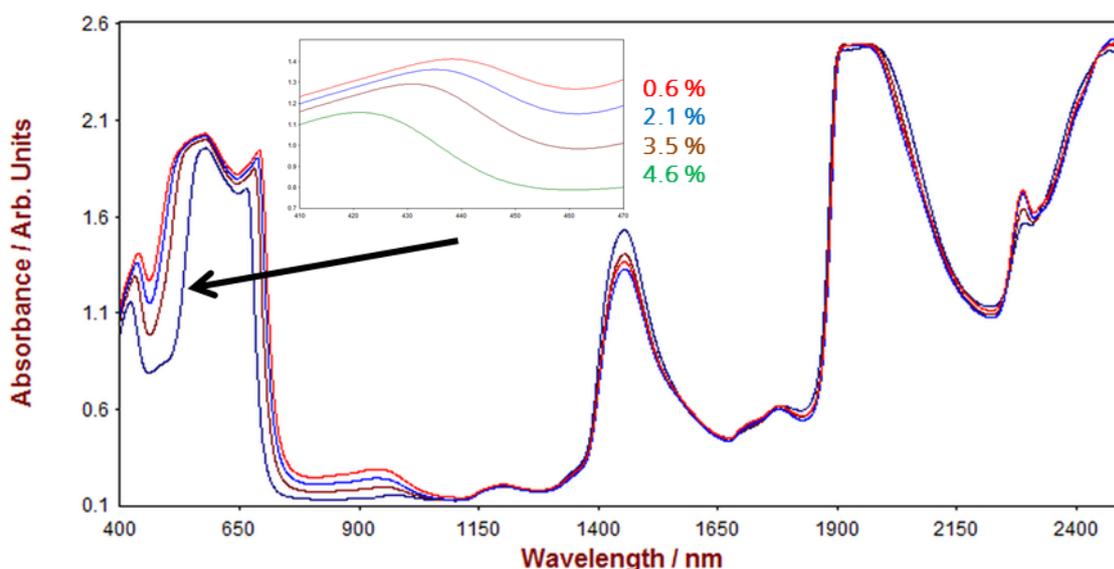# Dye, diethylene glycol, water, and surfactant content in ink

This Application Note shows that Vis-NIR spectroscopy can be used to quantify four important parameters – dye, diethylene glycol (DEG), surfactant and water content – of the ink industry. The determination of these parameters, commonly achieved using time-consuming wet chemistry methods, can be easily performed by Vis-NIR spectroscopy. The latter measures all parameters of interest in a single measurement and can be performed even by untrained users.

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Method description

Introduction

Even in times of digital media, handwriting and signing using ink pens is still popular. In former times, fountain pens were most commonly used, but are nowadays largely replaced by ball pens. While ink for fountain pens is purely based on water and dyes, ball pen ink contains multiple ingredients to produce the desired behavior. The dye (e.g., triphenylmethanphenazin or azo dyes) gives the ink its color, diethylene glycol (DEG) is used as a solvent and prevents clogging and drying out, and surfactants control the texture and prevent the ink from foaming. Monitoring the concentrations of additives is of high importance to ensure a constant product quality. The determination of these parameters, commonly done by time-consuming wet-chemistry methods, can also be achieved by Vis-NIR spectroscopy. The latter determines all parameters of interest within a single measurement and can be operated even by untrained users.

Configuration

The following equipment was used (**Tab. 1/Fig.1**).

Tab. 1:

Equipment	Metrohm order number
NIRS DS2500 Analyzer	2.922.0010
NIRS transflection vessel, optically flat	6.7401.000
NIRS gold diffuse reflector, 1 mm total path length	6.7420.000
Vision 4.03 Software	6.6069.102

Fig. 1: NIRS DS2500 analyzer equipped with liquid sample transflection

Experimental

A NIRS DS2500 analyzer (transflection measurement) was used to collect the spectral data of 20 blue inks for ball pens. The samples were placed in an optically flat quartz glass vessel and covered with a gold reflector of 1 mm optical path length.

Metrohm's Vision chemometric software was used to develop quantitative prediction models for four parameters (**Tab. 2**). The prediction model was tested on 8 independent samples.

Tab. 2:

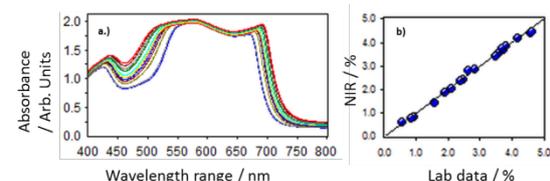| Parameter	Concentration range [%]
Dye content	0.6– 4.6
DEG content	12.5–29.8
Surfactant content	0.0–1.0
Water content	63.6–82.1%

Results

Dye content

Fig. 2: a.) Spectra of 20 blue ink samples with varying dye content from 0.6 to 4.6% over the wavelength range of 400–800 nm. b.) Correlation of reference data to values predicted by NIR using the newly developed quantitative method.

Math pretreatment	-
Regression model	PLS with 2 factors
Wavelength range	420–800 nm
R^2	0.9961
SEC	0.0835
SEV	0.0949
F-value	2056
PRESS	0.171

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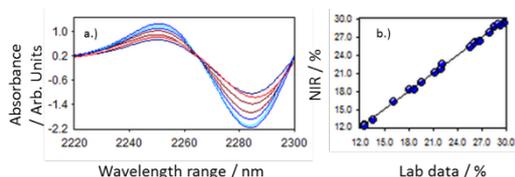# Method description

DEG content:

Fig. 3: a.) Spectra of ink samples with DEG contents varying from 12.5% to 29.8% over the wavelength range of 2220–2300 nm which were used to create a quantitative prediction model for DEG content. b.) Correlation of reference data to values predicted by NIR using the newly developed quantitative method.

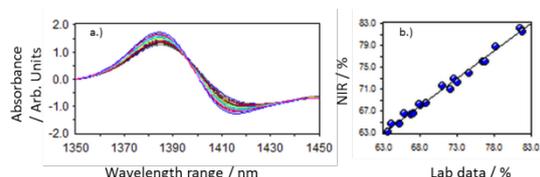| Math pretreatment	2nd derivative + SNV
Regression model	PLS with 2 factors
Wavelength range	2220–2300 nm
R^2	0.9934
SEC	0.5037
SEV	0.5888
F-value	1210
PRESS	6.5865

Water content:

Fig. 5: a.) Wavelength (1300–1550 nm) for the creation of a quantitative prediction model for water content varying from 63.6–82.1%. b.) Correlation of reference data to NIR predicted values as a result of quantitative method development.

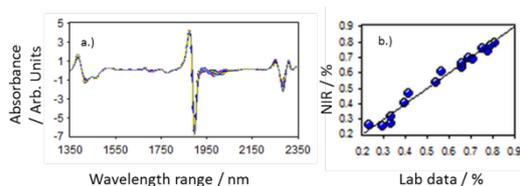| Math pretreatment	2nd derivative
Regression model	PLS with 3 factors
Wavelength range	1300–1550 nm
R^2	0.9909
SEC	0.5571
SEV	0.9614
F-value	546
PRESS	17.5618

Surfactant content:

Fig. 4: a.) Wavelength (1350–2350 nm) for the creation of a quantitative prediction model for surfactant content varying from 0.0–1.0%. b.) Correlation of reference data to NIR predicted values as a result of quantitative method development.

Math pretreatment	2nd derivative
Regression model	PLS with 5 factors
Wavelength range	1350–2350 nm
R^2	0.9774
SEC	0.0368
SEV	0.1316
F-value	86
PRESS	0.2772

External validation:

Sample ID	Dye [%]	DEG [%]	Surfactant [%]	Water [%]
1	-	-	0.95	74.03
2	-	-	0.69	73.17
3	-	-	0.82	81.32
4	-	-	0.85	73.50
5	4.20	20.82	-	-
6	4.19	20.39	-	-
7	2.52	13.81	-	-
8	2.48	19.17	-	-

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