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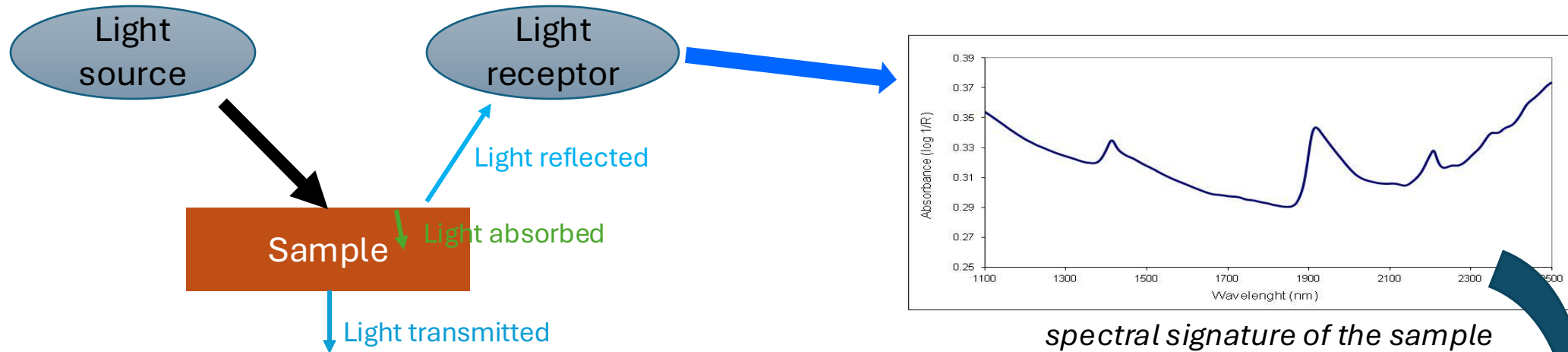
Determination of the clay content of soils

25 June 2024

Near-infrared spectroscopy

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What is soil spectroscopy?



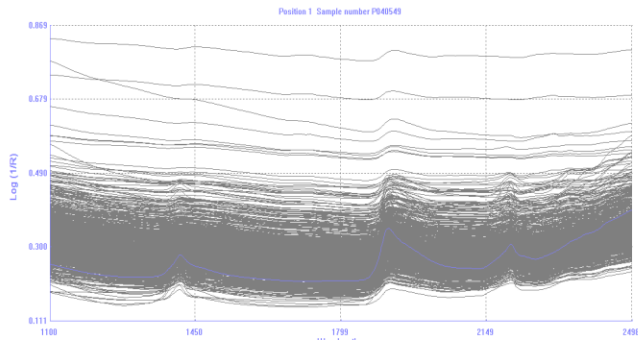
- 1) Molecular vibrations and electronic transitions associated with soil constituents
- 2) The way the light is divided into these 3 parts depends on the molecules that constitute the sample
- 3) Wavelength ranging between 800 and 2500 nm
- 4) Spectral signature responds to soil mineral and organic composition

%TOC, %Clay,...?

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What is soil spectroscopy?

Interpretation model



REF = sieving
and
+ sedimentation
method



Spectral library

3 steps :

Pre-treatment

- Derivative (1st, 2nd,...)
- MSC
- SNV
- Combinaisons
- ...

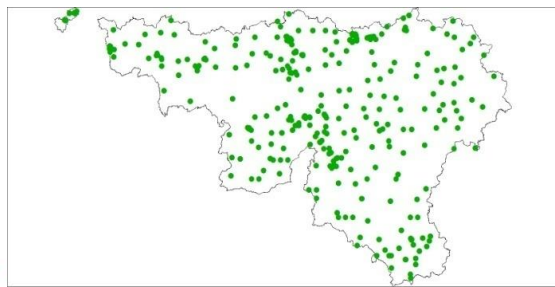
get a better view on the spectrum, get rid of the background noise, etc

Building of the predictive model

- MLR
- PCR
- PLS
- Global/ Local
- ...

Validation

- Leave-one-out
- Leave-more-out
- ...



Representativeness of the values of the parameters (Clay, TOC,...)
Representativeness of the land uses (cultures, grasslands, forests, etc)
Representativeness of the spectral diversity
Mainly composite surface samples

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What is soil spectroscopy?

Validation

- Leave-one-out
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- ...

Indicators of performance

R^2	Determination coefficient between the REF values and the predicted values
RMSEP	Root Mean Square Error of Prediction
RMSECV	Root Mean Square Error of Cross Validation
RPD	Ratio between Standard deviation of REF values/RMSEP or RMSECV
...	

The performances of the model can be improved by adding couples REF value – NIR spectrum in the spectral library.

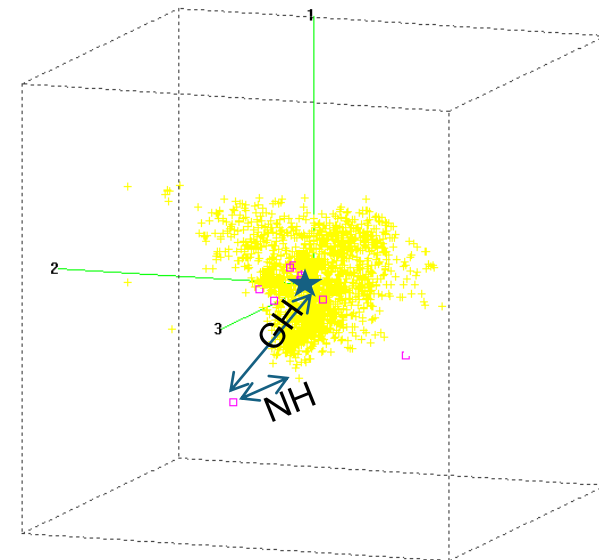
Why to invest in soil spectroscopy?

The main advantages of this method are : 

- rapid & low-cost
- reproducible
- easy to proceed
- non-destructive
- environmentally friendly
- multi-parameters
- prediction of parameters that are hardly measurable in a lab (CEC : hazardous chemicals, clay : time-consuming, etc) but useful for fertility advice

Soil spectroscopy in Wallonia

- Since end 2000's, REQUASUD is working on the development of NIR analyzes on dried and fresh soils.
- A general methodology has been developed :
 - analytical protocol, easy to proceed and repeatable
how to fill in the cells, how to take the measure, 2 mm sieved, dried samples, quarter cup, sample scanned in duplicate, etc
 - tests and choice of best pre-treatments and predictive models for each analytical parameter *1st derivative, SNV, local PLS, etc*



Preparation of the spectrometer

Every day :

- **Performance tests** (measurements of an internal reference, verification of RMS – Root Mean Square – and bias)
- **Check cell**



Preparation of the sample

Dried soil	Fresh soil
ISO 11464 : air-dried (or in a stove) at max. 40°C sieved at 2 mm	sieved at 8 mm
No destruction of organic matter and carbonates Homogenize the sample	

Fill in 2 quarter cups/sample
+ control sample



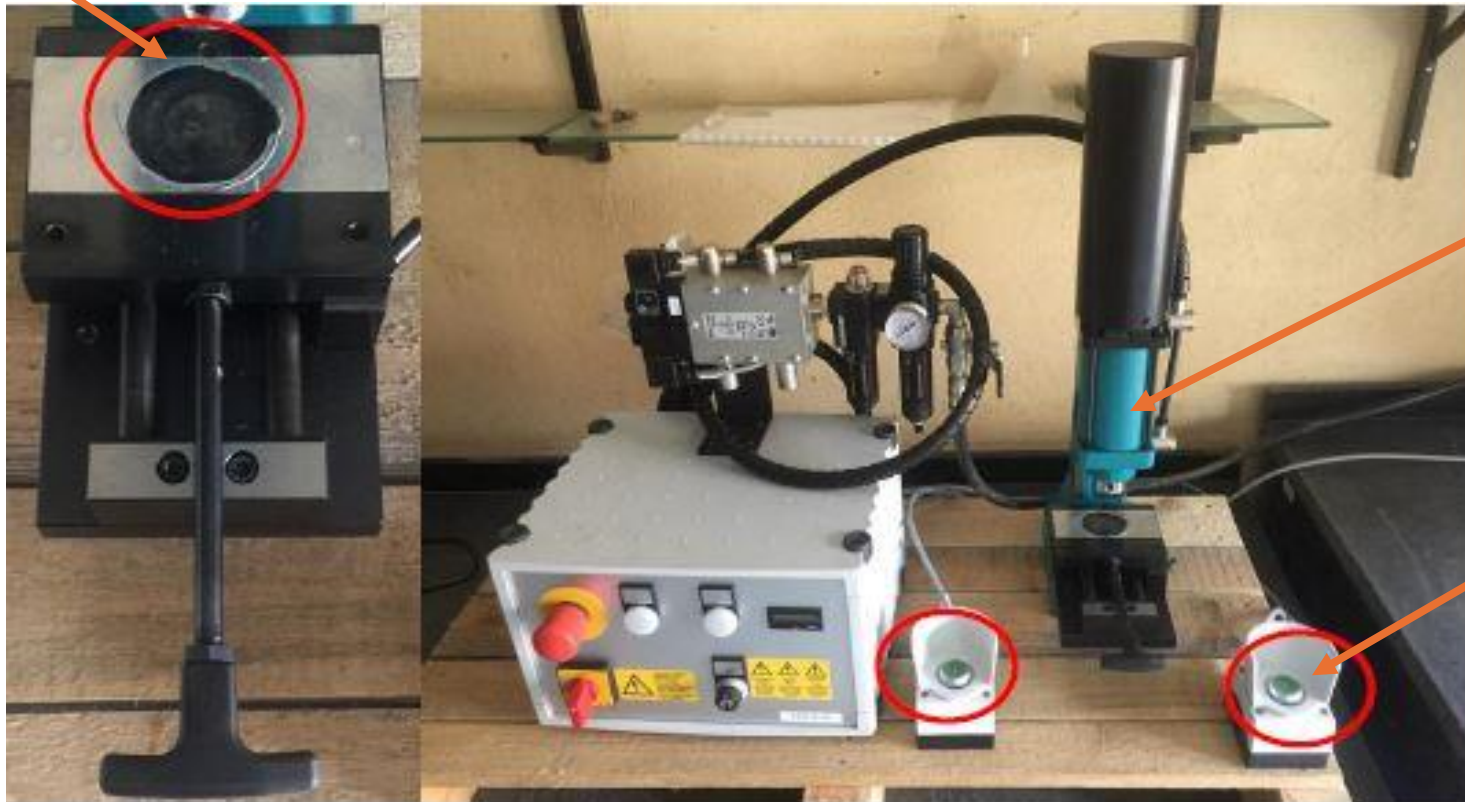
Fill in 1 ring cup/sample
+ control sample





Preparation of the sample

Ring cup and **fresh soil**



Measurement of the sample

Dried soil

Fresh soil



Empty and clean the cups (vacuum cleaner, tissue, smooth brush, possibly distilled water then dry properly)

Attention points

- The whole window must be covered by soil
- The light of the spectrometer reach only a thin layer of soil after the window
- Room temperature stable, same temperature as the samples, approx. 20°C ($\pm 5^\circ\text{C}$)

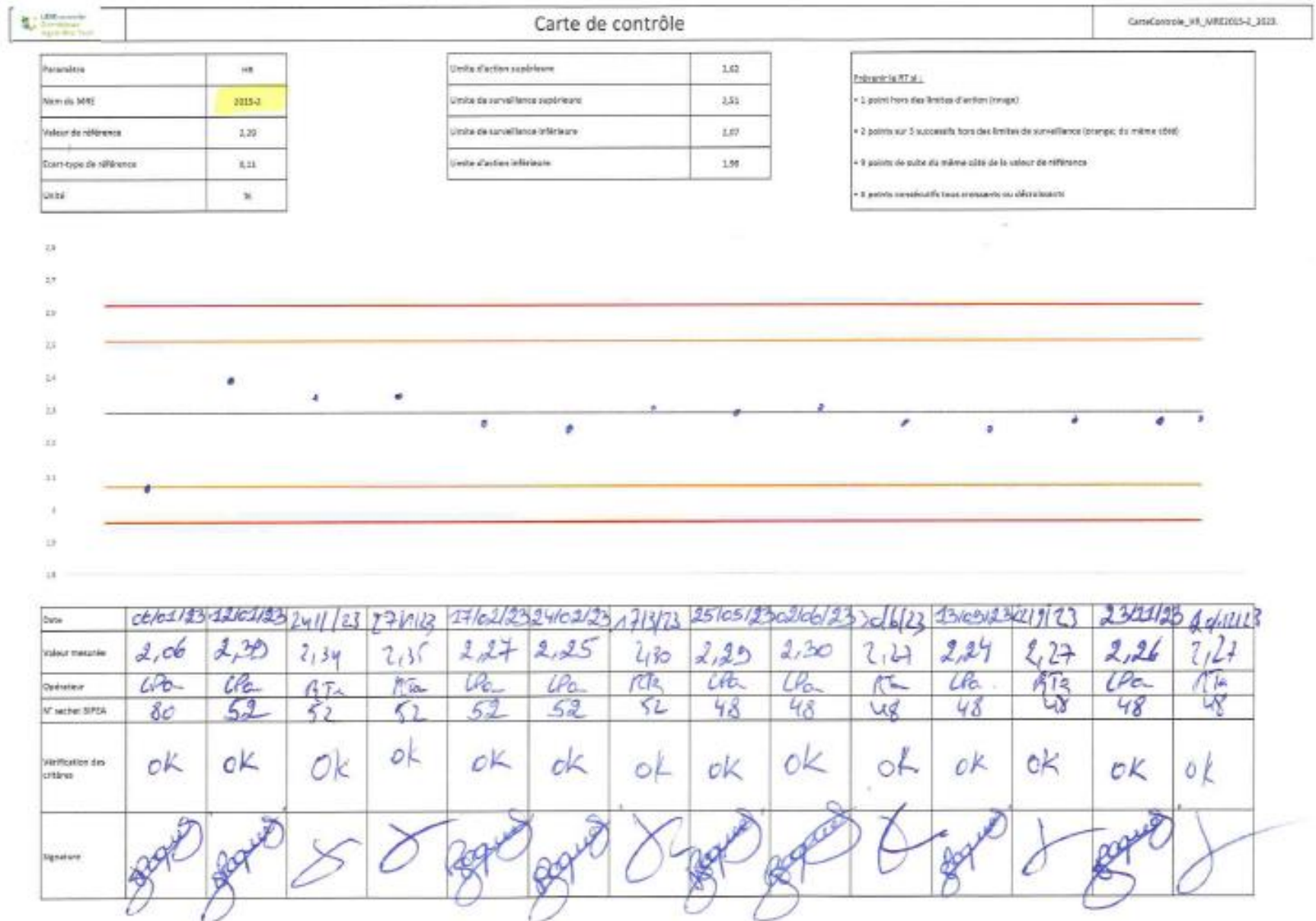
Validation of the measurement

Dried soil	Fresh soil
<p>2 quarter cups/sample calculate RMS (root mean square) :</p> <ol style="list-style-type: none">1. $RMS < 10\ 000 \Rightarrow$ ok, calculate mean of the 2 spectra2. $RMS > 10\ 000 \Rightarrow$ Nok, re-scan the sample	<p>1 ring cup/sample /</p>



Validation of the measurement

- Clay content (predicted) of the control sample

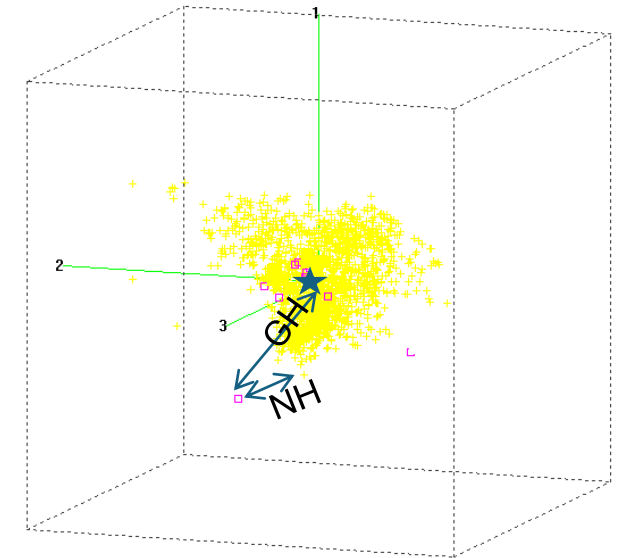


Interpretation of the measurement

- Prediction of clay content, using :
 - the mean spectrum (2 cups)
 - one of the database (cropland, grassland, vegetable garden)
 - the defined pre-treatment and prediction parameters, local mode
 - a « Repeatability file » : nir-file containing the same samples scanned in the different labs/apparatus of the network. Aim : limit the differences between the labs.

Interpretation of the measurement

Sample Number	Clay	GH-Clay	NH-Clay
gGP9384	16,11	0,31	0,27
gGP9386	28,25	1,44	0,57
gGP9389	30,50	0,40	0,32
gGP9391	31,43	0,29	0,28
gGP9393	25,19	2,51	1,66
gGP9396	33,35	0,37	0,27
gGP9398	14,99	0,60	0,32
gGP9400	17,72	0,35	0,32
gGP9402	28,66	0,73	0,59
gGP9405	18,24	0,40	0,48



- **GH** (global *H* or Mahalanobis distance) is the distance between the unknown sample (to be predicted) and the gravity centre of the closest samples (from spectroscopy point of view) of the library **Max = 3**
- **NH** (neighbor distance) is the distance between the unknown sample (to be predicted) and the closest of the closest samples (from spectroscopy point of view) of the library **Max = 1**

Interpretation of the measurement

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Measure validated

- Measure **not** validated :
- the database doesn't contain samples similar enough to predict this sample correctly
 - the sample must be analyzed with the REF method
 - this value by REF method is integrated in the database

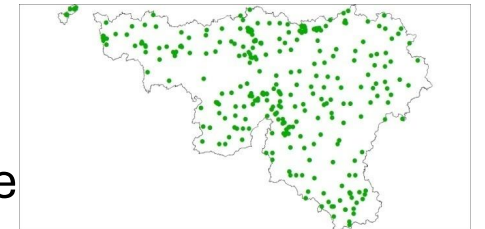
GHmax = 3 (global H or Mahalanobis distance)

NHmax = 1 (neighbor distance)

Soil spectroscopy in Wallonia

Currently

- Foss XDS instruments
- Annual calibration/verification of the apparatus and database update
- Growing the initial spectral library and its representativeness's
 - The contributors :
 - are the analytical laboratories of REQUASUD network : spread on the region, they are the most able to feed the library on an efficient way;
 - work with the same reference methods and the same equipment. That way, the so built spectral library is homogeneous and usable by every lab of the network, using the same predictive model.



Soil spectroscopy in Wallonia

Currently

	Croplands	Grasslands	Vegetable gardens		Ideal		
n	10.814	5.291	431			n	1.970
R ²	0,81	0,67	0,55		> 0,7	R ²	0,62
RMSEP	5,18	5,76	4,47		<<<	RMSEP	1,88
RPD	2,30	1,74	1,48		> 2	RPD	1,63

Improvements possible

Approximation of the uncertainty



dried soil



fresh soil



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Thank you



Avec le soutien de
la

