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Miniaturized MIR and NIR Sensors for Medicinal Plant Quality Control
Christian W. Huck
This work shows that methods based on miniaturized near- and mid-infrared spectroscopy can be used effectively for the quality control of herbal medicines.

Tracking Microplastics in the Environment via FT-IR Microscopy
Michael Bradley, Suja Sukumaran, Steven Lowry, and Stephan Woods
Microplastics from clothing, abrasive action on plastics, or engineered microbeads as found in some exfoliating cosmetics are showing up in many environmental systems. FT-IR microscopy is a useful tool in the analysis of microplastics, providing visual information, particle counts, and particle identification.

Assessing False Noncompliance and False Compliance with Limits Stated for Physicochemical Parameters in Red Wines Using a MIR-PLS Model
M.C. Ortiz, L.A. Sarabia, M.E. Meléndez, and M.S. Sánchez
When analytical techniques that depend on multivariate calibration models are used to analyze commercial products, the risks of false compliance and noncompliance must be assessed. This study presents an example from the application of a mid-IR partial least squares model to the analysis of seven parameters in red wine.

Solving Polymer Problems Using IR Spectroscopy
An interview with Naoto Nagai
Naoto Nagai focuses on solving problems for industry. In this interview, he explains his research to determine the cause of resin cracks in polyoxymethylene mold plates using IR spectroscopy.

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Miniaturized MIR and NIR Sensors for Medicinal Plant Quality Control

The trend in analytical method development follows two different strategies. One is the further improvement of spectrometers toward higher performance; the other is to develop small, portable, and relatively cheap instruments that can be easily used by anyone. Today, small near-infrared (NIR) sensors are either based on microelectromechanical systems (MEMS) or linear variable tunable filters (LVTF) for appropriate wavelength selection. For measurement in the mid-infrared (MIR) region, handheld attenuated total reflection (ATR) spectrometers give great promise based on the improved signal-to-noise ratio enabled by the evanescent field principle. Both instrumental technologies can be used to monitor quality and determine the ideal harvest time of medicinal plants, such as *Verbena officinalis* and *Rosmarini folium*. For method development, quantum chemistry for band assignment and two-dimensional correlation spectroscopy for a better understanding of the factors that determine the partial least squares (PLS) regression models can be conducted. From these studies it can be shown that the performance of NIR and MIR spectroscopy with miniaturized devices as a fast and noninvasive technique is able to replace time- and resource-consuming analytical tools, being of high interest for the continuous growing phytopharmaceutical industry and its quality control.

Christian W. Huck
mation extracted from overlapping peaks with NIR intensities that are 10–1000 times lower (5). The further development of portable and handheld NIR spectrometers based on microelectromechanical systems (MEMS) (6) and linear variable tunable filters (LVTF) (7) is making NIR spectroscopy very popular. Attenuated total reflection (ATR) is the most suitable form of applying MIR spectroscopy, benefiting from the evanescent field and penetrating at least a few micrometers into the sample of interest providing high signal-to-noise ratio (S/N) (8). Two-dimensional (2D) spectroscopy, first proposed by Noda in the 1980s (9), has been further developed (10) to be applied for quality control in food and agricultural products (11). Defined by two independent spectral axes, the 2D correlation spectrum is generated by applying correlation analysis to the dynamic fluctuations of spectral signals caused by external perturbations, for example, chemical, thermal, or mechanical stimulations. One notable feature of 2D correlation spectroscopy (2D-COS) is that the cross-peaks can potentially be used to characterize intermolecular interactions. The advent of 2D-COS brings about a new avenue for the investigation of intermolecular interactions.

In many applications it is impossible to correctly assign the corresponding vibration bands. Quantum chemical calculations are a great method for band assignment not only in the MIR, but also in the NIR region (12). Vibrational spectroscopy with multivariate statistical analysis (MVA) is a powerful, synergistic combination enabling the extraction of the required information from the spectrum in the first step (13,14). Clustering methods such as principal component analysis (PCA), hierarchical cluster analysis (HCA), fuzzy-C-means clustering, and others enable the reduction of the number of variables enabling qualitative analysis (15). For the establishment of quantitative regression models, partial least square regression (PLSR) (16) is the preferred method, correlating spectral data with those obtained from reference methods such as liquid.

Figure 1: Schematics showing (a) microelectromechanical system (MEMS) and (b) linear variable tunable filter (LVTF) construction.
chromatography (LC) (17) and LC–mass spectrometry (MS) (18), solid-phase extraction (SPE) (19,20), gas chromatography (GC) (21), capillary electrophoresis (CE) (22), capillary electrochromatography (CEC) (23), and MS (24).

In this article, the potent combination of these methodologies as a novel strategy in medicinal plant analysis is introduced and summarized for the highly efficient quality control of Verbena officinalis (25) and Rosmarini folium (26), two typical plants used in folk medicine.

**Methods**

**NIR Spectroscopy**

The inexorable trend toward the miniaturization of spectrometers has been continuing since the 1970s. The development of one of the smallest NIR spectrometers, with a current weight of only 60 g, was enabled by introducing MEMS in 2008 (Figure 1a) and LVTF in 2012 (Figure 1b) (27,28). A MEMS device is made up of components 1–100 μm in size, and it generally ranges in size from 20 μm to 1 mm (29). The active circuit contains upper and lower mirrors acting as wavelength-tunable parallel plates: The width of the air gap controls the resonant frequency of the cavity and allows certain wavelengths to pass. The upper mirror’s movement is controlled through an applied voltage. Applying different voltages results in transmitting a spectrum of wavelengths to be detected by the InGaAs PIN photodiode (30). This technology was added to smartphones in 2014; remote systems using wireless hyphenation to smart computers were created in 2016 (31).

LVTF are small-wedged Fabry-Perot etalons manufactured in a thin-film optical coating process. The bottom layer is made of several Bragg reflectors forming a dielectric mirror. The next layer forms the cavity of the resonator. Atop this resonator, a second layer of Bragg reflectors is applied as the upper dielectric mirror (7,32,33).

**2D-COS**

This method offers a higher resolution where even strongly overlapping peaks can be distinguished and explicitly assigned (9). Applying a second dimension enables the interpretation of the sequential order of intensity change and offers the possibility of correlating different spectroscopic methods via hetero-spectroscopic correlation. For the generation of the needed dynamic data sets of spectra an external perturbation, such as temperature, time, or pressure, should be applied during the IR measurements. The following correlation analysis generates a synchronous and an asynchronous 2D spectrum (10). While the synchronous spectrum features so-called auto-peaks along the diagonal and the sign of its cross-peaks indicates a rise or fall in intensity, the asynchronous spectrum lacks in auto-peaks, but its cross-peaks provide the information of the sequential order of the intensity change.

**Quantum Chemical Calculations**

The generalized second-order vibrational perturbation (GVPT2) theory approach on a density functional theory (DFT) level for the calculation of the NIR spectrum was used, with the aim of obtaining a better explanation of the observed experimental patterns. For small molecules in a diluted phase this theoretical approach gives excellent results (37).
Results and Discussion
Application of Benchtop and Portable NIR for Predicting the Optimum Harvest Time of Verbena officinalis

The applicability of NIR spectroscopy to determine the ideal harvest time of Verbena officinalis was demonstrated (25). As a reference method a novel high performance liquid chromatography–diode array detection–electrospray ionization-mass spectrometry (HPLC–DAD–ESI-MS) method was developed and validated. HPLC analysis of the leaf drug showed that the content of the quality-determining compound verbenalin fluctuated throughout the flowering period. Verbascoside as a further pharmacologically important ingredient increased from the middle of flowering, but was also subjected to certain variations. The causes of these fluctuations can have various reasons, such as insolation, humidity, fertilization, and soil

<table>
<thead>
<tr>
<th>Table I: PLS regression results for benchtop referred to the fresh and dried plant material*</th>
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<tr>
<td><strong>Verbenalin</strong></td>
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<tr>
<td></td>
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<tr>
<td>CV</td>
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<td>TSV</td>
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<tr>
<td><strong>Verbascoside</strong></td>
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<td></td>
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<td>CV</td>
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<td>TSV</td>
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</tbody>
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*Unit of SEP and range is %, $R^2$ refers to validation, in TSV SEP $\equiv$ SECV.

<table>
<thead>
<tr>
<th>Table II: PLS regression results for MEMS (miniaturization) referred to the fresh and dried plant material*</th>
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<tr>
<td><strong>Verbenalin</strong></td>
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<tr>
<td></td>
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<tr>
<td>CV</td>
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<tr>
<td>TSV</td>
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<tr>
<td><strong>Verbascoside</strong></td>
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<tr>
<td>CV</td>
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<td>TSV</td>
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*Unit of SEP and range is %, $R^2$ refers to validation.
Table III: Band assignments in NIR spectrum of rosmarinic acid, based on quantum chemical (DFT-B3LYP/N07D) calculation

<table>
<thead>
<tr>
<th>Wavenumber (cm$^{-1}$)</th>
<th>Major Contributions</th>
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<tr>
<td>Exp.</td>
<td>Calc.</td>
</tr>
<tr>
<td>1</td>
<td>6854.9</td>
</tr>
<tr>
<td>2</td>
<td>6767.2</td>
</tr>
<tr>
<td>3</td>
<td>~6680</td>
</tr>
<tr>
<td>4</td>
<td>~6044</td>
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<tr>
<td>5</td>
<td>5986.5</td>
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<tr>
<td>6</td>
<td>5929.7</td>
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<td>7</td>
<td>5752.5</td>
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<td>8</td>
<td>5128.0</td>
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<td>9</td>
<td>5075.8</td>
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<tr>
<td>10</td>
<td>4994.9</td>
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<tr>
<td>11</td>
<td>4923.8</td>
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<td>12</td>
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<td>4788.3</td>
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<td>15</td>
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<td>16</td>
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<td>17</td>
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<td>18</td>
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<tr>
<td>19</td>
<td>4233.3</td>
</tr>
<tr>
<td>20</td>
<td>4179.4</td>
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Abbreviations: aliph: moiety connected to aliphatic chain; ar: moiety connected to aromatic ring
condition. For these reasons, the development of a quick and noninvasive NIR method was essential for the determination of the optimum time to harvest *Verbena officinalis*.

PLS regression results obtained from the benchtop polarization interferometer–based FT-NIR spectrometer demonstrated that NIR spectroscopy is suitable for the characterization of *Verbena officinalis* (see Table I). PLS results of cross and test set validation models for verbenalin and verbascoside reached $R^2$ values ranging from 0.82 to 0.85. A maximum of five latent variables (LVs) was needed. The standard error of prediction–standard error of calibration (SEP–SEC) quotient achieved values near to 1 (0.8–1.2) attesting to the robustness of the developed models. The residual predictive deviation (RPD) was $>2$, confirming a satisfactory quality. A quick and simple determination of the ideal time to harvest *Verbena officinalis* was possible.

To allow a laboratory-independent applicability of the NIR analysis, the portable MEMS was evaluated for its performance. Regression results for verbascoside (see Table II) reached an $R^2$ of 0.8. A maximum of four LVs was required. The SEP–SEC ratio ranged between 0.95–1.09 and the RPD values from 2.21–2.23, which indicate the robustness and quality of the developed PLS models. The results of this study clearly revealed that the prediction of verbascoside applying the portable device could be performed with nearly the same accuracy as with the benchtop spectrometer. PLS models for verbenalin were less precise showing a value for $R^2$ between 0.69 and 0.75 and RPD values < 2. Therefore, the quality of the calibration using the handheld device was not satisfactory. Reproducible quantifications of verbenalin applying NIR spectroscopy are quite feasible as the aforementioned study using the benchtop device has shown. The low resolution and the limited spectral range of the mobile NIR device seem to be the responsible spectral features necessary for a successful quantification of verbenalin that were not sufficiently captured. Therefore, the application of a portable spectrometer with a different spectral range and

<table>
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<th>Table IV: Results of all PLS regression models</th>
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<tr>
<td>Spectrometer</td>
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<tr>
<td>Samples</td>
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<tr>
<td>Outliers</td>
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<tr>
<td>Validation method</td>
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<td>$R^2$</td>
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<tr>
<td>SECV (%)</td>
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<td>SEP (%)</td>
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<td>Factors</td>
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<td>RPD</td>
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higher resolution may lead to further improved calibration models.

**Evaluation of Spectral Information of Benchtop Versus Portable Spectrometers for the Quality Control of *Rosmarini folium***

The theoretical NIR spectrum of rosminic acid (RA) allowed a detailed band assignment for the molecule throughout the NIR region (8000–4000 cm$^{-1}$), which is presented in Table III (26).

For the comparison of benchtop, MEMS, and LVTF devices, the mea-

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**Figure 2:** 2D spectra obtained using (a) a benchtop system (NIRFlex N-500), (b) a MEMS-based system (microPHAZIR), (c) an LVTF system (MicroNIR 2200) with the pretreatment of the moving average transform, (d) an LVTF system without the pretreatment of the moving average transform, (e) hetero-correlation between the benchtop system ($x$-axis) and the LVTF system ($y$-axis), and (f) heterocorrelation between the benchtop ($x$-axis) and MEMS ($y$-axis) systems. The $x$- and $y$-axes are wavenumbers (cm$^{-1}$).
sured spectra of Rosmarini folium were investigated by syn 2D-COS plot using MSC pretreated spectra. Figure 2a shows the syn spectrum of the entire range of the benchtop FT-NIR system (NIRFlex N-500, Buchi Corp.), which is compared to the spectrum of the MEMS system (microPHASIR, Thermo Fisher Scientific) in Figure 2b. The major discrepancies are the peaks at 4678 cm\(^{-1}\) (region of appearance of \(\delta_{\text{CH}^+}\nu_{\text{O}H}\) bands of RA) and 4785 cm\(^{-1}\) (\(\nu_{\text{C}C^+}\nu_{\text{O}H}[\text{ar}], \delta_{\text{C}CH^+}\nu_{\text{O}H}[\text{carboxyl}], \delta_{\text{C}H[\text{ar}]}\nu_{\text{O}H[\text{car}]}) that appear in the 2D plot of the miniaturized device. Although these bands are unnecessary for the benchtop-based analysis, it is a crucial region for the calculation of an expedient PLS regression model for MEMS. Between 5600 and 6000 cm\(^{-1}\) (\(\delta_{\text{C}CH^+}\nu_{\text{O}H[\text{carboxyl}]}\) and \(\nu_{\text{asCH}_{2}^+}\nu_{\text{asCH}_{2}^+}\)), two peaks in the spectrum of the benchtop device are accompanied by one peak of the MEMS device. The conclusion of no further significant differences can be supported by the application of a hetero-2D-correlation between the spectra of the benchtop (x-axis) and the MEMS device (y-axis) (Figure 2f). By comparing the benchtop device (Figure 2a) with the LVTF system (MicroNIR 2200, Viavi Solutions) (Figure 2c), the significant differences are identified as the negative crosspeaks of the LVTF system in the range of 5400 to 7300 cm\(^{-1}\) (\(2\nu_{\text{OH}}[\text{ar and carboxyl}], 2\nu_{\text{CH}_{2}^+}, \nu_{\text{asCH}_{2}^+}\nu_{\text{asCH}_{2}^+}\)) that appears positive in the 2D benchtop system spectrum. If this result is investigated with a hetero-2D-correlation, these opposite signs are shown in the resultant spectrum as well (Figure 2e). Hence, the negative crosspeak in the mentioned range of the LVTF system at the y-axis signals a contrary behavior to the crosspeak of the benchtop device at the x-axis. In accordance with the homo-2D-correlation, the hetero-2D-correlation shows no other difference between the two devices.

In Figure 2d the 2D spectrum without moving average transform is illustrated, while Figure 2c includes the pretreatment. This shows that 2D-correlation provides an explanation about why moving average transform was a necessity for an improved PLS model calculation.

The results of the PLS regression models were ranked because of RPD and \(R^2\), respectively, evaluated by coefficient of variation (CV) (see Table IV). The best NIR-PLS regression model was achieved by the benchtop NIR spectrometer (RPD = 3.27; \(R^2 = 0.91\)), which covered the widest spectral range (10,000–4000 cm\(^{-1}\)) with the highest resolution (\(\Delta\nu = 8\) cm\(^{-1}\)) compared to the other two NIR devices. The miniaturized LVTF NIR spectrometer was the second best (RPD = 2.46; \(R^2 = 0.84\)), performing satisfactorily. The performance of the miniaturized MEMS NIR spectrometer (RPD = 1.88; \(R^2 = 0.73\)) can be improved.

Conclusions

Miniaturized spectrometers show great promise for quality control in the phytopharmaceutical industry. The evaluation of the results leads to the conclusion that it is necessary to prove the applicability of NIR devices in dependence on recording wavenumber ranges and resolution.
References


Continued on Page 32
Tracking Microplastics in the Environment via FT-IR Microscopy

Microplastics are particulates, roughly 20–1000 μm in size, originating from materials such as clothing, abrasive action on plastics, or engineered microbeads as found in some exfoliating cosmetics. The microplastics enter aquifers where the particles can be consumed by filter feeders. Microplastics are chemically stable, giving them a long lifetime in the environment and making excretion or digestion difficult. Analytically, the size and polymeric nature of microplastics makes Fourier transform infrared (FT-IR) microscopy an ideal tool for detection and identification. Standard analyses typically start with a filtration step, extracting the material from the matrix. The analysis can proceed directly on the dried filter without further sample preparation. This simplicity in both sampling and analysis enables the rapid assessment of microplastic encroachment and can assist in the development of remediation techniques. We show examples from both prepared and field samples using microattenuated total reflection (ATR) FT-IR.

Michael Bradley, Suja Sukumaran, Steven Lowry, and Stephan Woods

Macroscopic plastics are showing up in many environmental systems, including mid-Pacific zones (1) and tropical islands (2). Microplastics, 20–1000 (or 5000) μm fibers or granules of polymer, are more insidious because they can’t be picked up simply as trash. Microplastics originate both from engineered materials, such as the microbeads found in some exfoliating creams, and from abrasion or wear of polymeric materials, even from laundering of synthetic fabrics (3). Microplastics live a long time in the environment and unfortunately they are exactly the right size for filter feeders to consume them. From there, they can move up the food chain and are now found in fish, birds, and other wildlife.

The microplastic materials are small enough to be highly mobile in the environment, carried most easily by flowing water. Adsorption of chemical or biological toxins on the microplastics can then enable transport of those toxins from one
biome to another (mobilizing materials). More directly, the microplastics clog critical digestive pathways. The chemical environments in the digestive paths are insufficient to dissolve these clogs, resulting in incapacitation or death of the organisms (3,4).

Remediation requires answering two critical questions: What are the particles and how many particles are

Figure 1: Fiber on filter from a river water sample. The top spectrum was collected (Ge-ATR) directly using a Nicolet iN5 microscope; the lower spectrum is a library search result.

Figure 2: Two particles from water samples, again using Ge-ATR, with search results.
present (number density)? Polyethylene, polypropylene, polystyrene, and nylon are some of the more commonly found materials, from food packing, toys, and many other sources. The number density varies from negligible in isolated lakes and streams to severe in some lakes and estuaries. Changes in the microplastic population (type or number) indicate a new set of conditions, such as those generated by flooding which augments transport of these materials over a large area.

In the aquifer, simple filtration (sieves or simple filters) is typically sufficient to isolate representative populations of microplastics. Sampling within an organism requires separation of the microplastics from the organism, such as the digestive tract of fish (4). This generates a solution that is then filtered. Observation of these samples under a standard light microscope can lead to particle counts, but identification of the materials using visible microscopy alone is problematic at best. Microplastics have been analyzed by gas chromatography–mass spectrometry (GC–MS), scanning electron microscopy with energy dispersive spectroscopy (SEM–EDS), and combustion analysis. Each of these techniques has strengths and weaknesses—sensitivity, specificity, time for analysis, and destruction of the sample being considerations. However, vibrational spectroscopy, both Fourier transform infrared (FT-IR) and Raman, can provide insights quickly and nondestructively with a high degree of confidence.

FT-IR microscopy is an excellent tool for the analysis of these materials. Filtration of a known volume of
liquid (river water, for instance) followed by infrared identification and particle counting provides a thorough picture of the material present. Modern software automates many of the steps, enabling an analyst to answer those critical questions quickly and efficiently. This article starts with the basics of sample collection and single-particle identification and then examines techniques for the analysis of larger regions. We present data from both model systems using manufactured microbeads and real world filtrates.

**Experimental**

Samples prepared from both reference materials and environmental sources were examined. The primary goal of the work was to demonstrate the utility of FT-IR for this analysis; no extrapolation in a particular environment was made. Recently, we examined samples from the automotive, food, and cosmetics industries as well as environmental samples; here only a brief overview is provided.

Single particles were targeted and analyzed using the Thermo Scientific Nicolet iN5 FT-IR microscope, a point-and-shoot, small frame, manually operated FT-IR microscope. Larger images and a particle analysis were carried out on the automated Thermo Scientific Nicolet iN10 FT-IR microscope with OMNIC Picta software. Both microscopes used a single element MCT-A liquid-nitrogen-cooled detector for speed, although a room temperature detector would be sufficient for the point and shoot studies. All data shown in this paper were collected using germanium-attenuated total reflection (Ge-ATR) modes, though we have successfully used both reflection and transmission in other studies. Identifications were made using commercial libraries and standard searching or the OMNIC Specta multicomponent search.

Figure 4: Visual image from filter showing particles (polyethylene beads). The image is a mosaic of over 200 individual images.
Results and Discussion

Most environmental studies of microplastics involve filtration of a liquid sample (typically but not always aqueous), followed by drying. This results in a sample that can be placed directly into the microscope—there is no need to pick off pieces for analysis. The visible capabilities of FT-IR microscopes enable location and selection of a particle or a region for analysis.

Four point-and-shoot examples are shown in Figures 1–3. The fiber or particle was located visually and then centered in the aperture. The Ge-ATR accessory was then brought into contact with the particle. With Ge-ATR, there is a fourfold increase in magnification (because of its high index of refraction). The FT-IR microscope has a 10× magnification; with the Ge-ATR accessory the magnification increases to 40×. The standard 1-mm circular pinhole thus results in an effective aperture at the sample of 25 μm. As most microplastics are this size or larger, this ensures good spectral purity.

The fiber in Figure 1 was filtered from a river sample. The spectrum matches with acrylic (the strong nitrile peak at 2243 cm⁻¹ because of

![Figure 5: An IR image constructed by profiling the IR data from the sample in Figure 4. (a) Spectra of the beads and the filter paper. (b) Correlation map to the spectrum of the beads.](image-url)
–C≡N is strongly indicative of this), suggesting a fabric source as the likely origin. The two particles in Figure 2 were separated from another (different origin) water sample. Again, the ATR results are unambiguous, with the identification as polyethylene and polypropylene, respectively. These are common from many sources. Excluding sample preparation time, the analysis of these three samples took less than 2 min each even with a user not trained in microscopy.

Microplastics circulate in the aquifer readily. This means particles from many origins can be present in a single location. Field sampling (such as sieving) results in an inhomogeneous (in size and composition) filtrate, but each particle is typically one material. This was the case in the first three examples. In contrast, single microplastic particles deriving from complex origins like in moving automobile or food processing parts may consist of multiple components. Figure 3 shows a particle filtered from shock absorber fluid and the resulting FT-IR spectrum.

This sample shows two characteristics of many real world samples. First, it consists of several components—a mixture—meaning the software (or analyst) must combine multiple pieces of information to achieve a satisfactory result. Second, and more critically, the compounds present in the material have undergone changes because of physical, thermal, and chemical wear. These may change the molecular structure in subtle ways, causing peaks to shift and change shape. A fallacy of spectral searching is the expectation that all “good” results will give a high search metric, say 90 or 90% or 0.9, depending on the definition of the metric. The induced changes seen in materials, especially those exposed to high temperatures and strong solvents, will lower the resulting metric. In such cases, search results become indicative rather than definitive.

Such is the case here. The use of standard (single component) searching provides some clues, but this is clearly a mixture. The multicomponent searching shown in Figure 3 identified three classes of material: PTFE (the core composition of the component) along with an acrylic coating (maybe from paint) and a siloxane agent (a lubricant, most likely). The final composite search result is good but not perfect, assigning most of the peaks. Even with the discrepancies, the origin of the particle was easily tracked to a sliding joint in the device.

These first few examples entailed point and shoot analysis (find, target, collect, analyze). Point and shoot can be done on simple, manual FT-IR microscopes. Automation enables the analyst to obtain a wider picture—a map or image—of a sample, such as the one shown in Figure 4. For this study, a liquid suspension of standard polyethylene spheres was made and then filtered. The filter, after drying, was placed on a piece of double stick tape (to flatten and hold the filter), then inserted into an automated microscope with no further sample preparation. The visual image consists of more than 200 video captures combined into a mosaic covering approximately 1 cm². There were 17,500 spectra collected (50 μm steps, 0.1 s/spectrum resulting in ~30 min for collection). Example spectra from the
filter itself and the spheres are shown in the top of Figure 4.

The image in Figure 5 is a correlation map relating the polyethylene spectrum from a reference standard to each spectrum in the map. Correlation highlights the presence of a component, with a value of 1 being highly correlated (very similar to the reference) and 0 being uncorrelated (not similar to the reference). In this image, the blue field is uncorrelated to polyethylene while the red spots are strongly correlated. Extension of this to the general case would use multiple curve regression (MCR) to highlight and identify each particle in an inhomogeneous population as well.

The FT-IR microscope’s software can complete the analysis automatically, using a particle wizard. The software allows the user to select a region from the video image. The software then identifies the target particles (which the user can filter using a size based sieve) and the analysis proceeds to produce spectra for each particle. These are then searched and a report cataloging the number of particles in the inspection area with a specific identity is prepared. Those data can then be back extrapolated through the volume of liquid filtered to provide a semiquantitative measure of the particulate concentrations.

Conclusion
Microplastics are now deeply entrenched in the food chain. Rapid, reliable measurements are needed to assist in the assessment of the problem and the potential remediation. As evidence of the presence of microplastics even in our air is becoming available, this problem will grow in importance. FT-IR, and specifically FT-IR microspectroscopy, is a useful tool in the analysis of microplastics, providing visual information, particle counts, and particle identification. The prevalence of this problem is leading to further refinements in the techniques, which should improve the ability of FT-IR to address it.

References

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For more information on this topic, please visit our homepage at: www.spectroscopyonline.com
Assessing False Noncompliance and False Compliance with Limits Stated for Physicochemical Parameters in Red Wines Using a MIR-PLS Model

Several analytical procedures depend on multivariate calibration models, such as partial least squares (PLS) models fitted with near-infrared (NIR) and mid-infrared (MIR) spectroscopy signals. Most of these models are related to products that require the control of maximum (or minimum) legally defined limits so that assessing the risks of false noncompliance and false compliance when establishing the procedure is mandatory. This article shows an application in the field of oenology with red wines from the Spanish qualified denomination of origin (DOC) Rioja, with around 600 samples of wine available, enabling representative training-validation sets. Seven oenological parameters were studied, namely alcoholic degree, total acidity, pH, density, reducing sugar, volatile acidity, and malic acid.

M.C. Ortiz, L.A. Sarabia, M.E. Meléndez, and M.S. Sánchez

Mid-infrared spectroscopy (MIR) shows great potential for the noninvasive measurement of quality parameters. Consequently, its use is spreading in the world of industry, covering many fields such as agriculture, biotechnology, food and beverages, forensic science, clinical chemistry, pharmaceuticals, and more. The wine sector clearly requires this technology that offers rapid results and low cost.

Fourier transform infrared spectroscopy (FT-IR) is already widespread in wine laboratories and cellars. So much so that the International Organization of Vine and Wine (OIV) has published a guide (1) where the performance of FT-IR partial least squares (PLS) models are explained. This technique
can identify the spectral features that can provide information on the factors that affect the quality of alcoholic beverages and, combined with multivariate analysis, increases the chances to obtain quality parameters and their control (2,3). A search in Scopus (01/25/2017) with the keywords “wine” and “MIR” provided 87 publications since 2001, with 33 of them devoted to the determination of parameters in wine. However, no one evaluates the capability of PLS models for fulfilling the legal limits.

Regardless of whether it is a maximum or a minimum limit, when a regulated property is being measured, it is necessary to evaluate the probabilities of false noncompliance and false compliance. For univariate signals, the International Organization for Standardization (ISO) and the International Union of Pure and Applied Chemistry (IUPAC) defined a procedure for determining the detection capability of an analytical method, evaluating the probabilities of false positive and false negative (when an analyte is forbidden). The procedure was generalized for PLS in the literature (4,5) and also adapted to fulfill the official regulation for Zamorano cheese that demands only minimum permitted limits (6).

The MIR spectra combined with PLS regression could be used for the control of the maximum and minimum limits established for the certification of the red wines from Rioja, Spain (7). For this purpose, the new figures of merit $CD_\alpha$ and $CD_\beta$, analogous to decision limit and detection capability, must be evaluated.

**Figure 1:** Minimum permitted limit (PL). Probability of false compliance, $\beta$, as a function of the concentration in an accuracy line (left top corner) and operating characteristic curve (below to the right). The probability of false noncompliance, $\alpha$, is fixed.
The PLS regression method is based on latent variables computed as the linear combinations of the predictor variables that are highly correlated with the response variable and, at the same time, explain the variation in the predictor variables (8).

**CDα and CDβ**
To formalize this section the notation of Commission Decision 2002/657/EC (9) for univariate signals will be used. In particular, permitted limit (PL) is the maximum residue limit, maximum level, or other maximum tolerance for substances established in any normative. When PL = 0, the decision limit (CCα) is the concentration limit at and above which it can be concluded with an error probability of α that a sample is noncompliant when it is. In the case of forbidden substances, for which no permitted limit has been established, the detection capability (CCβ) is the lowest concentration at which a method is able to detect truly contaminated samples with a statistical certainty of 1–β. In the case of substances with an established permitted limit, this means that the detection capability is the concentration at which the method is able to detect permitted limit concentrations with a statistical certainty of 1–β.

The capability of detection in the case of univariate calibration models is well established in the ISO standard 11843 (10) and IUPAC (11). Interestingly, it can be generalized to any type of calibration models (multivariate, multiway, nonlinear, neural-network based, and so on). This generalization is based on the mathematical proof that the capability of detection as is defined by ISO and IUPAC for univariate calibration (signal versus concentration) is invariant for linear transformations of the response (the...
signal). In particular, the capability of detection is determined by using the concentration predicted with PLS versus true concentration, which is called the accuracy line \( y = a + bx \) (4,5).

\( \text{CC}_\alpha \) and \( \text{CC}_\beta \), which are defined for \( \text{PL} = 0 \), are thus generalized for any other value of \( \text{PL} \), and named in this paper as \( \text{CD}_\alpha \) and \( \text{CD}_\beta \) to distinguish them from the terms already established in other regulations (9–11). Precisely, if it is about a minimum limit, \( \text{PL} = x_0 \), stated for a given parameter, \( x \), the following hypothesis test is posed:

\[
\begin{align*}
H_0 : x &= x_0 & [1] \\
H_a : x &< x_0
\end{align*}
\]

In equation 1 the null hypothesis \( H_0 \) is in fact stating that the parameter \( x \) is greater than or equal to \( x_0 \) (that is, the sample is compliant), and thus the alternative hypothesis \( H_a \) says that the sample is noncompliant because the parameter \( x \) is less than the minimum limit admissible \( x_0 \).

The decision limit (\( \text{CD}_\alpha \)) of the method is the value of the parameter, below which it can be decided with a probability \( \alpha \) that \( x_0 \) was not reached when it was truly exceeded. Thus, \( \text{CD}_\alpha \) is related to the probability of false noncompliance, \( \alpha \). That is, \( \alpha \) is the probability of deciding that the tested sample is noncompliant when it was, the significance level in the hypothesis test of equation 1.

For a given \( \alpha \), the probability \( \beta \) of false compliant decision is the probability of wrongly affirming that the tested sample has a value of the parameter greater than or equal to \( x_0 \), that is, to conclude that it is compliant, when it was not. The detection
capability (CDβ) is the value of the parameter related to this decision.

This is precisely computed as

$$CD\beta = x_0 - \frac{\Delta(\alpha,\beta)\sigma_{\bar{x}}}{\hat{b}}$$ \[2\]

where $\Delta(\alpha,\beta)$ is the value of noncentrality parameter of a noncentral $t$-distribution related to the probabilities $\alpha$ and $\beta$, $\sigma$ is the residual standard deviation of the accuracy line, and $b$ its slope, and

$$\sigma_{\bar{x}} = \frac{1}{K} + \frac{1}{I} + \frac{(x_0 - \bar{x})^2}{\sum_{i=1}^{I} (x_i - \bar{x})^2}$$ \[3\]

depends on the position of the standards in the accuracy line ($x_i$), the number $K$ of replicates and the number of standards $I$.

When the established limit, $PL = x_0$ is a maximum limit, a similar hypothesis test is used but in this case the alternative hypothesis is $H_a: x > x_0$ (the parameter is greater than $x_0$, so the sample is noncompliant). Thus, the capability of detection CDβ (analogous to CCβ when $x_0 = 0$ in the Commission Decision and unnamed in the ISO 11843-2) is

$$CD\alpha = x_0 + \frac{\Delta(\alpha,\beta)\sigma_{\bar{x}}}{\hat{b}}$$ \[4\]

In both cases (minimum or maximum permitted limit, $x_0$), the limit of decision CDα (analogous when $x_0 = 0$ to CCα in the Commission Decision and unnamed in the ISO 11843-2) is

$$CD\alpha = y_c - \hat{a}$$ \[5\]

where $\hat{a}$ and $\hat{b}$ are the intercept and the slope of the accuracy line and $y_c$ is the value that satisfies either equation 6 for a minimum permitted limit

$$\alpha = \text{probability}(y < y_c / x = x_0)$$ \[6\]

or equation 7 for a maximum permitted limit.

$$\alpha = \text{probability}(y > y_c / x = x_0)$$ \[7\]

Figure 1 shows a schematic representation for an oenological parameter with a minimum limit established, for example the total acidity in Spanish qualified denomination of origin (DOC) Rioja. In this case, the test of equation 1 is applied. As it can be seen, setting the probability $\alpha$ is in practice setting a critical value, $y_c$, (equation 6) that allows the computation of the probability of deciding that a sample does not reach the minimum value when it is false. This statement is related to concentrations CDβ (equation 5) and CDβ (equation 2). The concentration CDβ, besides assess-

### Table I: Oenological parameters, reference techniques, and equipment

<table>
<thead>
<tr>
<th>Oenological Parameters</th>
<th>Reference Technique</th>
<th>Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alcoholic degree</td>
<td>Near infrared (NIR)</td>
<td>Technicon infraanalyzer 450, Bran Luebbe, S.L.</td>
</tr>
<tr>
<td>Total acidity and pH</td>
<td>Automatic potentiometric</td>
<td>Automatic tritator H-PLUS, Bran Luebbe, S.L.</td>
</tr>
<tr>
<td>Density</td>
<td>Electronic densimetry</td>
<td>Electronic densimeter Anton Paar, DMA 48</td>
</tr>
<tr>
<td>Reducing sugars, volatile acidity, and L-malic acid</td>
<td>Air-segmented continuous-flow analysis (SFA)</td>
<td>TRAACs 2000, Bran Luebbe, S.L.</td>
</tr>
</tbody>
</table>
ing the probability of false noncompliance (α), also assesses false compliance (β). Note that in the value CD<sub>α</sub> itself, the probability β<sub>1</sub> to falsely assert that the wine exceeds the required minimum values is 0.5, while in the concentration corresponding to CD<sub>β</sub> this error is reduced to a much smaller amount β<sub>n</sub>.

Figure 2 shows the corresponding diagram for a maximum limit. Once the probability α is set, now the critical value y<sub>c</sub> is determined by equation 7 and CD<sub>β</sub> is determined with equation 4. In CD<sub>α</sub>, the probability β<sub>1</sub> of asserting that the wine does not exceed the maximum required limit when it is false is 0.5, whereas in CD<sub>β</sub> this probability is largely reduced, β<sub>n</sub>.

In any case, the researcher chooses both risks. The operating curve of the test, the representation of β versus the concentration (for a fixed α) could be used to comparatively examine the performance in the detection capability of several procedures of analysis (or several analyzed parameters).

### Experimental

More than 600 samples of red wine are available, all of them analyzed in the official laboratory of the Oenological Station of Haro (La Rioja, Spain) according to official controls with the accredited methods under the norm EN/ISO 17025, number 183/LE407 (Table I). The same samples were scanned, between 1500 and 1000 cm<sup>-1</sup> each 5 cm<sup>-1</sup> (101 predictor variables), with a Spectrum One FT-IR spectrometer (PerkinElmer) to obtain the MIR spectra. PLS models were computed with PLS Toolbox 5.8.2 2. (11) over Matlab software. CD<sub>α</sub> and CD<sub>β</sub> were estimated using an in-house written program.

### Procedure to Build the PLS Models

The procedure consists of the following steps:
1. To build a PLS model for each parameter:
   - Preprocess the data matrix X by standard normal variate (SNV).
After that, both predictor and response variables are centered.

- Determine the number of latent variables by cross-validation (10 splits data) and compute the root mean squared error in cross-validation (RMSECV).
- Remove those samples with standardized residual (in absolute value) greater than 2.5 or with both Q and Hotelling $T^2$ values larger than their corresponding threshold values at a 99% confidence level.
- Repeat the two previous steps until there are no outliers.

2. To evaluate the predictive capability of the PLS models by using an external set of data with 150 wines. The threshold values of Q and $T^2$ obtained in the calibration step are used to detect outliers by assessing the similarity of the spectra of the evaluation samples with the calibration ones. Moreover, the samples with spectrum similar to those of the training samples but with different values of the calibrated property were also considered outliers. This last assessment is made with the least median of squares regression (LMS) line computed between the PLS predicted values versus the true values of the corresponding parameter. The outliers thus detected were removed and a least squares (LS) regression line was finally fitted. This regression line is the accuracy line, whose characteristics are in the last three columns in Table II for the parameters analyzed.

3. By using the data from the accuracy line, to determine CD$_\alpha$ and CD$_\beta$ with probabilities of both false non-compliance and false compliance fixed at 0.05.

### Results and Discussion

The characteristics corresponding to the different PLS models are shown in Table II, in which the slope, intercept, and $R^2$ values corresponding to the accuracy lines obtained with the predicted and true values can be observed too. In all PLS models, the RMSECV values are similar to their corresponding root mean squared error of prediction (RMSEP) values, which indicates stability of the models built. Also, notice that the variance of the oenological parameters ($Y$) explained by the models is high, ranging from 87.1% to 94.4%, except for the reducing sugar (60.8%).

On the other hand, the determination coefficients $R^2$ (explained $Y$ variance) of the accuracy lines with the wines in the external data set are greater than 84.5% (except for reducing sugar). Figure 3 depicts these accuracy lines,

<table>
<thead>
<tr>
<th>Permitted Limit</th>
<th>In Fitting</th>
<th>In Prediction</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># Wines</td>
<td>CD$_\alpha$</td>
</tr>
<tr>
<td>Alcoholic degree*</td>
<td>447</td>
<td>11.34</td>
</tr>
<tr>
<td>Total acidity*</td>
<td>426</td>
<td>3.34</td>
</tr>
<tr>
<td>Reducing sugar†</td>
<td>398</td>
<td>4.28</td>
</tr>
<tr>
<td>Volatile acidity‡</td>
<td>450</td>
<td>0.87</td>
</tr>
</tbody>
</table>

*Minimum and †maximum limit required for the certification of the red wines from DOC Rioja (7)
only for the models corresponding to the four parameters with compulsory limits. Table III shows the permitted limits and the values obtained for CDα and CDβ for both fitting (the accuracy line is made with the training set) and prediction (wines in the external data set). Also, the number of samples used to compute the values, after deletion of outliers, is shown in Table III.

In the case of alcoholic degree the minimum limit established is 11.50% v/v. According to the values in the first row of Table III, the PLS model for this parameter has a CDα in prediction equal to 11.35% v/v. This means that for values less than 11.35% v/v we say that the alcoholic degree of the wine is non-compliant with a 0.05 probability that this assertion is false. But in this case the probability β, to assert that a wine has an alcoholic grade greater than or equal to 11.50% v/v when it is false is 0.50. However, if the PLS model for a wine sample provides a value of 11.23% v/v (equal to CDβ) then β is reduced to 0.05 (see Figure 1).

For a parameter with a maximum limit established—for example, the volatile acidity (last row in Table III)—the values of CDα and CDβ in prediction are equal to 0.87 and 0.93 g/L, respectively, when the PL is 0.80 g/L. The interpretation is the same for the rest of the parameters with their corresponding values. CDα and CDβ in prediction and fitting are very similar, which means that the values are stable for the future routine use of the PLS models.

**Conclusions**

All the MIR-PLS models built are highly predictive and stable, for the determination of alcoholic degree, total acidity, pH, density, reducing sugar, volatile acidity, and malic acid in red wines.

This procedure is general, can be applied to products that require the control of maximum (or minimum) legally established limits, and should be taken into account when assessing the risks of false noncompliance and false compliance.

**Acknowledgments**

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You have been investigating the IR spectra of polyoxymethylene (POM) mold plates to determine the submicrometer-scale morphology and molecular orientation (1). What factors led you to perform this research?

We work in the business of informing companies, by using various analytical techniques, about the causes of problems such as resin cracks. In this work, we referred to the many papers published so far on the topic. However, we were not able to understand the cause of POM resin cracks in some cases. The measurements of IR in the papers referred to were usually conducted using KBr or Nujol methods, but curiously its spectral shapes more closely resemble the spectral shapes of specular reflection than those of the attenuated total reflectance method. We think that this phenomenon may be attributed to incorrect assignment due to optical effects. Since the problem in polymers seems to relate somewhat to their higher-order structure and orientation, we prepared a molding plate sample in which the flow of the melt can easily be imagined. We investigated how the spectral features changed from the gate position, in addition to observing the scanning electron microscopy (SEM) images. Furthermore, we applied the specular reflection method to avoid changes in the morphology resulting from pretreatment before the IR measurement. When we used the SEM images, we found cellular structures on the surface. Thus, as a first step, we would like to research the relation between the cellular structures and IR spectral shapes to consider the problems of POM.
Can you please briefly describe the method used in your investigation? Considering the change in orientation by the specular reflection method, it is necessary to rotate the polarizer and perform a reflection measurement at each site. Analysis of the spectra obtained in this way revealed that there is a region where the real part of the dielectric function (RPDF) is greatly negative in the main chain direction of the molecule. We also found that the strange peaks found in earlier work in other laboratories coincided with the wavenumber range where the RPDF intersects with zero, and I thought that it was a polariton due to the delay effect of electromagnetic wave. To confirm this hypothesis, we assembled an experimental setup to observe the Berreman effect; namely, we performed reflection measurements of a thin film on a metal plate with a large incident angle. The Berreman effect should be observed as a reflection response in p-polarization only at the position of the LO mode.

What conclusions did you draw from the study and why are they significant? So far, polaritons have been observed in inorganic ion crystals and polar semiconductors, and there have been no reports that surface phonon polaritons have been observed in polymers. Although POM is a crystalline polymer, it seems surprising that polaritons appear in weak ordered materials like polymers. Furthermore, we pointed out that we face the risk of misinterpreting the measured spectra obtained by the destructive techniques in the framework of the ordinary vibration analysis. Therefore, it is necessary to reconsider the interpretation of large peaks in polymers in connection with higher-order structures. Thus, IR spectroscopy has potential for use when investigating the higher-order structures of polymers. In addition, we show that polar polymers with high flexibility can also be candidates as materials for low-loss nanophotonic devices that require structures generating surface phonon polaritons.

What are the next steps in your research? We predict that a similar polariton mode will exist for polymers with a large dipole moment and relatively high-order structure. We would like to search for materials that behave similarly in polymers other than POM. Also, we will investigate the intermediate state that changes from ordinary vibrations to polaritons. Polymers are considered to be the most suitable materials to investigate the condition of changing from ordinary vibrations to polaritons in IR spectroscopy. Moreover, since it is believed that some IR peaks reflect higher-order structures, we would like to analyze such structures using finite-difference time-domain (FDTD) methods.

Reference

This interview has been edited for length and clarity. To read the full interview please visit: www.spectroscopyonline.com/solving-polymer-problems-using-ir-spectroscopy
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