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A guide to estimating soil characteristics using the Agri-Spec NIR analyser

Sustainable Soils Programme

Internal Report IR/06/034



BRITISH GEOLOGICAL SURVEY

SUSTAINABLE SOILS PROGRAMME

INTERNAL REPORT IR/06/034

A guide to measuring soil characteristics using the Agri-Spec NIR analyser

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Figure 1 Principal Component Analysis of samples analysed by NIR spectroscopy for organic C. Note that the PCA found the outliers in the left hand bottom corner and when checked these were all found to be sub soils, thus optically different from the top-soil.	8
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Summary

This report describes a step-by-step methodology for using the AgriSpec NIR (near infra red) spectrometer, purchased by the BGS in 2005. The instrument has considerable potential for a survey organisation with large soil archives but there are limitations and constraints of which the user needs to be aware. The guide includes sections on the requirements for creating a calibration model and its validation, as well as describing limitations in its use. A step-by-step guide to the use of the instrument is included so that new users will require minimal supervision. Information on potential statistical approaches to obtain quantitative estimates of soil properties from their NIR spectra are included.

1 Introduction

The use of Near Infra Red (NIR) spectrometry for determining soil characteristics has increased in recent years. Although the technique has been available for many years, the increasing sophistication of statistical packages has broadened its capabilities in estimating soil parameters as diverse as pH, organic C and N, exchangeable cations, available P and particle size. Viscarra Rossel *et al.* (2006) include a table that extensively reviewed the parameters that have been measured by various authors. This is a useful resource when requiring literature to examine the feasibility of new measurements. NIR is a tool that rapidly collects spectrums of analysed samples across the NIR wavelengths of the electromagnetic spectrum. The rapidity (typically 1 second) at which these spectra can be collected means that the technique has huge potential within a Survey organisation. One of the benefits is that it can allow spatial analysis of soil properties over large areas where cost may be prohibitive if wet chemistry methodologies had to be employed.

The method works by scanning samples in the NIR (750-3000 nm) part of the electromagnetic spectrum. Specific features in the spectra must then be identified using chemometric statistical packages. There are many reviews on the physics of NIR spectrometry including those by George & McIntyre (1987) and Colthup et al. (1975). A basic and very good introductory guide to the technique is provided by the instrument manufacturers and can be found on the BGS NIR pages.

This short guide is designed to help the user operate and understand the potential of NIR spectrometry as well as recognise the limitations of its use.

2 The use of NIR spectrometry

There are some basic principles that must be understood so that NIR spectrometry can be used and these are as follows.

2.1 KNOWING WHAT SOIL PROPERTIES YOU CAN ESTIMATE

It is important to understand that NIR estimates soil properties based on the spectra of a soil sample and it does not directly measure the property. This is a very important concept to understand in the use of NIR spectroscopy. At present it is only possible to analyse samples from a set of soils that have similar characteristics e.g. a soil series or a particular soil horizon. This means that the parameter that you want to examine (e.g. Organic C) has to be measured from a calibration curve constructed using soils of a similar type or horizon. It would not be possible, for example, to create a calibration curve for organic C in clay soils and then use it on a sandy soil. Separate calibrations for separate soils need to be made. This even applies to top and sub soils where the differences in soil properties may cause large differences in optical properties. For example, Figure 1 shows Principal Component Analysis (PCA) for data collected to analyse organic C in Newport Series sandy soils overlaying the Sherwood Sandstone. It is noticeable that

the outlying points are all from the subsoil where there is very little organic matter and the soil particles are mainly sand. This demonstrates an occasion when a separate calibration curve should be constructed for the subsoil because the optical properties are very different to those of the calibration range. Areas where particular care may need to be taken, for the reasons stated above, are on contaminated sites or urban soil surveys. Contaminated sites may produce problems because there is potential for large amounts of industrial waste mixed in with the soil. These soils, although collected within a small area may therefore have very heterogeneous properties. Urban soils, could exhibit similar degrees of heterogeneity because of areas of made-ground, bought-in top-soils and mixing of top and sub-soils in the building process.

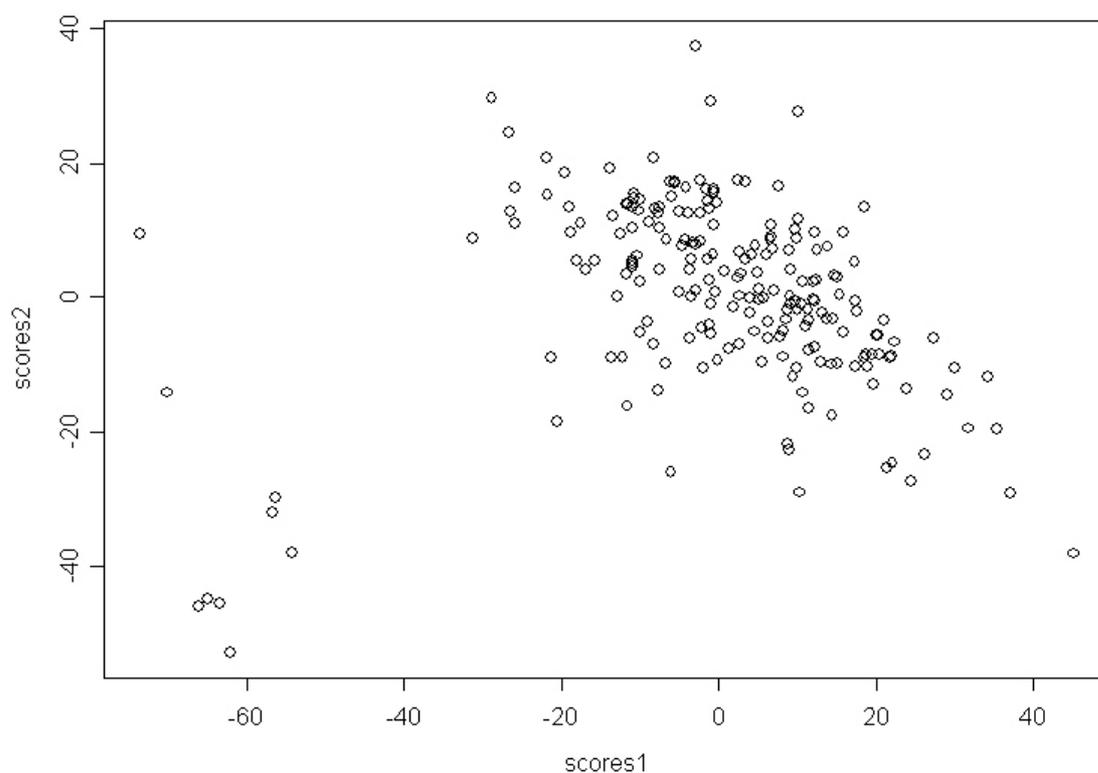


Figure 1 Principal Component Analysis of samples analysed by NIR spectroscopy for organic C. Note that the PCA found the outliers in the left hand bottom corner and when checked these were all found to be sub soils, thus optically different from the top-soil.

2.2 PREPARING A CALIBRATION GRAPH

Calibration curves need to be constructed using a subset of the samples to be analysed. The samples selected should span the expected range of concentrations. Once the appropriate samples have been selected, sub-samples will need to be submitted to a laboratory where the parameter to be measured can be analysed using accepted techniques. For example, organic C can be measured using the Walkley-Black method or by combustion methods. The remaining calibration samples can then be scanned using the NIR, so a group of spectra for the calibration is acquired.

2.3 VALIDATION OF THE MODEL

Results will be generated for the bulk of the samples set from the calibration curve. In effect the calibration curve is a model and will need to be tested to validate the results. It is therefore necessary, once results have been obtained, to take a further subset of samples and submit these to a laboratory for analysis of the property under study. Samples chosen for analysis should come from the complete range of results obtained; a useful reference that discusses validation requirements is Brown et al. (2005)

3 Using the Agri-Spec NIR spectrometer

3.1 BOOKING THE INSTRUMENT

The Agri Spec NIR spectrometer was bought primarily for the mobile laboratory, although it is housed in the E Block lab. A booking form has been created for users to record availability and use. This form is currently located on the W drive and the directory location is:
W:\CBH\EnvironHealth\projectInformation\NIR booking.xls

3.2 NIR SCANNER OPERATION – BASIC GUIDE TO USING THE AGRI-SPEC NIR SPECTROMETER

1. Switch the AgriSpec analyser and lamp on first and leave for at least half an hour to warm up.
2. Switch the Computer on. No password is required, user name is 'administrator'.
3. After warm up, double click on 'Indico pro' icon.
4. Goto File – new project. The directory route for the project will be, unless changed, C:\Program files\indicopro\projects\.
5. Enter project name.
6. Add project description (optional).
7. Goto Spectrum – Sample count / average – change the number of readings to 25. This means the saved spectrum will be an average of 25 readings.
8. Goto Spectrum.
9. Take a new reference spectrum by clicking on this command on the pull down menu. Check the Optimise First Box. Collect a new reference using white reference disc. In reflectance mode this should give a straight horizontal line with a value of 1. Save white reference file in DN (Digital Number) format.
10. Put soil sample in muglight.
11. Check that spectrum will be produced in 'Reflectance' format by checking on the button in LH corner of the tool bar.
12. Goto Spectrum – Take a new spectrum.
13. Goto Spectrum – Save – give the file name etc.
14. Carry out this procedure for each sample with 2 readings @ 90° angles on muglight by turning the sample holder. Duplicate readings should have 'a' and 'b' suffixes.
15. Take a white reference reading every 20 readings. Save in DN format.
16. At the end of a set of readings save the project under File pull down menu.
17. Download the data as in section 3.4.

3.3 SIGNAL TO NOISE RATIO

Once the samples have been scanned it is important to carry out a set of readings to determine the signal to noise ratio. These readings can be incorporated into the statistical processing of the data. The signal to noise ratio is the ratio of the radiance measured to the root mean square (rms) noise created by the detector and associated electronics. To do this the following is undertaken:

1. Goto File – new project. The directory route for the project will be, unless changed, C:\Program files\indicopro\projects\.
2. Enter project name.

3. Add project description (optional).
4. Goto Spectrum – Sample count / average – change the number of readings to 25. This means the saved spectrum will be an average of 25 readings.
5. Goto Spectrum.
6. Take a new reference spectrum. Check the ‘Optimise First’ box. Collect new reference using white reference disc. In reflectance mode this should give a straight horizontal line with a value of 1. Save white reference file in DN (digital number) format.
7. Put the soil sample in the muglight.
8. Check that the spectrum will be produced in ‘Reflectance’ format (Buttons in LH corner of tool bars).
9. Goto Spectrum – Take a new spectrum.
10. Goto Spectrum – Save – give file name etc.
11. Repeat for the same sample in the same position (i.e. not changing the angle 90°) sixty times.
12. At end of the readings, save the project under File pull down menu.
13. Download data as in section 3.4.

3.4 DOWNLOADING DATA FROM THE AGRI-SPEC NIR SPECTROMETER

Once spectrums have been taken they will need to be converted to Ascii files.

1. File open project.
2. File View spectrum if the user wants to view a specific spectrum.
3. Otherwise go to Run ASD to ASCII converter.
4. Go to Select – you can do multiple samples at one time (use shift on top and bottom samples of list you want to convert).
5. Press convert to ASCII button – Output to a single file if enough columns exist in an Excel spreadsheet for the number of files you want to produce. The maximum number of of columns in an Excel spreadsheet is 256. Otherwise split the list.
6. Save as Text files.
7. Load into Excel.

4 Converting Agri-Spec NIR spectrometer scans to results

This section deals with how the downloaded scan data from the AgriSpec NIR spectrometer is converted into results.

4.1 PROCESSING THE SPECTRAL RESULTS

There will be two spectra for each sample that has been scanned if the standard procedure described above has been followed. An algorithm has been written by Dr David Kinniburgh at BGS in the R environment (www.r-project.org) to automatically process the spectra. The R program can be downloaded for free from the internet and installed on networked PC's. The user should start the R program, then open the script called "spectra_processing.R" stored on the w:drive at the following location:

```
W:\ssm\sp2\Data\critical_zones\SSG_parent\dave_k_R_code\spectra_processing.R
```

The user then needs to edit the datapath and datafile entries to point at their spectral file. The code can then be run by selecting all the code (Ctrl+A) and run by Ctrl+R.

The code undertakes the following steps:

- 1) It reads in the data.
- 2) It removes the pathname for each spectra (e.g. it truncates the "c:\folder1\subfolder1\filename.txt" entry for each entry in the spectral file.
- 3) It removes the two "steps" between the three detectors using a simple extrapolation, splicing the spectra together.
- 4) It checks to see that there are two spectra for each sample – and will inform the user if there is a mis-match.
- 5) It then averages the paired spectra, or in the case of there being only one spectra simply returns it.
- 6) It plots both the paired spectra for each sample, and then the averaged spectra.
- 7) Finally it writes the averaged spectra to a file which can be exported for further statistical analysis.

The user must alter the filename at the end of the code to return their file in a tab delimited text (.txt) format. At the time of writing the procedure for incorporating the features of the signal to noise ratio of the spectrometer into the processed spectra has not been included in the R code. It is likely that this will be achieved in the next few months and will be flagged in the file:

```
W:\ssm\sp2\Data\critical_zones\SSG_parent\dave_k_R_code\spectra_processing.R
```

4.2 CONSTRUCTING A CALIBRATION MODEL FOR SOIL PROPERTIES

The spectral data must be analysed statistically in conjunction with a set of calibration data and independent validation data so that predictions of a measured property can be made based on a sample spectra. There are numerous chemometric processing techniques available; one of the most popular is partial least squares regression (PLS-R). A researcher at Sydney University has developed a software package called (PaRLeS) that uses PLS-R to develop calibration models for soil properties based on their spectra. The package can be downloaded from:

<http://www.usyd.edu.au/su/agric/acpa/people/rvrossel/soft01.htm>

There is a .pdf file with instructions on using the package to perform PLS-R based on a set of soil spectra and measured soil properties. There are a series of options in terms of pre-processing then the partial least squares regression is applied to the calibration data. In the final step validation data can be loaded to generate predictions, and finally other soil sample spectra to generate predictions. It is important to note that partial least squares regression is just one of the chemometric techniques available for developing calibration models for NIR spectra.

References

Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

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