Consolidated Vis-NIR-MIR soil measurements

Application Note M177

Soil analysis is a fundamental cornerstone of modern agriculture and environmental science, providing crucial insights into soil composition, fertility, and health.

One of the most powerful and efficient tools in this realm is the integration of spectroscopy to uncover valuable information about soil characteristics. Within this landscape, the fusion of Visible-Near Infrared-Mid Infrared (Vis-NIR-MIR) spectroscopy, coupled with Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTs), has emerged as a new approach, offering an unparalleled depth of analysis, and understanding.

Unraveling Comprehensive Soil Insights

The amalgamation of Vis-NIR-MIR spectroscopy with DRIFTs technology provides a multidimensional view into soil properties. The Visible range provides information about soil color and organic matter content. As the analysis shifts into the Near Infrared region, DRIFTs reveal intricate details about soil moisture, texture, and even the presence of specific chemical compounds. Further progress into the Mid Infrared region unveils mineral composition, carbon-nitrogen ratios, and structural attributes.[1,2]

Enhanced Efficiency through DRIFTs

The incorporation of DRIFTs augments the efficiency of soil analysis by offering non-destructive, in-situ measurements. Unlike traditional methods that might necessitate sample preparation or alteration by chemical treatment, DRIFTS enables direct analysis of unprocessed soil samples. This future expedites the analytical process and eliminates potential biases introduced by introduced sample handling, ensuring that the obtained insights are as close as possible to the soil's natural state.

Why Single-run Measurement is Beneficial?

Employing a single spectrometer that covers the entire spectral range, combined with DRIFTs technology, offers a multitude of benefits that elevate the accuracy, efficiency, and affordability of soil analysis to unparalleled levels. The integration of Vis-NIR-MIR spectroscopy and DRIFTs within a single instrument not only eliminates the requirement for multiple devices but also simplifies calibration and maintenance processes. This comprehensive approach enhances cost-effectiveness by reducing expenditures related to equipment, calibration standards, and technical assistance. The economic feasibility of this technology paves the way for broader adoption, positively impacting both research institutions and agricultural enterprises.

INVENIO for Multispectral Soil Analysis

The highly innovative INVENIO X with patented INTEGRAL™ interferometer, stands at the forefront of multispectral R&D spectrometers, with unparalleled automation of optical components. By seamlessly integrating MultiTect™ technology and the INTEGRAL™ interferometer with an incorporated automatic beamsplitter changer, it offers unrivaled versatility and performance for soil analyses in an extremely broad spectral range. In the applied configuration, the instrument facilitates precise measurements from 350 to 26,000 cm^{-1} (28.5 µm-385 nm), ensuring a robust S/N ratio. The comfortable OPUS Measure Multiple Experiments function goes a step further by allowing for a single push button measurement, thereby eliminating the necessity of any further user interaction.

Sample Preparation

Sample preparation methods can vary based on the chosen measurement protocol and available reference data for comparison[3]. Typically, the process involves air-drying and sieving, while samples can also be mixed with dry KBr or other transparent salts to enhance penetration. However, such enhancement should be meticulously considered, since it also can introduce distortions[4]. Bruker application experts can provide suitable guidance.

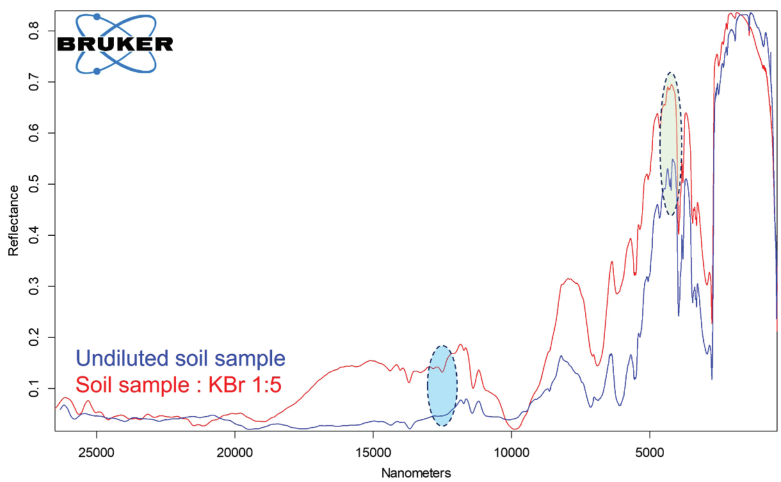


Fig. 1
Effect of dilution on soil samples. Spectra were normalized. Circles indicate the regions of distortions. In this experiment sample was air-dried and sieved through 5mm mesh.

Experimental Protocol

The general workflow is presented in the Figure 2.

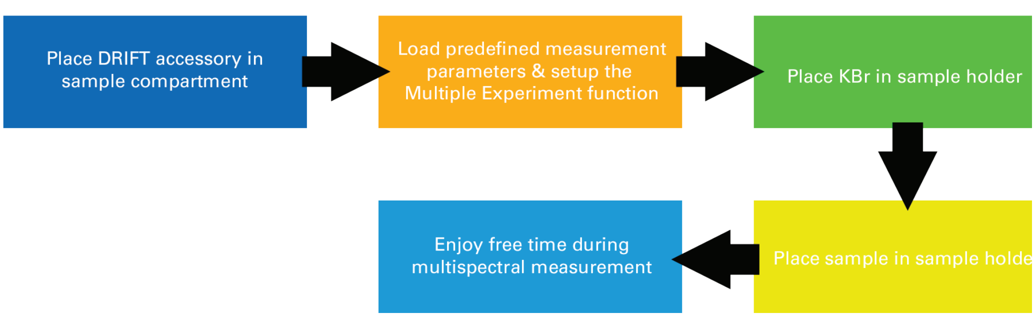
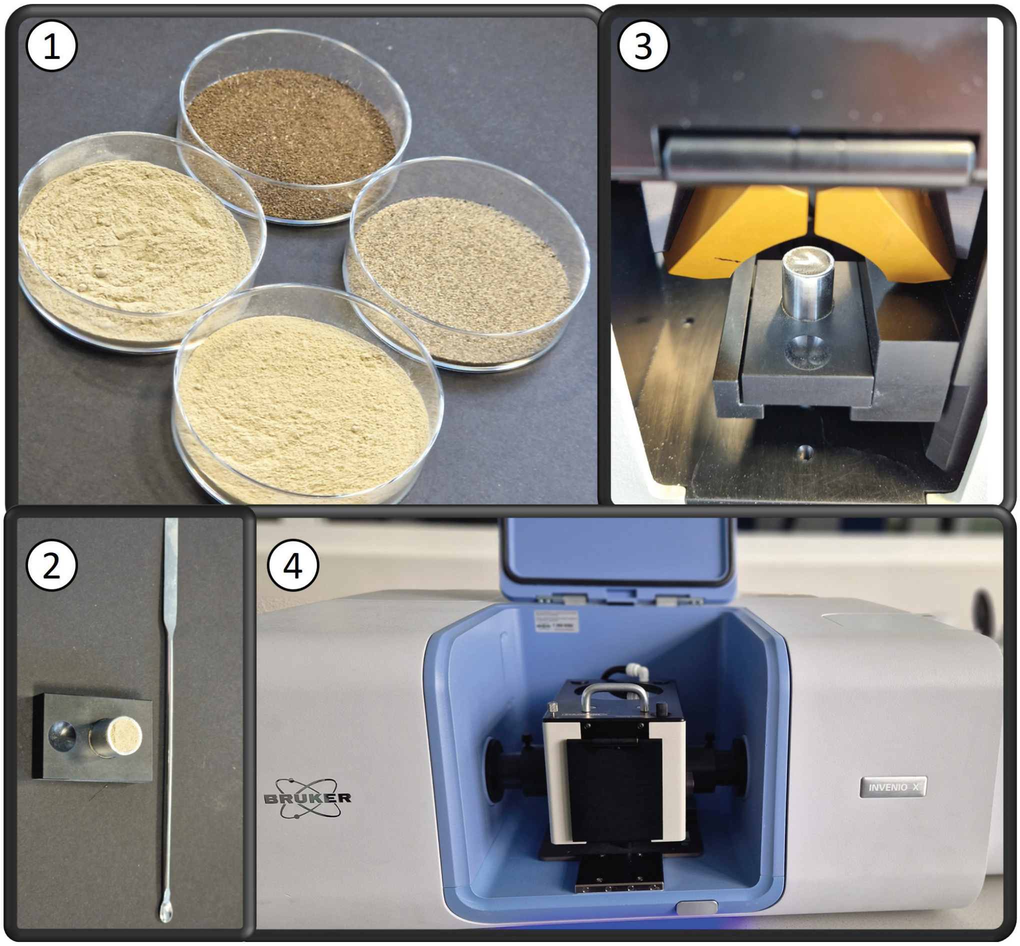


Fig. 2
General workflow of consolidated Vis-NIR-MIR soil measurements.

Fig. 3
Sample preparation and sample compartment setup.



The procedure involves the following steps:

1. Choose measurement parameters (XPM files*) for multirange measurement with recommended parameters:

   Resolution: 4 cm^{-1}
   Number of scans: 128/256
   Scanner velocity: 10kHz
   Aperture: 6 mm
   Results spectrum: Reflectance/Kubelka-Munk

	MIR	NIR	Vis
Source	MIR	NIR/Vis	NIR/Vis
Beamsplitter	KBr	Quartz-Vis	Quartz-Vis
Detector	RT-DTGS	Te-InGaAs	Quartz-Vis
Range	5,000 - 380 cm^{-1}	12,000 - 5,000 cm^{-1}	26,000 - 12,000 cm^{-1}
Optical filter	OPEN	OPEN	Custom Filter 1 (F505)

   Depending on the need, experimental parameters can be adjusted and stored by modification of the existing or creation of the new XPM files. Once created XPM files can be reused.

2. Apply parameters to „Measure Multiple“ feature in OPUS. Check the „Merge Spectra“ option to consolidate data into a single spectrum.

*Sample xpm files can be provided.

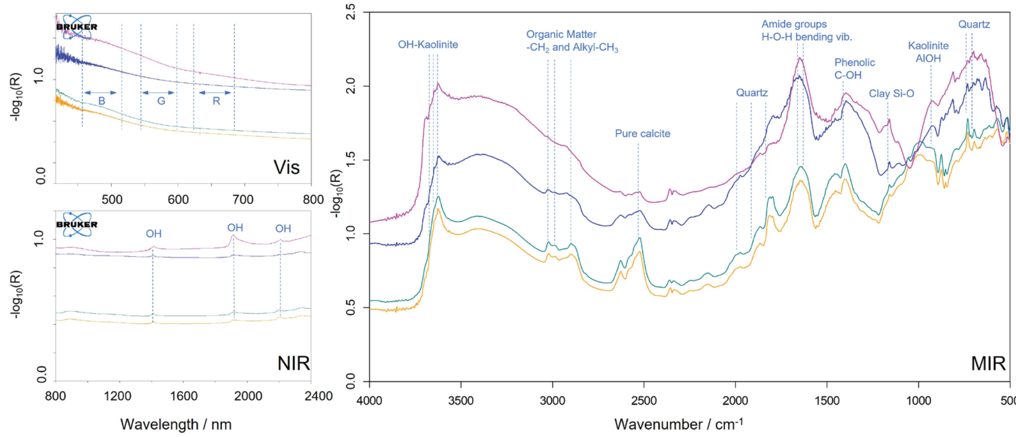


Fig. 4
The most important spectral regions in soil analysis. Red (R), green (G) and blue (B) bands are shown in the VIS spectrum; The VIS–NIR–MIR spectrum is annotated with some dominant soil components and absorption peaks; OH indicated features of free water and lattice minerals.[1,4] Measurements were conducted on undiluted samples.

3. Conduct a background measurement using dry KBr with similar grain size as samples for optimal results.

4. For sample preparation and measurement, both the sample and KBr can be placed directly inside the DRIFT sample holder, as illustrated in Figure 3 ❷ and ❸.

The measurement time per sample is about 7 min. The results obtained in these measurements are shown in Figures 4 and 5, respectively. All spectral regions important for both qualitive and quantitative soil analysis are included. The clear differences between spectra of different soil samples are consistent with their appearance (Figure 3 ❶) and several significant spectral features can be justified.

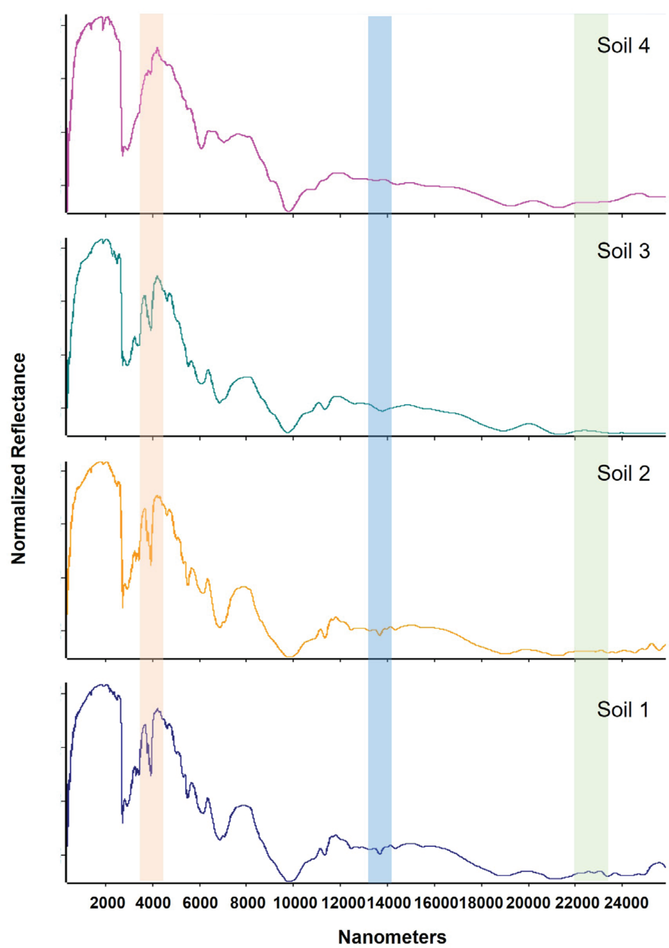


Fig. 5
Example spectra of 4 different soil samples collected in different locations in Europe. The colored areas indicate the ranges with the largest observed changes. Samples were 5 times diluted with KBr.

Data Analysis

Quantitative analysis of soil through Vis-NIR-MIR reflectance spectroscopy necessitates advanced statistical approaches for extracting soil attributes from spectral characteristics. In literature, various techniques have been employed to establish connections between soil spectra and attributes. For instance, multiple regression analysis (MRA) is utilized to correlate specific bands in the NIR range with diverse soil properties. Principal components regression (PCR) and partial least squares regression (PLSR) stand as prevalent methods for spectral calibration and prediction. Additional methodologies encompass multivariate adaptive regression splines (MARS) for soil property estimation from spectral libraries, radial basis function networks (RBFN) for linking soil organic matter to NIR spectra, and artificial neural networks for predicting soil attributes like organic matter, phosphorus, and potassium from the VIS-NIR spectrum[1]. More recently, more sophisticated methods based on machine learning have been also applied.[6]

The extensive capabilities of OPUS facilitate various analysis approaches and the integrated Python API enhances the accessibility of consolidated experimental data using further analytical methods. Calibration data can be self-done or come from public sources.[5]
O/QU-N OPUS quantitative analysis software (multivariate PLSR analysis package) for building calibrations from spectral and reference data is available as an option.

Alternatives

Depending on the need Bruker also offers solutions for routine-like soil analysis in MIR:

- ALPHA II Soil Analyzer for individual samples
- INVENIO HTS-XT Soil Analyzer for multiple samples

For more information, please refer to Product Note M195.

Conclusions

Utilizing a single Vis-NIR-MIR spectrometer for soil analysis provides the advantage of capturing a broad range of spectral information in a consolidated manner, enhancing precision, reducing operational complexity, and enabling cost-effective and rapid assessment of soil properties.
For more information, please contact our specialists and ask for the application package INV-X/SOIL.

Compenents used in this analysis

- INVENIO-X Fourier Transform Infrared Spectrometer INV-X with INTEGRAL™ interferometer, automatic beam splitter exchanger, and MultiTect™ unit with mid infrared DTGS detector. The standard spectral range covers 8,000 to 350 cm^{-1}
- A528/Q Harrick Research Diffuse Reflectance Accessory "Praying Mantis"
- D427/M Thermoelectrically-cooled InGaAs detector on MultiTect™ unit.
- W121/I-S Changer for second internal NIR/VIS source including focusing optics
- Q428/I7 Tungsten source
- F505 Optical filter with holder
- T502/IX VIS-beamsplitter, Quartz
- D510/M Si-diode installed on MultiTect™ unit
- F103/IX laser blocker
- OPUS software

References

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[6] R. A. Viscarra Rossel, T. Behrens, E. Ben-Dor, S. Chabrillat, J. A. M. Demattê, Y. Ge, C. Gomez, C. Guerrero, Y. Peng, L. Ramirez-Lopez, Z. Shi, B. Stenberg, R. Webster, L. Winowiecki, and Z. Shen, Diffuse reflectance spectroscopy for estimating soil properties: A technology for the 21st century. European Journal of Soil Science,73 (2022) e13271

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