Archaeological Pottery – FAME Experiment

Day 1: The goal is to extract the oily residue from the pores of the ceramic material.

1. Examine sherd and record all pertinent information.
2. Crush sherd (bits smaller than 3 mm)
3. Transfer to larger vial (black lid). (You will need the mass of the crushed sherd sample.)
4. In hood, pipet 2.5 mL of the extracting solvent into the vial and then recap tightly. The extracting solvent is dichloromethane and anhydrous methanol in 2:1 ratio by volume.
5. Let vial sit in hood until next class meeting.

Day 2: The goal is to separate the solvent from the crushed sherd bits. Hopefully the solvent contains the extracted oily residue. Then we can evaporate the solvent and claim the oily sample.

1. Make observations on vial contents.
2. With a beral pipet, withdraw the liquid portion from the vial and transfer to a smaller vial (white cap). Cap the smaller vial tightly.
3. Pipet 2.5 mL of the extracting solvent into the original vial to wash the crushed sherd sample.
4. Allow the crushed sherd bits to settle.
5. With the beral pipet, withdraw the liquid portion again and transfer it into the smaller vial.
6. Place the smaller vial into the centrifuge and spin at setting "4" for 5 minutes.
7. With a clean beral pipet, withdraw the clear liquid solvent portion away from the settled ceramic dust and place into a dry, clean and massed white vial.
8. Cap your vial and give it to your instructor. They will place it uncapped into the ventilation hood. The evaporation of the solvent will take place overnight.

Day 3: The goal is to complete the hydrolysis and transesterification of the triglycerides in your oily residue to produce a FAME mixture (fatty acid methyl esters).

1. Make observations on your vial contents.
2. Using a beral pipet, transfer 3 drops of your oily residue to the bottom of a weighed dry test tube. Remass the test tube on the analytical balance and determine the mass of the oily residue in the tube.
3. Working in the hood, add 1 mL toluene to the tube to dissolve the oily residue. Then add 2 mL of 5%v methanolic HCl to the tube. Place the cap on the tube.
4. Place the tube in the sand bath. The temperature of the sand bath is at approximately 50^{o}C. The reaction should take place at this elevated temperature overnight.

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Day 4: The goal is to perform a liquid-liquid extraction to prepare the sample that now hopefully contains a FAME mixture to inject into the gas chromatograph.

1. The sample needs to be at room temperature.
2. In the hood, pipet 1 mL of hexane and 6 mL of saturated NaCl (aq) into the test tube. Swirl the tube contents for 2 minutes and then let settle. The intent is to extract the FAME mixture into the hexane layer.
3. With a beral pipet, remove the top hexane layer and place it in the tiny vial with the blue cap.
4. Repeat steps 2 and 3 a second time so that you have at least 1.5 mL of sample in your tiny vial.
5. Before capping, add a pinch of solid sodium sulfate to the small vial and then cap tightly. Na_2SO_4 is a drying agent. It removes any water from the liquid sample.
6. Mrs. Ford will store these samples in the refrigerator until our trip to UC to run the samples through the gas chromatograph.

Day 5: The goal is to inject our prepared samples into the gas chromatograph to obtain a chromatogram for analysis.

1. Withdraw a two-microliter sample from the tiny vial and inject into instrument – Hewlett-Packard G1530A Plus..
2. The column is a Supelco Omegawax 250 Fused silica capillary column (30m X 0.25mm X 0.25 micron film thickness).
3. The instrument settings are as follows:
   Injector temp 250^{o}C
   Injection vol 1.0 uL
   Injection mode: split/split less
   Split ratio 50:1
   Starting oven temp 140^{o}C held for 1.0 min
   Oven temp ramp 10 deg/ min
   Final oven temp 240^{o}C
   Final oven temp hold time 10.0 min
   Detection mode Flame ionization detection (FID)
   Detector temperature 260^{o}C
4. Each run should take approximately 20 minutes.