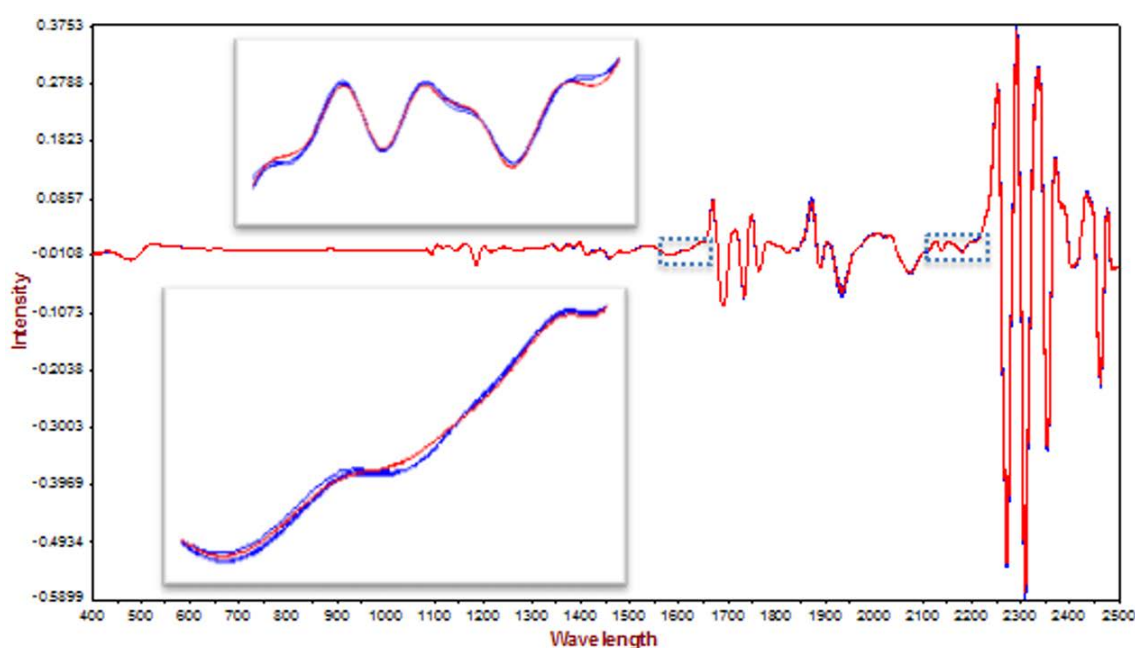


Quality control of an active ingredient in hair spray using near-infrared spectroscopy



Near-infrared spectroscopy (NIRS) was used as an analysis method for quality control of hair spray samples. A model for an active ingredient within hair sprays was developed, enabling fast and reliable out-of-specification analyses.

Method description

Introduction

Hair sprays are cosmetic products used for styling hair. Pump hair sprays mainly contain solvents and copolymers which are distributed upon the hair for fixing the hairstyle. Besides those main ingredients, active ingredients can also be found in the formulation in order to fight dandruff or head lice. The right dosing of such active compounds is crucial in order to guarantee high efficacy. Therefore, control of these quality parameters is necessary and the concentration of the actives has to be determined on a regular basis.

The primary methods to determine active ingredients in liquids is LC-MS or LC-MS/MS. However, this method requires chemicals, thus generating waste, and can take 30 minutes or longer.

In contrast, near-infrared spectroscopy (NIRS) is a rapid, non-destructive analysis method applicable in routine quality control. Using NIRS, the quality of incoming materials, intermediates and final products can be determined in a matter of seconds without sample preparation.

Experimental

The set of customer samples used consisted of 15 hair spray samples of 5 batches. The concentration of the active ingredient varied from 0.0% to 8.4%. Approximately 3 g of sample material was poured in the bottom of the transfection vessel. A gold reflector was pressed onto the sample inside the LiquidKit, then the sample was placed into the DS2500 analyzer (see figure 1). Spectra were acquired on a DS2500 analyzer (see Figure 1) over the full wavelength range from 400 nm to 2500 nm in transfection mode. After approximately 3 g of sample material was poured in the bottom of the transfection vessel, a gold reflector was pressed onto the sample inside the LiquidKit, followed by placing the sample into the DS2500 analyzer for the measurement. The transfection mode works in such way that the light that passes through the sample reflects via the gold reflector back into the detector.

For data acquisition and developing the quantification method the Vision Air Complete software was used.

Tab.1: Used equipment and software.

Equipment	Metrohm code
NIRS DS2500 Analyzer	2.922.0010
LiquidKit for NIRS DS2500	6.7400.010
Vision Air 2.0 Complete	6.6072.208



Fig. 1: The NIRS DS2500 Analyzer was used for spectral data acquisition over the full range from 400 nm to 2500 nm.

The spectra were pre-treated using a 2nd derivation and a Partial Least Squares Regression (PLS) was performed over the spectral regions 1590–1720 nm and 2090–2220 nm. The spectral variations arising due to concentration changes of the active ingredient were apparent (see Figure 2).

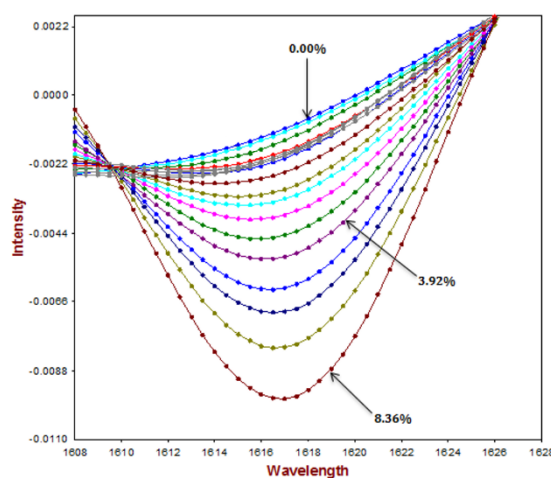


Fig. 2: 2nd derivative spectra of the hair spray samples with various concentrations of active ingredient ranging from 0.0% to 8.4% in the range 1608–1628 nm. The concentration increases from top to bottom. The selected spectral ranges showed distinct changes with concentration. The traces also compare the acceptable lots (grey traces) with the traces of various concentrations (colored).

Method description

Results

Fitting the model with 3 factors yielded a Standard Error of Calibration (SEC) and a Standard Error of Cross Validation (SECV) of each 0.034% (see Figure 3).

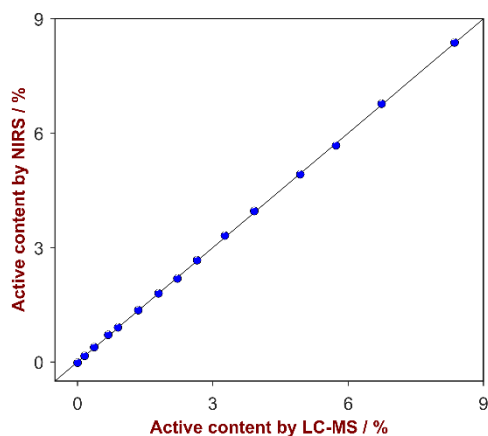


Fig. 3: Correlation plot of the active ingredient's concentration determined by LC-MS and by NIRS.

These results show that NIRS is excellently suited to reliably determine the concentration of active substances in hair sprays and it can be used in routine quality control for out-of-specification analysis. Thus, NIRS relieves time intensive and waste generating primary analysis methods, such as LC-MS.

Summary

Vis-NIR spectroscopy can be successfully used for qualification and quantification of active ingredients in hair sprays. Therefore, it can be used as a high throughput quality control analysis method to distinguish between the good lots (with acceptable concentrations of the active ingredient) and the bad lots (containing too high or too low active ingredient concentration). Vis-NIR spectroscopy solutions offer a number of unique advantages over traditional analysis methods, most notably: it generates reliable results within seconds, it does not need any sample preparation, it generates no waste and its non-destructive nature allows the sample to be re-used.

Although the work described herein has been performed on a DS2500 solid analyzer with the Transflection Kit, an alternative is the use of the XDS RapidLiquid Analyzer with sample measurement in the transmission mode. The advantage of that instrument is that it allows the use of disposable glass vials, which means that the measurement window of the DS2500 LiquidKit does not need to be cleaned after each measurement.