammage greater than 225 g/m^2, only one layer removed should suffice. Depending on the size of the lot, or in this case the reel, the minimum number of samples is between 10 to 20 sheets. Since moisture content is to be determined, the samples have to be quickly sealed into plastic bags and delivered to the laboratory. (ISO 2002).

ISO 287:2017 is the standardized method for the determination of the moisture content of a paper or board sample. From the sample bag, the three outermost and all damaged sheets are discarded. At least four consecutive sheets are cut into desired size strips and a mass of at least 50 grams. The container and the sample are weighed and the mass at the initial sampling is recorded. (ISO 2017).

The test piece is dried in an oven within its container. The oven temperature must be maintained at 105 ± 2 °C. The drying period must be at least 60 minutes for grammages greater than 225 g/m^2. After drying, the samples are cooled down in a desiccator. The

container and its content are weighed, and the mass of the dried test piece is calculated by subtracting the mass of the container from the total weighed mass.

The procedure is repeated until a constant mass is reached. The mass is considered constant when the mass of the sample between two consecutive weighings does not differ more than 0.1 % of the initial mass. (ISO 2017).

aching constant mass, the moisture content w_{H_2O} can be calculated according to equation 1:

$$w_{H_2O} = \frac{wet\ mass - dried\ mass}{wet\ mass} * 100\ \% \qquad 1$$

This procedure is performed on each of the samples and their duplicates.

The major disadvantage of oven drying is the time consumption. Samples must be oven dried for several hours for accurate results, which means that the results cannot be used to make real-time adjustments to the process. If the results from oven-drying are alarming, a great number of produced rolls must be sorted and treated, which causes a large amount of manual labor. (ISO 2017).

The sampling procedure is also very important, and the plastic bags must be sealed perfectly for delivery, so the samples cannot dry or draw moisture from the ambient air and provide false results. If the sample bag is discovered to be punctured or torn, the sample should be discarded without measurement. (Siisiäinen 2023).

3.2 Capacitive measurement

The dielectric properties of a material depend greatly on its moisture content. Dielectric capacitance measurement methods have been found to be suitable and efficient in quickly determining the moisture content of cellulose-based materials, such as wood, paper, and board (Silveira et al. 2021; Ek et al. 1997; Skaar 1988). The method is also suitable for many other materials, such as solid biofuels (Jensen et al. 2006) or even soil (Bobrov et al. 2019; Eller & Denoth 1996).

The capacitive measurement method to measure moisture content is based on measuring the changes in the electric field through capacitance. The changes are caused by the dielectric properties of the paper when radiated with alternating current (AC) at a high frequency and

oscillating voltage (Skaar 1988). The frequency for moisture capacitance-based measurements can vary from the radio frequency wavelengths at 100 kHz up to 1 GHz without severely impacting the dielectric constant for water, and thus the result of the moisture measurement. At lower frequencies, the high conductivity of free ions from salts affects the measurement (Baxter 1997). However, this issue does not interfere with the moisture measurement of paper or board since there should not be any dissociated electrolytic salts present in the board or the modern coating polymers.

Along with the moisture content, other parameters affecting dielectric properties include density, temperature, and the frequency of alternating electric fields in the measurement. (Skaar 1988; Nelson 1981). Since temperature is easy to measure and is often ruled out by meters' automatic adjustment, density is relatively constant in a batch of product, and the frequency is set before measuring, the unknown changing moisture content can be deduced to have the greatest impact on the dielectric properties of paper or board product in a continuous process with little variation. It is important to remember to adjust and calibrate the measurement correctly when the product specifications and especially the density change.

3.2.1 Theory behind capacitance measurement

The dielectric properties are expressed as the relative permittivity to the permittivity of a pure vacuum. This relative permittivity is also known as the dielectric constant:

$$\varepsilon'_r = \frac{\varepsilon_m}{\varepsilon_0} \qquad 2$$

where ε'_r is the relative permittivity or the dielectric constant, ε_m is the permittivity of the measured material and ε_0 is the permittivity of vacuum, which is $8.854 \cdot 10^{-12}$ F/m (Prastiyanto 2016).

The dielectric constant for material is related to its capacity for storing energy in its electric field (Nelson 1981). The dielectric constant can be determined from capacitance measurements. The formula for capacitance is presented below. The relative permittivity can be determined from this equation via basic algebra.

$$C = \frac{\varepsilon'_r \varepsilon_0 A}{d} \quad (3)$$

where C is the capacitance in farads, A is the surface area of the two electrode plates in square meters, and d is the distance between the two electrode plates in meters. The dielectric constant for wood is roughly 4–5 (Skaar 1988), for paper and board 2–10, and for water roughly 78 at 25 °C (Baxter 1997). Polyethylene has a dielectric constant of about 2 (Baxter 1997, appendix II), and PLA used in bio-based coatings has a dielectric constant between 2.1 and 2.9, depending on the used frequency (Dichtl et al. 2017). From this, it is justified to claim that polymer coating should not have a significant effect on moisture content measured from the dielectric constant. This hypothesis is examined more in the experimental part of this thesis.

Complex relative permittivity is a parameter often used when describing dielectric properties. Relative complex permittivity is described by the equation:

$$\varepsilon_r = \varepsilon'_r - j\varepsilon''_r \quad (4)$$

where ε_r is the relative complex permittivity, ε'_r is the dielectric constant from equation 2, j is the imaginary parts operator and ε''_r is the loss factor. The loss factor describes the fraction of the signal lost due to dissipation of the energy in the dielectric field (Thierauf 2010).

While most dielectric moisture meters determine the water content from the dielectric constant, some meters which measure the combined effect of the dielectric constant and the loss factor exist (Skaar 1988).

3.3 Microwave-based measurement

Another common method to measure the moisture content is using microwave loss or absorption. The method itself is somewhat similar to capacitive measurement, except it involves the use of higher frequencies between 300 MHz and 300 GHz. Low-power electromagnetic waves are introduced to the sample and measured again when exiting the sample from the other side. The reduction in the wave power, often referred to as attenuation, is measured in decibels, which is then calculated by the device to provide moisture

percentage. Microwave loss or absorption in the sample rises logarithmically with the moisture content. (Anderson 1992).

Due to the high frequency, the effects of ion conductivity from coating or filling agents are negligible. The microwave loss method ignores the dielectric effect of loadings which influence the AC capacitance measurements. Microwave measurements are also not affected by the color or surface reflection of the sample. (Anderson 1989).

There are several types of methods to use microwaves in moisture measurements: Free space method, reflection method, and resonance method among others. Many industrial suppliers utilize the resonance method with their modern sensors (ABB 2023; Valmet 2023). The moisture content is measured from the resonant frequency of a resonator and its quality factor. The relative permittivity and thus the moisture content can be then calculated from the measured frequencies (Anderson 1992).

Despite the ability to measure deeper layers of the sample, microwave-based measurements are often implemented as online configurations by modern suppliers. (ABB 2023; Valmet 2023).

3.4 Infrared-based moisture measurement

At around 300 GHz frequency, where microwaves end, the infrared spectrum is considered to begin. The IR spectrum is divided into parts which are named based on the wavelength ranges. Short-wave infrared, or near-infrared (NIR) region is considered the wavelength span of 0.7–2.5 μm, mid-IR (MIR) is 2.5–20 μm and the far-IR (FIR) is 20–500 μm. (Derrick et al. 2000). IR spectroscopy is a versatile method that can be used to measure various properties, such as chemical composition, density, viscosity, particle size, or the well-known temperature (Pasquini 2003). The working principle is similar to that of microwave techniques: high frequency, short-wave radiation is directed to the material, and the amount of radiation absorbed is measured. Water molecules absorb IR radiation which causes the bonds in the molecule to bend and stretch causing vibration.

NIR is the main method used to measure moisture in different materials. Since absorption occurs on several wavelengths, the NIR measurement utilizes several wavelengths in the spectrum. (Derrick et al. 2000). IR absorbance linearly increases with increasing moisture

content, whereas with microwaves this increase is logarithmical. (Anderson 1992). In practice, an IR spectrum is measured and a certain spot of the spectra, depending on the measured property, is examined. Water molecules vibrate at the wavelength of 1.9 µm, which is utilized to determine moisture. The value of intensity at this wavelength is then compared to the intensity at one or more definite wavelengths, where the measured property does not absorb radiation, but the other background material properties remain constant as in the first one. This second wavelength acts as a reference point where the water content is known and is usually zero. Moisture content is then calculated from the ratio of the intensities at the measured point and the reference point. (Pasquini 2003).

IR techniques are most often utilized in online measurements, rather than as manual off-line measurements. One reason for this is that IR measurement is not able to measure deep beneath the surface of the material. For NIR the depth of penetration generally varies from 0.5 to 2 millimeters and is proportional to wavelength (Padalkar & Pleshko 2015; Derrick et al. 2000). Penetration depth is usually not an issue in online measurements, where only one thin layer of the moving web is measured. However, when measuring polymer-coated boards, and especially colored non-transparent materials, the penetration is greatly reduced.

Since online measurements are performed on the moving web, it is impossible to perform standardized measurements (Maijanen 2021). Effects of the environment, such as temperature, humidity, sensor fouling, changes in the surface material, or dust contamination affect the online measurement. Results are also not directly comparable with measurements done in the laboratory because of these environmental factors.

Online meters are either transmissive or reflective. In transmissive measurement, the detector is on the opposite side of the board than the light source, and in reflective measurement the detector is on the same side but slightly differently angled. Transmissive measurement is suitable for paper and thin uncoated board, but not for the polymer-coated board as the coating interferes with the IR waves. The effect of the coating is lesser for IR reflection measurement but still significant. (Siisiäinen 2023). An example of an IR transmission moisture measurement system manufactured by ABB is shown below in Figure 7.

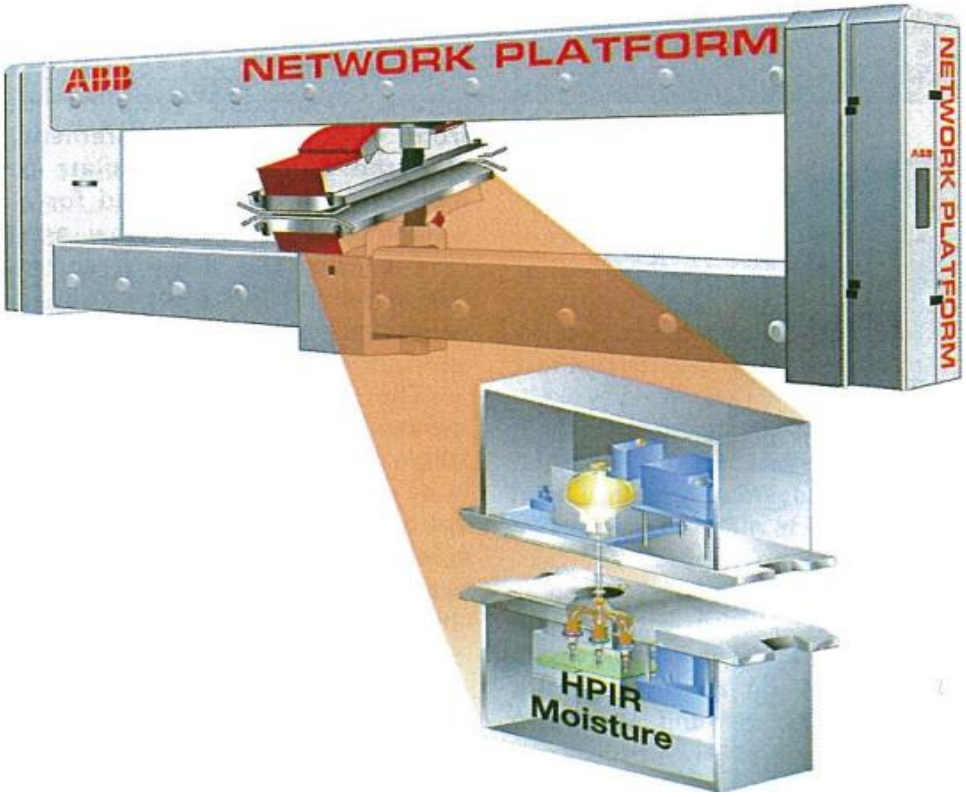

Figure 7. Online IR transmission moisture measurement system by ABB (paperAsia 2010).

The system consists of the frame and the sensing head. The material web to be measured is directed through the gap between the two sensor heads. The sensors move along the frame and constantly measure the moisture content, which allows a moisture profile to be constructed from the data. The configuration in the figure utilizes transmitting measurement, where the amount of IR waves absorbed is detected and recorded on the bottom sensor head.

When the moisture content of a finished product reel must be determined, IR analysis would require separating a single layer for analysis. There are a few off-line IR moisture meters, manufactured for example by Kett US in the 2010s. Some handheld models exist (Figure 8) but most of the meters are stationary, meaning a sample must be separated from the board web to analyze it (Kett 2011).

Figure 8. Universal Kett KJT-130 moisture meter for multiple different materials (Kett 2011).

These IR absorbance meters also have a smaller measurement area compared to other methods, meaning more measurements have to be done in order to cover the same area. To determine the moisture profile deeper inside the reel, more layers should be removed and analyzed individually, meaning more manual labor and time consumption. There is also great potential for error when manually taking samples from the product reel due to sample conditioning to the surrounding environment. These same issues also occur with other meters analyzing only a single surface layer, such as the Moistrex MX8000 microwave meter.

3.5 Method comparison

Below is a comparison chart summarizing the advantages and disadvantages of the moisture measurement methods studied in this literature part.

Table I. Comparison chart of the moisture measurement methods described in this work.

Method	Advantages	Disadvantages
Oven-drying	+Most accurate +Standardized measurement	-Long time consumption -Destructive -Prone to errors when handling samples
Capacitive	+Fast measurement +Non-contact measurement +Non-destructive +Ability to measure deeper beneath the surface layer	-Prone to the interference of electrical conductivity -The least accurate of the methods studied by Forsén & Tarvainen (2000) -No known online measurement configurations for paper and board
Microwave	+Fast measurement +Non-destructive +Manual and online configurations exist	-Online configurations generally need laborious calibration -Even though non-destructive, manual measurement requires taking separate samples
Infrared	+Fast measurement +Non-destructive +Possible to measure several properties with a single sensor by inspecting different wavelengths. +Penetrative measurement is possible for uncoated paperboard.	-Online configurations generally need laborious calibration -Poor penetration depth: < 2 millimeters with NIR, less for MIR and FIR -Polymer coating distorts the measurement results -Only less accurate reflective measurement is possible for coated material

Not one of the methods described can be declared superior compared to the others. Each method has its advantages, but also its disadvantages in terms of performance, suitability for specific products, and time efficiency. In the end, the accuracy and performance depend on the measurable material and the environmental conditions (Forsén & Tarvainen 2000). Proper custom calibration would most likely greatly improve the absolute accuracy to a sufficient level for each of the methods described above (Davis et al. 2017; Silveira et al. 2021). This would mean that the final decision of which method to pick most likely boils down to more practical factors such as availability, need for service, equipment size, cost, and preference.

4 Measurement error and uncertainty

Willink (2012) defines measurement so that there is a unique unknown true value that is the ideal result, and the measurement act itself provides an estimation of this unknown value. Measurement error then is often perceived as the deviation between the measured estimated value and the ideal true value (Viswanathan 2005; Yi 2017; Willink 2012; Kimothi 2002).

In their book, Kimothi (2002, p.97) quotes The International Vocabulary of Basic and General Terms in Metrology (VIM) to define measurement uncertainty as follows:

"*A parameter associated with the result of a measurement that characterizes the dispersion of values that could reasonably be attributed to the measurand*."

Measurement uncertainty, or as the quote states "the dispersion of values" should not be mistaken for purely measurement error. Kimothi (2002) explains the relationship between measurement error and uncertainty as the cause and the effect. Measurement uncertainty can be considered as the interval between which the result is at a certain confidence level or probability. 95 % is the confidence level used most often in chemical and physical metrology. Willink (2012) suggests the view that uncertainty is an indicator of the potential size of the measurement error. This fits together with the idea by Kimothi (2002), where the measurement error is unknown and unmeasurable, but it is utilized with statistical methods through factors like deviation, which are used to report the actual known uncertainty.

Standard deviation is a term used to describe the variation of data when it is normally distributed. Standard deviation represents how the measured data differs from the mean value (Lee et al. 2015). The formula for calculating standard deviation is:

$$SD = \sqrt{\frac{\Sigma_{i=1}^{n} (\bar{x} - x_i)^2}{n - 1}} \qquad 5$$

where *SD* is the standard deviation, *n* is the number of measurements, $\bar{x}$ is the mean value of the measurements and x_i is the measured value for measurement *i*. Standard deviation is usually automatically calculated by a statistical software. Standard deviation should be utilized together with the measurement accuracy for best results.

In their book, Kimothi (2002) lists factors that influence the measurement process and cause uncertainty in metrology. The factors are used equipment, operator, time interval, place, and environment. They also mention chemicals and reagents, but those are excluded, since the purity level and characteristics of chemicals are not relevant in this work. Levlin & Söderhjelm (1999) also list similar factors in their book, with the addition of material and sampling. These mentioned factors are briefly studied in this work as well.

Scientifically valued measurement results can be considered incomplete without a proper explanation of its margin of error or uncertainty. Without proper expression of uncertainty, satisfactory comparison with earlier or future measurements is not possible. Thus, the results may not be considered reliable either. (Kimothi 2002).

4.1 Types of error

In their book, Willink (2012) discusses several sources of error in measurement. It is a common conception that there are two sorts of error: systematic error and random error. The systematic error remains the same between replicate measurements or changes in a predictable way. Random error on the other hand does not depend on the measured value. The relationship between the estimated or measured value and the true value can be described with the equation:

$$x = \theta + e_{sys} + e_{ran} \qquad 6$$

Where x is the measured value, θ is the true value, e_{sys} is the systematic error and e_{ran} is the random error. This equation is usually used when the systematic components remain constant, rather than varying predictably. An example of a varying or *moving error* would be a temperature-dependent error when the temperature is uncontrolled and not measured. This kind of error can often be neutralized with relative ease. The random error is usually characterized by the frequency distribution function. The Gaussian frequency distribution function is used to characterize the random error in physical and chemical measurements by two parameters: the mean and the standard deviation. The standard deviation is often used to estimate the random error as it is used to measure the variability of data (Kimothi 2002).

Statistical error is related to the fact that it is only possible to take a finite number of samples of a lot. There are multiple types of statistical error in statistical mathematics, but the most

relevant kind of statistical error in physics or chemistry is inhomogeneity error This type of error is associated with the concern that a sample may not be representative of the whole lot. Statistical errors are considered random errors. (Willink 2012). Since the random error is of unrelated value and it can be positive or negative, the average value for random error is usually stipulated as zero when enough repetitive samples are taken (Willink 2012; Kimothi 2002).

Calibration error is another important factor to consider when measuring. Equipment usually must be calibrated in some way for it to provide realistic and accurate results. Mistakes or errors during the calibration procedure may lead to initial statistical errors, which later turn into constant unknown error through subsequent uses (Willink 2012). In a continuous production process, proper and frequent calibration of measuring equipment is extremely important. The faster the possible calibration errors are discovered and corrected, the less of the product is categorized wrong and even delivered to the customer. A redeeming quality of calibration error is that the error inside the calibration limits is usually constant between measurements. This means that the error can be corrected relatively easily by utilizing a correction factor, as long as the magnitude of the error is known (Willink 2012).

Discretization error, also known as the digitization error, is caused by a finite unit of resolution in measuring and recording equipment. The size of the error depends on how the device rounds data or numbers. There are usually two possibilities: either the device rounds to the nearest unit or it simply truncates decimals (Willink 2012). This error often is dependent on the specifications of the measuring device. Many handheld measuring devices used in industries are not particularly accurate. Forsén & Tarvainen (2000) reported that in their study for solid wood moisture measurement, slightly less than 50 % of the moisture measurements with capacitance-based moisture meters were within ± 1.0 % of the reading of oven-dried readings. However, meters have greatly improved since the 1990s, and the effect of discretization error is said to be often negligible (Willink 2012).

The relative error is the ratio of the measured value and the absolute error, which is the difference between the true and measured value (Wilson et al. 2008). Relative error is a handy tool to review the size of the error compared to the measured value itself. Relative error can be used regardless of units or scales, which makes it a very useful tool for comparing different measuring instruments or methods (Glen 2023). It also demonstrates the magnitude of an error very well. For example, a 1% difference in moisture content might not

seem large at first, but when comparing it to an example goal moisture content of 8 %, the relative error is 12.5 %, which may be considered significant, depending on the measurement criteria.

There are some actions one can take to attempt to minimize the error in a measurement. A few of the most important of these are proper calibration of measurement equipment, understanding and minimizing the effect of environmental factors, and lastly proper sampling and measuring procedure (Willink 2012).

4.2 Environmental factors

There are a few moments when the environment may affect the moisture percentage result in this work. First of these is the time period between the moment when the coated product reel is finished and the moment when it is measured. Despite cooling down during the post-treatment and online quality measurements, the temperature of the board coated with hot polymer is slightly higher than the ambient temperature.

Temperature and time are the two most important factors when drying various materials (Caparanga et al. 2017; Chua et al. 2002). Thus, the rate of drying is the fastest right after finishing the reel. This leads to the conclusion that it is important to measure the moisture and, if necessary, to take samples for oven-drying, as soon as possible to avoid excessive drying. Air velocity is known to speed up drying, but the effect was determined negligible in drying of arrowroot starch at air velocities up to 0.6 m/s (Caparanga et al. 2017). The air velocity in the production hall may locally be considerably higher than that, so the effect of air velocity should not be neglected completely. However, only the outer layers of the reels suffer from drying as determined by Leskelä (2008), since the reels are laid open to ambient air only for short periods.

Another case when the environment may impact the results is in the laboratory when determining the reference moisture content via oven-drying. The ISO standard 187 (ISO 2022) sets the standard atmosphere conditions for the testing of pulp, paper, and board, which is the atmosphere in testing laboratories. This standard atmosphere for non-tropical countries must have a temperature of 23 ± 1 °C and a relative humidity of 50 ± 2 %. Since oven-drying is usually done in a laboratory environment, the atmosphere may potentially

affect the samples and the results, although the polymer coating greatly enhances the water vapor barrier (Kuusipalo 2008). When conditioning to a certain relative humidity, board with two-sided PE-coating must be conditioned for several days, up to a week to ensure equilibrium.

Environmental errors should be minimized as well as possible since the magnitude of them is difficult to determine. Although Willink (2012) explains that unmeasured fluctuation of environmental factors leads to a spread in the results, which will act as a simple source of pure error.

4.3 Measurement system analysis and Gage R&R

Measurement system analysis (MSA) should always be performed on measurement systems to ensure that their variability is within acceptable limits to produce satisfying results. While there are many studies in MSA, Gage repeatability and reproducibility (R&R), is the one used most. By Gage R&R it is possible to determine whether the error in a measurement is caused by the operator, variability in the product, or the meter (gage) itself. Different sources for process variation and the ones Gage R&R can be used to inspect are presented below in Figure 9. In the study, the variability of the gage or the operator is determined by the term repeatability. Repeatability describes the variability in the results when a single operator repeats the same measurement several times. Reproducibility is another factor of the R&R study, and it represents the variability in the results caused by different operators, measurement setups, or measuring times. (Barrentine 2003).

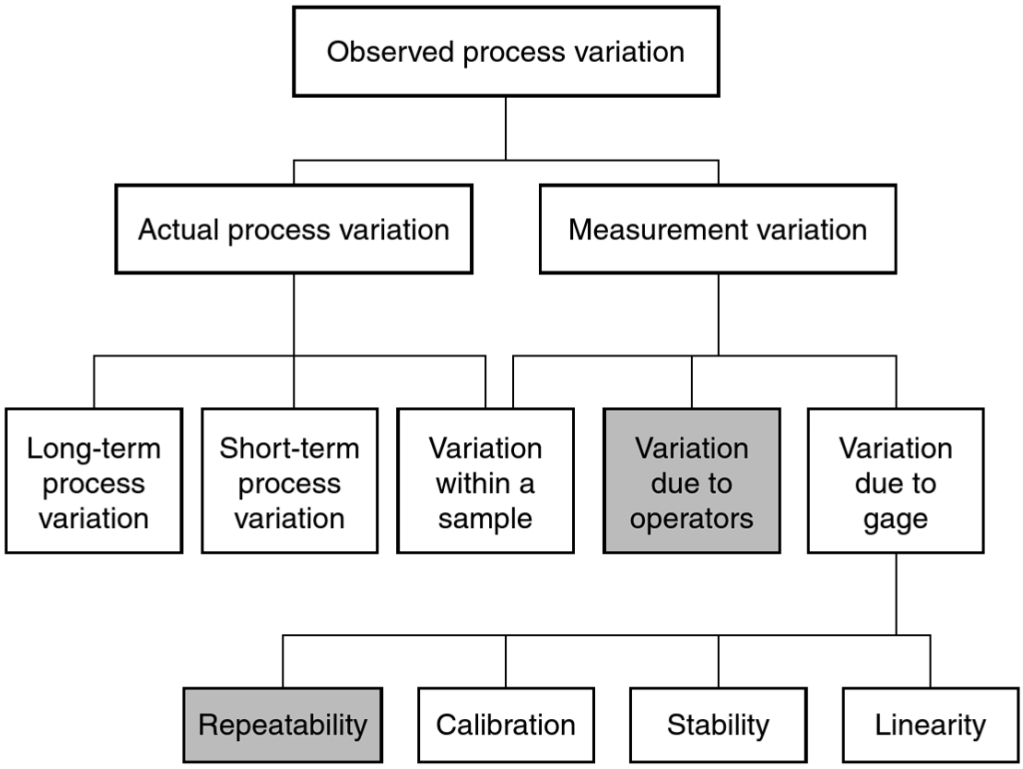

Figure 9. Factors contributing to variation in a process and the ones inspected by Gage R&R (dark boxes). Adapted from (Barrentine 2003)

Gage R&R study is often analyzed by using the analysis of variance (ANOVA) approach. ANOVA is widely used and provides results of the data interaction between the operator and part, which cannot be achieved with other methods. (Soares et al. 2022). The ANOVA method for Gage R&R can only be applied to univariate data with a single variable at the time (Peruchi et al. 2014). This one-way ANOVA compares the means of a minimum of two groups, for one dependent variable. Sample size is one of the most important factors when determining the confidence interval, and generally equal sample size is recommended for all the sample groups (Lai & Kelley 2012).

4.4 T-tests

Another, and probably the most popular method of statistical testing is the t-test (MacFarland & Yates 2021). The t-test compares the mean value of a sample to a target value, which is usually a theoretical deduction. The null hypothesis is that there is no statistically significant difference in the data between a group and its target value. After that,

the hypothesis is tested by determining the t-value. The t-value for a one-sample t-test is calculated by using the following formula:

$$t = \frac{\bar{X} - \mu}{\frac{SD}{\sqrt{n}}} \quad (7)$$

where $\bar{X}$ represents the mean value of the sample dataset, μ is the expected value or the population mean, *SD* is the sample standard deviation and *n* is the number of cases or samples. The resulting t-value is then compared to a critical t-value in a t-distribution table at a desired level of significance, which is usually 95 %. The null hypothesis can be rejected if the calculated value is less than the significance alpha-value, which is 0.05 for 95 % confidence. If the calculated value is greater than the alpha-value, there is not enough evidence to reject the null hypothesis. (MacFarland & Yates 2021). However, this does not necessarily mean that the null hypothesis is true, which is why the situation should always be evaluated carefully.

In two-sample t-tests, the mean values of two different groups are compared. Normal distribution does not necessarily have to be assumed if the number of samples is high enough, around 25. In the traditional two-sample t-test, standard deviations and variances are considered to be equal between the groups. However, with equal sample sizes, the effect of differing variances is not necessarily critical (Wilson et al. 2008). If the variances are considered different, the following formula is used to determine the t-value in a two-sample test (Pandis 2015; Wilson et al. 2008).

$$t = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{{SD_1}^2}{n_1} + \frac{{SD_2}^2}{n_2}}} \quad (8)$$

If comparing more than two groups, t-test cannot be used, and for example, ANOVA has to be used instead (Wilson et al. 2008).

4.5 Data normality and Anderson-Darling test

Normal or the Gaussian distribution is the most commonly established model used to characterize the variation of data. It is generally presented as a probability density function (PDF), where the horizontal axis represents the measurement data, and the vertical axis represents the probability or the frequency of that certain measurement. The highest point in the PDF curve is the mean value of the dataset. (Brereton 2014). In a normal distribution, the mean value paired with two times of standard deviation covers roughly 95 % of the distribution. This $\bar{X}$ ± 2SD is the most often used confidence interval, but other intervals such as $\bar{X}$ ± 3SD for about 99.7 % confidence may also be used. (Limpert & Stahel 2011). Below in Figure 10 is an example of a normally distributed data PDF curve.

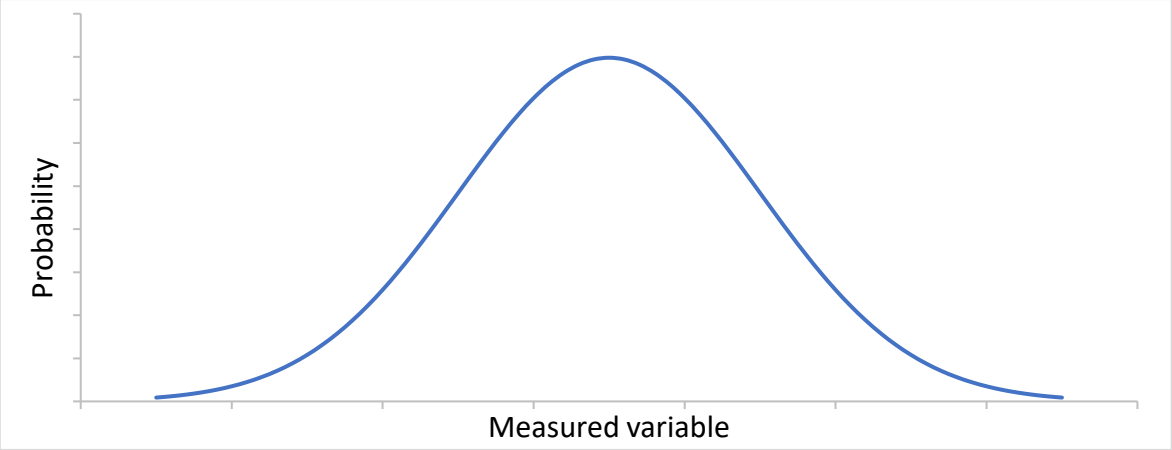

Figure 10. Bell-curve of normally distributed data

However, this symmetric range of variation of the standard deviation can be misleading and even false in some cases. For example, if the standard deviation is greater than the mean value, the lower end of the data could extend below zero, which in many cases is impossible. This issue only occurs when the original data does not adequately fit normal distribution, for example when the dataset tends to have a longer tail on the right side. For this kind of data, log-normal distribution theory is often better. In log-normal distribution, the variable's logarithm is normally distributed. (Limpert & Stahel 2011)

Anderson-Darling test is used to examine how well an empirical dataset from a certain population corresponds to a normal distribution (Dodge 2008). It is a modified version of the Kolmogorov-Smirnov test, but it gives more weight to the tails of the distribution. The

Anderson-Darling measures the deviation between the observed data and the theoretical distribution being tested by comparing critical values for a specific distribution. Hypotheses for the Anderson-Darling test are: H_0: The data follows the specific distribution, H_1: The data does not follow the specific distribution. (NIST/SEMATECH 2012b).

The Anderson-Darling test statistic is defined as:

$$A^2 = -N - S \tag{9}$$

Where N is the number of sample species and S is defined as:

$$S = \sum_{i=1}^{N} \frac{(2i-1)}{N} \left[\ln F(Y_i) + \ln\left(1 - F(Y_{N+1-i})\right)\right] \tag{10}$$

Where F is the cumulative distribution function for the specific defined distribution and Y_i is the ordered data.

The Andersson-Darling test statistic A^2 is compared to the critical value of the specified distribution. If A^2 is greater than the critical value, the null hypothesis is rejected, and thus the data does not follow the specific distribution. (NIST/SEMATECH 2012b).

Experimental part

In the experimental part, the suitability of a modern paper and board moisture meter, Humimeter PM5, is examined on extrusion coating lines producing different types of board products.

The first main goal of the experimental part is to determine the measurement uncertainty and reliability for Humimeter PM5 by using oven-drying as a comparable standard. Different product specifications are studied here, including the base board grammage per square centimeter (gsm) and the amount of polymer coating. The second goal is to determine the standard error and uncertainty for all the product types studied. The third goal is to develop a reliable but efficient and quick measurement method, sequence, and routine for everyday life in the coating line.

Throughout the whole experimental section, different sources of error are tracked and their effect on the result is evaluated. The errors of other measuring methods are also estimated and compared to Humimeter PM5.

5 Moisture meters

Two different measurement systems are examined in this experimental part. These are the older microwave based Moistrex MX8000 and the newer Humimeter PM5. Oven-drying is used as a standard reference method for comparing results. Oven-dried moisture content is also considered the “true” value during the uncertainty calculation.

5.1 Moistrex MX8000

Moistrex -family is a series of moisture analyzers that utilize microwaves at a frequency of about 10 GHz to determine the moisture content for paper and board (NDC Infrared Engineering Ltd 2005). Moistrex MX8000 measures a certain area of the paper at a known temperature and thus is capable of accurately determining the moisture content with little to no error. The measuring electromagnetic wave in the device is entirely enclosed, which prevents leaks or loss from affecting the results. Earlier versions of Moistrex MX8000 MX2000 meters are from the 1980s (Anderson 1989), and the more recent MX8000 (Figure 11) model dates to the mid-2010s (NDC Infrared Engineering Inc. 2018).

A downside of the analyzer is that it requires separating a sample from the finished product, which is then analyzed. There is always an error present when taking, transporting, and handling samples. While the method is directly not destructive, the sample sheets removed from product reels are considered waste. Online analyzers and measurements performed on the surface of the paper reel do not have this issue.

There are several measurement channels for Moistrex MX8000, roughly one for each product grade. These channels are created by comparing the result of one of the default channels to the oven-dried result, after which the Moistrex MX8000 channel is finely adjusted manually to give the same result as oven-drying. The accuracy is monitored yearly, and the device is calibrated as needed. The moisture percentage result from Moistrex MX8000 and oven-drying should not differ more than 0.3 % on average, as per the quality standards in the facility.

Figure 11. Moistrex MX8000 tabletop moisture meter (NDC Infrared Engineering Inc. 2018)

In the Moistrex MX8000, product types are by default separated by their type, whether they are made from mechanical or chemical pulp, as well as grammage, and ash content. These predefined paper types can then be trimmed to fit all product types as well as possible. This is done by comparing the results of a Moistrex MX8000 measurement to the results of oven-drying and manually changing the result to match, which simultaneously teaches the meter for future measurements of the same product. The polymer coating is not taken into account in the Moistrex MX8000, and only the paper grammage is fed into the system. (NDC Infrared Engineering Ltd 2005)

5.2 Humimeter PM5

The Humimeter PM5 is a capacitance-type dielectric moisture meter. A capacitance-type meter consists of two electrodes, between which the electric high-frequency field in the material forms. The material with the higher water content is reflected in a higher capacity. The evaluation electronic system calculates and converts this measured capacity into water

content as a weight percentage. The measurement device Humimeter PM5 is shown in Figure 12 and Figure 13 below. (Schaller Messtechnik GmbH 2019)

Figure 12. Picture of the Humimeter PM5 paper moisture meter. 1. Selection button, 2. Handle, 3. Display, 4. USB port, 5. Reset button, 6. LED battery indicator (Schaller Messtechnik GmbH 2022)

Figure 13. The rear side of the Humimeter PM5. 1. Infrared temperature sensor, 2. Sensor bars (Schaller Messtechnik GmbH 2022).

The meter's sensor bars shown in Figure 13 are lightly but firmly pressed against the side long side of the reel. The display shows the measured moisture value, which is also recorded

in the storage of the device. The meter can be moved along the reel to get descriptive results from the entire length of the reel. Measurements can also be taken from moving reels during winding.

According to the manufacturer, the Humimeter PM5 is capable of measuring up to 50 millimeters deep into the sample due to the dielectric nature of the measurement. The device has automatic temperature measurement and compensation, which eliminates the effect of changing temperature on the measured result. (Schaller Messtechnik GmbH 2022).

5.2.1 Calibration and measurement setting

Humimeter PM5 adjusts itself automatically to the ambient environment when the power is turned on. This calibration should always be done in the same space as the measurement is to be done in to ensure proper calibration.

Selecting the proper paper grade is based on the material density in Humimeter PM5. Different product grades are by default described by their densities at the increments of 50 kg/m^3 in the range of 300 – 1100 kg/m^3, and from there in increments of 100 kg/m^3 up to 1600 kg/m^3. Density is automatically calculated for the product by using the grammage per square meter and the paperboard thickness:

$$density\ \left[\frac{kg}{m^3}\right] = \frac{grammage\ \left[\frac{g}{m^2}\right] * 10^3}{board\ thickness\ [\mu m] * 10^6} \qquad 11$$

The values for grammage and thickness are considered constant from the product property table. However, these may in reality vary by small amounts and thus cause the actual density to change. For example, a board with a grammage of 214 g/m^2 and thickness of 334 µm has a density of roughly 641 kg/m^3, which would be rounded up to 650 kg/m^3 for the meter. If the actual grammage was for example 4 g/m^2 lower and the thickness 3 µm higher, the density would be 623 kg/m^3 and then could be rounded down to 600 kg/m^3. According to preliminary testing at the coating line, a change of 50 kg/m^3 in the meter settings leads to a 0.6 – 1.0 % difference in the measured moisture content. Such error is significant, so it is especially important to use the most suitable settings for the product grade and to determine what is the standard principle for rounding the numbers up or down.

6 Methods

Paperboard products with four different polymer coatings were examined in the experimental part. For confidentiality, these product grades were named in this work as follows: Grade 1, Grade 2, Grade 3, and Grade 4. Each product grade covers a range of different paperboard grammages and different amounts of polymer coating.

Moisture readings and samples were taken from reels whenever possible. The production at the coating line changes in cycles and the moisture data was collected throughout many cycles. This means that some changes in environmental factors such as ambient air temperature and humidity are present, as they are impossible to negate completely. However, this should not severely affect this work, since the goal is to study and compare the accuracy of the different methods and not the actual moisture content percentage.

6.1 Humimeter PM5 density channel

Since the Humimeter PM5 settings are adjusted based on the density of the product, it was necessary to determine the right density channel for the product board. Initial tests were carried out with the same density setting on the device as the density of coated board is reported to be in the production planning system. The measurements with these settings, however, led to unrealistic results with moisture content being more than 1 % higher than oven-drying, or the Moistrex MX8000 meter determined.

The higher the density setting in Humimeter, the lower the measured moisture percentage is. A more fitting density setting was pinpointed by testing several different settings in the range of 0-250 kg/m^3 higher than the initially proposed setting. The best density channel setting was determined and later used for each product type individually.

Results from Humimeter PM5 were compared to moisture content results from oven drying. The number of tested density channels varied from three to five for each product grade. From the first few results, the channels providing the least accurate results were excluded and future tests were carried out with only three or two most accurate settings. The number of

tests or samples varied depending on product grade, production cycle, and the results of the first measurements.

6.2 Sampling and measurement data

Measurement error for Humimeter PM5 was determined by measuring the moisture content from specific product reels with Humimeter PM5 and taking samples for oven drying. The samples were delivered to the laboratory, where the laboratory technicians and assistants performed the oven-drying analysis as per ISO standard 287. The Humimeter PM5 measurements were taken by the machine line operator personnel to simulate the real-life process conditions and their effects on accuracy. Measurements and samples were taken around the clock by the workers on every shift.

A large amount of moisture data was treated in a spreadsheet. The largest and most apparent outliers were excluded from each data set, but no more than 20 % of the data was left out. A data point was considered an outlier if either the oven-dried moisture content or the Humimeter PM5 reading at a specific setting differed greatly from the rest of the data with no evident explanation, such as different machine reel diameters or malfunctions during the process. Data outliers were also discarded if Individual-Moving Range (I-MR) charts implicated serious deviation from the average. Interpreting this chart is described later in chapter 9.1.1

It was assumed that every measurement was done on the most optimal density setting for that current product. This means that if a reel was measured with multiple Humimeter PM5 density settings, the moisture result that resulted in the least absolute error was used in the analysis.

6.3 Correlation between absolute error and certain product properties

The effect of different product properties on the absolute measurement error was studied. These properties were paperboard density, the amount of polymer coating, and the product total grammage. The Humimeter PM5 moisture data was used for this study. The readings were compared to results from oven-drying, and the absolute error was the difference

between the two measurements. Minitab statistical software was used in the study to draw the graphs.

6.4 Analysis route for the two-sample t-test

The measurement uncertainty was studied via the t-test described earlier in the literature part. To use the two-sample t-test, the system and data normality variances had to be determined.

First, the measurement data was studied via the Individual-Moving Average (I-MR) chart. The moving average chart is used to monitor the variation of a process with continuous data. Since the moisture data collected in this experimental section was not exactly continuous, but rather randomly collected whenever possible from the machine reels, the MR chart was not especially informative. The MR chart is more suitable for inspecting data when the difference between consecutive measurements is important.

The individual values -chart plots individual data observations and it helps to visually determine points when the process mean, or in this case, the measurement is out of control. The I-chart was quite useful to detect outliers of the dataset, especially on the lower end of the moisture range.

Next, the data normality was inspected with the Normality Tool in Minitab. Anderson-Darling normality test was selected. The P-value from the Anderson-Darling normality test was then compared to the confidence interval alpha-value of 0.05 to determine whether the data is normally distributed or not. If the P-value is higher than the alpha-value, there is no evidence that the data is not normally distributed. Thus, it was decided to treat the data as normally distributed.

For the two-sample t-test, it is important to determine if the variances can be considered equal since it impacts the results and the reliability of the t-test. This was done with the 2 Variance tool in Minitab. The data from Humimeter PM5 at a specific setting was Sample 1 and the equivalent moisture data from oven drying was Sample 2. The confidence level used was 95 %. The null hypothesis for the 2-variance test was that the variances are equal: $H_0: \sigma_1 / \sigma_2 = 1$, and the alternative hypothesis was that the variances are not equal: $H_1: \sigma_1 / \sigma_2 \neq 1$.

The Minitab statistical software utilizes two different methods to determine the statistical difference between the variances of two samples or populations. Levene's test method dates back to the 1960s, and it utilizes the mean values of the subgroups to determine the Levene's test statistic, which is compared to the alpha-value. Levene's method is not especially sensitive to data's deviation from normality. (NIST/SEMATECH 2012a). Brown and Forsythe (1974) modified Levene's test and extended it to also use either the median or trimmed mean values in addition to the mean. Utilizing median or trimmed mean values in the test improves the accuracy especially with lower sample sizes with high deviation between the data points. In trimmed mean test method, 10 % of the largest and smallest values are deleted and the mean value of the result is used in the Levene's test. This method is the most suitable for data where the distribution is long tailed. (Brown & Forsythe 1974). Minitab statistical software uses Levene's test based on Brown and Forsythe's modification in testing for equal variances.

Bonett's method is more suitable for normally distributed data with little or no tailing or skewing. It considers the range that the data is distributed in and is suitable for small sample sizes of less than 30. Bonett's test also utilizes a trimmed mean, which's proportion depends on the sample size. Bonett's test is based on the pooled kurtosis estimator for Layard's test statistic from 1973, which is only consistent when the variances of populations are equal. (Bonett 2006). Minitab uses a modified version of the Bonett's test, where a misstep leading to false confidence intervals caused by unequal populations is corrected. (Banga & Fox 2013).

If the data was determined to be normally distributed earlier, the P-value from Bonnett's test was compared to the confidence interval value of 0.05. If it was determined that the data is not normally distributed, Levene's test P-value was used instead. If the P-value is greater than the test's alpha-value, the two datasets were considered to have equal variances.

Finally, the 2-sample t-test was performed in Minitab. Depending on whether the variances are determined to be equal, the option is toggled on or off prior to the analysis. Data from Humimeter PM5 and oven drying were compared to each other at a 95 % confidence interval. The null hypothesis was that there is no statistically significant difference between the mean values of moisture measured by Humimeter PM5 and oven drying: $H_0: \mu_1 - \mu_2 = 0$. The

alternative hypothesis was that there is a statistically significant difference between the mean values: $H_1: \mu_1 - \mu_2 \neq 0$.

This procedure described above was performed on each of the four polymer coating types studied in this thesis.

6.5 Gage Repeatability and Reproducibility

The Gage Repeatability and Reproducibility MSA was performed on the Humimeter. Three operator personnel were selected to participate in the experiment. Three machine reels were measured right after they finished at the pope winder. All normal measurements were performed, and necessary samples were taken before the Humimeter PM5 Gage R&R experiment. The moisture was measured from three sections of the long side of the reel: front, middle, and back, which is better demonstrated below in Figure 14.

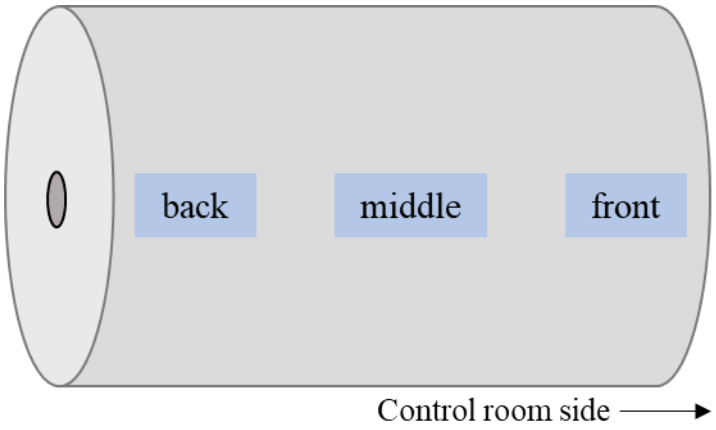

Figure 14. Visualization of the sampling locations on the long side of the reel

The average value of these measurements was used in the analysis. An operator measured all the reel sections once, and then the meter was passed to the next person. Two measurement rounds were done so that the operators did not perform the two measurements in a row. A total of three machine reels were measured.

Data for this study was gathered all during the same day, and all of the machine reels had similar properties in terms of board and polymer types, board grammage, and coating amounts. The coating machine's web speed and thus the capacity remained constant

throughout the whole test. The Gage R&R analysis was executed in Minitab statistical software with the Crossed Gage R&R tool. The selected method of analysis was ANOVA.

6.6 Comparison between Humimeter PM5 and Moistrex MX8000

It was necessary to compare the accuracy of the current Moistrex MX8000 meter and the newer Humimeter PM5 to be implemented. This was done by running the 2-variance and two sample t-tests on the Moistrex MX8000 as well in a way demonstrated in Chapter 6.4 The results from these tests were then compared to the results for the Humimeter. The comparison was only performed on the results of product Grades 1 and 2, as they were the most reliable and successful.

7 Selecting the optimal density setting in Humimeter

For Humimeter, the optimal density channel was determined as demonstrated in chapter 6.1. By using the automated paper and board testing system, the actual board density was calculated from measured board mass per square meter and thickness with equation 11. From a few different experiments, it was determined that the actual density did not vary for more than 10 kg/m^3 in either direction. From this quick review, it was decided that no further inspection is needed for the product's density, and the density value from the production planning system was used for the rest of the experiments.

The optimal Humimeter PM5 density channel was determined for each coating type individually. This proved difficult, since the product densities for one polymer type varied greatly throughout the range of, for example, 700 – 775 kg/m^3. Products that have a density of 705 kg/m^3 or 745 kg/m^3 are easily rounded up or down to the closest setting which also results in good measurements, but issues arise when the product density is somewhere in the middle, for example at 724 kg/m^3. In this case, it was not clear whether the value should be rounded up or down and which density setting should be used.

When the test results started to accumulate, it was discovered that the difference in results between two consecutive Humimeter PM5 density settings was on average about 0.75 – 0.80

%, depending on the product type and the setting. This meant that if the absolute error between the moisture reading from Humimeter PM5 and oven-drying differed more than 0.40 %, it would've been beneficial to use another density setting instead to achieve a lower absolute error. This is demonstrated below in Figure 15.

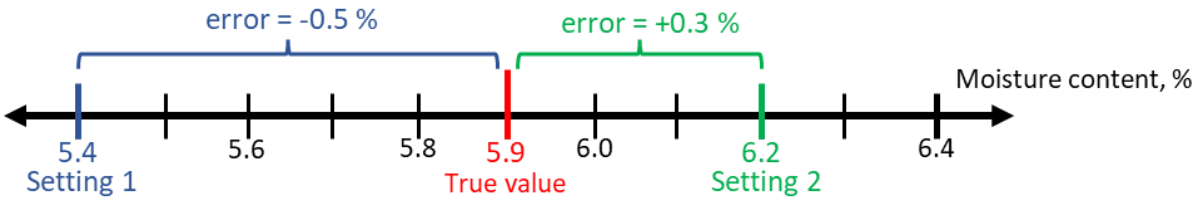

Figure 15. Illustration of the difference in the absolute error depending on the density channel used. Setting 1 (blue) uses a higher density value than setting 2 (green), leading to lower density reading, and in this case greater absolute error.

This led to the conclusion that when operating Humimeter PM5 at an ideal density setting, the error could not be more than 0.40 %. However, determining the right density setting for every single product type with unique grammage, thickness, and coating amount is a laborious task, which means that some rarely coated products may occasionally have to be measured with a non-ideal density setting.

8 Product properties' effect on absolute error

It was determined how three different product properties affect the error in the measurement. The properties were product density, amount of polymer coating, and product total grammage. This inspection was done on product Grades 1 and 2. Below is presented a Main effects plot demonstrating the error for product Grade 1 (Figure 16**)** and Grade 2 (Figure 17)**.**

Figure 16. Visual representation of how product density, amount of coating, and product grammage affect the absolute error of Grade 1 product. The mean value of the error is marked on a dashed line at +0.04 %.

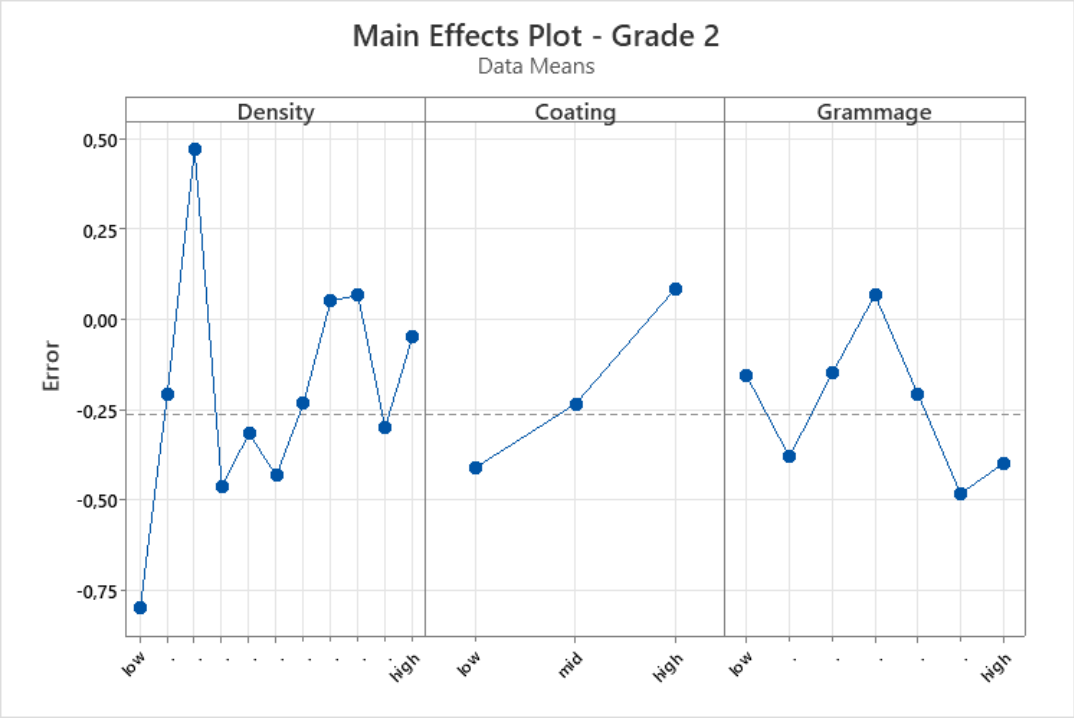

Figure 17. Visual representation of how product density, amount of coating, and product grammage affect the absolute error of Grade 2 product. The mean value of the error is marked on a dashed line at -0.26 %.

From the graphs, it was quite evident that there is no clear correlation between density or grammage and the magnitude of absolute error, as the variation in the error appears to be quite random with no evident trend. The amount of polymer coating showed a slight incline with increasing coating and error. However, this may be caused by several factors, so additional inspection would be needed to draw any conclusions on that part. One explanation for this phenomenon might be that the polymer interferes with the measurement, as it has different dielectric properties than the board. Increasing the amount of the polymer coating increases the polymer-to-board ratio, which again increases the error through the interference. In the literature part of this thesis, the polymers were theorized to not have a significant effect on the capacitance-based measurement as the dielectric constants are very low and overlap with the values of the board, but it seems like that, at least with this data, the theory does not apply.

9 Results from the T-test

The same procedure demonstrated in Chapter 6.4 was performed on each of the four product Grades. The results are demonstrated and discussed more specifically for Grade 1 and Grade 3 products, and for the rest, the charts and graphs are listed in Appendix I.

9.1 Grade 1

The number of Grade 1 measurements was 35, which is more than enough to achieve reliable and descriptive results. The variation of product specifications in this grade was not very high, and the ideal density setting for the Humimeter PM5 was found quickly. The Humimeter PM5 results for this grade were the best of the four.

9.1.1 I-MR Charts for Grade 1 products

I-MR Chart of the oven-dried moisture data for product Grade 1 is presented in Figure 18 below.

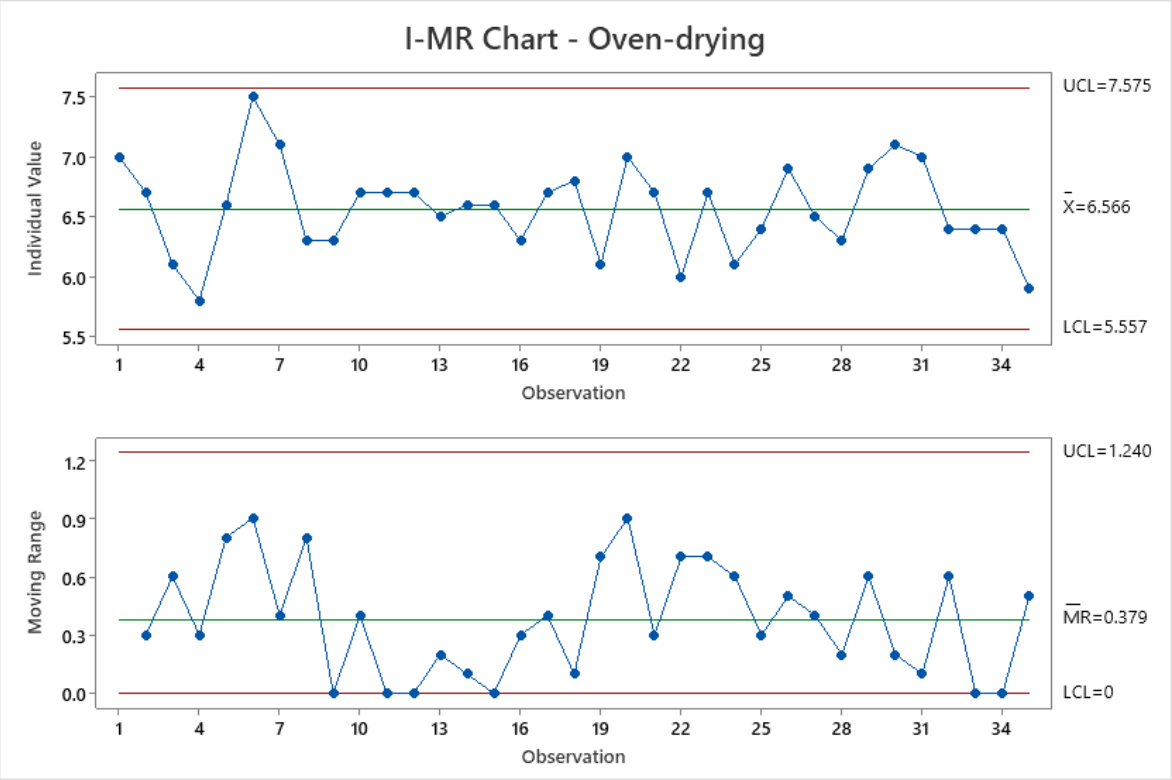

Figure 18. Product Grade 1 Oven-dried moisture data I-MR chart

The I-MR charts in Figure 18 shows that the data points are relatively well scattered around the mean value and that they are well within the upper and lower control limits. A single individual value is near the upper control limit (UCL). This is likely caused by an exceptionally moist machine reel on which the oven-drying measurement is done. The MR-Chart is of less importance since it compares and draws based on consecutive data points, which is irrelevant in this case since the measurements were not necessarily done on consecutive machine reels.

A similar I-MR chart for Humimeter PM5 moisture data is presented below in Figure 19.

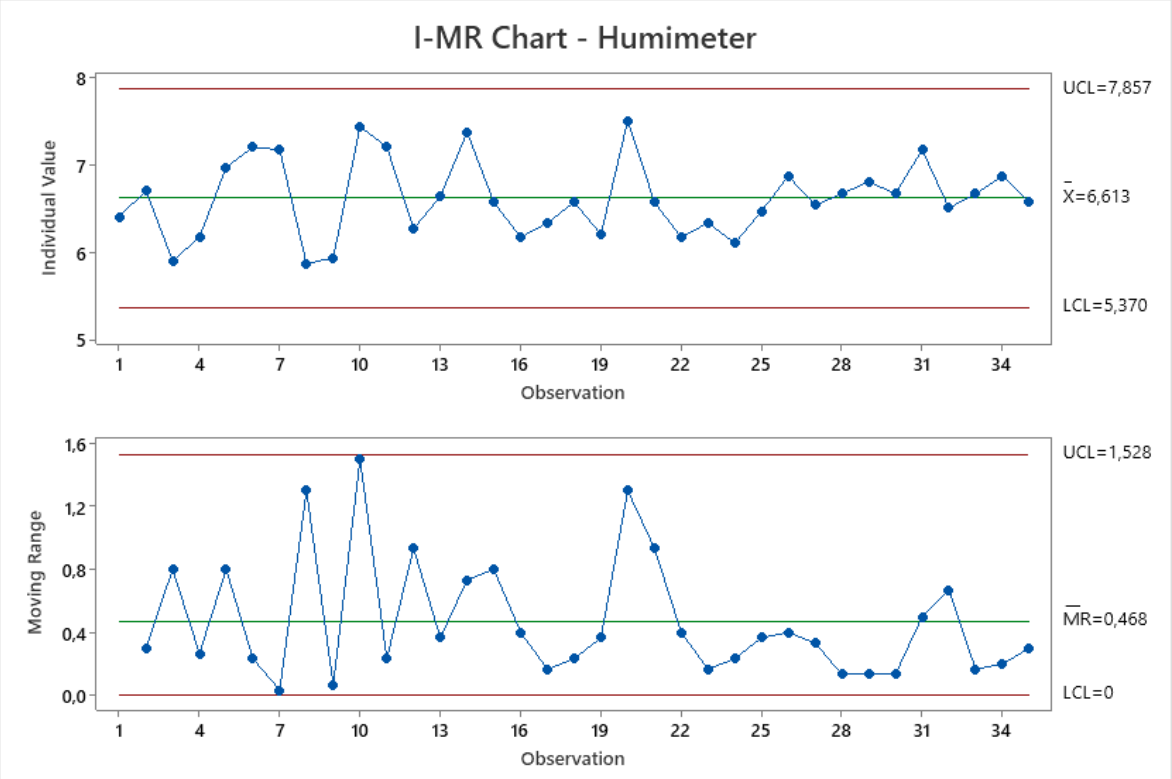

Figure 19. Product Grade 1 Humimeter PM5 moisture data I-MR chart

In the I-chart for the Humimeter PM5 moisture data in Figure 19, the individual values are well scattered around the mean value of about 6.61 %. Overall, the data is reasonable. The MR-chart is within the control limits and there is nothing alarming about the continuity of the data.

Judging by the two I-MR charts for oven-dried and Humimeter-measured data, a conclusion was made that the data is good within the control limits and there are no clear outliers, and thus it is good for the normality test.

9.1.2 Normality test results for Grade 1 products

For product Grade 1, the normality test was done as described in Chapter 6.4 For oven-dried samples, the Normal Probability plot from the Anderson-Darling normality test is presented below in Figure 20.

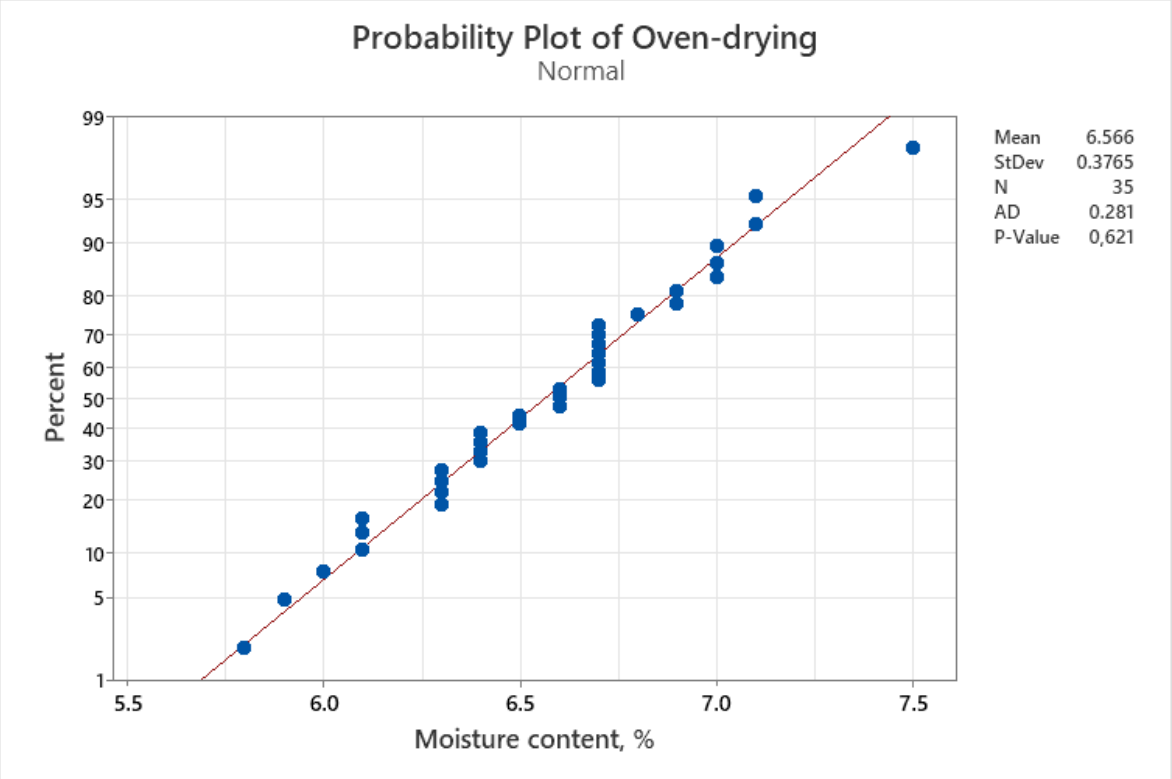

Figure 20. Anderson-Darling normality test for Product Grade 1 samples measured via traditional oven-drying. On the right-hand side is the resulting P-value of 0.621

The data from oven-drying is nicely distributed throughout the whole reference line with little outliers. Test results are listed on the right side of the chart. The mean value of all the oven-dried measurements was roughly 6.57 %, and the P-value from the test was 0.621. The confidence interval of 95 % was decided, so the alpha-value for this test is 0.05. Since the P-value of 0.621 is significantly higher than the alpha-value, there is no evidence that the measurement data is not normally distributed. Thus, the data is assumed to be normally distributed for the rest of this analysis.

The result of the probability plot from the normality test for Humimeter PM5 measurements is presented below in Figure 21.

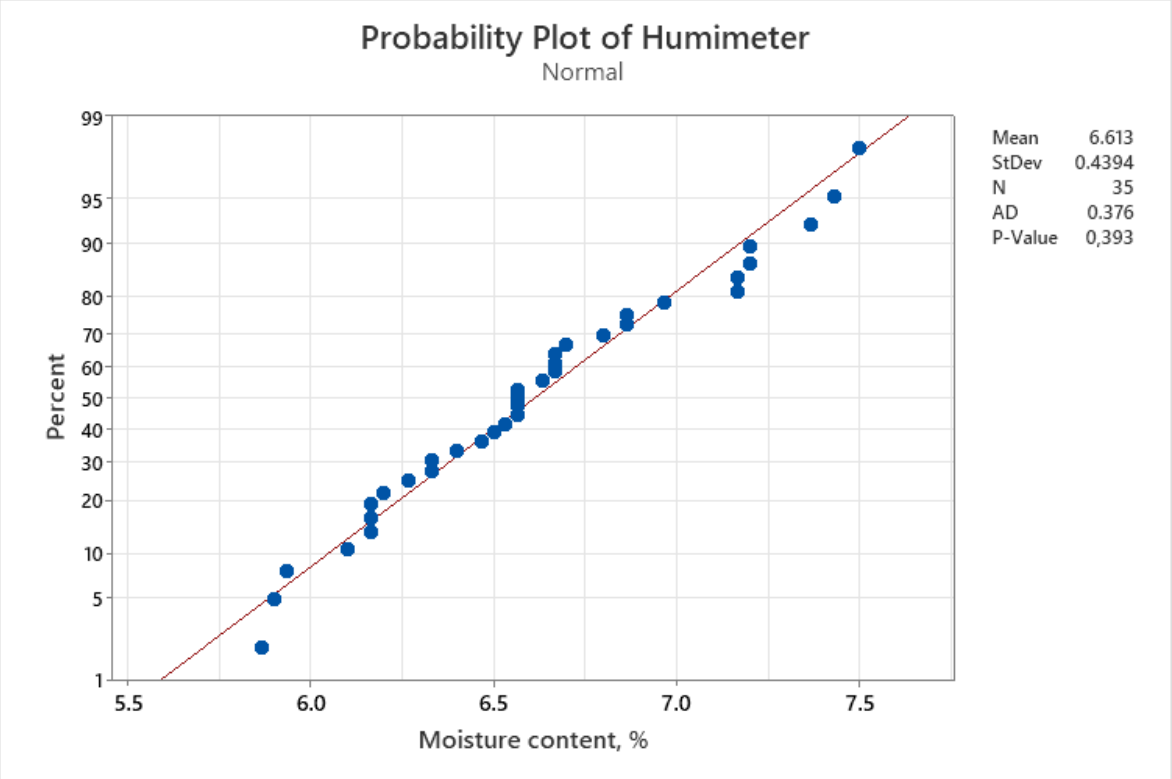

Figure 21. Anderson-Darling normality test for Product Grade 1. On the right-hand side is the resulting P-value of 0.393.

The mean value for Humimeter PM5 measurements was roughly 6.61 %, which is very close to the results from oven-drying. Since the P-value of 0.393 is significantly higher than the alpha-value, there is no evidence that the measurement data is not normally distributed. Visually, the data is also divided across the x-axis, and it follows the red reference line quite well. Thus, this data was also considered normally distributed.

9.1.3 2-Variance test for Grade 1 products

Before the 2-variance test, the skewing of the data was determined by a simple histogram plot. This is presented below in Figure 22.

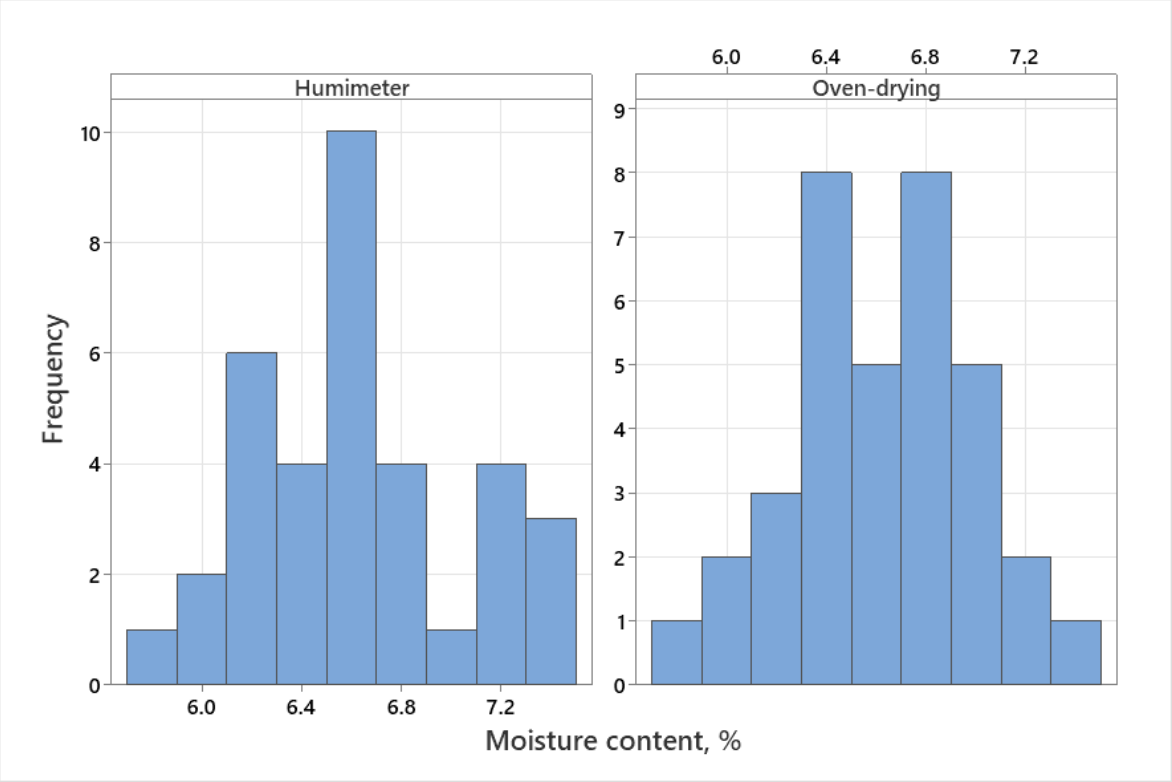

Figure 22. Histogram plot of frequencies of different moisture readings for Product Grade 1.

In these histograms, neither of the data appears to be severely skewed in either direction. On the contrary, both histograms are quite center-focused. Thus, Bonett's test was used in the 2-variance test.

The results from the 2-variance test and more importantly Bonett's test were as follows:

Null hypothesis	$H_0: \sigma_1^2 / \sigma_2^2 = 1$			
Alternative hypothesis	$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$			
Significance level	$\alpha = 0.05$			
Method	Test Statistic	DF1	DF2	P-Value
Bonett	0.97		1	0.324
Levene	0.55		1	68 0.460

The P-value from Bonett's test is 0.324, which is greater than the significance level of 0.05. Thus, the null hypothesis fails to be rejected, and it cannot be concluded that the standard deviations between the two measurements are significantly different.

Using Bonett's method, the ratio of the variances is:

Ratio of Variances		
Estimated Ratio	95% CI for Ratio using Bonett	95% CI for Ratio using Levene
1.36233	(0.722; 2.719)	(0.608; 2.845)

This result means that we can be 95 % certain that the ratio of variances is between 0.722 and 2.719. The average ratio according to the test was 1.36.

Finally, a summary plot from Minitab is presented below in Figure 23.

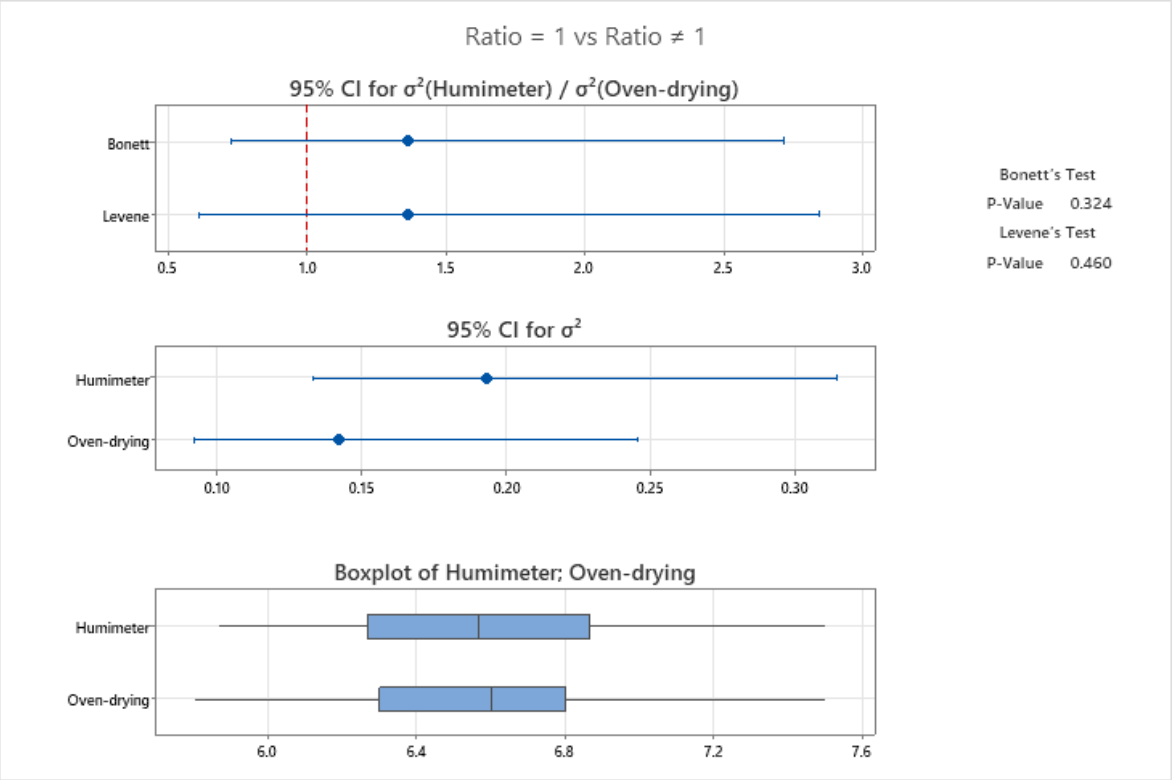

Figure 23. Summary plot of the 2-variance test for Product Grade 1

The summary plot of the 2-variance test in Figure 23 provides a visual representation of the 95 % confidence interval for the variances (middle graph), the ratio of the variances (top graph), and finally a boxplot (bottom graph) of how the moisture data is skewed. As demonstrated earlier in the histogram, the data is not skewed so it would affect the results.

9.1.4 2-Sample t-test for Grade 1 products

For the 2-sample T-test for Grade 1 products, equal variances were assumed, as the null hypothesis was not rejected earlier, and the P-value was relatively high.

The most important result of the 2-sample t-test tool is the estimation for difference. At a 95 % confidence interval, the result is:

Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
0.0476	0.4092	(-0.1476; 0.2428)

This means that calculated from the data in this thesis, the difference between the Humimeter PM5 moisture reading, and Oven-dried result is at a 95 % confidence level between -0.15 % and +0.24 % for Product Grade 1. On average, the difference is 0.05 %. This result is very satisfactory, as the error between the two methods is so small.

The null hypothesis states that there is no statistical difference between the mean values of the two measurement methods. The result of this test is:

Null hypothesis		$H_0: \mu_1 - \mu_2 = 0$
Alternative hypothesis		$H_1: \mu_1 - \mu_2 \neq 0$
T-Value	DF	P-Value
0.49	68	0.628

Since the alpha-value is again 0.05, and the P-value is considerably higher, the null hypothesis fails to be rejected. Thus, it is assumed that the means are of similar values. Altogether, the results deem that the Humimeter PM5 is very accurate for measuring the moisture content of Grade 1 products.

9.2 Grade 2

The number of measurements in this dataset was 28. The array of Grade 2 products is much wider in terms of grammage, coating amount, and product density than Grade 1. This was also reflected in the results, as there was more deviation in the measurement results and the determined error.

The data points were distributed well in the individual value and the moving range charts for Grade 2, and there was no reason for concern in terms of the reliability of the data. All the charts from the test for Grade 2 products are listed in Appendix I.

For the normality test for both oven-drying and Humimeter, the data was well distributed along the red reference line quite well. For oven-drying the P-value was 0.122 and for Humimeter PM5 it was 0.305. Both P-values exceeded the alpha-value of 0.05, and thus the null hypothesis could not be rejected. Both datasets were assumed to follow the normal distribution.

9.2.1 2-Variance test for Grade 2 products

The skewness of the data was again checked before the variance analysis via histogram graphs, which showed very little and negligible skewing for both methods, so Bonett's test was used as the main method for variance analysis.

The results from the 2-variance test are as follows:

Null hypothesis		$H_0: \sigma_1^2 / \sigma_2^2 = 1$		
Alternative hypothesis		$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$		
Significance level		$\alpha = 0.05$		
Method	Test Statistic	DF1	DF2	P-Value
Bonett	2.00	1		0.157
Levene	0.76	1	54	0.387

The P-value from Bonett's test is 0.157, which is greater than the significance level of 0.05. Thus, the null hypothesis fails to be rejected, and it cannot be concluded that the standard

deviations between the two measurements are significantly different. A similar conclusion would be made if Levene's test was used instead, as the P-value in that is 0.387.

The ratio of variances according to the test is:

Ratio of Variances		
Estimated Ratio	95% CI for Ratio using Bonett	95% CI for Ratio using Levene
0.590976	(0.297; 1.279)	(0.295; 1.695)

This result means that according to Bonett's test, we can be 95 % certain that the ratio of variances is between 0.297 and 1.279. The result from Levene's test is quite similar in the lower end, and the ratio range is 0.295 and 1.695.

9.2.2 2-Sample t-test for Grade 2 products

Equal variances were assumed for the 2-sample t-test for the Grade 2 product as well since the P-values from the 2-variance tests were greater than the alpha-value of 0.05. With this assumption, the result for the Estimation for Difference was:

Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
-0.2642	0.3695	(-0.4622; -0.0662)

So, at a 95 % confidence interval, the difference between the Humimeter PM5 measurement and the result from oven drying for a certain sample of Grade 2 product is likely to be between -0.46 % and -0.06 %. The difference of -0.2642 means that the Humimeter PM5 measures on average 0.26 % lower moisture content than oven-drying. This result is good, especially when considering that for the current Moistrex MX8000 meter, the goal deviation between it and oven drying is between -0.3 % and 0.3 %.

However, when testing for the statistically significant difference, the result is:

Null hypothesis		H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis		H_1: $\mu_1 - \mu_2 \neq 0$
T-Value	DF	P-Value
-2.68	54	0.010

The P-value from this test is 0.01, which is less than the alpha-value of 0.05. This means that the null hypothesis is rejected, and there is a statistically significant difference between the measurements, even though it is quite little.

9.3 Grade 3

A potential issue arose during the measurements for product grade 3. The same polymer from different suppliers, with only slight differences in its properties, was used in the coating process, but with the second polymer, the results in Humimeter PM5 measurements and the oven-dried results were on average different than the results from the first polymer. The second polymer clearly had a different effect on moisture retention, as there were no significant differences in the coating process parameters such as the intensity of the flame treatment.

Despite this change, all the statistical analyses were made for the whole batch of product Grade 3, instead of dividing the two slightly different polymer types for individual analysis, as the sample size would have been small, and the reliability of the result would’ve been compromised. The difference in the measurement accuracy for these different polymers should be inspected more thoroughly in the future. The total number of data points was 26.

9.3.1 I-MR Charts for Grade 3 products

The I-MR chart of the oven-dried results for the Grade 3 product is presented below in Figure 24.

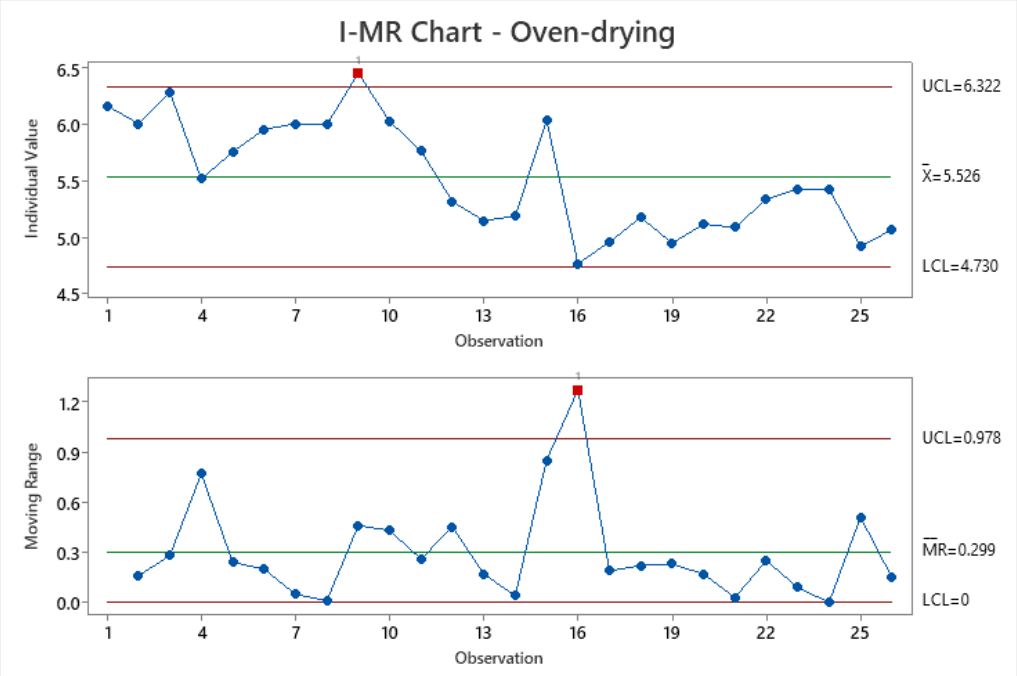

Figure 24. I-MR Chart of oven-dried moisture data for product Grade 3. The transition point of the two polymers is evident in observation 16.

The I-MR chart of the Humimeter PM5 for product Grade 3 measurement is presented below in Figure 25.

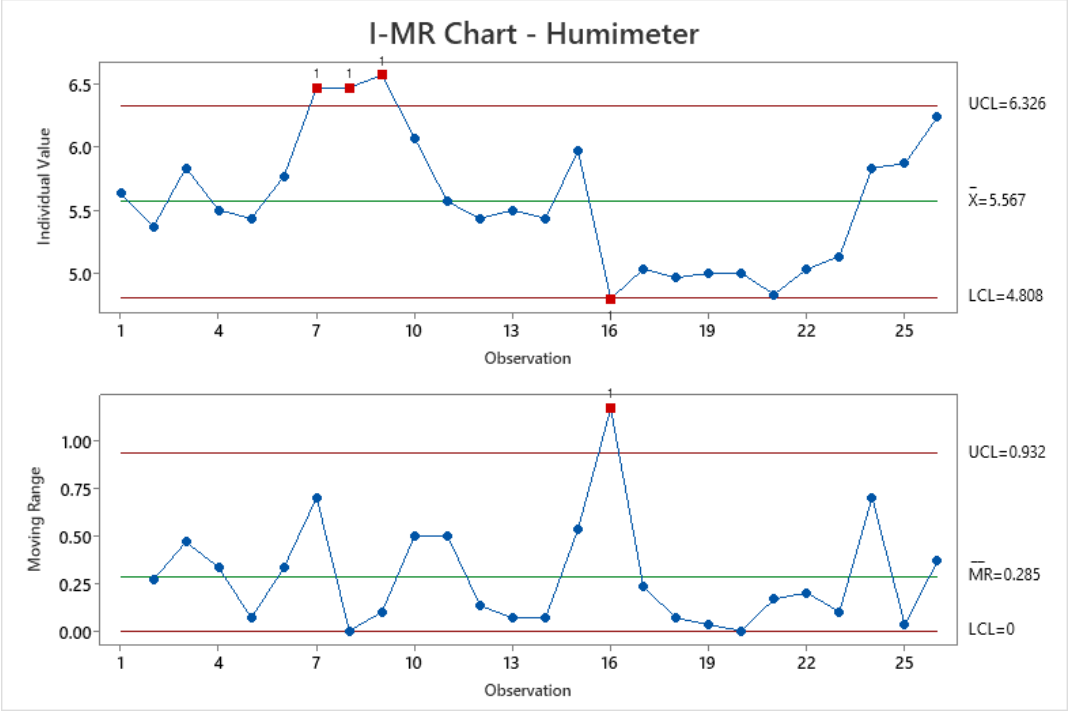

Figure 25. I-MR Chart of Humimeter PM5 measurements for the two product Grade 3 polymers.

Both Individual Value charts have some data points exceeding the upper control limit. This is not a serious issue, since the second type of polymer lowers the mean moisture percentage, which also lowers the upper control limit. Since the individual values exceeding the UCL for Humimeter PM5 are also high or even exceeding the UCL for oven-drying, there is no need for concern as the absolute error is not particularly high.

The red data points in the Moving Range chart were again caused by the sudden change in the individual values, which is not an issue since we were not inspecting the continuity of the process values.

9.3.2 Normality test results for Grade 3 products

The normality of the moisture data of the whole Grade 3 product batch was tested. The normal probability plot of the results from oven-drying is presented below in Figure 26.

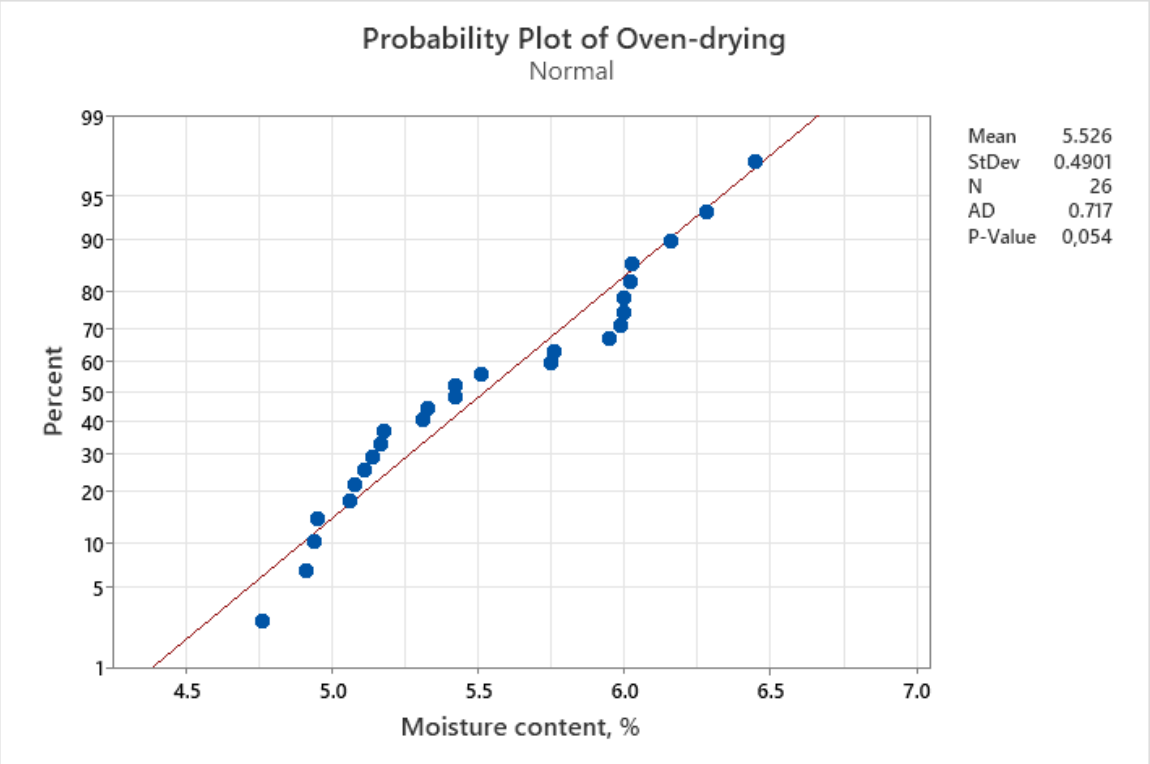

Figure 26. Product Grade 3 normal probability plot of the moisture data from oven-drying. The P-value is 0.054.

This time the data points did not fit on the red reference line as well as before. The P-value of 0.054 was barely over the alpha-value of 0.05, which meant that the null hypothesis of

the normal distribution cannot be rejected with this analysis, but the result should not be considered extremely reliable either.

For Humimeter, the normal probability plot is presented below in Figure 27.

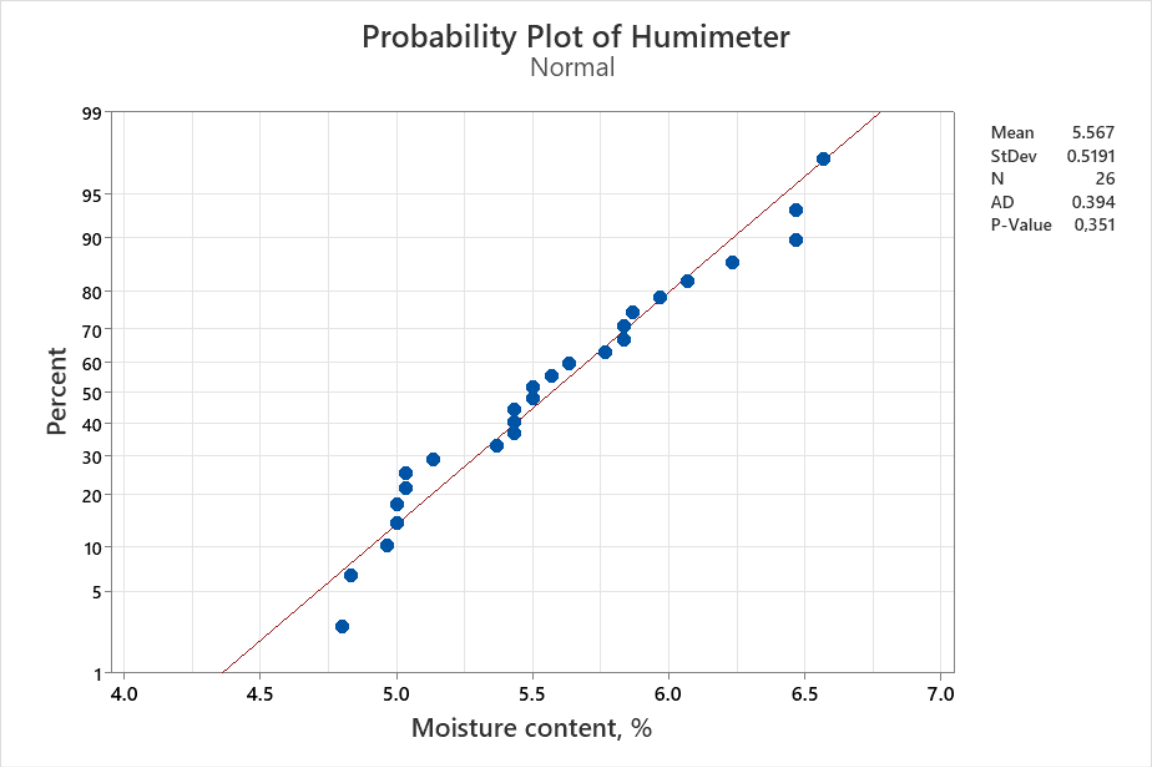

Figure 27. Product Grade 3 normal probability plot of the moisture data from Humimeter. The P-value is 0.351.

For Humimeter PM5 the data also curved slightly but resided better on the reference line than with oven-drying. The P-value of the normality test was 0.351, which was quite strong evidence that the null hypothesis stands, and the data is normally distributed.

9.3.1 2-Variance test for Grade 3 products

The data was examined for skewing by plotting a histogram. This is presented below in Figure 28.

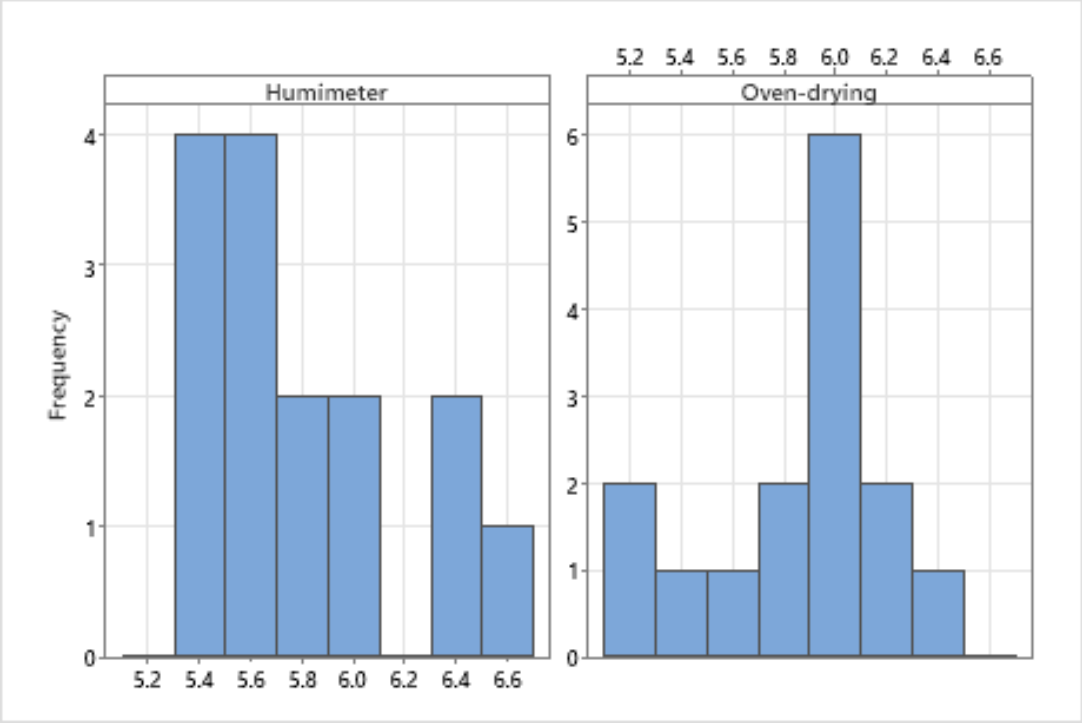

Figure 28. Histograms demonstrating the possible skewing of Grade 3 product moisture data.

This time from a visual inspection it could be said that the data from Humimeter PM5 is slightly skewed towards the left-hand side, but the data from oven-drying is more center focused. This is again due to the second polymer type having on average lower moisture content than the first one. Considering the magnitude of the frequencies the skewing was not that significant, so Bonett's test was used. The result from the variance ratio test for product Grade 3 was as follows:

Null hypothesis		$H_0: \sigma_1^2 / \sigma_2^2 = 1$		
Alternative hypothesis		$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$		
Significance level		$\alpha = 0.05$		
Method	Test Statistic	DF1	DF2	P-Value
Bonett	0.14	1		0.708
Levene	0.01	1	50	0.938

Both Bonett's and Levene's tests resulted in P-values greater than the alpha-value of 0.05, so it was justified to say that the null hypothesis is not rejected. The P-values are the largest of all tests done for all product grades.

The ratio of variances -test results are presented below:

Ratio of Variances		
Estimated Ratio	95% CI for Ratio using Bonett	95% CI for Ratio using Levene
1.12195	(0.566; 2.042)	(0.431; 2.064)

The ratio of standard variances was estimated to be on average 1.12. However, at a 95 % confidence interval, the range of the possible ratios is quite broad according to both tests: 0.566 – 2.042 for Bonett's test and 0.431 – 2.064 for Levene's test. The results from both tests were close to each other. These kinds of broad ranges often mean that there is a lot of uncertainty in the system. Despite the broad range, due to the high P-values, there was no evidence to reject the null hypothesis of equal variances.

9.3.2 2-Sample t-test for Grade 3 products

Equal variances were assumed in the 2-sample t-test. Results from the estimation of difference were:

Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
0.041	0.505	(-0.241; 0.322)

According to this, the average absolute difference in the moisture reading would've been 0.041 %, which was extremely good. However, at 95 % confidence interval the range for the difference between oven-drying and Humimeter PM5 was from -0.241 % to +0.322 %. The range was broad which was caused by the high standard deviation in the measurements. Thus, the average result may be good, but the total uncertainty remained quite high. Looking from another perspective, the result meant that it was 95 % likely that the moisture content measured with Humimeter PM5 does not differ more than 0.322 % from the oven-dried reading, which is acceptable when compared to the current limit of 0.30 % for Moistrex MX8000.

The result from the null hypothesis test was:

Null hypothesis		H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis		H_1: $\mu_1 - \mu_2 \neq 0$
T-Value	DF	P-Value
0.29	50	0.774

As the P-value from the 2-sample t-test is 0.774, the null hypothesis fails to be rejected. Thus, it is assumed that there is no statistically significant difference between the Humimeter PM5 and oven-drying for Grade 3 products.

9.4 Grade 4

Grade 4 product had the smallest sample size of the four, at only 13 with the clear outliers removed. This was due to difficulties caused by the production cycle timings, which lead to the fact that enough measurements could not be done. Some of the moisture measurements with the Humimeter PM5 were also done in a non-ideal density setting, which caused an absolute error of about 0.80 % in many of the individual data points. However, this test was useful for inspecting the impact and consequences of a natural operating mistake. The charts for Grade 4 products are listed in Appendix I.

In short, the I-MR charts for Grade 4 products were good for oven-drying and Humimeter PM5 datasets. There were no clear outliers, and the data was well divided around the mean values with very little deviation.

The result from Anderson-Darling normality tests was that the data both from oven-drying and Humimeter PM5 was evenly distributed along the reference line and the p-values were high and well above the alpha-value 0.05. The data was considered normally distributed.

When the skewing of the data was inspected before the variance test, minor skewing towards the right side of increased moisture content was detected. However, since the number of samples was so small and the skewing was not particularly serious, Bonett's test was used.

9.4.1 2-Variance

The skewing of the data was inspected from a histogram. This is presented for both Humimeter PM5 and oven-drying below in Figure 29.

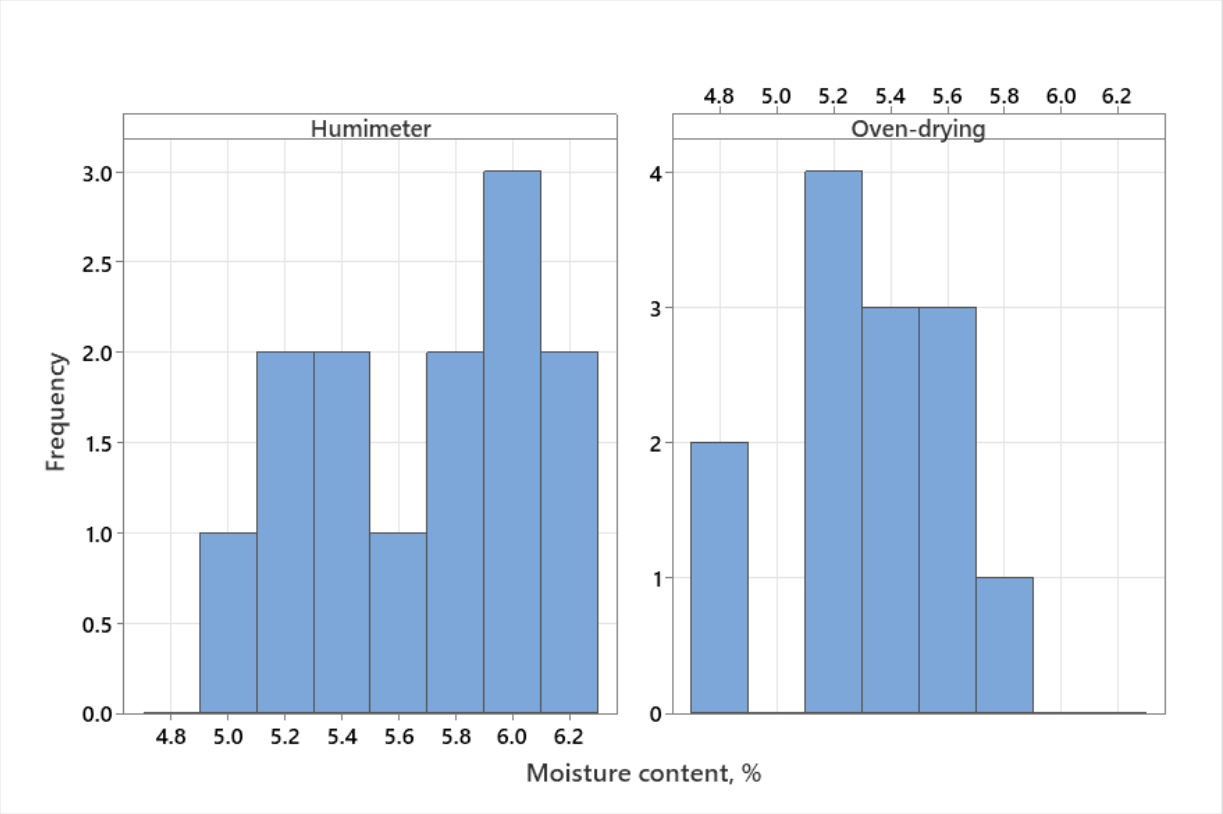

Figure 29. Histogram plot of frequencies of different moisture readings for Product Grade 4. Humimeter PM5 data is on the left and oven-drying data is on the right-hand side.

The histogram for Humimeter PM5 was slightly skewed to the right with increasing frequency of the high moisture content, whereas for oven-drying the histogram is center focused. From these graphs, it was deduced that on average, Humimeter PM5 measures slightly greater moisture readings than oven-drying for Grade 4 products. The skewing of the Humimeter PM5 data was considered small enough to still use Bonett's test.

The results from Bonett's test are presented below.

Null hypothesis		$H_0: \sigma_1^2 / \sigma_2^2 = 1$		
Alternative hypothesis		$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$		
Significance level		$\alpha = 0.05$		
Method	Test Statistic	DF1	DF2	P-Value
Bonett	3.60	1		0.058
Levene	3.09	1	24	0.092

The resulting P-values were both barely above the 95 % significance level alpha-value of 0.05. Bonett's test at the P-value of 0.058 was in theory enough to not reject the null hypothesis, but since the number of samples was so small and overall uncertainty in this test was very high, it was smarter to proceed with more caution and not assume equal variances for the t-test.

The result of the ratio of variances test is presented below.

Ratio of Variances		
Estimated Ratio	95% CI for Ratio using Bonett	95% CI for Ratio using Levene
2.45768	(0.965; 8.403)	(0.817; 11.380)

The estimated average ratio was 2.46. The confidence interval for the ratio calculated by both tests was very large, 0.965 – 8.403 for Bonett's test and 0.817 – 11.380 for Levene's test. This broad ratio meant that the result was very uncertain, which was very likely caused by too little number of samples.

9.4.2 2-Sample t-test

Equal variances were not assumed for the t-test as the P-values in the 2-variance test were so low. The result of the estimation for difference test is:

Estimation for Difference	
Difference	95% CI for Difference
0.337	(0.029; 0.645)

The average difference between Humimeter PM5 and oven-drying according to the t-test was +0.337 %, which by itself was acceptable. The confidence interval was +0.029 % – +0.645 %, which was broad. This was most likely due to the relatively high standard deviation combined with a little number of samples.

At 95 % confidence, the maximum absolute error according to this test was the upper limit of 0.645 %. This acts as further evidence of the fact that a non-ideal density setting was used in measurement since the average difference between two density settings is 0.8 %, which means that any absolute errors over 0.40 % could be mitigated by choosing a different density setting for a specific measurement.

The 2-sample t-test result is presented below:

Null hypothesis		$H_0: \mu_1 - \mu_2 = 0$
Alternative hypothesis		$H_1: \mu_1 - \mu_2 \neq 0$
T-Value	DF	P-Value
2.28	20	0.033

The P-value of this test was 0.033, which was less than the alpha-value of 0.05. Thus, the null hypothesis was rejected, and it was concluded that there was a difference of statistical significance between the results of oven-drying and Humimeter PM5 on Grade 4 products.

9.5 Results of the t-test for the Moistrex MX8000

The same tests were performed on Moistrex MX8000 for product Grades 1 and 2. The charts of the 2-variance and 2 sample t-tests for the Moistrex MX8000 are listed in the appendix I.

For Grade 1, the variance test signaled that there was no statistically significant difference between the variances of Moistrex MX8000 and oven-drying. Thus, equal variances were assumed in the t-test. The result of the t-test is presented below.

Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
-0.434	0.433	(-0.641; -0.228)

The average difference between the Moistrex MX8000 and oven-drying was -0.43 %, and the confidence interval is from -0.641 % to -0.228 %. This means that on average, the moisture readings from Moistrex MX8000 are significantly less than the actual or oven-dried moisture content.

The P-value from the hypothesis test was 0.000 (Appendix I), meaning that it is below the alpha-value of 0.05. The null hypothesis was rejected, and it was concluded that there is a statistical difference between the Moistrex MX8000 and oven-drying for Grade 1 products.

For Grade 2, the variance test again showed no statistical difference, and equal variances were assumed in the t-test. The t-test result is presented below.

Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
-0.577	0.375	(-0.780; -0.375)

Similarly, the average difference was significant at -0.577 %, and the confidence interval was from -0.780 % to -0.375 %.

The resulting P-value from the hypothesis test was 0.000 (Appendix I), and thus it was concluded that there is a significant statistical difference between the Moistrex MX8000 and oven-drying for Grade 2 products as well.

10 Comparison between Humimeter PM5 and Moistrex MX8000

The results from the t-tests for the Humimeter PM5 and the Moistrex MX8000 are presented in Table II below.

Table II Comparison between the t-test results against oven-drying for the Humimeter PM5 and the Moistrex MX8000.

	Grade 1		Grade 2	
Error	**Average, %**	**Range, %**	**Average, %**	**Range, %**
Humimeter	+0.048	−0.148; +0.243	−0.264	−0.462; −0.066
Moistrex MX8000	−0.434	−0.641; −0.228	−0.577	−0.780; −0.375

From the table, we can see that on average the Humimeter PM5 had significantly less absolute error than the currently used Moistrex MX8000. For Grade 1, the confidence interval of the Humimeter PM5 was narrower, and for Grade 2 the intervals were nearly equal. However, for Grade 2 the average error for Humimeter PM5 was over 0.3 % less than for the Moistrex MX8000, which makes Humimeter PM5 a far more accurate and reliable choice in terms of these tests.

11 Results of the Gage R&R measurement system analysis

Crossed Gage R&R analysis in Minitab provides results on how well the measurement system can handle and separate parts from each other, as well as whether the operators are using the measurement system uniformly. The analysis was done to inspect the uncertainty factors for the Humimeter PM5.

The components of the variation graph demonstrates which factors are the main source of the measurement uncertainty. Total Gage R&R is the sum of the repeatability and reproducibility variance components. Repeatability is the variability in results when the

same operator measures the same part multiple times, and reproducibility demonstrates the variability when different operator measures the same part. Total Gage R&R is the sum of the repeatability and reproducibility variance components. Finally, part-to-part variability is the variability in the results due to different parts with different properties. The Components of Variation graph for this study is presented below in Figure 30.

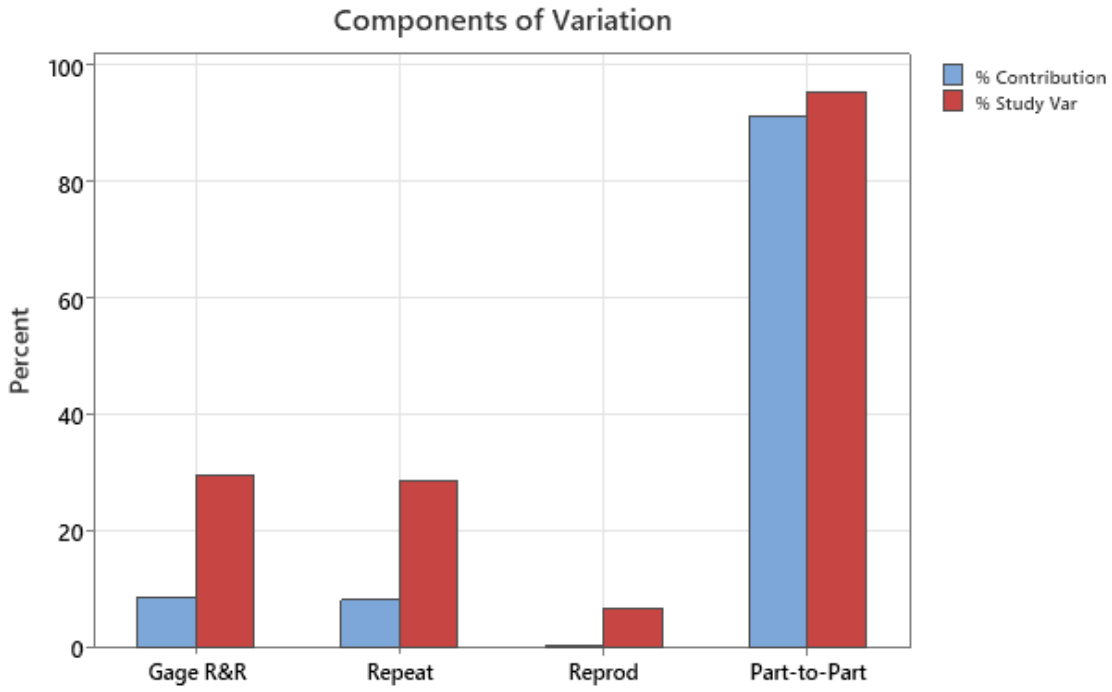

Figure 30. Components of Variation graph from Minitab for the Humimeter PM5

The graph clearly shows that the largest component was part-to-part variation. This is a good result, and it means that the measurement system is reliable in terms of repeatability and reproducibility.

R-Chart was used to study repeatability in the measurement system. If a point exceeds the upper control limit it implies that the operator may have some difficulties with uniform measuring between different parts. The Xbar chart compares changes in part-to-part variation to repeatability. Since part-to-part variation should be the largest, most of the data points should reside outside the control limits. R- and Xbar charts are presented below in Figure 31.

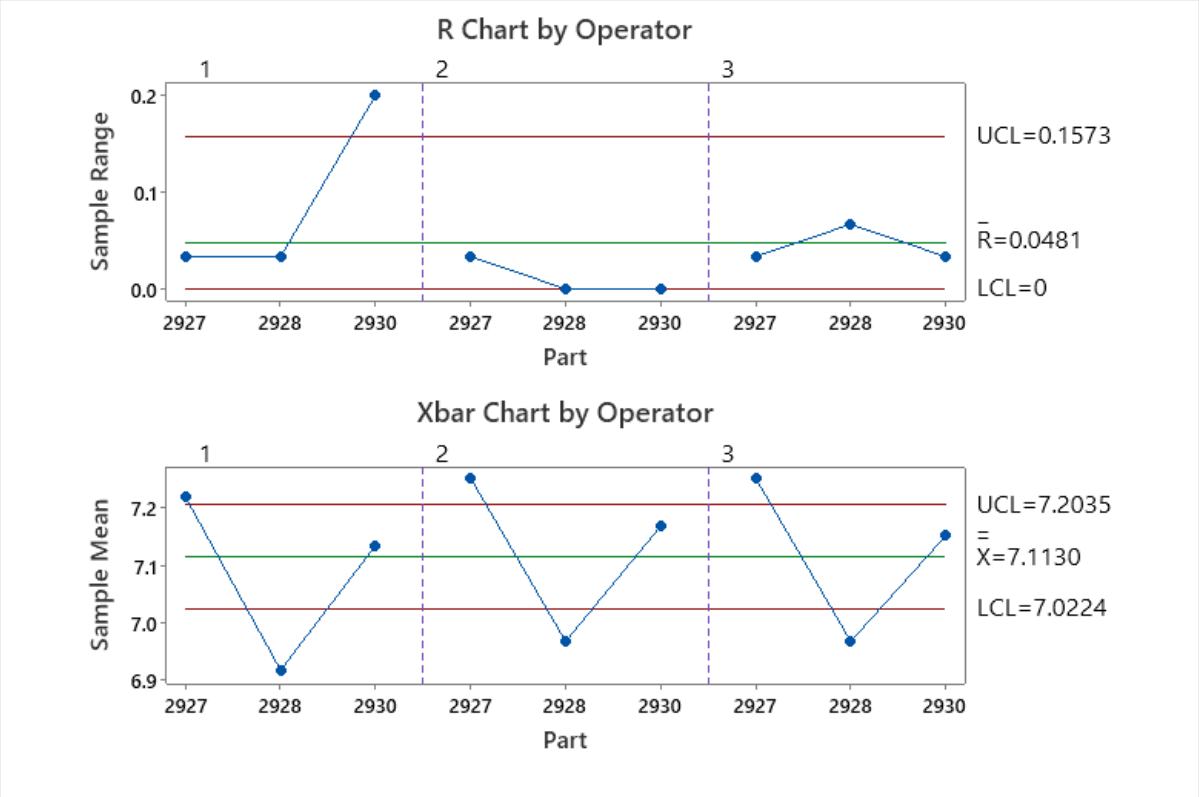

Figure 31. R- and Xbar charts of the Gage R&R analysis.

From the R-chart above we can see that a single point from a single operator exceeded the upper control limit. However, the data point was at 0.2 % and the UCL was at 0.157 % moisture content, which is very small when compared to the sample mean of about 7 %. Even though the number of parts was quite little, it was assumed that this single point exceeding the UCL was not particularly significant.

From the Xbar chart in Figure 31 above, it was clear that the majority of the data points were outside the control limits. Also in this analysis, the upper and lower control limits were relatively very close to each other with a difference of roughly 0.2 %. When considering the meter's resolution of 0.1 %, it was determined that the result was satisfactory.

Part-Operator interaction chart demonstrates the average measurements performed by each operator on each part. Ideally in this chart, all the connected lines follow the same pattern. If the patterns differ from each other, it means that the operators' accuracy depends on the part. The Part-Operator interaction chart is presented below in Figure 32.

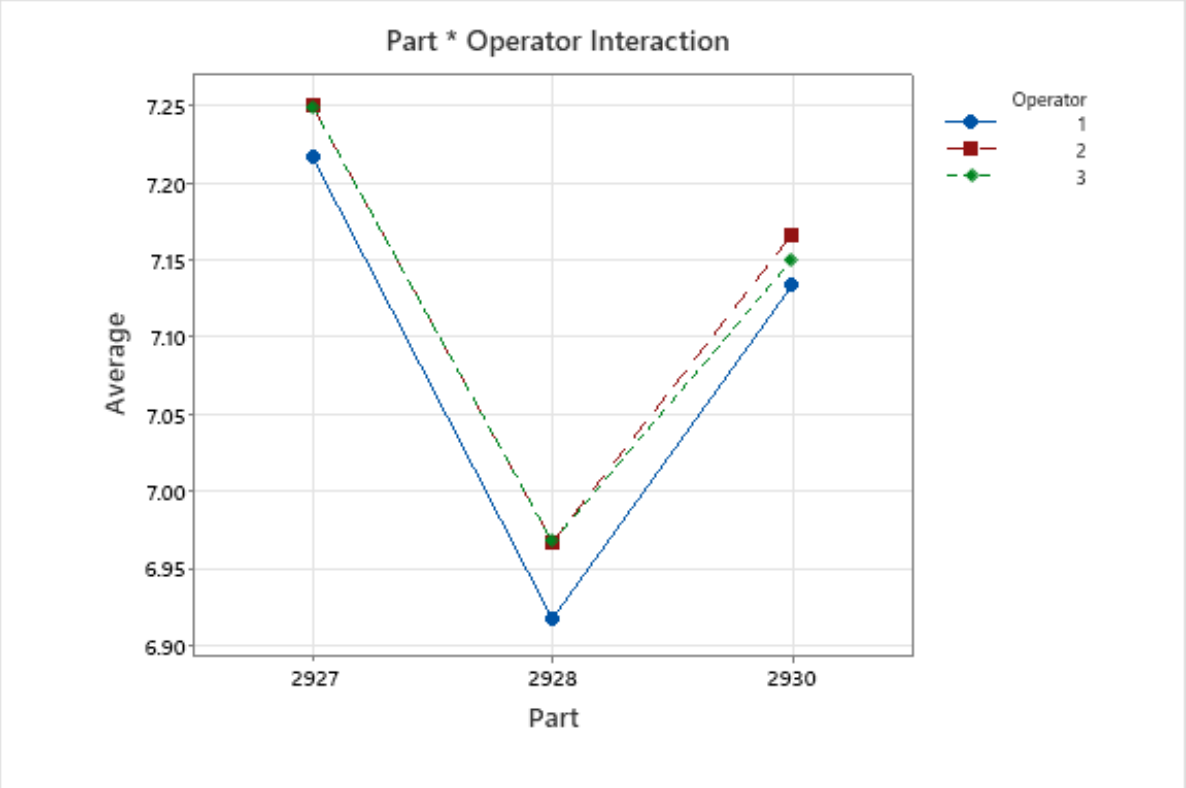

Figure 32. The part-Operator interaction chart in Gage R&R analysis

In this figure, it was evident that there was only a minor difference between the three operators. It seems that Operator number one measured on average slightly lower moisture contents than the two others. All the lines follow the same pattern, which made the result ideal.

12 Error estimation

An important factor causing uncertainty in the measurement is the uneven moisture profile of the board. Sometimes the board is drier in some parts and some sections contain more water. An example of this is demonstrated in Figure 33 below.

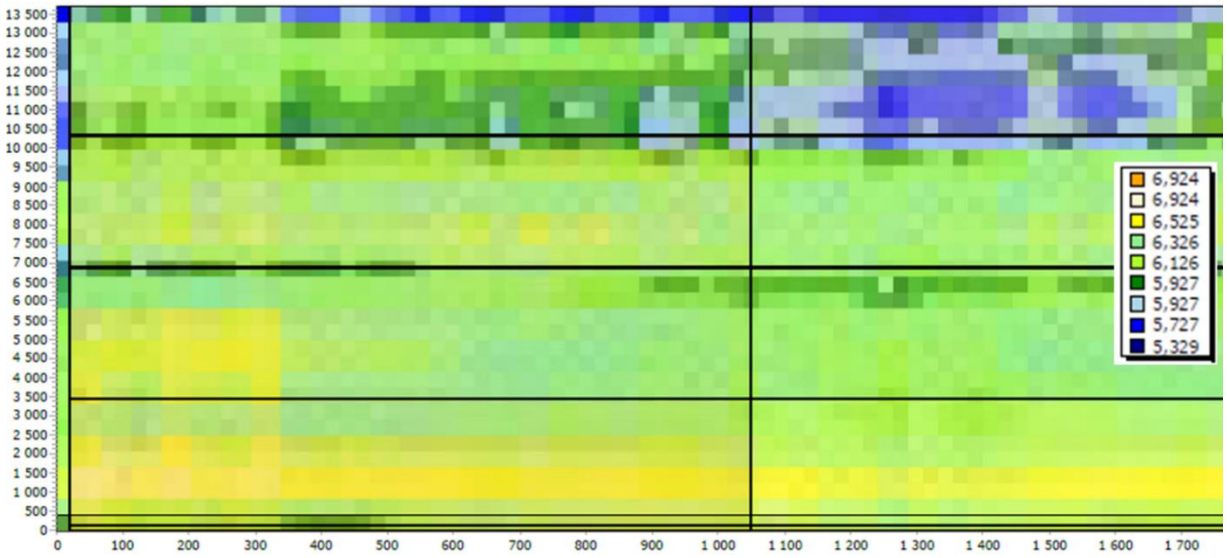

Figure 33. Moisture profile (%) measured by an online scanner for a machine reel. Reel width is on the x-axis in millimeters and the board web length on the y-axis in meters.

From the figure, it can be seen that there is a moisture content difference of about 0.4 % in the top right section of this specific machine reel. Similarly, the measured moisture content at the beginning of the reel, or the lowest section, was slightly higher than the average moisture content. When manually measuring, these local points may distort the average result in either direction and thus cause error in the measurement result. This is why it is extremely important to create clear instructions on which spot the measurement and possible samples for oven-drying should be taken from.

Errors are very likely when taking samples for oven-drying. It is extremely important that the samples are taken as soon as possible and sealed thoroughly in plastic bags to prevent drying. This is not always possible, as the operator often must prioritize sampling, controlling the process, phone calls, and possibly other tasks as well. It was also possible that the samples were contaminated during handling from grease or dirt from equipment, gloves, or bare hands, which then could cause error in the oven-drying result. Some exceptionally low oven-dried results were excluded from the analyses, but not all since they did not all quite exceed the control limit. Exceptionally high results from oven-drying were not excluded, since the likelihood of an error that causes oven-drying to overestimate the moisture content was considered very little.

The Humimeter PM5 is of sturdy build, and it is exceptionally simple to use, which reduces the risk of error caused by the operator. However, it was noted that the Humimeter PM5

results slightly depended on the force that the device was pressed against the reel with. Pressing the device harder resulted in a slightly higher moisture reading of about 0.1 % – 0.3 %. This was also discovered by Forsén & Tarvainen (2000) when they compared multiple different capacitance-based handheld wood moisture meters. They also reported that many capacitance-based meters report too low moisture contents, but that issue is easily negated by selecting the most accurate density channel.

13 Conclusions

This thesis aimed to determine the suitability of a new modern handheld capacitance-based moisture meter for measuring the moisture content of polymer-coated packaging paperboard. The new meter was compared to standardized oven-drying and the older microwave-based tabletop meter, and the uncertainty and confidence interval was calculated for different product grades with Minitab statistical software. Gage Repeatability and Reproducibility measurement system analysis was performed on the new meter to determine the main cause of variation in the results.

The new Humimeter PM5 performed well for most of the product grades and the resulting accuracy was on average better than for the currently used Moistrex MX8000. The confidence interval was also narrower, which meant that there was less deviation in the results.

According to Gage R&R analysis, the Humimeter PM5 is a reliable measurement method in terms of repeatability and reproducibility as the biggest variation in the measurements was caused by the changing part that was measured and not the operators using the device.

The new meter shows great promise, but there is still work to be done. The biggest challenge through the experimental part was the occasional too little amount of data caused by challenging production cycles and occasional lack of sampling personnel, as continuous production with good quality was the priority number one. The little number of samples led to broad confidence intervals and more unreliable results. Especially for the last two product grades 3 and 4, the sample pool should be increased significantly, and the analysis should be repeated to determine the error margins with greater confidence.

As the meter is still new, its long-term suitability for the coating mills is to be evaluated. A maintenance program will have to be created, as well as a standard method for determining correct calibration on-site.

As the step size between the density channels is 50 kg/m^3, the measurement is necessarily non-ideal for some product types. It would be beneficial to contact the supplier and discuss the option to decrease the interval to 25 kg/m^3, which at least in theory would decrease the maximum possible error on an ideal measurement setting, as well as enable more accurate measurements for different product grammages.

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Appendix I

Graphs during the t-test analyses

Grade 2 products

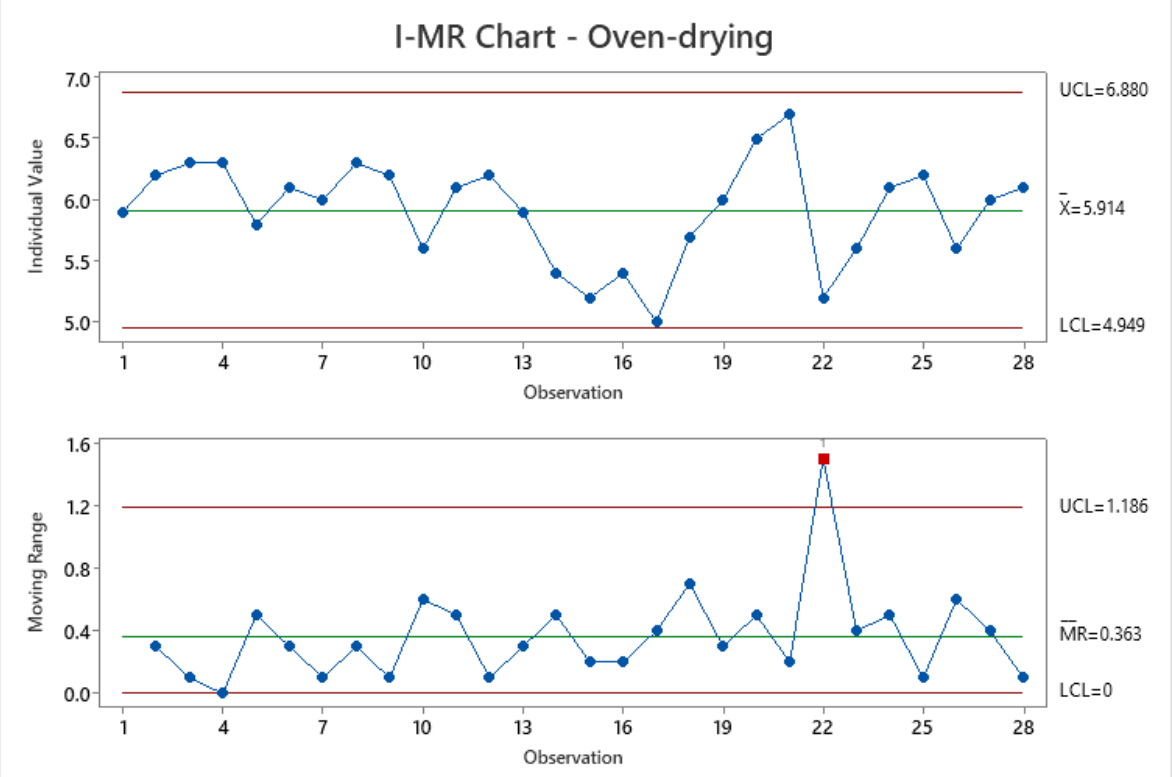

Figure 34. I-MR Chart of oven-dried moisture data for product Grade 2

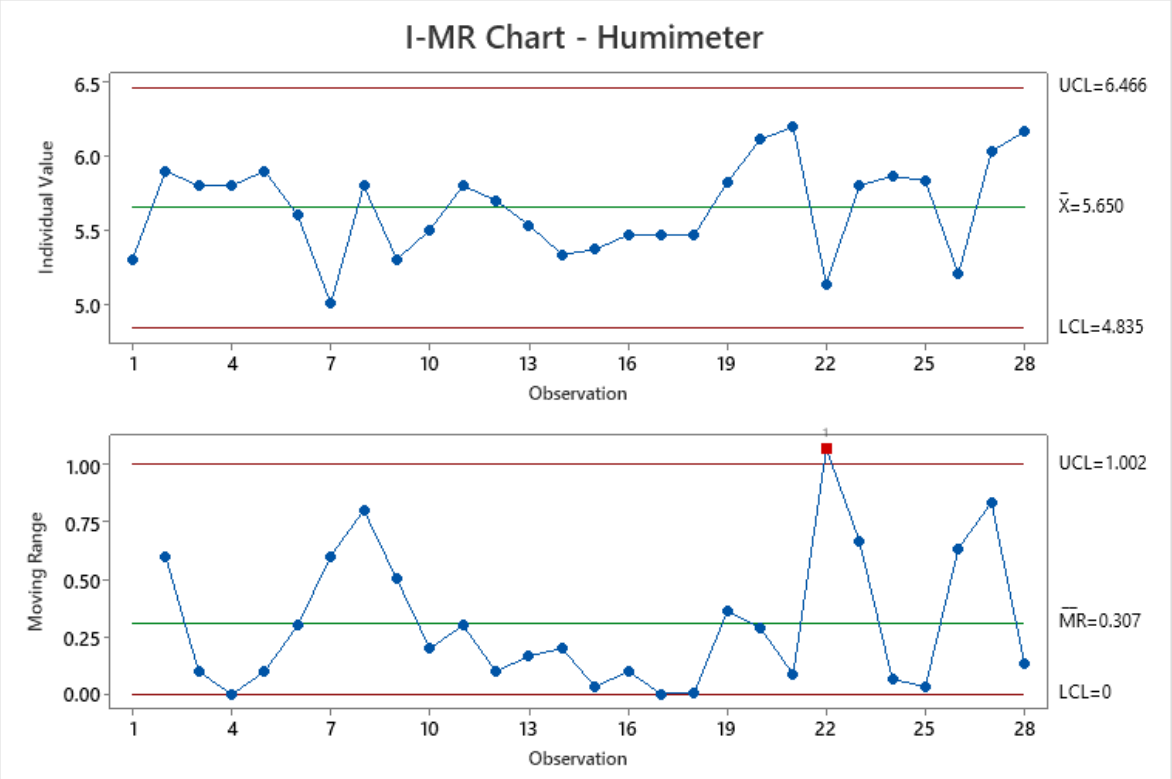

Figure 35. Product Grade 2 Humimeter PM5 moisture data I-MR Chart

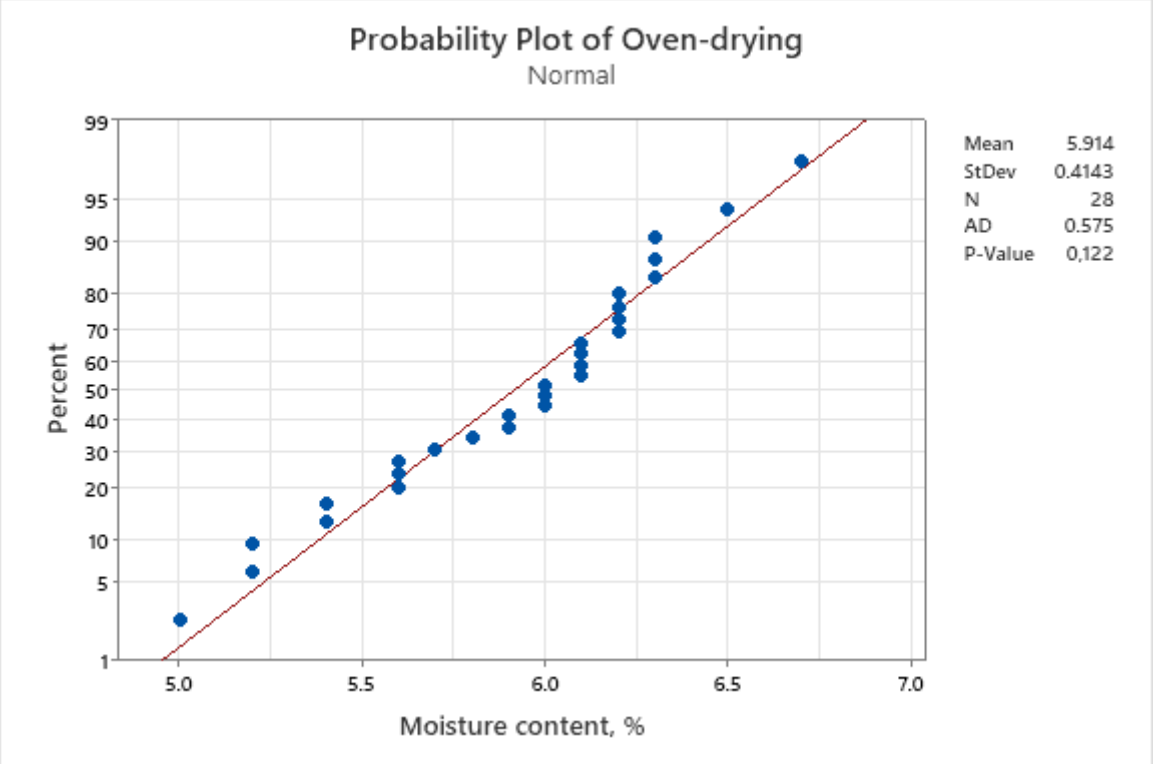

Figure 36. Product Grade 2 normal probability plot of the moisture data from oven-drying. The P-value is 0.122.

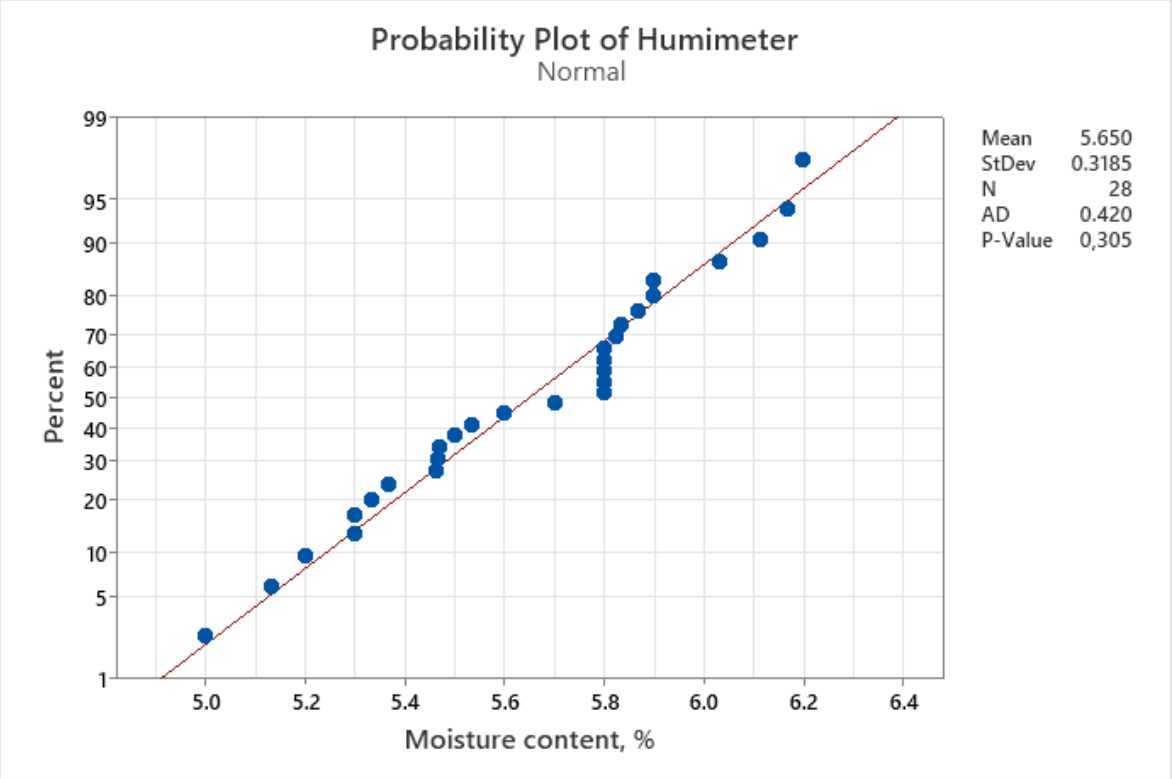

Figure 37. Product Grade 2 normal probability plot of the moisture data from Humimeter. The P-value is 0.305.

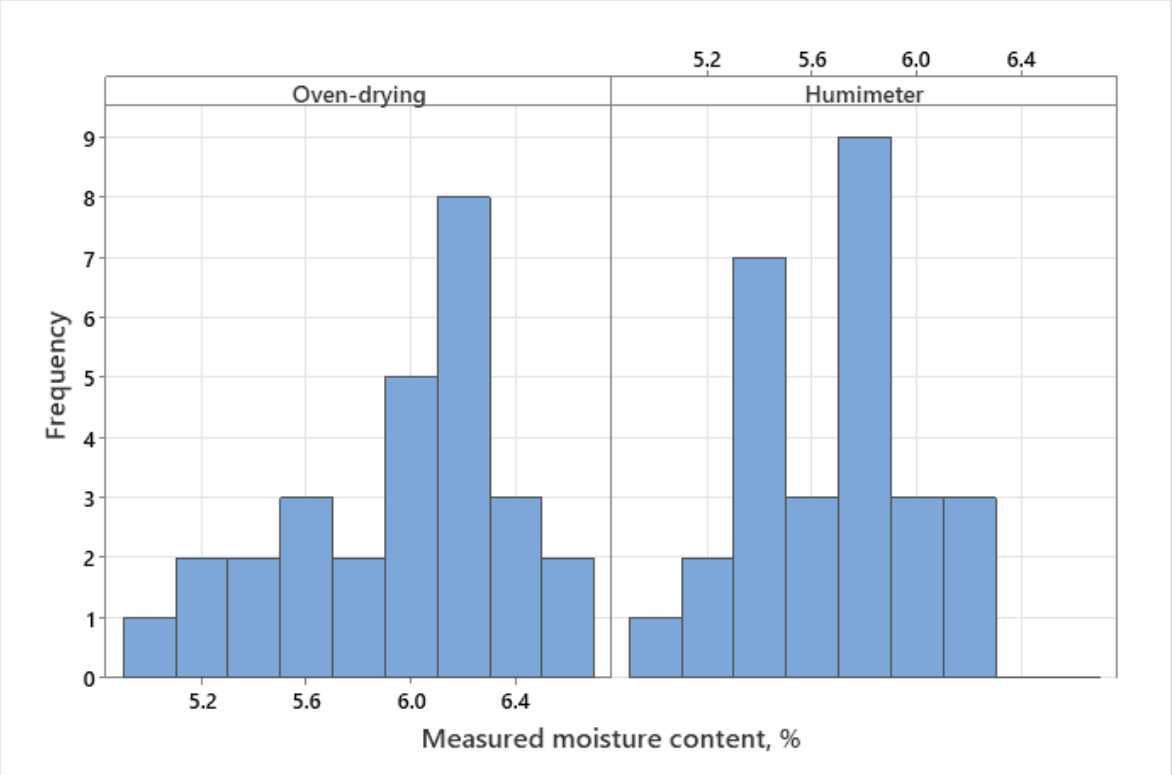

Figure 38. Histograms demonstrating the skewness of product Grade 2 measurement data.

Grade 4 products

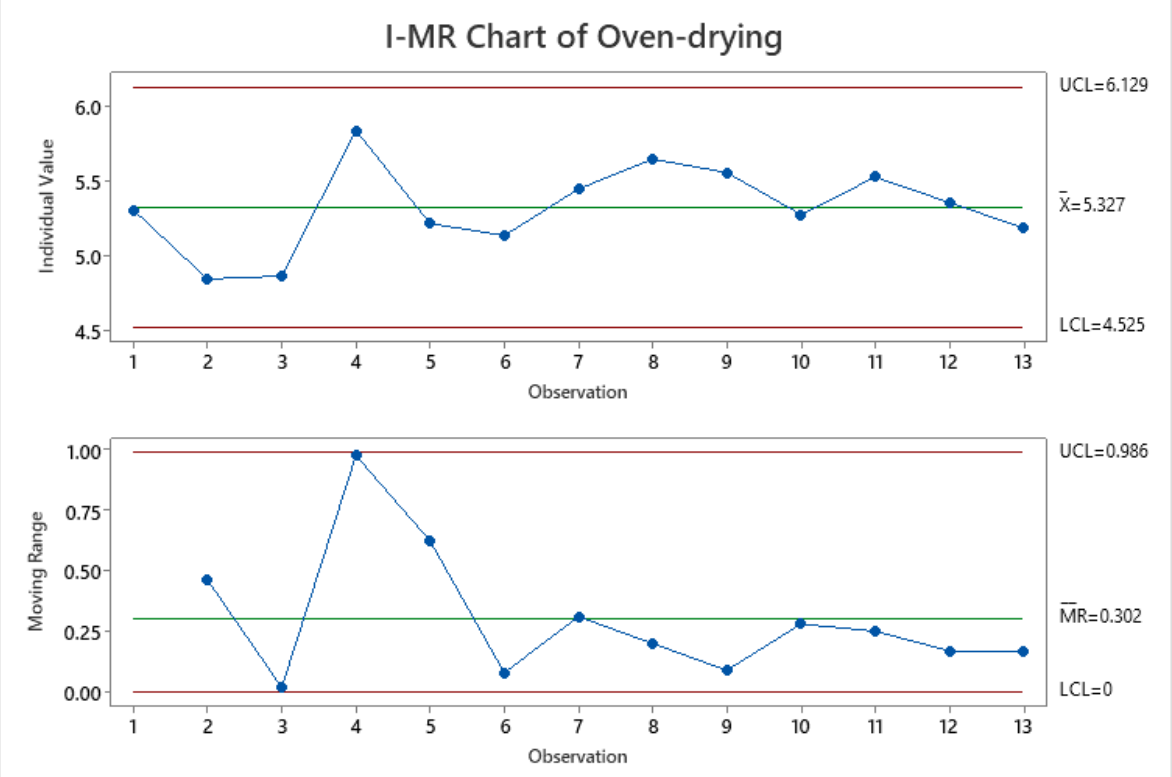

Figure 39. I-MR Chart of oven-dried moisture data for product Grade 4

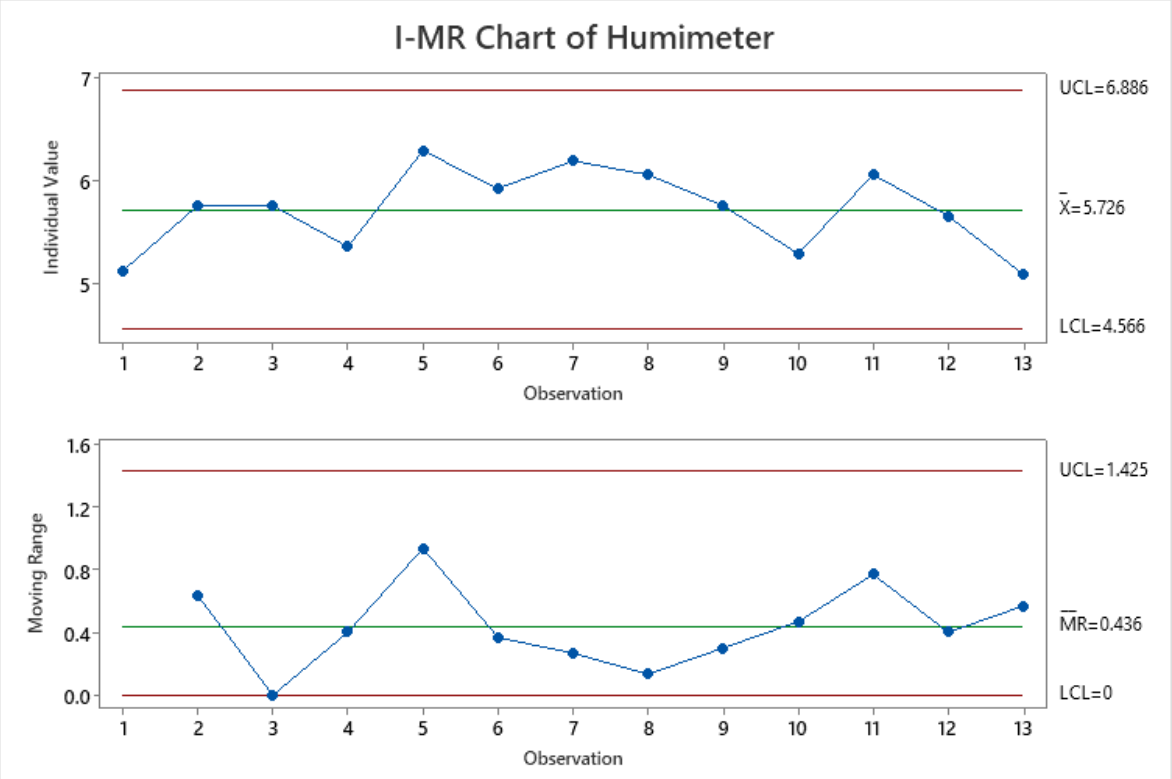

Figure 40. I-MR Chart of Humimeter PM5 measurement data for product Grade 4

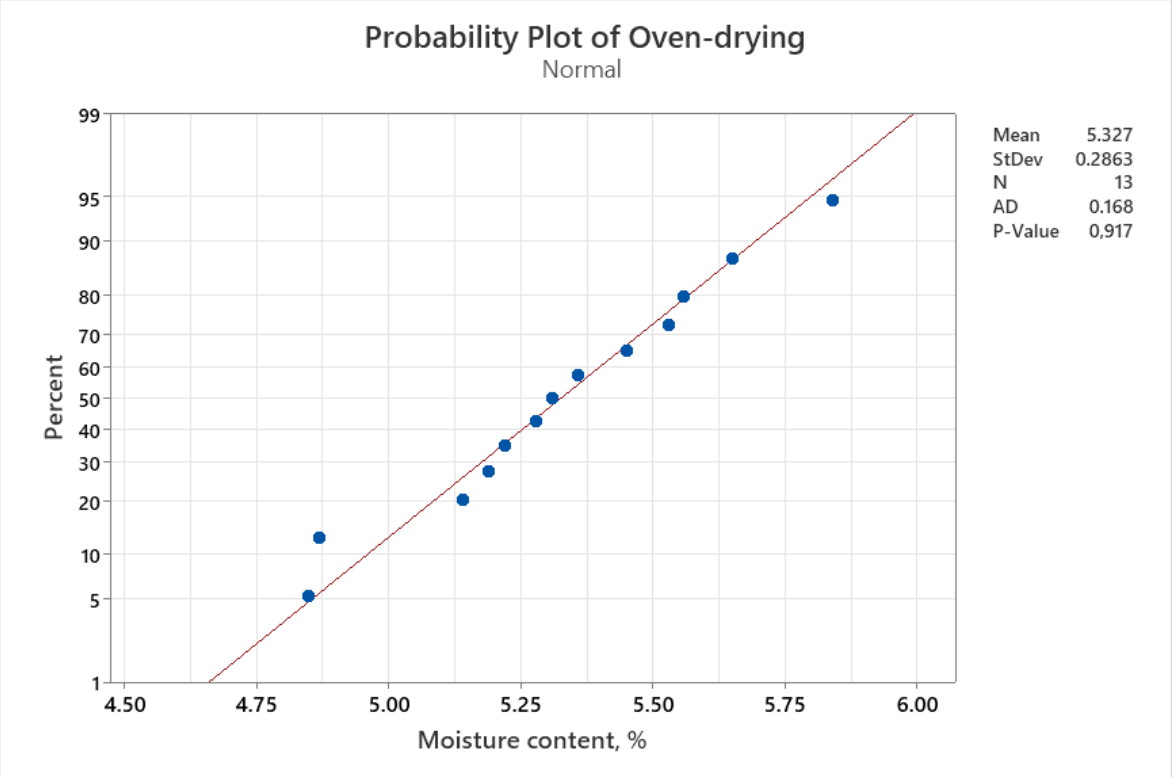

Figure 41. Product Grade 4 normal probability plot of the moisture data from oven-drying. The P-value is very high at 0.917.

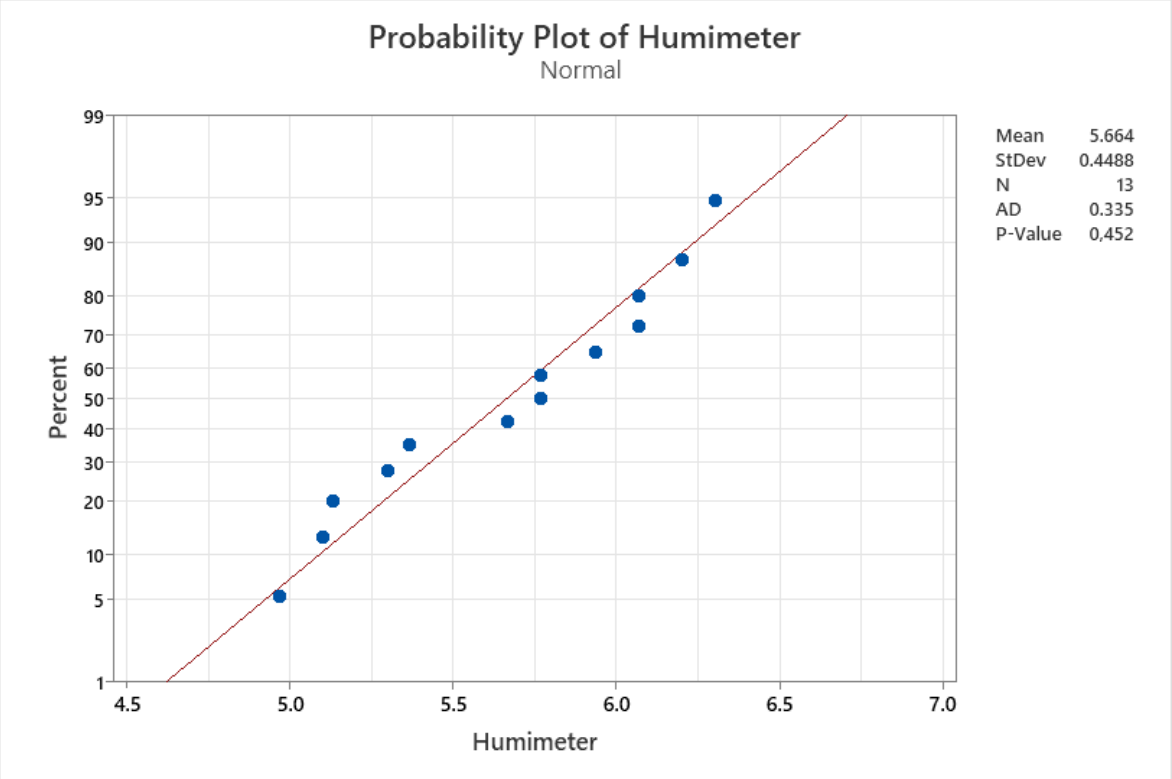

Figure 42. Product Grade 4 normal probability plot of the Humimeter PM5 moisture data. The P-value is 0.452.

Moistrex MX8000 2-variance and 2-sample t-tests

Results from the 2-variance test for Moistrex MX8000 and oven-drying:

Ratio of Variances		
Estimated Ratio	95% CI for Ratio using Bonett	95% CI for Ratio using Levene
0.628094	(0.286; 1.403)	(0.299; 1.705)

Null hypothesis	$H_0: \sigma_1^2 / \sigma_2^2 = 1$			
Alternative hypothesis	$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$			
Significance level	$\alpha = 0.05$			
Method	Test Statistic	DF1	DF2	P-Value
Bonett	*			0.227
Levene	0.76	1	53	0.387

Results from the 2-sample t-test for Moistrex MX8000 and oven-drying

Alternative hypothesis	$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$	
Estimation for Difference		
Difference	Pooled StDev	95% CI for Difference
-0.577	0.375	(-0.780; -0.375)

Null hypothesis	$H_0: \mu_1 - \mu_2 = 0$	
Alternative hypothesis	$H_1: \mu_1 - \mu_2 \neq 0$	
T-Value	DF	P-Value
-5.71	53	0.000