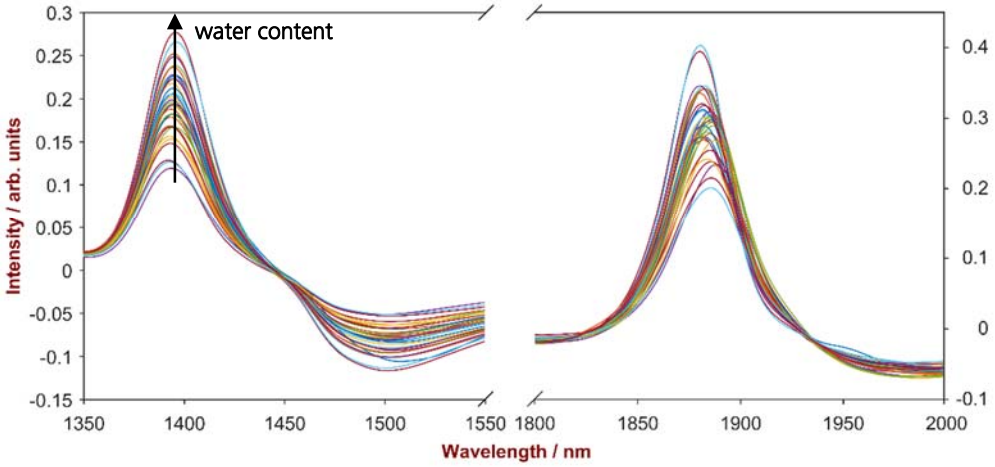
NIR Application Note NIR–055

Determination of water content in moisturizing skin creams using near-infrared spectroscopy



Near-infrared spectroscopy (NIRS) was used as an analysis method for quality control of skin creams. A model for the quantification of the water content was developed based on Karl Fischer titration (KF), enabling fast and reliable at-line analyses and final product quality control.

Metrohm NIRSystems

Method description

Introduction

Creams are applied on the skin of different body parts and can fulfil various tasks. They can be carriers of active ingredients and facilitate the absorption into the dermal layers, but they also normalize the humidity of the skin. Creams can be counted among personal care products, cosmetics or pharmaceuticals. Therefore, creams have to fulfil stringent quality control. One of the most important quality parameters is water content because it contributes to a major extent to the texture, feel and moistening characteristics of creams. The traditional method to determine water content is Karl Fischer titration. The creams need to be dissolved in anhydrous methanol which can be accomplished by heating them in an ultrasonic bath. Dissolving certain creams can be problematic due to their ingredients. In such cases, solvent mixtures (methanol, chloroform, decanol) and solubilizers may be required. Afterwards, the prepared samples are titrated with the Karl Fischer reagent.
In contrast, near-infrared spectroscopy (NIRS) is a rapid, non-destructive analysis method applicable in routine quality control. There is no sample preparation/dissolution step needed. Once a method was developed, the quality of incoming materials, intermediates and final products can be determined in a matter of seconds without sample preparation.

Experimental

The set of samples used in this study consisted of 42 different commercially available creams of several manufacturers (see Fig. 1).

Fig. 1: Moisturizing creams used in this study.

The water content was determined by Karl-Fischer titration in triplicate using the procedure described in the Karl-Fischer application note AN-K-011 [1]. The water content was determined to range from 60% to 80% and these values were used as primary values in the NIRS model development. In order to analyze the samples with NIRS, approximately 0.5 g cream were applied on the NIRS Slurry Cup. The NIRS Diffuse Reflectors, Gold 1 mm was pressed on the sample; creams were analyzed in transflection. The transfection mode works in such way that the light that passes through the sample reflects via the gold reflector back into the detector. The Slurry Cup was placed in the Metrohm NIRS DS2500 Analyzer and the spectra were acquired over the spectral range from 400 nm to 2500 nm (see Fig. 2). After the measurement, the gold stamp and the Slurry Cup were carefully cleaned using dry paper towels.

Tab.1: Used equipment and software.

Equipment	Metrohm code
NIRS DS2500 Analyzer	2.922.0010
NIRS Slurry Cup	6.7490.430
NIRS Diffuse Reflectors, Gold 1 mm	6.7420.000
Vision Air 2.0 Complete	6.6072.208

For data acquisition and developing the quantification method the software package Vision Air 2.0 Complete was used. The instrument management, such as the assignment of the operating procedures was conducted in the office using Vision Air Manager Network, whereas the actual measurements took place in the quality control laboratory using Vision Air 2.0 Routine. The acquired spectra were automatically uploaded to the database, where they were accessible for method development. The quantitative method development was performed in the Vision chemometric software.

Fig. 2: The Metrohm NIRS DS2500 Analyzer was used for spectral data acquisition over the full range from 400 nm to 2500 nm (left) and the used NIRS Slurry Cup and 1mm Gold Diffuse Reflector (right).

Metrohm NIRSystems

Method description

Method development

The analysis results obtained by the primary method (Karl-Fischer titration) in combination with the NIRS spectra were used in the subsequent method development. The spectra were pre-treated using a Detrend function (2nd order polynomial baseline correction) in combination with a 1st derivative (see Fig. 3). The multivariate regression method Partial Least Squares Regression (PLS) was performed over the spectral regions 1350 – 1500 nm and 1800 – 2000 nm, which represent the two most prominent water bands. The spectral variations arising due to concentration changes of moisture were apparent (see Fig. 3).

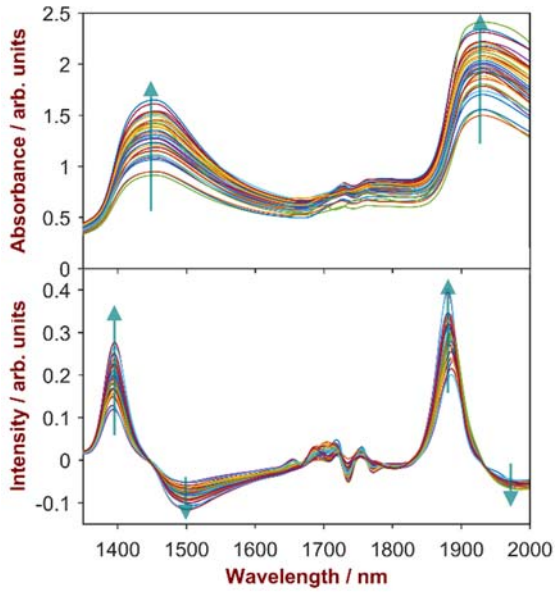


Fig. 3: The raw spectra (upper trace) were pre-treated using a Detrend function with a subsequent 1st derivative (lower trace). The arrows indicate the trend to higher water concentration.

Three samples were identified as outliers. Those samples were more wax-like than creams and did not fit to the other sample population. The remaining samples were split into a calibration set (31 samples) and a validation set (8 samples).

Results

Because of the varying sample matrices (each sample is a unique product with different ingredients), the model was fitted using 4 factors. A Standard Error of Calibration (SEC) of 0.565% and a Standard Error of Cross Validation (SECV) of 0.745% was obtained (see Figure 4). These Figures of Merits of the internal cross validation (leave-one-out method) indicate the model is robust and provides accurate results. The defined validation set was used for external validation, yielding. A Standard Error of Prediction (SEP) of 0.819% was obtained (see Tab. 2).
These results show clearly that NIRS is excellently suited to reliably determine the water content in moisturizing creams and it can be used in quality control for out-of-specification analysis and final product inspection[1].

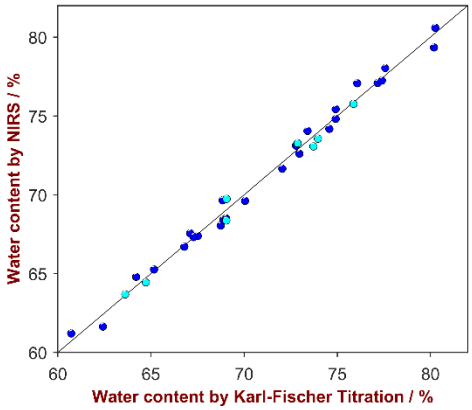


Fig. 4: Correlation plot of the moisture concentration determined by Karl-Fischer titration and predicted by NIRS. The blue marks stand for samples used in the calibration set, the turquois marks are samples used in the validation set.

Tab.1: Results of the quantitative method for water content in skin creams.

Regression model	PLS with 4 factors
Pre-treatment	Detrend, 2nd order polynomial, 1st derivative, 10 nm segment size
Wavelength range	1350–1550 nm 1800-2000 nm
R^2	0.992
SEC	0.565%
SECV	0.745%
SEP	0.819%

[1] Using the full spectral range of the Metrohm NIRS analyzers shines light to even more sample characteristics, such as whether a cream contains UV-block ingredients, and even how much thereof.

Metrohm NIRSystems

NIR Application Note NIR-055
Version 1, first published November 8, 2017

Method description

Conclusion

Vis-NIR spectroscopy can be successfully used for quantification of water content in moisturizing creams. Vis-NIR spectroscopy offers a number of unique advantages over traditional analysis methods: it generates reliable results within seconds without the need for any sample preparation or chemicals and does not generate any waste. The accessories are cleaned easily and are readily available for the next measurement.

References

[1] KF Application Note No. K-11, Water in moisturizing creams (cosmetic products)

NIR Application Note NIR-055
Version 1, first published November 8, 2017

Metrohm NIRSystems