Procedure for the Soxhlet Extraction and Gravimetric Determination of Bitumen

1. Introduction

This procedure is used to extract bitumen (solvent-soluble organic matter) from a wide variety of unaltered and altered source and reservoir rocks such as coal, lignite, oil shale and tar sands. The concentration of bitumen in total parts per million in the rock is determined gravimetrically.

2. Interface with Other Methods

Bitumen extracts can be used in the following methods:

1. **EGL Method 04** *Fractionation of Oil or Bitumen by Column Chromatography and Gravimetric Determination of Saturated, Aromatic, Resin and Asphaltene Compounds*
2. **EGL Method 05** *Elemental Analysis of CHNS/S/O*
3. **EGL Method 08** *Analyses of Whole Oil, Saturate, Aromatic, or Bitumen Extracts by Gas Chromatography Flame Ionization Detection.*
4. **EGL Method 09** *Determination of Biomarkers in Geologic Materials by Gas Chromatography Mass Spectrometry*

This method uses the following methods:

1. **EGL Method 25** *Method for Sample Login, Control, and Disposition*
2. **EGL Method 29** *Calibration of Laboratory Scales and Analytical Balances*

3. Materials and Equipment

1. Solvents; Chloroform, High Purity HPLC grade, Optima[1] or equivalent.
2. Glass wool; Pyrex brand or equivalent. Baked at 420 ˚C for 24 hours
3. 7mL clear glass vials with foil-lined caps; Baked at 420 ˚C for 24 hours
4. Sheet of Copper cut into strips approximately 2.5inch by .75inches; J.T. Baker 99.9% pure or equivalent
5. Soxhlet thimbles; Whatman high purity glass microfibre. Internal diameter 43mm, external length 123mm. (cat. #2814432), Baked at 420 ˚C for 24 hours
6. Glass filter; Whatman grade GF/A glass microfiber filters, 55mm diameter. (cat. 1827 055) or equivalent.
7. Boiling stones; Chemware brand PTFE or equivalent.
8. 500 mL glass round bottom flask
9. Soxhlet apparatus; 55/50 top 24/40 bottom, matching reflux condenser,
10. Heating mantle, fits 500ml round-bottom flask

[1] Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

11. Mounting stand with appropriate clamps and hardware, variable transformer, plumbing, Neslab CT-33 refrigerated recirculator or equivalent.
12. Long metal hemostatic clamp or tweezers approxamatly 12inch long
13. Funnel/Filtration system; fritted glass filter support base for 55mm filters with integral vacuum connector and 24/40 joint, matching funnel and clamp, mounted on a metal stand with appropriate clamps, connected to a vacuum line inside fume hood.Rotary evaporator apparatus; Buchi Rotavapor-R, Neslab RTE-101 refrigerated recirculator and a Savant Speed Vac water jet vacuum.
14. Nitrogen evaporator; Organomation N-evap
15. Class A volumetric pipets
16. Class A volumetric flasks
17. Glass Pasteur pipets

4. Procedure

1. Place a copper strip and 4 to 7 boiling chips in the bottom of a 500 ml round-bottom flask. Transfer 300 ml of chloroform into the flask. Insert the appropriate sized ground glass stopper and place the flask on a heating mantle.
2. Using a beaker as a stand, weigh out sample into thimble. Place glass wool in thimble on top of sample to prevent sample from splashing out.
3. Using long tweezers (or a hemostatic clamp) place the loaded thimble into the Soxhlet apparatus. Remove the ground glass stopper from flask, and connect it to the Soxhlet apparatus (condenser, soxhlet, round-bottom with solvent, and heating mantle).
4. Start the water circulation for the condensers. Turn on the heating mantles via a power strip/surge protector, and variac set to the 120V position. The variac is typically set up for chloroform extractions (input set at 50) and should not need adjustment. After several minutes, check to make sure solvent is boiling and is cycling through the Soxhlet siphon tube.
5. Run the extraction for at least 24 hours or until the solvent in siphon tube appears colorless.
6. Turn off the heat and water circulation. Wait for the apparatus to cool to room temperature.
7. To drain the Soxhlet, cycle as much solvent as possible through the siphon by tipping the Soxhlet. Using the long tweezer or hemostatic clamp, carefully lift the thimble a few centimeters without tearing the material and allow the remaining chloroform in the thimble to drain into Soxhlet. Alternately, use alligator clip hangers to hang the thimbles on the edge of the Soxhlet until each thimble stops dripping solvent.
8. Remove the thimble from Soxhlet and place in a beaker. Place a label on the beaker and set it in a fume hood to completely dry the thimble. Transfer the dried rock powder into labeled plastic bags for further analysis.

9. Assemble the filter apparatus consisting of a funnel, a glass fiber filter, a fritted glass support base, a 500ml round-bottom flask, and connection to the vacuum system.
10. Rinse the Soxhlet three times with approximately 5 ml of chloroform, and transfer the rinses to the filtration system, collecting the filtrate into a clean round-bottom flask. A moderate vacuum is usually sufficient.
11. Transfer the bitumen extract from the round-bottom flask to the filter system and filter using gentle vacuum. Rinse the round bottom flask three times with approximately 5 ml of chloroform and transfer each rinse to the filter system and filter.
12. In the Fracs_Template_1(Ver4.3) spreadsheet, note the change in color of the copper strip. The color change ranges from none (copper color), to faint steel gray (minor) to black (major).
13. Reduce the volume of the extract to 3 to 4 ml using a rotary evaporator under a moderate vacuum (about 18 in Hg), a water bath temperature of about 38 °C, and condenser coolant temperature of 0 ˚C. Avoid bumping or boiling during the solvent reduction process.
14. Transfer the extract to a volumetric flask (usually a 25 or 50ml) using a Pasteur pipet. Rinse the round bottom flask three times with 2-4 ml of chloroform and transfer to the volumetric flask. Bring the volumetric flask to full capacity with chloroform and seal the flask by placing an appropriated sized ground glass stopper in the opening.
15. Take a known volume (e.g. 1 ml) aliquot of extract using a volumetric pipet and place in a clean tared vial. Place the vial under nitrogen evaporator with nitrogen flowing until the solvent completely evaporates. Weigh the vial. Place the vial back in nitrogen evaporator again for 10 minutes. Weigh again. Repeat evaporation step until a constant weight is obtained (less than 1% per minute change).
16. Calculate the concentration of bitumen in the volumetric flask, and total ppm (wt/wt) in the rock. Given the calculated concentration of bitumen in the volumetric flask, another aliquot may be taken from the flask for column chromatography in a volume equivalent to a bitumen mass of 50 to 100 mg.

5. Calibration and Quality Control Samples

1. A method blank consisting of 50 to 100 grams of quartz sand baked at 420˚C for 24 hours is analyzed every 24 samples, when reagent lot number changes, or if a contaminant is suspected. The blank extract is analyzed by gas chromatography with flame ionization detection to check for contaminants.
2. Any deviations from the standard operating procedure are documented in analyst's lab notebook and in the LIMS.

6. Limits, Precautions, and Interferences

1. Health, Safety, and Waste;

- Chloroform is a suspected carcinogen and has been proven to have mutagenic effects on mammalian somatic cells. In humans, chloroform affects the central nervous system (brain), liver, and kidneys after a person breathes air or drinks liquids that contain large amounts of chloroform. If you breathe air, eat food, or drink water containing elevated levels of chloroform, over a long period, the chloroform may damage your liver and kidneys. Large amounts of chloroform can cause sores when the chloroform touches your skin.
- Always wear proper laboratory personal protection equipment such as nitrile gloves, safety goggles, lab coat and always work in a ventilated fume hood while working with chloroform.
- Disposal of all organic solvents and samples must be performed in accordance with the current waste disposal procedure.

2. Interferences

- Errors in weight measurements can arise from changes in ambient conditions within the lab or balance drift. For this reason is has been found useful to take the final bitumen wgt and vial tare wgt on the same day.
- If the extract is not properly filtered, calculated bitumen concentrations may be high.
- Skin oil is a possible contaminant. Glass fibre thimbles and filters should only be handled with tweezers. Glassware should be clean and baked (except volumetric flasks and pipets).

7. Acceptance of Data

1. In the event of that the method blank is determined by GC-FID to be contaminated, efforts will taken to determine and eliminate the source of contamination.
2. The event of contamination and the solution to the problem are to be documented in the analyst's lab notebook and in the LIMS.
3. All glassware used is Class A, Class A glassware is calibrated to specifications established by ASTM i.e. ASTM-E969-02 and ASTM E228-10.
4. No standard is used in this method, extractions are considered complete when the solvent t in the siphon tube becomes colorless.

8. Data handling and Transfer

The total ppm (weight/weight) in the rock is calculated using an Excel™ spreadsheet (Fracs_Template_1 (ver4.3)), transferred to the proper spreadsheet template for submittal into the LIMS via a shared network folder.

Once in the LIMS, results are validated by the analyst and approved by an appropriate QA official before being released to the submitter.

9. References

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10. Attachments

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11. History of Changes

R0: Initial Issue