SAMPLE HANDLING AND TRACE ANALYSIS OF POLLUTANTS pdf

1: Analysis of Soil Heavy Metal Pollution and Pattern in Central Transylvania

The book treats different aspects of environmental analysis such as sample handling and analytical techniques, the applications to trace analysis of pollutants (mainly organic compounds), and quality assurance aspects, including the use of certified reference materials for the quality control of the whole analytical process.

Part 12 By Paul Gaines, Ph. Overview The lack of negative side effects is unfortunately limited to the inorganic side of the table. The ability of nitric acid to react with alcohols and aromatic rings forming explosive compounds nitro glycerine and TNT, to name two calls for caution when using nitric acid alone or in combination with other reagents in the decomposition of organic matrices. If your sample contains -OH functionality it is best to pre-treat the sample with concentrated sulfuric acid. When concentrated, the sulfuric will act as a dehydrating agent: Nitric and Perchloric Acid Digestions Nitric acid is rarely used alone. Care should be exercised and the literature consulted before attempting to use nitric acid in combination with other acids for organic sample digestions. The use of perchloric acid should only be attempted by those individuals well-versed in the safe use of this reagent. Consult "Perchloric Acid and Perchlorates"1 for safety guidelines. Organic matrices should always be pre-treated with nitric acid see exceptions above. Perchloric acid should never be used alone. Perchloric acid digestions should never be allowed to go to dryness. Hot perchloric acid should never be added to an organic matrix. Sample sizes should never exceed 1 gram dry weight for biologicals. Unknown organic matrices should be analyzed by molecular spectroscopy to determine primary structure before attempting the use of either nitric or perchloric acid. Sample Preparation Procedure The following preparation procedure was taken from our procedures manual for the acid digestion of biological samples. Only excerpts pertaining to the sample handling and digestion are included. Introduction and Scope Determination of Trace Metals in Biological Samples This procedure is intended for the determination trace metals excluding Hg in biological tissues and biological liquids involved in biologically related tests such as skin absorption studies. This method is applicable for the determination of trace metals down to the ppb concentration level. A mixture of nitric and perchloric acids is used to decompose the tissue and serum sample types. Yttrium is added to the sample prior to digestion and is used as an internal standard. Apparatus and Chemicals 20 by mm borosilicate test tubes with Teflon-lined screw caps, cardboard holder for weighing, and stainless steel racks for holding. Borosilicate glass digestion beads. Aluminum heating blocks with bore size of 20 mm equipped with thermometer and aluminum heating plate. An all plastic metal-free acid digestion hood. ICP spectrometer capable of making simultaneous internal standard measurements and storing spectra on computer disk. High purity 18 megaohm water. Plastic disposable weighing dishes. Two boiling beads are added to each vessel and it is capped with the Teflon lined screw on caps. No heating of caps or volumetric glassware is allowed -- only air drying is acceptable. Sample Handling, Identification, and Storage Immediately upon arrival, all samples are to be inspected and compared with the chain of custody information to confirm the proper shipping procedure, labeling procedure, and number of samples i. After signing off on the chain of custody document s it is required that each sample be assigned an ID number. The number assigned is to be written using permanent magic marker on a plastic bag into which the sample is placed and entered into the laboratory notebook along with the submitter ID The sample and the plastic bag are to accompany one another throughout their existence at the lab. The assigned numbers are always to be in sequence starting with 1 so it will be obvious to the operator if a sample is missing, was not prepared for analysis, etc.. The assigned number is to never appear in any laboratory notebooks, reports, or other documents without the accompaniment of the identification given by the submitter. This guarantees that no mix-ups will occur. The tissue and serum samples are stored in a freezer until hours before they are scheduled for analysis, at which time they are allowed to thaw at room temperature. Any remaining sample is stored in the freezer. Plastic forceps and plastic spatulas are used to handle the tissue samples. Plastic pipettes are used to withdraw the serum samples. If the whole tissue is not to be taken for analysis, it is transferred from the sample container to a disposable PE dish where it is divided with plastic forceps or spatulas. Sample Preparation of Tissues and Serum Place a digestion vessel holder on a 4-place analytical balance and tare.

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Uncap a cleaned digestion vessel and add two boiling beads. Label the digestion vessel using a graphite pencil with the assigned ID. Obtain the vial weight using a 4-place analytical balance. Record the digestion vessel weight in the analytical notebook in the space provided across from the submitter and the assigned IDs written in the notebook as described in step 2 under "Sample Handling, Identification, and Storage" see above. At no time is the vessel cap to be included in the vessel weight. Use only plastic forceps, spatula, or dropping pipettes to handle the tissue and serum samples. Tissue samples are removed from their shipping container to a plastic HDPE dish for tearing if necessary just prior to weighing. After recording the digestion vessel weight, tare the balance. It should read 0. Record the weight in the analytical notebook and tare the balance. A group of samples consists of a full digestion block of samples. One group is equal to 24 vessels 22 samples and 2 controls. Since the NIST liver is dried, the sample weight should not exceed 0. Brown nitrogen dioxide fumes should be observed within 5 minutes. Do not leave the digestion for the first 15 minutes. The sample should be completely dissolved within 15 minutes. Swirl the digestate to render homogeneous. With the sample weights recommended, foaming should not be a problem. If foaming does occur, remove the sample from the digestion block periodically to cool until dissolved. Continue digesting the sample with nitric acid until the brown NOX fumes are barely visible. Place the explosion shield in front of the digestion block, put on a face shield and heavy rubber gloves. The digestate should appear a very pale yellow to water white. Allow the digestate to cool to room temperature. Calculate the weight of the digestate and record this value in the notebook. The density of the digestate has been determined to be 1. Calculate the volume of the digestate by dividing the digestate weight by the density. Enter this value in the notebook. The final volume of the digestate is brought to 10 mL using 18 meg-ohm water. Calculate the mL of water to add by subtracting the digestate volume from 10 mL and enter this value in the notebook. Calculate the weight of water to add by multiplying the calculated mL by 0. A 2-place analytical balance can be used. Mix the final sample solution after capping by hand-shaking. The sample is ready for analysis. The above procedure has been used at our laboratory for processing large numbers of biological tissues from animal feeding studies. For smaller sample numbers, a Kjeldahl digestion rack with a glass hood and caustic scrubber is more convenient and is very effective in removing any perchloric acid fumes. For samples that are harder to digest, higher temperatures reaching fumes of perchloric acid may be required. Microwave Digestion References Dr. Skip Kingston has devoted a significant portion of his career to microwave chemistry. I recommend several books by Dr. Kingston for researchers with interest in acid chemistry, digestion, and safety. Introduction to Microwave Sample Preparation: Theory and Practice; Kingston, H.

2: Field Sampling and Laboratory Resources â€“ Marine Pollution Studies Laboratory at MLML

Sample handling and trace analysis of pollutants Techniques, applications and quality assurance.

3: EPA Methods Laboratory | NPDES Compliance Testing

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