

## Initial Instrument Calibration and Demonstration of Capability

Each analyst must complete instrument calibration prior to performing this method. Calibration with OSS provided CSDs must take place: monthly; after instrument servicing; or after any changes to the method.

- Record a reference spectrum with OSS provided Calibration Standard Device Extractor “CSD #0.”
- Perform an analysis for each of the other CSDs (#1 to #7).
- Plot Concentration vs. Absorbance.
- Draw best-fit line, record slope and intercept.
- To update the calibration, contact an OSS technical advisor.

See Section 13 of the SOP for more details.

Each analyst must complete a Method Detection Limit (MDL) and Initial Precision and Recovery (IPR) study prior to reporting any data obtained from this method.

- Record a reference spectrum with a new and clean ClearShot™ Extractor.

**MDL: See Section 12 of the SOP**

- Test 7 replicates of a laboratory sample spiked with 4 mg/L oil and grease.†
- **The average measured concentration must be 1-5 times the calculated MDL.**

**IPR: See Section 11 of the SOP**

- Test 4 replicates of a laboratory sample spiked with 40 mg/L oil and grease.†
- **The average recovery of the four samples must be between 88% and 105% of the target and the %RSD must be ≤ 10.5%.**

## Daily Routine

New Blank, Laboratory Control Sample<sup>†</sup> (LCS), and Matrix Spike<sup>†</sup> (MS) samples must be analyzed each day and for each batch of 20 field samples.

### 1. Sample Collection and Homogenization

- Collect Sample in a cleaned glass container and acidify to a pH of 2 with HCl.
- Heat Ultrasonic Bath to 40°C.
- Shake Sample for 30 seconds.
- Submerge Sample in Bath and turn on vibration.
- Sonicate for 20 minutes or more until homogenous, shaking every 5 minutes.
- **At least 40 minutes sonication is required for LCS and MS samples.**

See Sections 9 & 14.1 of the SOP for more details.

### 2. Processing ClearShot™ Extractors

- Shake freshly homogenized sample for 30 seconds.
- Collect ~12mL of sample with syringe.
- Remove all air bubbles.
- Attach Extractor to syringe.
- Process Extractor upward.
- Remove water from Extractor's reservoir.
- Remove syringe while keeping Extractor's reservoir facing down.

See Section 14.2 of the SOP for more details.

### 3. ClearShot™ Extractor Drying

- Fix the Extractor to the Luer-Lok® fitting.
- Blow clean, dry air at 60 psi until Extractor is sufficiently dried. (5 to 40 min. required depending on sample's nature and compressed air dryness.)
- Use the backside drying attachment for samples with high particulate.
- Verify dryness in spectrum.

See Section 14.3 of the SOP for more details.

### 4. Spectroscopic Analysis

- Record a 200 scan reference spectrum with a new, clean ClearShot™ Extractor.
- Record a 50 scan Absorbance spectrum of the processed Extractor.
- Measure peak height at 2920 cm<sup>-1</sup> using a baseline from 2800 cm<sup>-1</sup> to 2990 cm<sup>-1</sup>.
- Calculate concentration using the Calibration slope and intercept.
- **The Blank must be ≤ 1 mg/L**
- **LCS recovery limits: 79% to 113%**
- **MS recovery limits: 70% to 126%**

See Section 14.4 of the SOP for more details.

† OSS recommends a spike solution composed of 4 mg/mL heavy mineral oil in acetone. See Section 10.1 of the SOP for more details.