SAMPLE PREPARATION OF NITROAMINES AND NITROAROMATICS IN SEDIMENT

Log-In

- Match the sample(s) to its Analytical Services Request form(s) and fill out a data sheet.
- Decant the excess water from each sample and dispose of it in the non-chlorinated waste stream.
- Place the samples in the sample storage refrigerator (4ºC ± 2º).

Pre-extraction Preparation

- Retrieve the samples from the refrigerator and allow them to equilibrate to room temperature.
- Assemble the paperwork for all the samples and create a database record for the set adding a set blank and spike.
- Rinse a spatula and two centrifuge bottles/cap assemblies for each sample with acetone and allow to dry. In addition, rinse two extra centrifuge bottles/cap assemblies for the blank and spike with acetone and allow to dry.
- Mix and homogenize each sample with a different rinsed spatula.
- Zero a balance. Place a bottle and cap assembly on the scale and transfer the sample from shipping container to the centrifuge bottle until a gross weight of 80-grams(g) is reached.
- Centrifuge 4 bottles together for 20 minutes @ 35%.
- Decant the excess water from each sample and dispose of it in the non-chlorinated waste stream.
- Again mix and homogenize each sample with its corresponding spatula.
- Remove a 1.8 to 2.2-g sample for determining the percent dry weight. This is calculated automatically by the drying balance. Record this number on the sample data sheet.
- Determine the wet weight needed for this sample by dividing 20-g by the percent dry weight. Record this number on the sample data sheet.

Extraction

- Tare the other rinsed centrifuge bottle.
- Weight out the appropriate sample wet weight. Record the weight on the sample data sheet.
- Tare scale and add 20-g of burned reagent grade sodium sulfate. Mix thoroughly. Add more sodium sulfate, if needed, until mixture is dry and loose. Record the total weight of the sodium sulfate added on the sample data sheet.
- Create a set blank and spike by adding 40-g of burned reagent grade sodium sulfate to two labeled rinsed centrifuge bottles.
- Dispense 10-µL (5000 pg/µL) of surrogate to all samples, blank, and spike using a 10-µL micro dispenser and baked glass bore. Note: Allow surrogate to come to room temperature before dispensing into samples. Vortex mixture thoroughly before using.
- Dispense 10-µL (5000 pg/µL) spike mixture to set spike only using a 10-µL micro dispenser and baked glass bore. Note: Allow spike mixture to come to room temperature before dispensing into samples. Vortex mixture thoroughly before using.
- Record the identification number and amount of spike and/or surrogate added on the sample data sheet.
- Soak each sample, blank, and spike with two or three milliliters of methanol and recap. Allow 20 minutes for the methanol to percolate through the sample.
- Add 50-mL of a 70% water / 30% acetone mixture.
- Recap and shake for 2 minutes each, venting often.
- Centrifuge sample @ 35% for 20 minutes. Let stand 30 minutes to equilibrate.
- Decant the extract into a rinsed graduated cylinder and record the volume on the sample data sheet.

Filter

- Rinse a 5-mL gas-tight glass syringe with acetone.
- Attach a 1-uM polytetrafluoroethylene (PTFE) membrane filter to the gas-tight syringe. Then attach a 0.2-uM PTFE membrane filter to the 1-uM PTFE membrane filter (i.e. piggybacked filters).
- Place this apparatus on a stand suitable to hold the syringe/filter setup.

- Transfer the centrifuged extract into the syringe barrel with a Pasteur pipette, being careful not to dislodge any centrifuged solids or to spill any liquid sample.
- Place the filter tip over a rinsed and labeled 100-mL volumetric flask and carefully insert the syringe plunger.
- Pass the extract through the filter.
- Using a Pasteur pipette, rinse down the sides of the graduated cylinder with 3-mL of the 70% water / 30% acetone mixture.
- Transfer the rinse to the syringe.
- Filter the rinse into the flask and repeat a second time.
- Bring to volume with reagent grade water.
- Carefully add a magnetic stir bar to each volumetric flask. NOTE: Use only disposable stir bars due to 2,4,6-trinitrotoluene absorption onto PTFE surface.
- Using a volumetric pipette, add exactly 1-mL of reagent grade toluene to each sample, blank, and spike.
- Place each sample on a magnetic stir plate and extract for 30 minutes. NOTE: The toluene should be pulled down by the stirring motion in tiny droplets. It should look like the motion of a tornado. Make sure stir bar is continuously stirring in the middle of volumetric flask.
- After the 30 minutes, turn off the stir plates and allow the toluene to move back up to the top of the sample. This should take about 15 minutes.

Vialing

- After the toluene has settled, use a disposable Pasteur pipette to transfer as much of the toluene fraction (top layer) into a correspondingly labeled vial (with insert) as possible. Record on the data sheet if an emulsion was present. NOTE: Avoid getting any of the water fraction (bottom layer) into the vial insert. Sometimes emulsions occur and must be broken. If they cannot be easily broken, pipette the emulsion into a centrifuge tube and centrifuge for 5 minutes. This should break the emulsion.
- Tightly cap the vial. NOTE: Always hold the vial so the insert does not fall out if the insert breaks the bottom of the vial.
- After all the samples are vialed, label the tray with the set number and schedule. Place this rack in the sample extract refrigerator (4ºC ± 2º).
- Ensure that all laboratory paperwork concerning this set is filled out. Complete any database entries and place the paperwork in the appropriate analysts' bin.

Further Enhancements

- A iso-amyl acetate extraction that will recover cyclotrimethylenetrinitramine (RDX) and provide higher extraction recoveries of 4-amino-2,6-dinitrotoluene, 2-amino-4,6-dinitrotoluene, and 3,5-dinitroanaline.
- A sample clean-up step that will remove chromatographic interferences.

Analyzing

- Performed exactly as the water method using 30 meter RTX-1 and RTX-5 columns with electron capture detectors. See SOP OMOO71.O Section 7.4.

NITROAMINES AND NITROAROMATICS IN SEDIMENT

Sample Preparation

Centrifuge the sample matrix and decant as much standing water as possible. Remove any large rocks or debris from the sample matrix and homogenize. After a moisture determination of the sample matrix, place 20 g., dry weight, of the sample matrix into another centrifuge bottle. Add 20-g. of burned reagent grade sodium sulfate to the centrifuge bottle. Thoroughly blend together the sample matrix and sodium sulfate. Repeat the process until the sample mixture is dry and loose. Surrogate the sample mixture with 10-µL's of a 5000-ρg/µL solution containing 3,4-dinitrotoluene. Dispense two or three milliliters of methanol onto the sample mixture and allow it time to percolate. Dispense 50-mL of a 70% water/30% acetone solution into the centrifuge bottle and shake it for 2 minutes, venting frequently. Centrifuge the sample mixture for 20 minutes and let it equilibrate undisturbed for 30 minutes. Decant the sample extract into a graduated cylinder. Filter the sample extract through a 0.2-µm polytetrafluoroethylene (PTFE) membrane filter into a 100-mL volumetric flask. Bring the flask to volume with reagent grade water. Add precisly one milliliter of toluene and a magnetic stir bar to the flask. Vortex the flask on a magnetic stir plate for 30 minutes and let it equilibrate undisturbed for 15 minutes. Remove the toluene or top layer in the flask containing the analytes of interest with a pipette and put it into a GC vial. The processed sample matrix is now ready for analysis. Future enhancements to this extraction method may include an iso-amyl acetate fraction to recover cyclotrimethylenetrinitramine (RDX) and to provide higher extraction recoveries of 4-amino-2,6-dinitrotoluene, 2-amino-4,6-dinitrotoluene, and 3,5-dinitroanaline. Additionally, a sample clean-up procedure may be implemented to remove some chromatographic interferences.

Sample Analysis

Performed exactly as the water method, 8371, using 30 meter RTX-1 and RTX-5 columns with electron-capture detectors. See SOP OMOO71.O Section 7.4.