Developing MIR Calibrations to estimate soil properties

# Setting up for calibrations

Start OPUS

Click <Setup Quant2 Method>

* Components Tab  
  Enter all the soil properties (“components”) in the table with laboratory data.   
  IMPORTANT: Components must be in same order as in the table of laboratory data
  + <Add Component>
    - Enter Name, Unit, Digits after the decimal point for each soil property
* Spectra Tab
  + <Add Spectra>
    - Select Spectra for calibration set
  + <Set Sample Numbers>
    - Enter “Number of Spectra per Sample”   
      (This is the number of replicate scans per sample. The recommended number is 4)
    - <Set>
    - <Exit>
  + Switch to .CSV file with laboratory data
    - Highlight all data values (NOT header row or columns with sample ID)
    - Copy (<Ctrl>c)
  + Switch back to OPUS
    - Highlight top left cell
    - Paste (<Ctrl>v) data just copied from .CSV file
  + CHECK DATA LINES UP CORRECTLY
  + CHECK AGAIN THAT DATA LINES UP CORRECTLY
* Parameters Tab
  + “Preprocessing in calibration regions”: No spectral data preprocessing
  + “Mean Centering”: Check
  + “Calibration Regions”: from 4000 to 600
* Validate Tab
  + Select “Cross Validation”
  + “No. of samples leaving out”: 1
* Settings Tab
  + “Select preprocessing options for Optimize” by highlighting:
    - No spectral data preprocessing
    - Vector normalisation (SNV)
    - First derivative
    - First derivative + Vector normalisation (SNV)
  + “Maximum test range:” 4000 to 600
  + Uncheck “Run optimization in background”
* Store Method Tab
  + Select “Store only Spectra List + Parameters”
  + <Store Method>
    - Filename: …\MIR\_Calibration\Sample & Component List.q2
* Click <X> to exit Quant 2
* You should now have a basic template to use for calibrating each soil property. Take care NOT to overwrite this file

# Calibrating a soil property e.g. Clay

Click <Setup Quant2 Method>

* Load Method Tab
  + <Load Method>
    - Open Sample & Component List.q2 (created in previous section)
* Spectra Tab
  + <Set Data Set>
    - <Special Settings>
      * Select <Blank>
      * Select soil property currently being calibrated e.g. Clay
      * <Set> (This excludes any samples with no data)
      * <Exit>
    - <Exit>
* Validate Tab
  + Uncheck all Validation parameters except soil property of interest
  + <Validate>
    - Give validation a name e.g. “Clay\_O” (O for ‘original’)
  + Once validation has run QUANT 2 will switch to the Graph Tab
* Graph Tab
  + Use graphs to evaluate the calibration. R2 should be as high as possible
  + Rank should be as low as possible (“Rec:” shows OPUS’ recommendation)
* Store Method Tab
  + Select “Clay\_O”
  + Check “Store validation results”
  + <Store Method>
    - Name “Clay.q2”

## Optimizing the calibration

* Settings Tab
  + “Maximum test range:” 4000 to 600

### Optimize using General A

* Optimize Tab
  + Select <General A>
  + <Optimize>
  + Wait for optimization to complete – may take several hours
  + When complete the optimization runs will ordered from best (i.e. lowest RMSECV) to worst (highest RMSECV)
  + Select run at top (i.e. the best) by clicking on its “Number”
  + <Use Parameters”
* Validate Tab
  + <Validate>
    - Give validation a name e.g. “Clay\_A1” (A for General A and 1 for first run)
    - When finished QUANT 2 will switch to Graph Tab
* Graph Tab
  + Use graphs to evaluate the calibration. R2 should be as high as possible
* Optimize Tab
  + Select next best run from top which has a lower rank by clicking on its “Number”
  + <Use Parameters”
* Validate Tab
  + <Validate>
    - Give validation a name e.g. “Clay\_A3” (A for General A and 3 for third best run)
    - When finished QUANT 2 will switch to Graph Tab
* Graph Tab
  + Use graphs to evaluate the calibrations. Choose the calibration that has the lowest rank that has an R2 > the greatest R2 of all the runs minus 1.  
    e.g. If Clay\_A1 has R2 of 80.00 and rank 10 and Clay\_A3 has R2 of 79.1 and rank of 8, then choose Clay\_A3

### Optimize using General B

* Optimize Tab
  + Select <General B>
  + <Optimize>
  + Wait for optimization to complete – may take several hours
  + When complete the optimization runs will ordered from best (i.e. lowest RMSECV) to worst (highest RMSECV)
  + Select run at top (i.e. the best) by clicking on its “Number”
  + <Use Parameters”
* Validate Tab
  + <Validate>
    - Give validation a name e.g. “Clay\_B1” (B for General B and 1 for first run)
    - When finished QUANT 2 will switch to Graph Tab
* Graph Tab
  + Use graphs to evaluate the calibration. R2 should be as high as possible
* Optimize Tab
  + Select next best run from top which has a lower rank by clicking on its “Number”
  + <Use Parameters”
* Validate Tab
  + <Validate>
    - Give validation a name e.g. “Clay\_B3” (B for General B and 3 for third best run)
    - When finished QUANT 2 will switch to Graph Tab
* Graph Tab
  + Use graphs to evaluate the calibrations.
  + Choose the best one using the same criteria as above, e.g. Clay\_B1
* Store Method Tab
  + Select “Clay\_B1”
  + Check “Store validation results”
  + <Store Method>
    - <Select All>
    - Name “Clay.q2”