Cost-effective System for Inline Coating Evaluation in R2R Processes

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Abstract

Monitoring the properties of coatings during the production process is a method to stabilize the process itself by ensuring the product quality in its early stage which enables the operator to optimize process parameters in real time. The paper will give a short overview of different methods to evaluate a coating during the production process (using color evaluation, physical model based and chemometric methods). In addition we will detail a cost effective measuring system for in-line and in-vacuo use. Setup: All methods described in the paper are based on the ThinProcess® WEB solution. These solutions are made possible by utilizing accurate measurements sensors with measurement capabilities which can be calibrated automatically during the production run. Methods: Different method to calculate or predict the coating thickness or coating weight will be shown in the paper. These methods can be combined to provide additional data for complex coatings. For simple layer designs, a correlation of the thickness and a color value may exist and this correlation needs to be found. The paper will show how this correlation is used to predict the coating thickness. Thickness results based on b* measurements. Additionally we will show how model based fits can be used to predict coating parameters like thickness.

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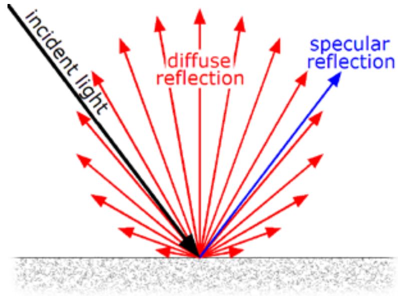
Background Information

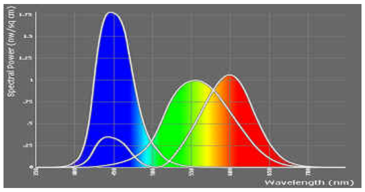


Real time solutions in the color appearance evaluation of plastic manufacturers is an important consideration to manufacturers and customers. Process automation permits the measurement of appearance parameters that are critical to customers; such as color and gloss, to be evaluated in real time. The benefits from real time assessment and the dynamic, continuous monitoring and closed loop color control permit the real time evaluation and correction and sorting or colored materials

The term color and appearance adds the additional geometric term to the classical color evaluation that is typically performed in the laboratory. Gloss is a parameter of appearance that quantifies the quantity and quality of the reflection component from a surface in the specular direction. The more mirror like the reflection component, the greater the specular reflection. Gloss is one of the important parameters used to describe the visual appearance of an object. Related words describing the phenomena are gloss and shininess. Factors affecting gloss are surface texture, index of refraction, surface roughness, surface haze and surface finish.

In color measurement, called colorimetry, the examination and quantification of color is based on the three-components of color vision. The three components of color vision are attributable to the primary receptors in the human eye; formally called long, medium and short, colloquially known as red, green, and blue. In colorimetry, these components are referred to as CIE $\bar{x}, \bar{y}, \bar{z}$, the color matching functions. All colors that are seen are primary colors or mixtures of these primaries.

Spectrometry measures the spectral reflectance factor of an object at each wavelength on the continuum of the visible spectrum and expresses the measurement in terms of a psychophysical specification called CIEXYZ. These values are normalized then converted to CIE *L*a*b** (CIELAB**),** which is a psychometric color space specified by the French *Commission internationale de l'éclairage.* The color space is dependent upon the color of the sample, the illuminant expressed as a Spectral Power Distribution, SPD, and the observer. The observer function is standardized as the 1931 2^{o} Standard Observer, X_2, Y_2, Z_2, and the 10^{o} Standard Observer X_{10}, Y_{10}, Z_{10}, in conformance with CIE 15:2004. CIELAB is the most widely used color system in the world. This CIELAB system describes all the visible colors and is device-independent.



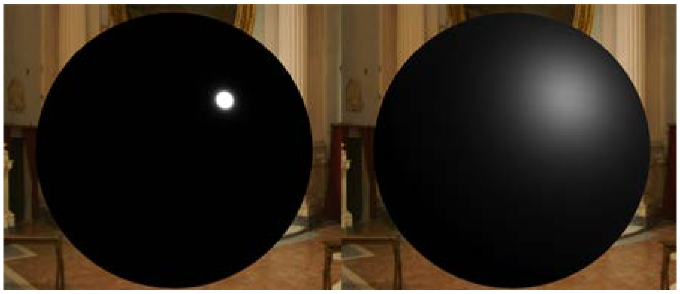
When light strikes an opaque object that has surface texture as shown in the figure, the interaction of light and its' characterization occurs at the surface of that object. The total reflection component consists of two phenomena. The first phenomenon is the diffuse body reflectance that we attribute to and evaluate for color. The second is a specular reflection component that attributable to and is evaluated for gloss or shininess.

When the smoothness of a surface changes resulting in higher micro-roughness, similar to a matte surface, that has a lower gloss, the light-reflection characteristics are more complex. This causes the color of the object to appear lighter and less saturated. Therefore, the color appearance of an object is affected by the surface roughness of the object and is independent of color.

In addition, plastics coated with a thin film, such as ZrO2 and SiO2, can also be evaluated as they affect not only the color appearance of the opaque and transparent plastics, but also the durability. The thickness of the film play an important role in both the color and hardness.

Important factors in manufacturing are the stabilization of color appearance critical variables, which may include the application of the film layer and number of layers that are responsible for product quality and the reduction of production and ultimately product costs.   These goals are often related and resultant by controlling processes in the manufacturing process. The analysis of these data are often multivariate. These data must be de-convolved for control of the desired parameters. There are on-line, at-line and laboratory instruments that provide the data necessary to derive the color appearance as well as chemometrics necessary to achieve significant enhancements in Operational performance parameters.

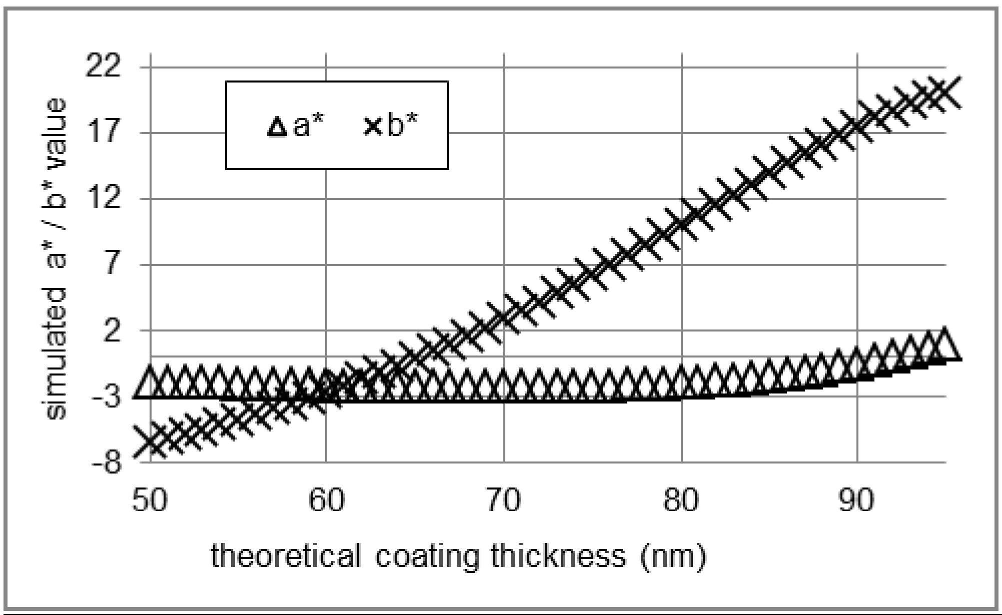
We examine and define the science of chemometrics[i] as the science of extracting information from chemical system by data driven means. As it is inherently interdisciplinary, using methods in core data-analytic disciplines such as multivariate statistics, applied mathematics and computer science, we will address how these disciplines can be used incorporating spectroscopic data in the context of this paper. This paper does not cover the rigor of the mathematical techniques used in the analysis; such as; matrix algebra, analytic geometry, experimental design, calibration regression, linearity, design of collaborative laboratory studies, comparison of analytical methods, noise analysis, use of derivatives, analytical accuracy or analysis of variance that are the classical tools used in the science of chemometrics. Rather, a small example is



used to illustrate some ways of working, mainly by using graphical analytical techniques.

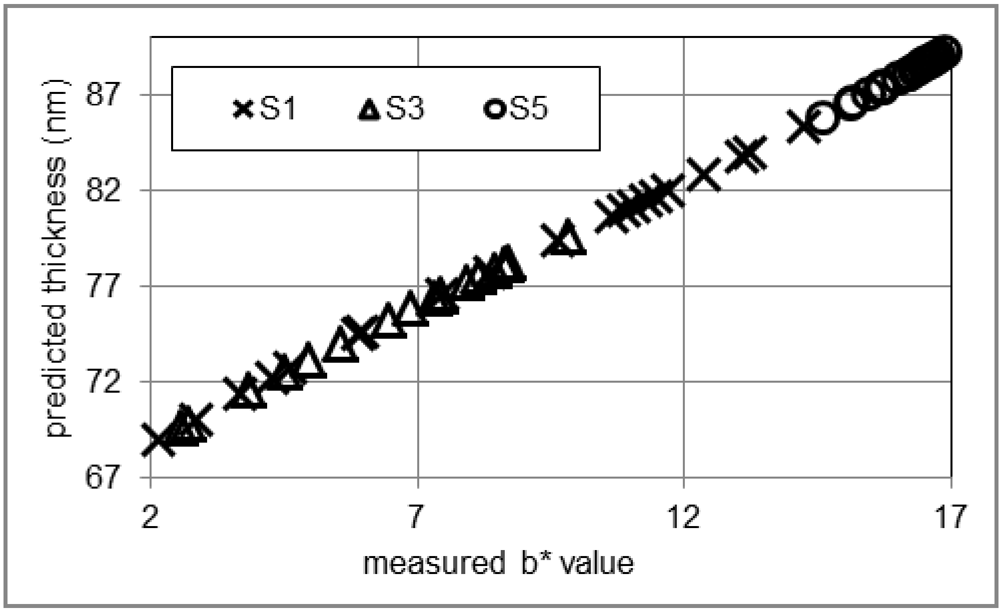
Selected literature references are given. Part 1 deals with classical chemometrics.

For simple layer designs a correlation of the thickness and a color value may exist and this correlation needs to be found. This theoretical calculation is performed using modeling software prior to any real measurement. The data shows a good correlation between thickness and the CIE b* value for this specific layer.



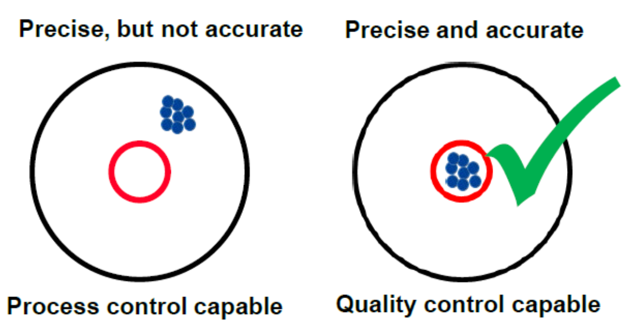
Part 2 presents some newer developments and includes some more elaborated examples

For process control a 3rd order polynomial based on the data was used to calculate the coating thicknesses from the measured CIE b* values. On six (6) ZnO coated glass panes (S1 – S6) the CIE b* values were measured and the predicted thickness results are shown.



Objective

In order to qualify the parameters to be controlled, outcome of the data must be determined. Data used for quality control must be precise and accurate while data used for in process control needs to be precise, but accuracy is not necessarily the objective.



Tolerances may be influenced by such factors as:

- Measurement System
- Reference Method
- Calibration Standards
- Process Conditions

Measurement Procedure

The stability of a sensor is largely determined by several factors. The measure of stability is typically assessed as drift. That is, the sensor's ability maintains a constant output given the same input. Drift is determined by several factors; calibration, stability, response time, self- heating, and sensor noise. The calibration of an on-line, optical sensor for the determination of optical properties involves a physical standard that is used to provide an optical reference for the instrument. This can be performed in two ways:

The traditional way to calibrate spectral measurements involves the use of a certified external calibration standard; such as a mirror or white standard, for instance.

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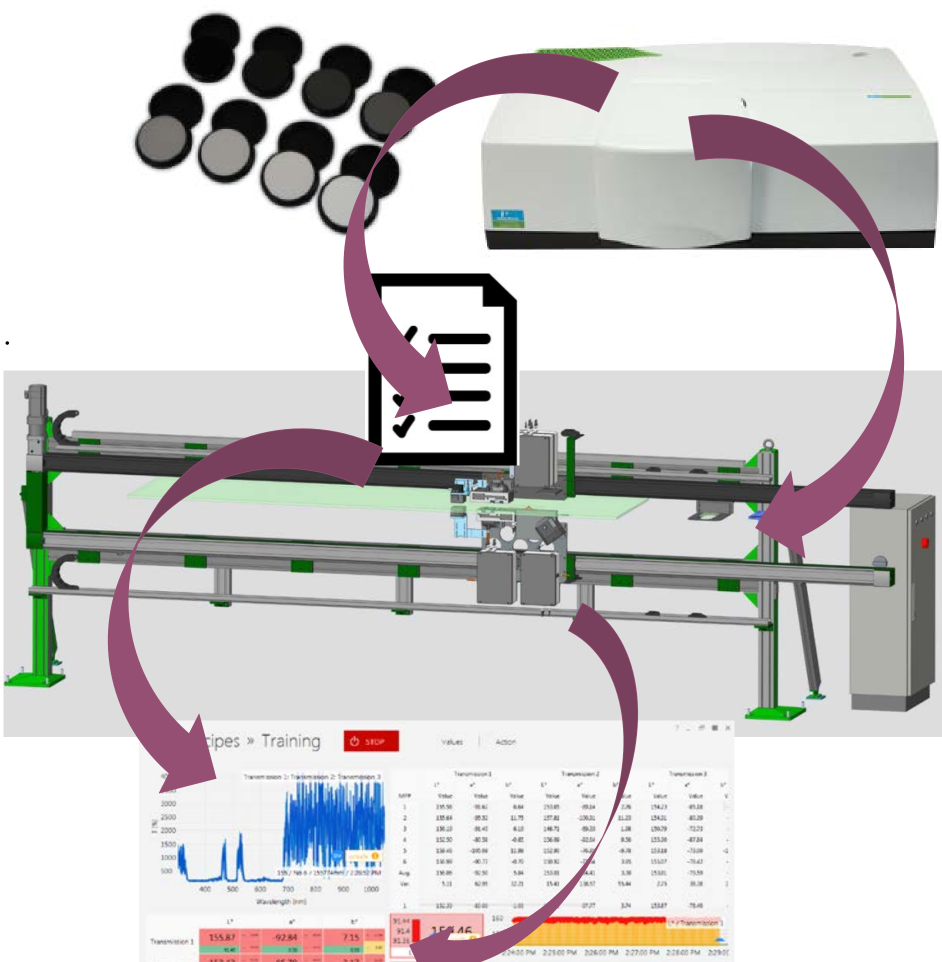


The challenge of moving measurements from the laboratory to the production environment include human and operational factors such

- Robust system
- Ease of use of the software and maintenance of the hardware
- Operator's skills and uptime training

Calibration

Complex calibration chain

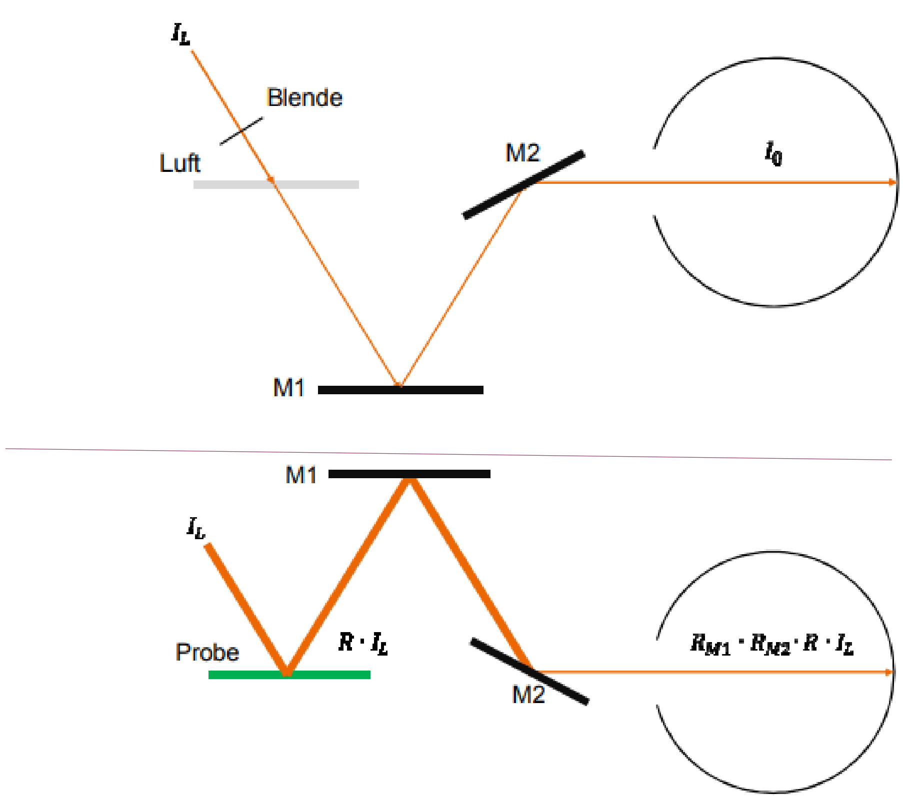
- diffuse or specular calibration standard
- reference instrument / reference method
- certificate file

This chain is usable in lab-instruments; but may be difficult to implement in in-line instrumentation (except traversing solutions)

- in-line instrument calibration
- measurement

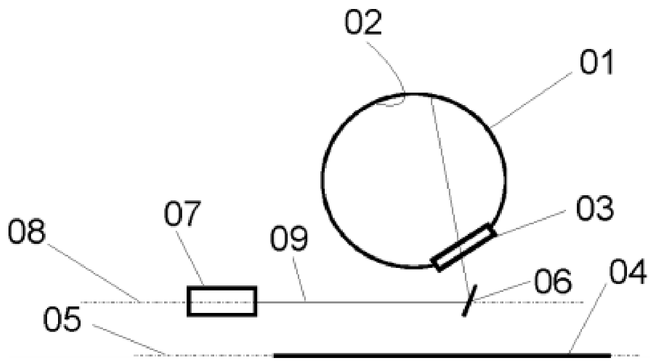
No "national standard" available

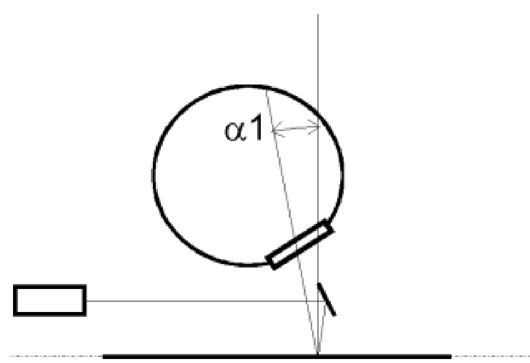
Fraunhofer IOF VN – Add-on for Perkin Elmer Lamda



Using this method we uses the same optical elements for both, calibration and sample measurement path. The Mirror M1 moves across sample plane simulating a moving sample. While this is usable for lab applications it is not usable for in-line applications

ZEISS OFR A10 Configuration

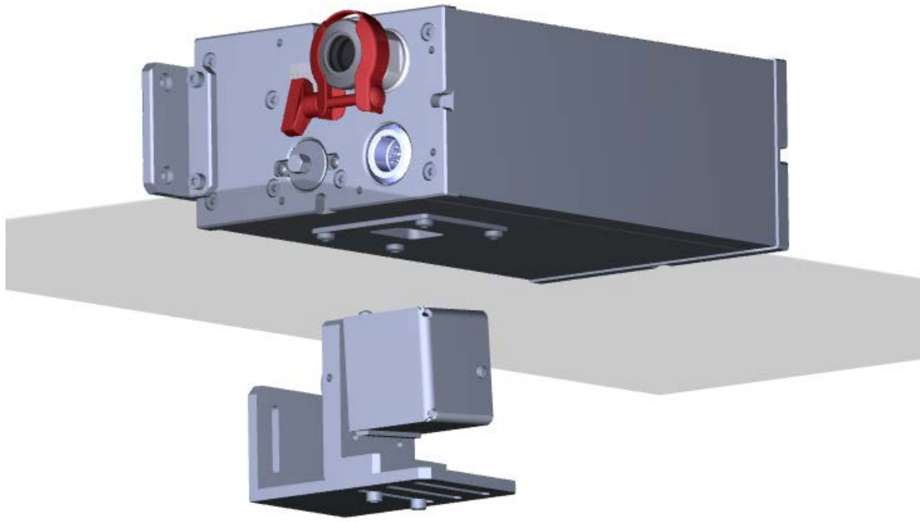




Only one mirror sliding and rotating

- initial position to calibrate for reflectance
- sliding and rotating to measure sample
- Extendable to transmission measurements
- Multiplexing (%R | %T | bypass

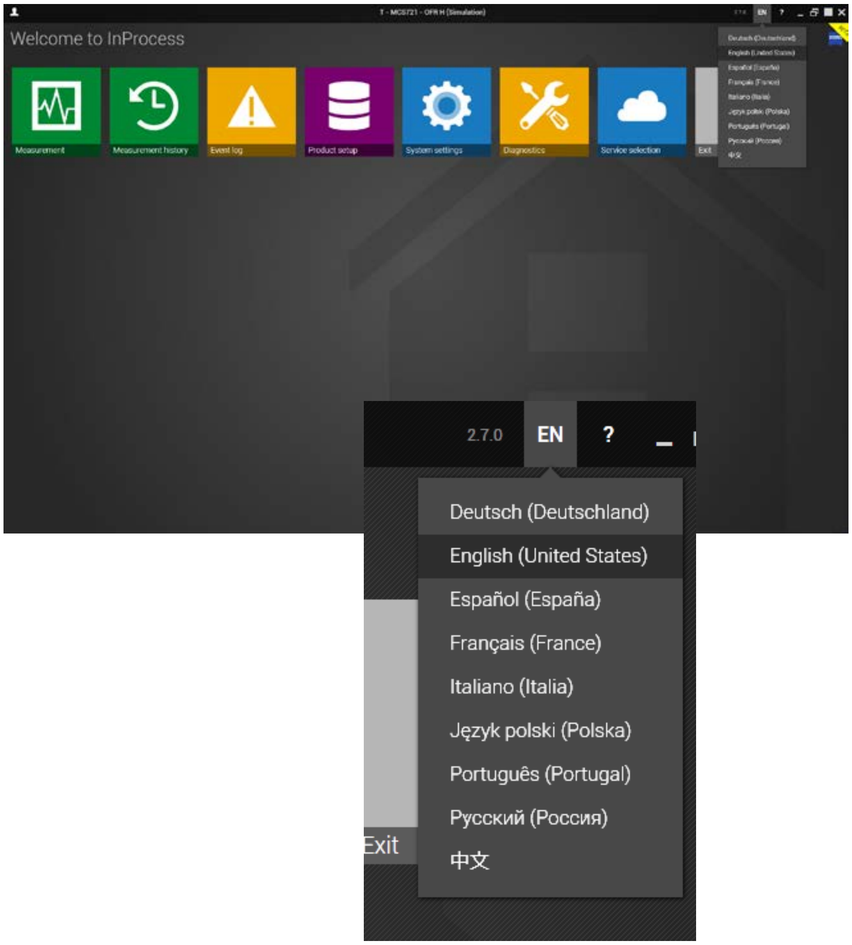
This reference allows the instrument to be standardized due to instrumental drift is a result of contamination, a change in the absolute light intensity, unstable electronics, or adverse environmental effects.



- **Multiplexing by mirror switching spectrometer for:**
  - Reflectivity
  - Transmissivity
  - Bypass channel
  - **Absolute measurements**

- **Continuous In-Line applications**
  - Time and Trigger Based measurements Measured values: Spectral reflectivity and transmissivity (360 nm – 1050 nm / 1650 nm)
  - Derived / calculated values: color metrics (L*a*b*),
  - coating thickness, spectra characteristics (min, max)
  - Time and Trigger Based measurements Measured values: Spectral reflectivity and transmissivity (360 nm – 1050 nm / 1650 nm)

A new paradigm can be realized in optical properties measurement of transparent and opaque plastic materials by utilizing this type of technology incorporated with software to include:

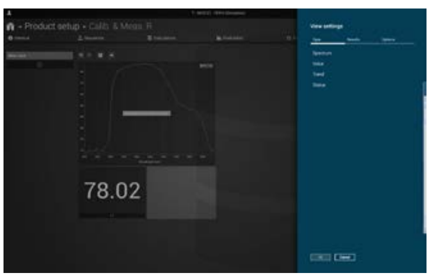


Touch Panel optimized software

- Multilingual
- Recipe Driven
- All configuration done with separated Management Console Program

- Freely configurable Measurement Sequences
- Configurable calculations

- Data and signal transfer to PLC



- Scalable diagrams, result views, trends

The layer thickness is derived from the maxima and minima of the interference spectrum. This method is very accurate and fast, however, noise-sensitive. It is suitable for single layers < 5 µm.

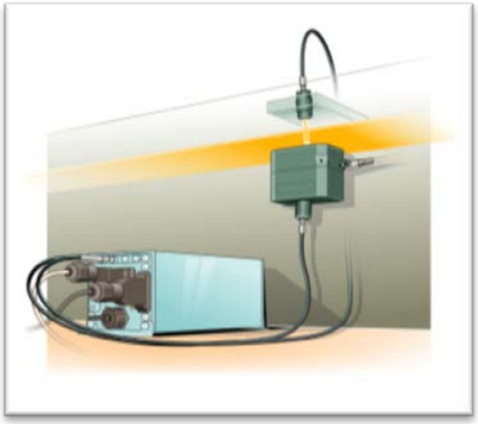
Based on the Hardware and software solution we investigated the following tasks

- Identify type of substrate
- Identify coating
- Identify if the substrate is running on the coated side or uncoated side
- Identify substrate thickness

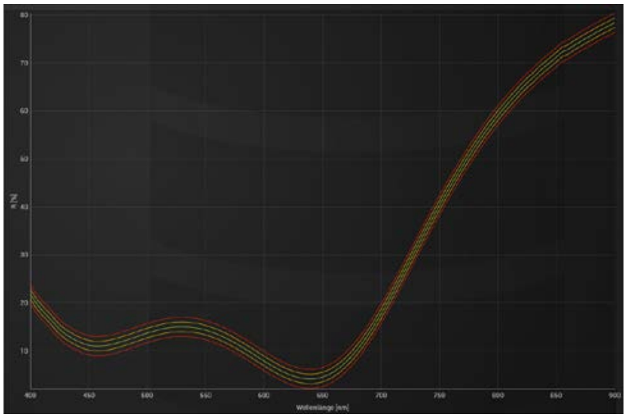
The following assertions are based on a set of samples:

#	Type	Strength	Qty
1	Sample a	SS	2
2	Sample b	DS	2
3	Sample c	TS	2
4	Sample e	SS	2
5	Sample f	DS	2
6	Sample g	TS	2
7	Sample h	SS	2

The tests were performed using a setup with spectral reflectance and transmittance measurement:



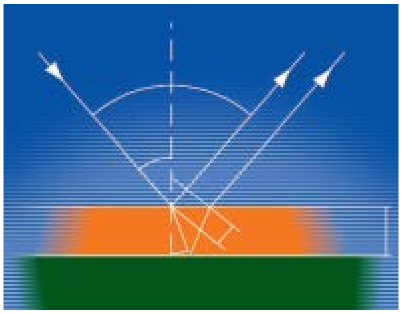
The software is able to decide, if a spectrum is within a given range of %T or %R values:



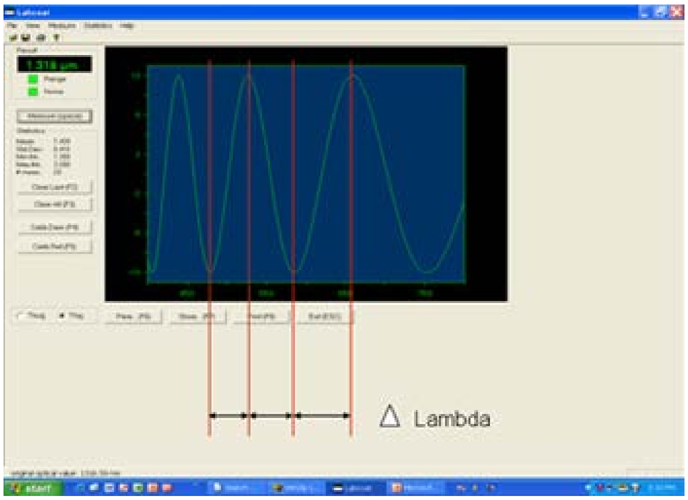
Thus it is possible to define rules to identify product types,

- *White light interference*

The use of white light interference offers an excellent method for measuring layer thickness. The principle is as follows: illuminating the sample with white light results in interference spectrums which depend on the geometric layer thickness and the index of refraction. This is caused by the superposition of the reflection spectrums from the top and bottom sides of the coating. Mathematical methods evaluate this interference in respect of optically transparent layers in the form of optical and geometric layer thicknesses.



- The layer thickness is calculated from the periodicity of the interference spectrum



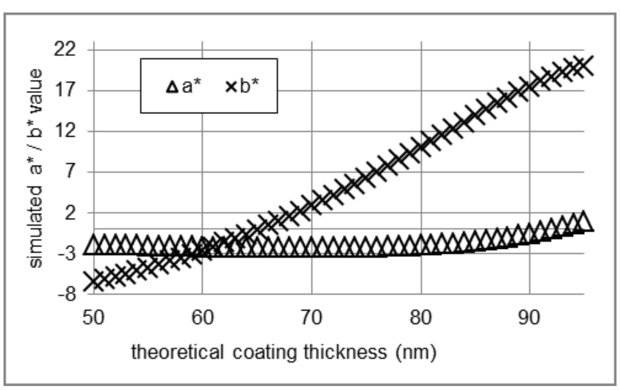
$$2 \cdot d \cdot n_2 = i \cdot \lambda_1 = (i+1) \cdot \lambda_2$$

$$d = \frac{1}{2 \cdot n_2} \cdot \frac{\lambda_1 \cdot \lambda_2}{\lambda_1 - \lambda_2}$$

$$= \frac{1}{2 \cdot 1.49} \cdot \frac{622 \cdot 550}{622 - 550} \; nm$$

$$= 1590 \; nm \approx 1.6 \; \mu m$$

- ***Color as a thickness indicator***
  - Color changes with the layer thickness



- *Model based approaches*

The layer thickness analysis is carried out by an algorithm that is proprietary. The model does not take the interference of the substrate material into account. Therefore, no fringes caused by the 23 µm PET material are modelled in the simulated spectra. The optical and physical parameters of the model are fitted to yield simulated spectra that match the measured spectra. The best match was achieved with a silicone layer thickness of 186 nm.

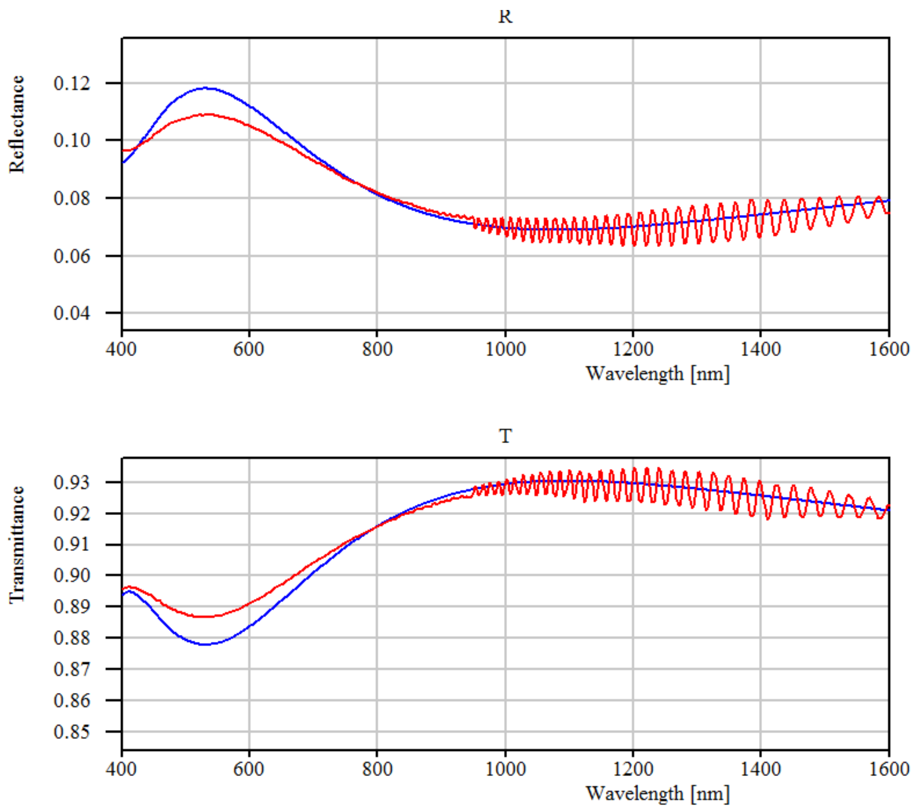


Figure 2: Measured (red) and simulated (blue) reflection and transmission spectra for sample 2. The best match of simulated and measured data was achieved with a silicone layer thickness of 186 nm.

Conclusion

Color as thickness indicator: Photometric accuracy is important for this method

Chemometrics prediction: Will work with non-accurate (but precise) systems as well: **BUT**: The developed methods will only work on the system they are developed with No scaling is possible. In case an instrument or parts of the system needs to be changed, the method needs to be adopted

Peak method: Only wavelength accuracy needed

White light interference: Only wavelength pitch accuracy needed

Recommendations

There is no price that can be put on a customer dissatisfaction. Losing faith in a product or in a company can result in a lost contract, loss revenues and litigation. Poor quality, long lead times, missed deliveries, returns, and rework are some of the quality costs that can significantly impact customer satisfaction and profitability. In manufacturing, there are claims filed against manufacturing. This occurs when the product is shipped outside of the customer specification from the factory. There are also manufacturing claims; that is where the product departs from its intended design even though all possible care was exercised in the prepartion and maketing of the product[ii].

References

[i] ***Chemometrics in Spectroscopy,*** *1st Edition*, Elsevier, Mark & Workman,

[ii] *Torts: Keyed to Prosser/Wade/Schwartz, Eleventh Edition, page*