



STEPPING UP IN THE NEW NORM

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Rapid Field Analytical Methods for Total Petroleum Hydrocarbons

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Project Background

Opportunity

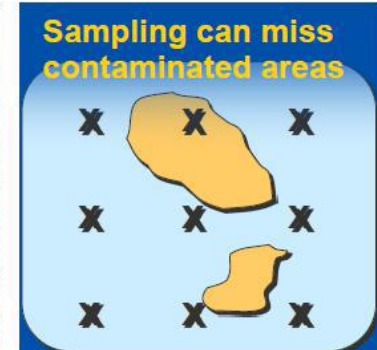
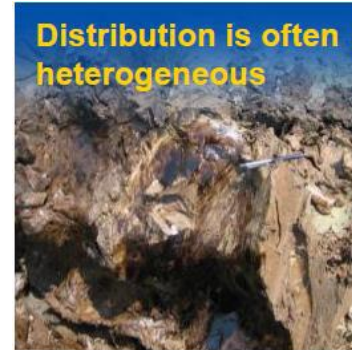
Soil samples from hydrocarbon impacted soil in CPI operations need to be tested for Total Petroleum Hydrocarbon (TPH)

- **Delays in sample analyses and decision making** due to thousands of soil samples per week needing analysis
- Lab analysis can take 2-4 weeks

Approach

Development of rapid TPH analytical method to increase accuracy and efficiency

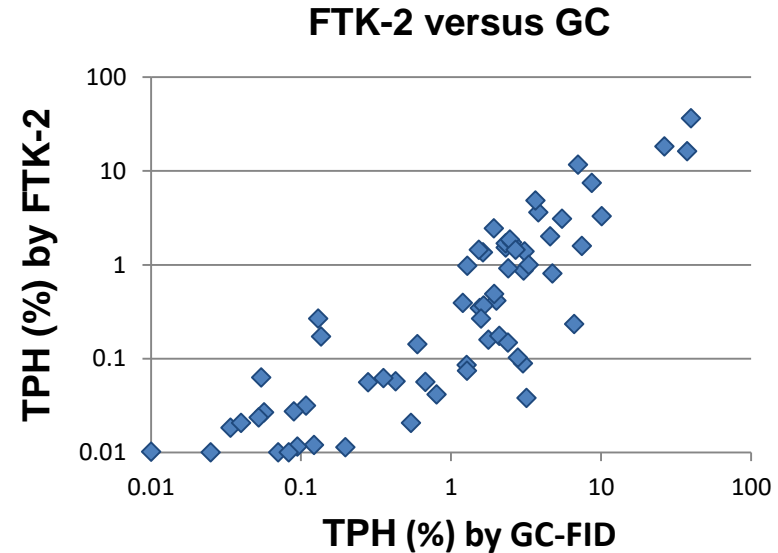
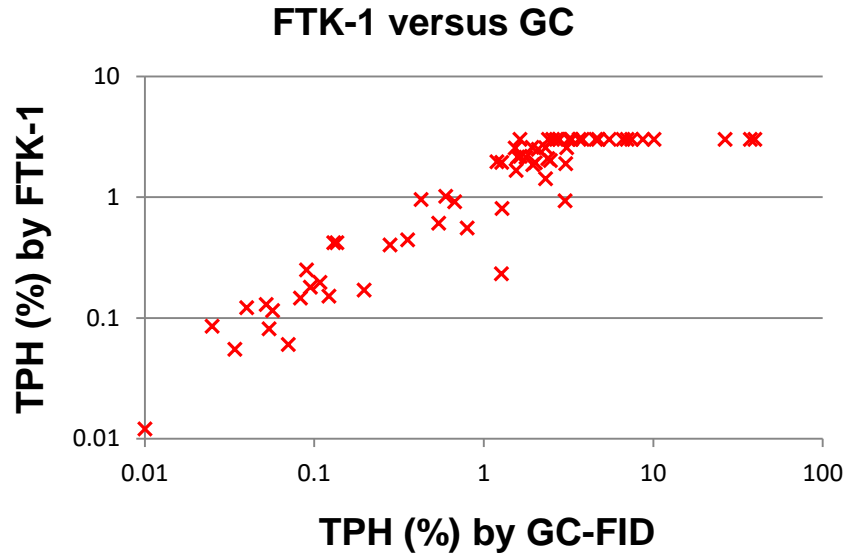
- 1) **real-time remediation process monitoring**
- 2) **reducing the number of samples going to lab**



Six FTKs have been Selected and tested with field soil samples

	Method	Extraction	Extraction Solvent	Data Quality
FTK-1	Turbidity	Yes	Methanol	semi-quantitative screening
FTK-2	Infrared	Yes	Hexane	quantitative
FTK-3	Ultraviolet fluorescence	Yes	Methanol	semi-quantitative screening
FTK-4	Colorimetry	Yes	Dichloromethane	semi-quantitative screening
FTK-5	Visual	Yes	Heptane	qualitative screening
FTK-6	Visual	No	Water	qualitative screening

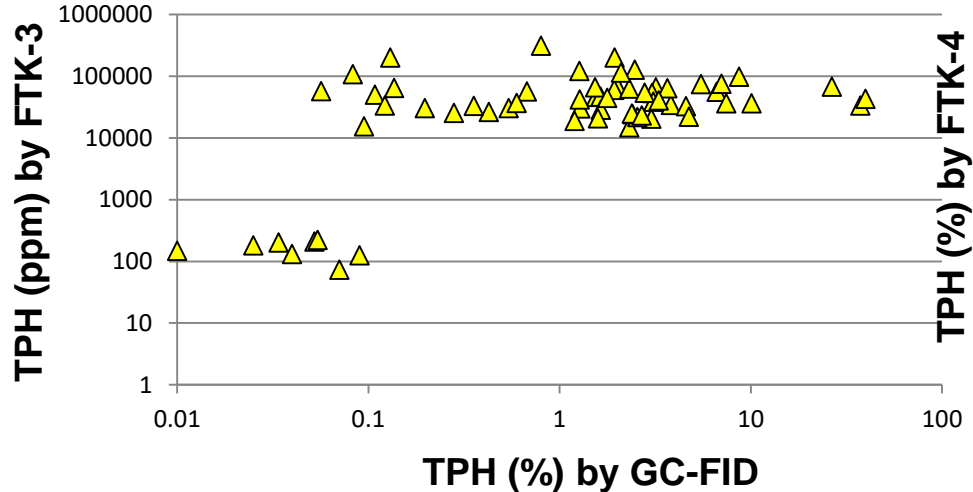
FTKs Performance Evaluation Results



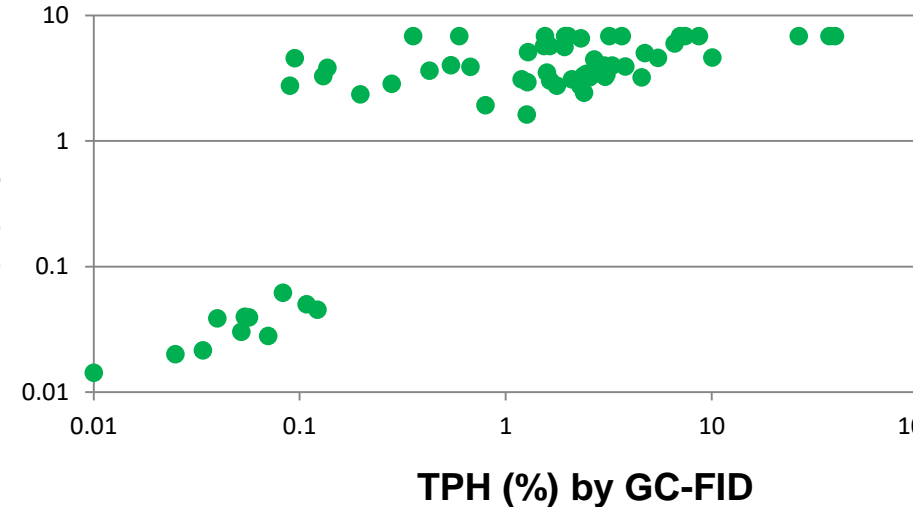
FTK-1 and FTK-2 demonstrated the good correlation with referenced lab GC data

Six FTKs have been Selected and tested with field soil samples

FTK-3 versus GC



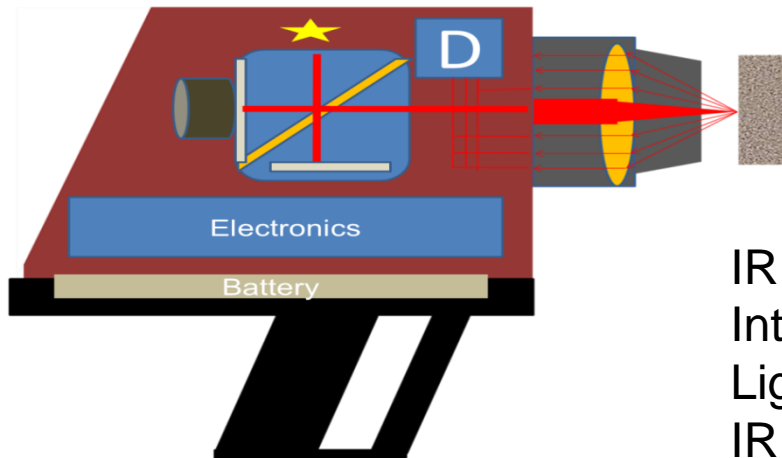
FTK-4 versus GC



FTK-3 and 4 might be able to use in field settings after proper calibration
 FTK-6 showed non-detect for all samples due to matrix effects

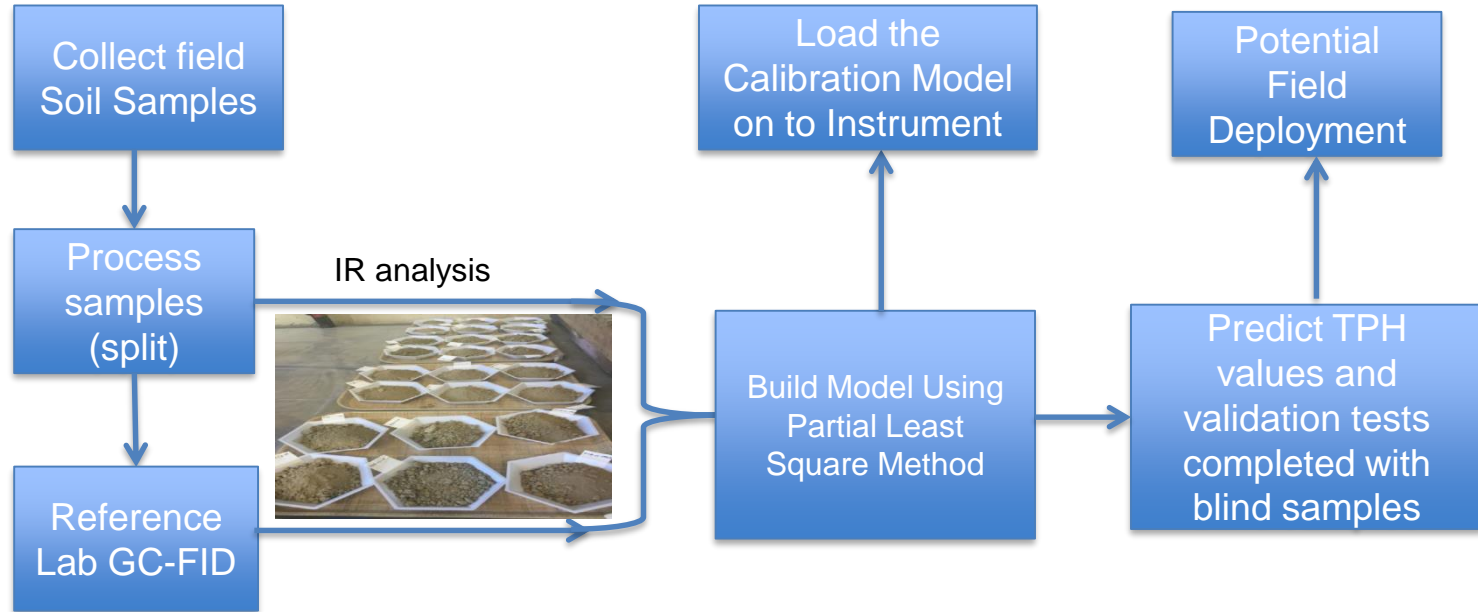
Handheld IR Instrument for non-destructive TPH measurement

- Portable handheld IR instrument
- Diffuse reflectance of IR light reflected from the sample
- The world's first handheld instrument for the direct measurement of TPH in soil
- User simply pulls the trigger for a 15 second reading of TPH (C_{10} - C_{36}) in mg/kg



IR light is emitted
Interacts with the surface of the sample
Light is diffusely reflected back to detector
IR spectrum (readout) is produced

Field Pilot Approach



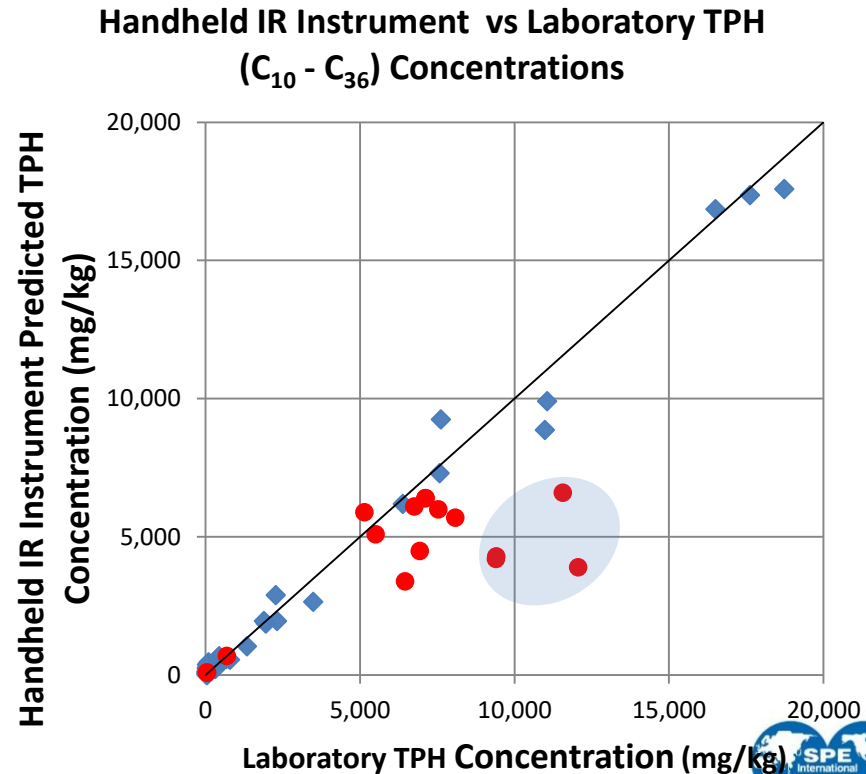
Pilot Studies Results Evaluation- Minas Field

Calibration model completed with 111 soil samples from Minas field at TPH range 0-120,000 mg/kg

Using Minas calibration model vs. GCFID Data for validation Test
Validation Samples (•) & Calibration Samples (◆)

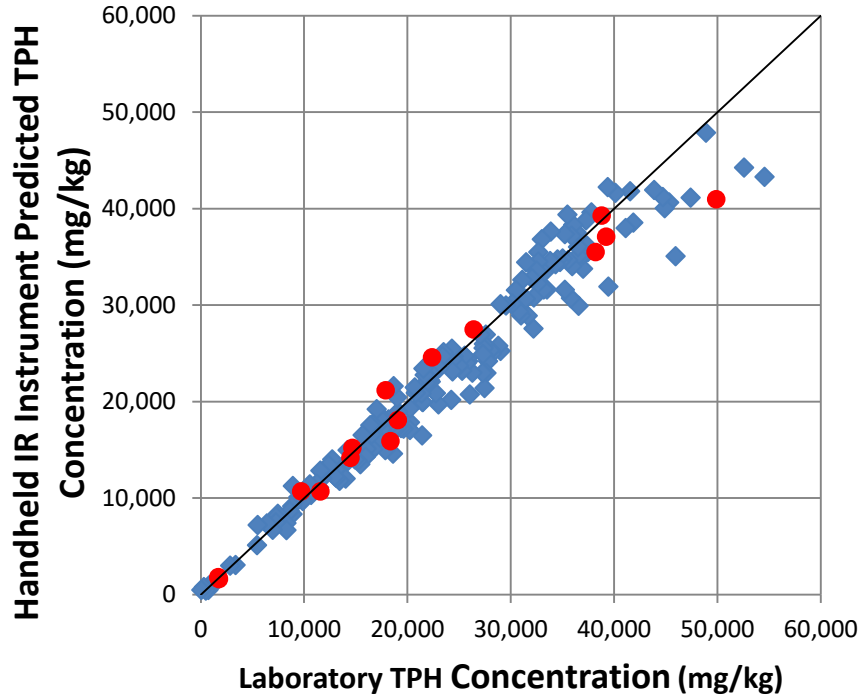
Outliner analysis- spectrum suggests the high clay contents of those samples

Detection limit of this model- 170 mg/kg



Pilots Studies Results Evaluation- Duri Field

Handheld IR instrument vs Laboratory TPH
(C₁₀ - C₃₆) Concentrations



Calibration model completed with 200 soil samples from Duri field at TPH range 0-50,000 mg/kg

Using Duri calibration model vs. GCFID Data for validation Test

Validation Samples (•) & Calibration Samples (◆)

Detection limit of this model- 380 mg/kg

Accuracy for various assay ranges for calibration samples for Duri field vs. Minas field

Duri site (limited calibration range up to 5%)

Assay Range (mg/kg TPH)	RMSECV (mg/kg TPH)	Relative Standard Deviation* (%)
0 - 5,000	376	n/a
5,000 – 15,000	930	≤ 19
15,000 – 20,000	1,390	≤ 9
20,000 – 30,000	2,107	≤ 11
30,000 – 50,000	2,815	≤ 9

Minas (wide range of calibration range up to 12%)

Assay Ranges (mg/kg TPH)	RMSECV mg/kg TPH	Correlation Coefficients (r^2)
0 - 3,000	170	0.92
3,000 - 5,000	184	0.96
5,000 - 15,000	410	0.98
15,000 - 30,000	803	0.99
30,000 - 120,000	2,375	0.99

RMSECV: Root-mean-square Error of Cross-Validation

Summary

- ❖ Portable handheld IR Instrument will enable rapid and accurate delineation of CPI sites & allows real time process monitoring for different remediation technologies
 - **Significant time reductions**
 - Real-time process monitoring
 - Rapid, field based testing
 - Improve data density for site assessment
 - Less waiting time for soil movement
 - **Improved Safety**
 - Prevents worker exposure and generation of waste by eliminating the use of solvents (used in the lab and in other field test methods)



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Acknowledgements

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