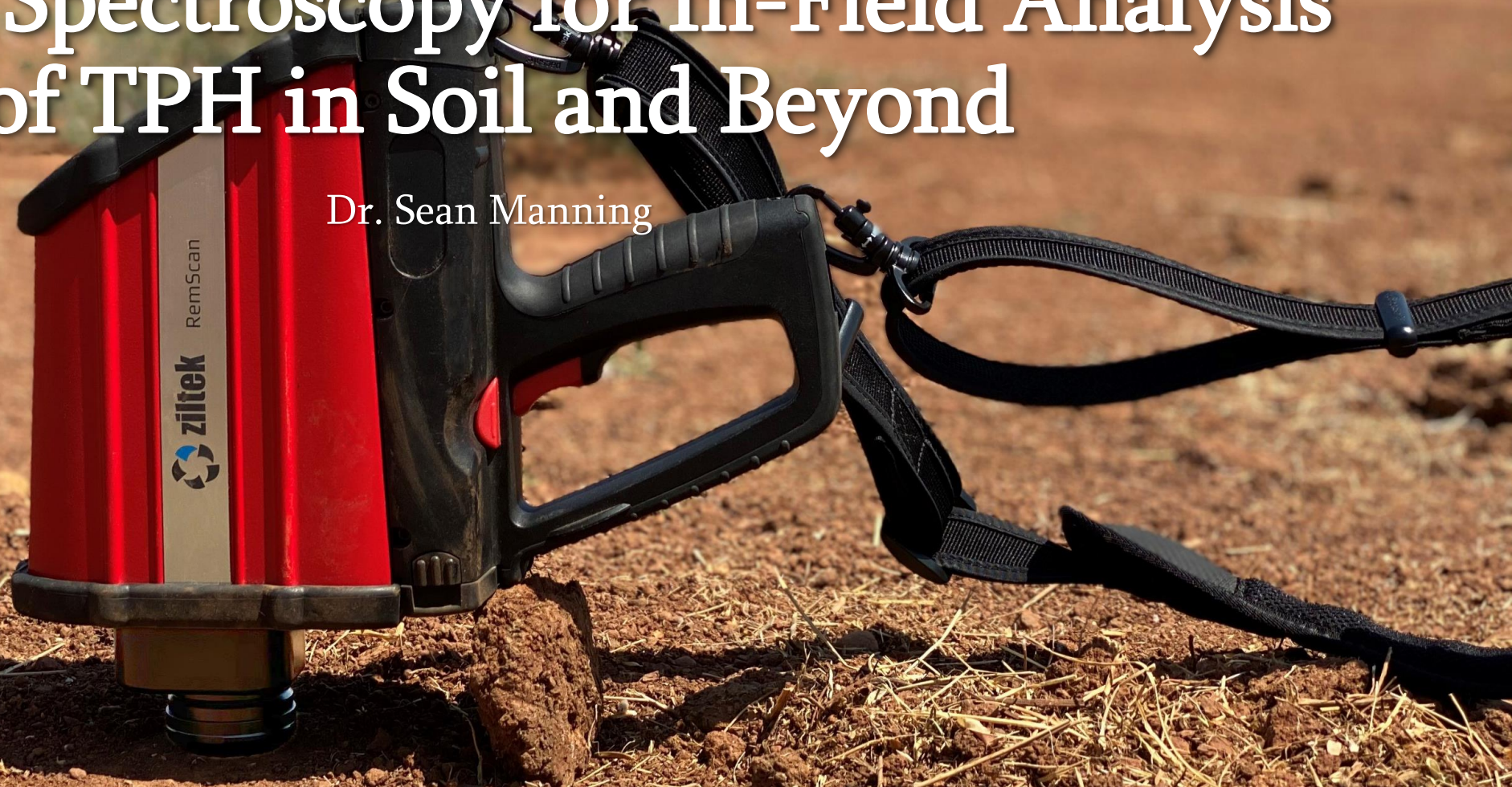


Infrared Spectroscopy for In-Field Analysis of TPH in Soil and Beyond

Dr. Sean Manning



Contents

- Company background
- MIR Spectroscopy
- Site specific calibrations
 - Concept
 - Process
- Universal calibration
 - Concept
 - Design
 - Validation
- Potential new directions
- Concluding Remarks



Ziltek

- Ziltek founded in 2008 by Dr. Richard Stewart
- Located in Thebarton, Adelaide
- Ziltek commercialised CSIRO patented technology
- Measurement of total petroleum hydrocarbons (TPH) in soil with mid Infrared spectroscopy
- RemScan was developed
 - Handheld MIR spectrometer (Agilent 4100/4300)
 - Field kit
 - Lab stand
 - Drying box
 - Calibration service
 - Training
 - Support



Benefits of RemScan

- On the spot results in under 20s
 - Make decisions in real time
- Measure multiple parameters at once
 - Spectra are information rich
 - TPH + moisture + soil texture
- Nil incremental cost of measurement
 - Make more measurements → increase confidence
- Non destructive
 - Retain sample for lab analysis / rescan
- Correlated to metadata
 - GPS location
 - Time/date
 - Operator
 - Analysis method
- Secure and auditable
- Indelible record of sample
 - Re-evaluate at any time using latest methods



Ziltek

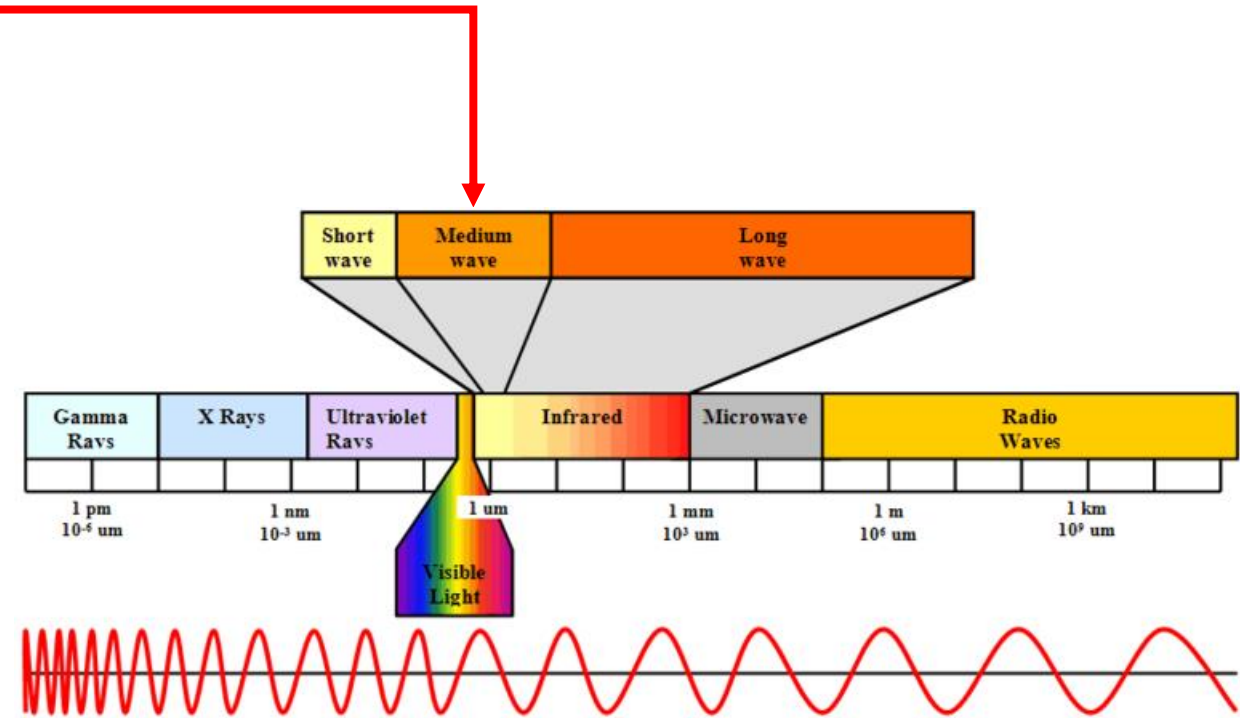
- Process control tool for TPH remediation
- Compliments laboratory analysis
- Many Customers
 - Oil companies: Chevron, Shell, Total, Sinopec
 - Analytical Labs: ALS Malaysia
 - Mining: FMG, Alcoa, Roy Hill
 - Environmental/Waste: Cardno, Envisol, Suez, SAES, PolyEco, AECOM, DEME
 - Universities: RMIT, Melbourne Uni, Concaawe, Jan Evangelista Purkyně University
 - Government: UN Italy, UN Congo, Indonesian Government
- RemScan has been use to remediate soils/sites from all over the world





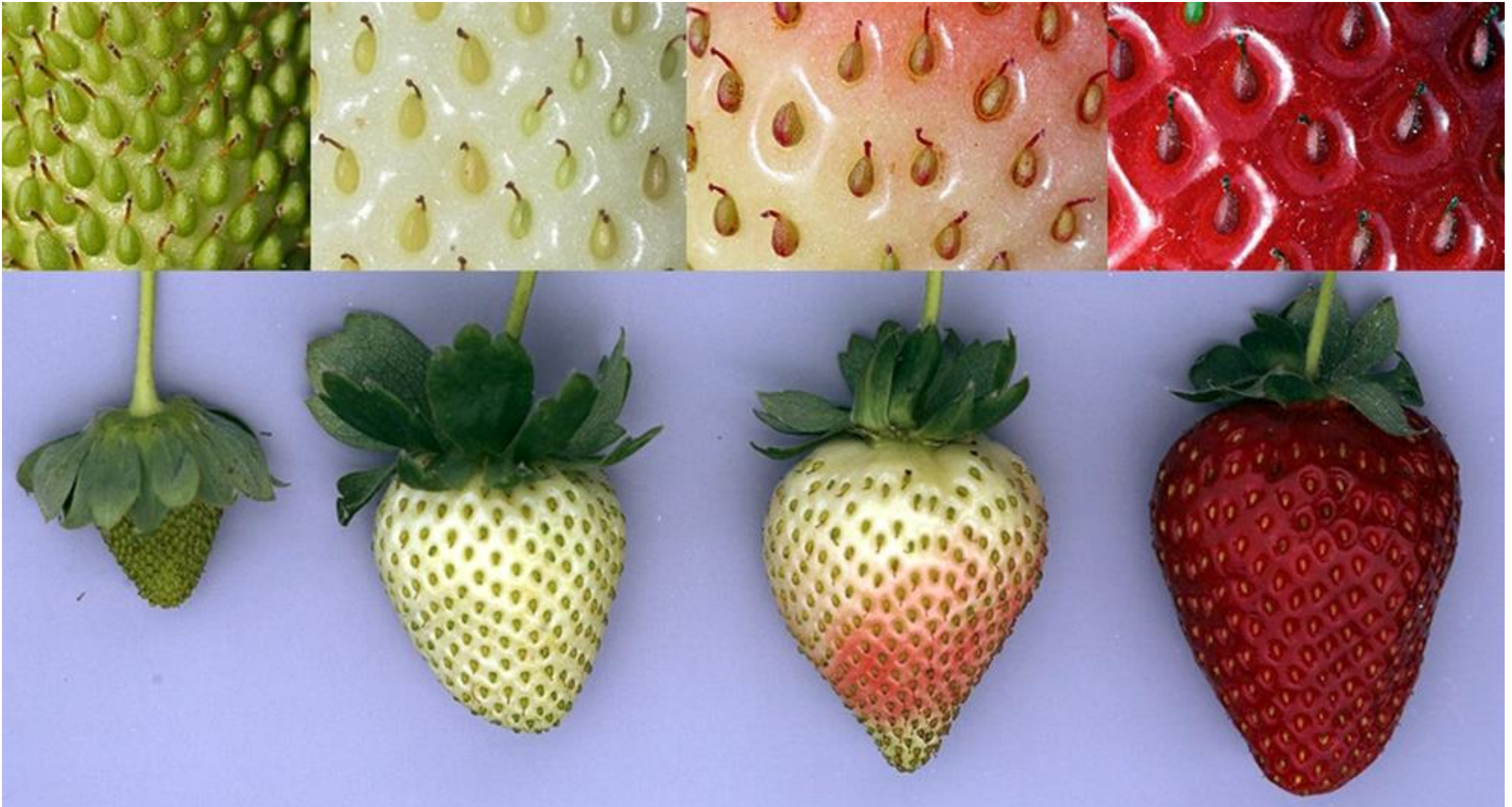
MIR Spectroscopy

- Mid infrared light = long wavelength light, 2-20 μm
- Causes molecular bonds to vibrate
- Reflected light from samples is missing certain colours
- Record of the reflected colours is a spectrum
- Spectra tell us what substances are in a sample
- Spectra + lab measurements can be turned into calibrations





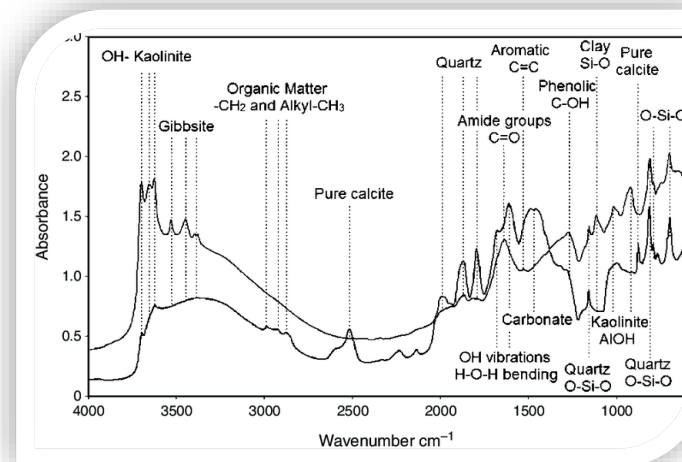




MIR Scanning



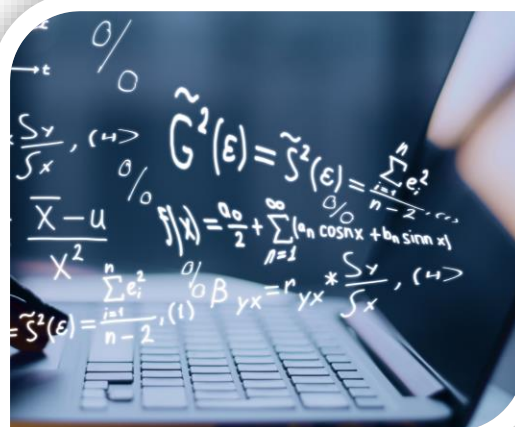
Identification



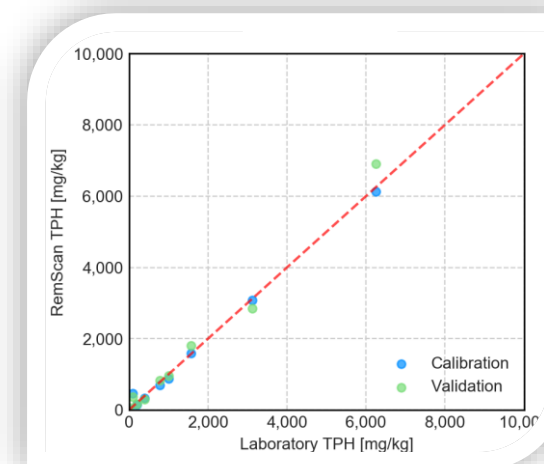
Laboratory Assay



Mathematics



Calibration



Site Specific Calibrations

- Prior to use RemScan must be calibrated
- Site specific calibrations provide the greatest accuracy
 - LOD as low as 50 mg/kg
 - Dependant on soil type, heterogeneity, interfering species (SOC, carbonate)
 - May require ongoing monitoring
- Collaborative process between customer and Ziltek



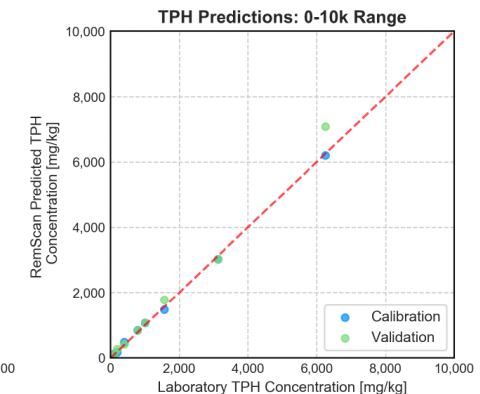
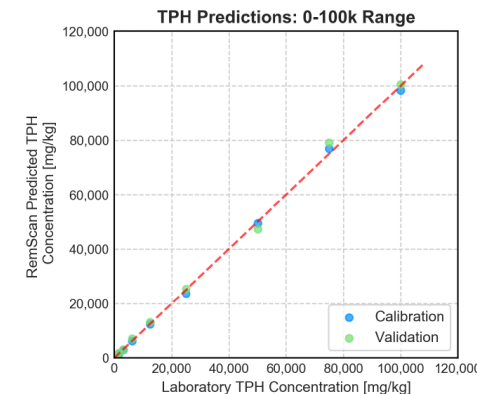
Site Specific Calibrations

- Customer
 - **Sampling** – guided by Ziltek staff
 - **Spiking** – Ziltek procedure
 - **Scanning** – with RemScan
 - **Send** – data to Ziltek
- Ziltek
 - **Analysis** – Determine the number of calibrations required
 - **Calibration** – Optimised cascaded PLS
 - **Report** – Regression statistics
 - **Monitoring** – Ongoing support to ensure the calibrations remain relevant

Calibration Report for Light Clay

Sub-Model	Model Cutoff	Samples	Spectra	PLS Factors	RMSECV (mg/kg)	r_{CV}^2	RMSEP (mg/kg)	r_P^2
1	100,000	14	115	2	802	0.999	1,224	0.999
2	50,000	12	105	2	384	0.999	895	0.998
3	20,000	11	100	2	553	0.995	576	0.99
4	12,000	10	95	2	82	1.0	286	0.99
5	6,000	9	85	2	50	0.999	160	0.998
6	3,000	8	75	2	68	0.997	139	0.99

Limit of detection: 46



RMSECV: Root mean square error for Cross-Validation

RMSEP: Root mean square error for Prediction

$$RMSE = \sqrt{\frac{\sum (x_i - \hat{x}_i)^2}{N-1}}$$

Light Clay Demo, Friday 27th August, 2021

Universal Calibration

- Small spills don't justify calibration setup time/cost (2-3 days)
- Need an “out of the box” calibration solution
- We developed a Universal calibration marketed as “Spill Response”
- Short setup time (<1 hour)
 - Representative clean soils are scanned to establish a site baseline
 - Site details are saved in software
 - Return visits require no further setup
- Fast results → Fast decisions



Regional soil sampling guided by CSIRO

- >200 samples
- Sourced primarily from Victoria
- Some South Australian samples



Customer Site Specific Data

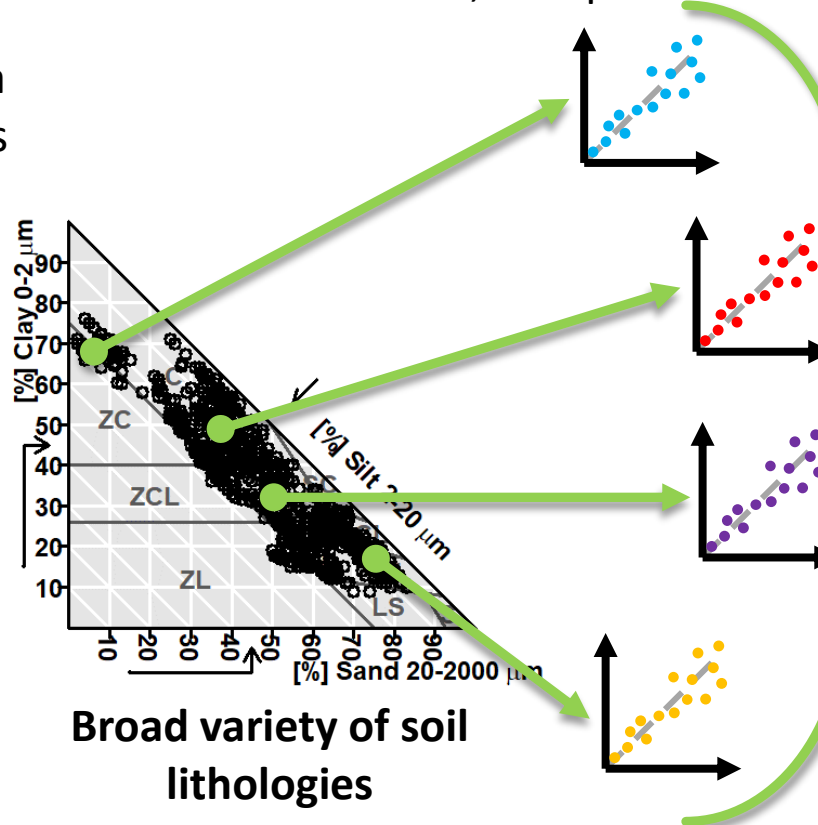
- International soils
- Screened for quality and suitability

In-house generated data

- Soil collection campaign
- Several soils added per month
- Looking to increase rate

Dilution series produced from samples

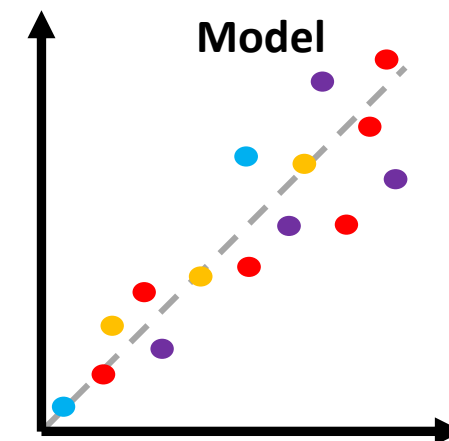
- Scanned on RemScan
- >10,000 spectra



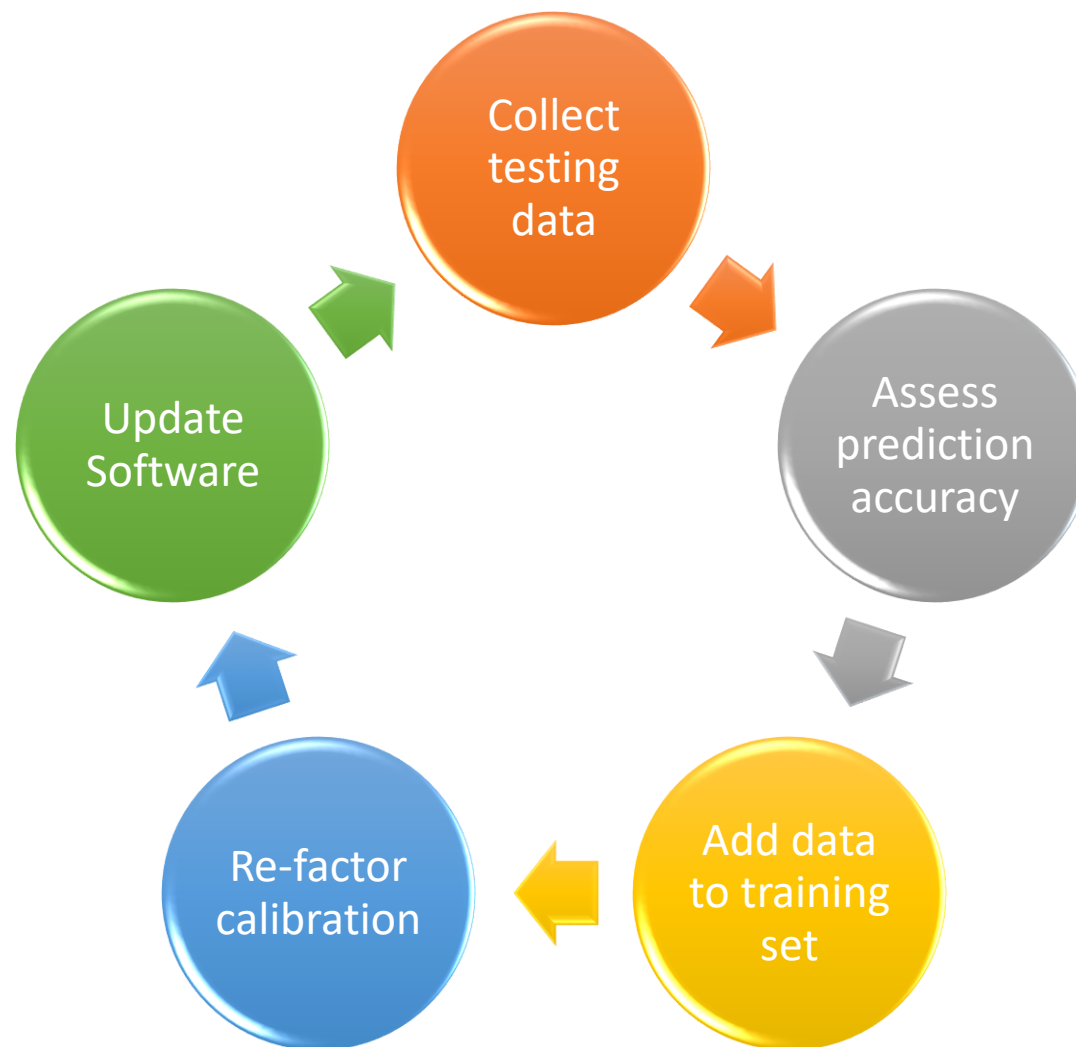
Machine Learning applied to dataset



Universal Calibration Model



Continual Improvement



Spill Response – Trial

- Suspected leaky pipe between waste oil hopper and storage tank
- Area was a steep, clayey slope with a small amount of vegetation and damp from rain
- Site baselining ≈10 min
- Samples collected
 - Jar samples for later lab analysis
 - Scanning samples
 - Dried on sample slides ≈30 min
- Dried samples scanned
 - Contamination results obtained
- Entire job completed in ≈3 hours
- Lab assay turn around ≈3 days
- Lab and RemScan results in agreement!



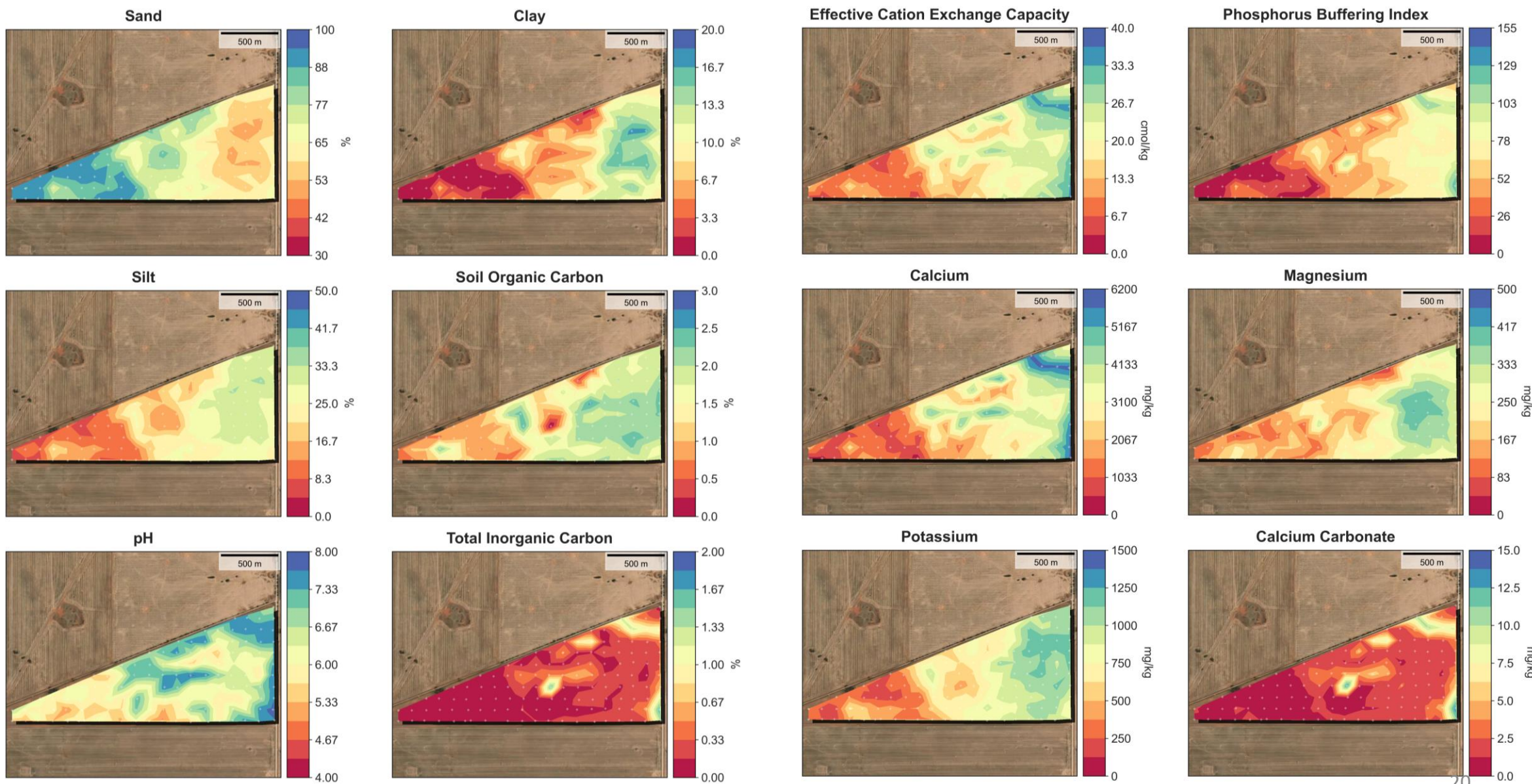
Where to now?

- Ziltek are leaders in in-field TPH measurement of soil
- Looking to expand the capabilities of RemScan / enter new markets
- Precision agriculture and soil carbon sequestration show potential
 - Scientific literature
 - Climate change
 - Food security
 - Water usage





Agricultural Field Trial Results



Thanks for listening

Questions welcome

Contact→

