LIF Frozen Core Analysis (LIFFCA) using UVOST® and TarGOST®

Introduction

Dakota has collaborated with colleagues at CH2M to develop a laser-induced fluorescence frozen core analysis (LIFFCA) technique that allows for direct comparison of LIF response to non-aqueous phase liquid (NAPL) saturation on frozen sample segments cut from intact cores. While “homogenized splits” of soil samples have been analyzed on the benchtop with LIF for decades, LIFFCA allows for direct comparison of LIF response to geotechnical and chemical properties of the exact same intact core segment. Because LIF is a non-destructive method, the frozen sample segments can be cut from the intact cores by a laboratory that specializes in petrophysical analyses and shipped to Dakota for LIFFCA. The core segments can be subjected to LIFFCA and the exact same core segments can then later be returned for geotechnical and/or chemical testing.

Figure 1 illustrates the overall concept of harvesting cores for LIFFCA. Both the top and bottom faces of each core segment are typically analyzed so that close spatial heterogeneity is accounted for as best as is possible without destruction of the core segment. Alternatively, if LIFFCA analysis of the core segment to be geotechnically analyzed is not possible, then the faces of the core segments immediately adjacent to the analyzed core segment are analyzed with LIFFCA. The assumption here is that the faces of the core segment immediately above and below the chemically/geotechnically analyzed core segments closely match the faces of the analyzed segment.

The LIFFCA method has the substantial advantage of nearly eliminating the errors commonly introduced during attempts to correlate downhole LIF data with data gathered from immediately adjacent core samples. This is because spatial NAPL distribution heterogeneity always occurs and is often significant, especially in the case of dense NAPLs (DNAPLs) like coal tar and creosote. Making the chemical/geotechnical and LIF measurements on the very same sample greatly reduces uncertainty caused by spatial heterogeneity.

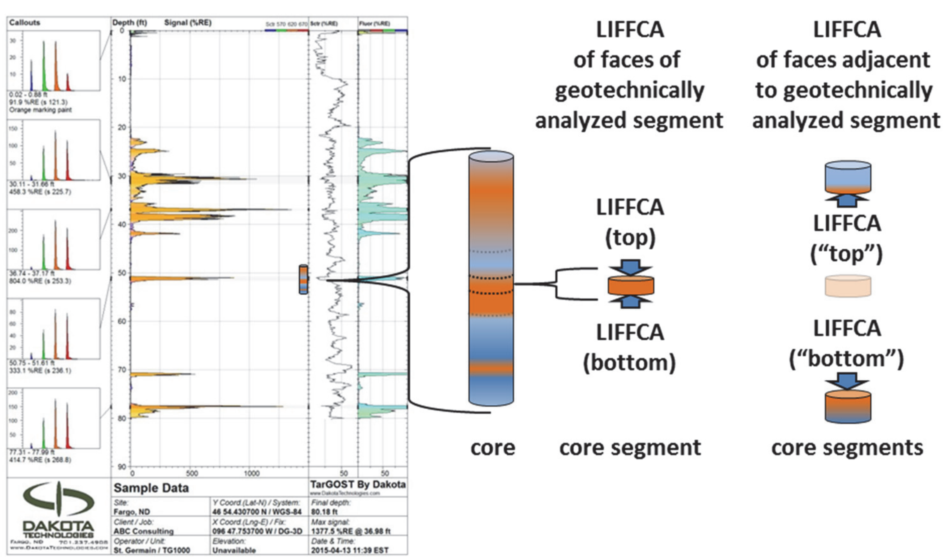

Figure 1. LIFFCA analysis overview. Core recovered next to TarGOST location is segmented and analyzed with LIFFCA.

The process begins in the field where cores are obtained, frozen on fry ice, and then sent to the lab that is responsible for chemical/geotechnical analysis where the cores are cut into segments using diamond saws. The segments to be “harvested” for LIFFCA analysis are selected by lab technician in coordination with the project engineer various lines of evidence such as: LIF field log data, visible inspection for NAPL, UV inspection/photography, and other feature. The core segments shipped to Dakota on dry ice in order to prevent thawing during shipping.

The relative affordability of LIFFCA allows testing of a relatively large population of core segments, from which a modest subset can be returned to the lab for more exhaustive geotechnical properties. For example, in some cases the focus is on the heaviest contamination (highest likelihood of being mobile) – so only those core segments showing modest to very high LIFFCA responses are selected for more exhaustive chemical/geotechnical testing. Alternatively, if the lower NAPL saturation range is the focus (finding the transition from low NAPL to non-impacted for instance) then those core segments shown to be highly NAPL impacted by LIFFCA can be disregarded and only the mid to lowest LIFFCA response samples need be returned to the lab for more exhaustive testing. In this fashion, LIFFCA can be used to minimize the number of core segments required for the relatively more expensive chemical/geotechnical analyses.

Apparatus

The frozen core segments are analyzed at Dakota’s facility on a LIF sample station built specifically for LIFFCA. Figure 2 shows the station being used to analyze a frozen core segment. The LIF analysis occurs in a small (ca $4mm^2$) area at the front face of the sapphire window (not visible in Figure 2 because it is hidden under the core segment in the technician’s hand). The station’s optics match those of Dakota’s standard percussion and CPT probes, so the data is directly comparable to downhole LIF. The station’s polished and seamless design allows for complete decontamination between samples, eliminating any risk of NAPL carryover between core segments.

Figure 2. A core segment being analyzed on the LIF sample station

Method

The core segments arrive at Dakota and are immediately transferred to a freezer until analysis. Just prior to analysis they are transferred in small batches to a cooler for transport to and from the LIFFCA sample station.

A typical LIFFCA analysis process is outlined below:

1. The technician turns on the LIF system, allows it to come to steady sate, and checks for proper function.
2. The technician checks the plate, sapphire window and mirror assembly for contamination or damage.
3. The Optical Screening Tool (OST) software (the same software used in the field) is set up by the technician to run in emulation mode where it records LIF readings sequentially over time as opposed to depth (emulating a push).
4. The technician calibrates the instrument with a Reference Emitter (RE) immediately before analyzing each core segment. This is the same RE that was used in the field, allowing for an “apples to apples” comparison between field and lab LIF response (%RE).
5. One at a time, core segments are removed from the cooler and unwrapped. A timer set to three minutes is started. Should the reading process for that core segment extend beyond three minutes (an unexpected delay of any sort) the core segment is returned to the freezer. The core segment is frozen for a minimum of one additional hour in order for the core to achieve fully frozen condition before another attempt at analysis.
6. The technician enters the sample ID into the Optical Screening Tool (OST) software.
7. If it is an “adjacent face” core segment being analyzed, the technician identifies the proper face (marked by the lab) to analyze and places that core segment face-down onto the LIF station’s sapphire window.
8. If this is the same core segment to be later analyzed for geotechnical properties the technician choose the “up” side of the core segment as the first to be analyzed (if so labeled) and places that face-down on the station.
9. The technician holds the core segment up against the window with modest downward force to ensure that minimal air gaps exist.
10. The technician cues the software to begin logging waveforms and simultaneously begins slow rotation of the core segment. To prevent “duplicating” interrogation paths the technician also translates the core segment back and forth across the sapphire-windowed region of the station. This scanning method results in roughly radial analysis paths that span the face of the core segment, in a manner similar to a phonograph record player. Large core segments might only be rotated once or twice, while small diameter core segments might be rotated numerous times in order to keep the sample continually “in motion” during the reading sequence.
11. Once the predetermined duration of reading has elapsed (typically 30 to 60 seconds resulting in 30 to 120 waveforms, depending on OST technology) the software stops acquisition and a pop-up message alerts the technician to position to the next face and continue scanning or end the logging sequence for that core segment.
12. Once both sides have been logged (or a single side in the case of adjacent core segment) the technician stops the recording and immediately repackages the still-frozen core segment. It is returned to the cooler and, shortly thereafter, the freezer. Only a minor “skin” of the core segment has thawed during this process.

13. The technician reviews the LIFFCA data in the OST software environment, typically highlighting the data from each core segment face so that the callout at left in the printout indicates the data attributable to each face. They also make notes under each callout concerning the alignment (top/bottom) or other noteworthy observations made during the analysis.

Data Output

The LIFFCA log is displayed in the familiar vertical field log format but what it actually represents are the spiral scans of the core segment's cut face (as illustrated in Figure 3). The technician highlights the data from a core segment's face for callouts (waveforms) to be displayed at left. The bold line along the left border indicates the data range spanned in the callout. The OST software displays the mathematically averaged waveform and the average and standard deviation %RE of the data that spans the region highlighted by the callout. In Figure 3 the average of all 30 LIF readings taken across the core segment's single face was 24.5%RE with a standard deviation of 29.1%RE. High standard deviations like this indicate heterogeneous NAPL distribution (as can be seen in the core segment face photo in Figure 3) while low standard deviations indicate homogenous distribution of NAPL (or an overall absence of detectable NAPL in the case of homogeneously low %REs). The data deliverables include the graphical log with callouts as shown in Figure 3 along with a text file containing all the readings. In some cases a scatter plot that allows easier viewing of the fluorescence nature of all waveforms recorded for the core segment is included.

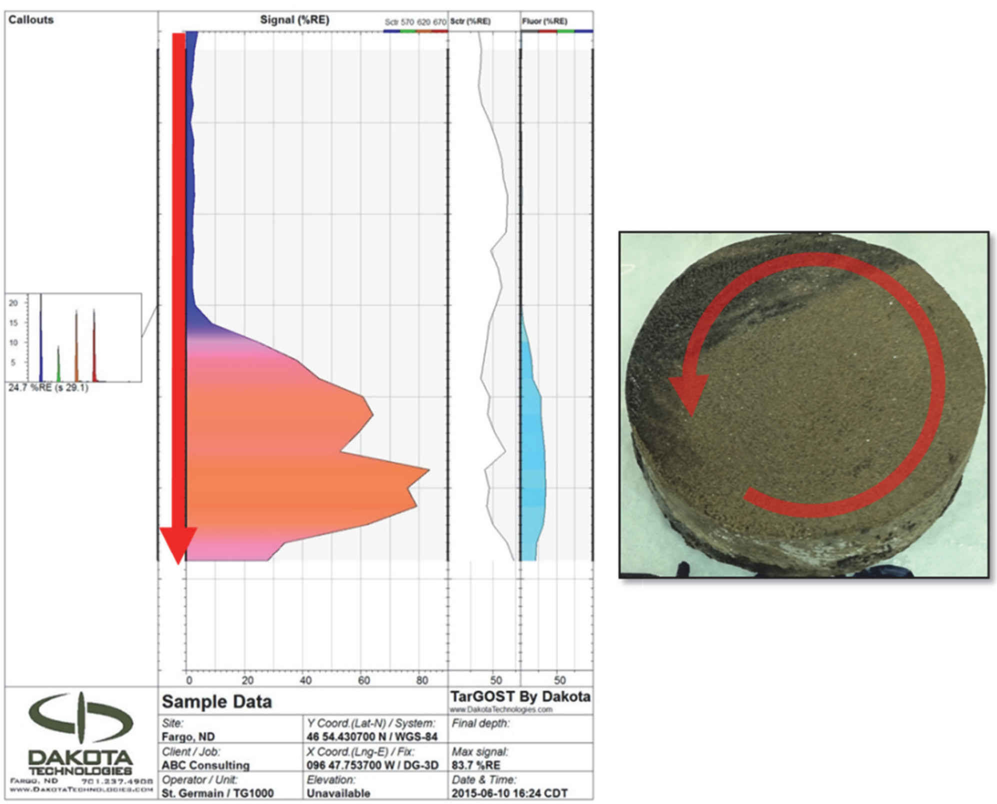

Figure 3. An example TarGOST LIFFCA log and photo of the core segment that produced the data.

Common Questions

Q: Dakota literature suggests that oxygen quenching lowers benchtop fluorescence response. Does this apply to core readings as well?

A: Recent testing by Dakota Technologies, Inc. (Dakota) on coal tar NAPLs that contains 20% oxygen and 0% oxygen reveal that “heavies” like coal tars (which are naturally self-quenching) are not affected by oxygen quenching - at least not to any degree detectable with the TarGOST. Studies on petroleum fuels (gasoline, diesel, kerosene) reveal that oxygen quenching of uphole samples is significant. While oxygen quenching studies of frozen fuel-impacted core segments has not been conducted we expect that oxygen quenching of core segment face NAPL will be significant enough to result in significant differences between LIFCCA response vs. downhole response (where little oxygen if any exists). One possible route to accounting for these differences is by doing oxygen quenching studies on site-specific NAPLs and subsequently applying the liquid NAPL oxygen quenching correction factor to the LIFFCA data.

Q: Does the fact that the cores are frozen or cold affect the response?

A: In recent testing of a number of cores from different sites where TarGOST was used in the analysis, the fluorescence data of the frozen cores was indistinguishable from thawed cores.

Q: The data looks very complicated. Do you have guides for basic LIF interpretation?

A: There are log reference guides attached to this LIFFCA description. Note that TarGOST and UVOST data, while similar in nature, are unique; have differing REs, laser and fluorescence wavelengths. In addition, TarGOST data incorporates scatter correction which is more complicated than UVOST data. For these reasons and others one should never attempt to directly compare UVOST %RE values to TarGOST %Res or vice versa.

Q: What are the maximum or minimum allowable core diameters?

A: The LIFFCA analysis relies on manual manipulation of the sample by the technician, so there are no size limits other than perhaps if the cores were so small they lack the mass necessary to stay frozen for the few minutes necessary for analysis.

If you have any additional questions please don’t hesitate to call Dakota Technologies, Inc. at 701-237-4908.

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