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Sample preparation techniques for AAS, ICP-OES and ICP-MS for regulated testing laboratories

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Introduction

This handbook contains examples of typical sample preparation methods recommended for AAS, ICP-OES and ICP-MS analysis of a variety of sample types. Disclaimer: The described sample preparation protocols require the use of hazardous chemicals. Appropriate personal protective equipment as recommended by local safety requirements should be used. A full risk assessment should be carried out prior to undertaking any activities.

Contents

Part 1 Environmental samples

1-1 Water samples:

Hot plate digestion for water samples:

a. General procedure:

This procedure is a suitable digestion method for the preparation of water samples for AAS, ICP-OES and ICP-MS analysis. If samples are being prepared for ICP-MS analysis, it is recommended to dilute them further due to the acid concentration in the final solution.

Step 1:

Add 25 mL of the water sample to a PTFE beaker and acidify with 2.0 mL of concentrated HNO_3 and 6.0 mL of concentrated HCl (trace metal grade acid for AAS and ICP-OES and high purity acid for ICP-MS). Heat the beaker on a hot plate located in a fume extraction hood until the sample is just below boiling. This should continue until the solution becomes clear and transparent.

Step 2:

After cooling to room temperature, transfer the sample to a 50 mL volumetric flask. Rinse the inner wall of the beaker with ultrapure water (resistivity of 18.2 MΩ·cm), then add the rinse water to the sample in the volumetric flask. Bring the sample up to volume with ultrapure water.

Equivalent procedures are described in the HJ700-2014[9] method.

b. U.S. EPA SW-846 Method 3010A[4]

This digestion procedure is used for the preparation of water samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals and is not suitable for volatile sample analytes.

Step 1:

Transfer a 100 mL representative aliquot of the well-mixed sample to a 150 mL Griffin beaker. Add 3 mL of concentrated HNO_3 to the beaker and cover with a ribbed watch glass or equivalent and place on a hot plate or equivalent heating source. Slowly evaporate the sample to a low volume (e.g., around 5 mL), without boiling and with avoiding taking the sample to dryness. Cool the sample and add another 3 mL portion of concentrated HNO_3 to the beaker. Cover the beaker with a non-ribbed watch glass and returned to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.

Step 2:

Continue heating, with additional HNO_3 added as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, uncover the beaker or use a ribbed watch glass, and evaporate to a low volume (3 mL), while not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add a small quantity of 1:1 HCl, cover the beaker, and reflux for an additional 15 min to dissolve any precipitate or residue resulting from evaporation.

Step 3:
Rinse the beaker walls and watch glass with ultrapure water and, when necessary, filter or centrifuge the sample to remove silicates and other insoluble material that could clog the nebulizer. Filtration (Whatman™ quantitative filter paper, ashless, Grade 41) should be done only if there is concern that insoluble materials may clog the nebulizer. This additional step can cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned. Rinse the filter and filter apparatus with dilute HNO_3 and discard the rinsate before filtering each sample. Filter the sample and adjust the final volume to 100 mL with ultrapure water and the final acid concentration to 10%. The sample is now ready for analysis.

Samples that are digested using the 3010A[4] digestion method can be analyzed using U.S. EPA SW-846 Method 6010D and Method 6020B.

Microwave digestion:

a. U.S. EPA SW-846 Method 3015A[5]

This microwave method is designed to perform extraction using microwave heating with HNO_3, or alternatively, with a mixture of HNO_3 and HCl. Due to the rapid advances in microwave technology, consult the manufacturer's recommended instructions for guidance on their microwave digestion system. This method is generic and may be implemented using a wide variety of laboratory microwave equipment.

Step 1:
Add a 45 mL aliquot of a well-shaken, homogenized sample using an appropriate volumetric measurement and delivery device to an appropriate vessel equipped with a controlled pressure relief mechanism.

Step 2:
Add 5 ±0.1 mL of concentrated HNO_3 or, alternatively, 4 ±0.1 mL of concentrated HNO_3 and 1 ±0.1 mL of concentrated HCl to the vessel in a fume hood (or fume exhausted enclosure).

Step 3:
Seal the vessel according to the manufacturer's directions. Properly place the vessel in the microwave system according to the manufacturer's recommendations and, when applicable, connect appropriate temperature and pressure monitoring equipment to vessels according to manufacturer's specifications.

Step 4:
The temperature of each sample should rise to 170 ±5 °C in approximately 10 min and remain at 170 ±5 °C for 10 min, or for the remainder of the 20 min digestion period.

Step 5:
At the end of the microwave program, allow the vessels to cool for a minimum of 5 min before removing them from the microwave system. When the vessels are cooled to near room temperature, determine if the microwave vessels have maintained their seal throughout the digestion.

Step 6:
Complete the preparation of the sample by venting the microwave containers in a fume hood before uncapping, to avoid a rush of acid vapor that may still be in the headspace. When sufficiently cool to handle, carefully uncap the vessels using the procedure recommended by the vessel manufacturer. Quantitatively transfer the sample to an acid-cleaned bottle. If the digested sample contains particulates that may clog nebulizers or interfere with injection of the sample into the instrument, the sample should be centrifuged, allowed to settle, or filtered.

Step 7:
Transfer or decant the sample into a volumetric flask and dilute the digest to a known volume. The sample is now ready for analysis.

Samples that are digested using the 3015A[5] digestion method are suitable for analysis by ICP-MS, ICP-OES, FLAA and graphite furnace AA.

1-2 Soil samples:

Hot plate digestion:

The described sample preparation protocol requires the use of hazardous chemicals, especially hydrofluoric acid (HF) and perchloric acid ($HClO_4$). Because of the ability of these acids to penetrate tissue, poisoning can occur readily through exposure to skin or eyes, or when inhaled or swallowed. Appropriate personal protective gear such as laboratory coat, safety glasses, and gloves specifically for handling HF and $HClO_4$ are required. When using HF, it is also essential to ensure that calcium gluconate gel is immediately available for application to any areas of skin that come into contact with this acid, after rinsing the affected areas with water and drying thoroughly.

a. General procedure:
This procedure is a suitable digestion method for the preparation of soil, sediment, and solid waste samples for ICP-OES and ICP-MS analysis. If samples are being prepared for ICP-MS analysis, it is recommended to dilute samples further due to the acid concentration in the digestate.

Step 1:
Homogenize and accurately weigh 0.1 g of the soil or sediment sample into a polytetrafluoroethylene (PTFE) crucible and moisten with 3 mL of ultrapure water. Add 4 mL of an acid mixture ($HF:HClO_4$ = 5:1,) to the crucible, and heat the sample to 260 ˚C on an electric hot plate digestion system in a fume hood. Add a further 4 mL of the acid mixture and heat until emission of white smoke has ended.

Step 2:
Add 2 mL of aqua regia ($HCl:HNO_3$ = 3:1) to re-dissolve the mixture. Then add 10 mL of 10% aqua regia to the extract; the sample solution should become clear and transparent. Cool the solution, add 2 mL HNO_3, transfer the sample extract to a 100 mL PTFE volumetric flask and make up to volume with ultrapure water.

Equivalent procedures are described in method HJ803-2016[10].

b. U.S. EPA 3050B[6]
For this digestion procedure, weigh to the nearest 0.01 g and transfer a 1–2 g sample (wet weight) or 1 g sample (dry weight) to a digestion vessel. For samples with high liquid content, a larger sample size may be used as long as digestion is completed.

Step 1:
For the digestion of samples for analysis by GFAA or ICP-MS, add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a watch glass or vapor recovery device. Heat the sample to 95 ˚C ±5 ˚C and reflux for 10 to 15 min without boiling in a fume hood. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the cover, and reflux for 30 min. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until no further brown fumes are given off by the sample indicating complete reaction with HNO_3. Using a ribbed watch glass or vapor recovery system, either allow the solution to evaporate to approximately 5 mL without boiling or heat at 95 ˚C ±5 ˚C without boiling for two hours. Always maintain a covering of solution over the bottom of the vessel.

Step 2:
After the first step is completed and the sample is cooled, add 2 mL of water and 3 mL of 30% H_2O_2. Cover the vessel with a watch glass or vapor recovery device and return the covered vessel to the heat source for warming and to start the peroxide reaction.

Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the vessel. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

Step 3A for ICP-MS:
Cover the sample with a ribbed watch glass or vapor recovery device and continue heating the acid-peroxide digestate until the volume is reduced to approximately 5 mL or heat at 95 ˚C ±5 ˚C without boiling for two hours. Always maintain a covering of solution over the bottom of the vessel. After cooling, dilute to 100 mL with ultrapure water. Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle. The sample is now ready for analysis by GFAA or ICP-MS.

Step 3B for ICP-OES:
For the digestion of samples for analysis by FLAA or ICP-OES, add 10 mL concentrated HCl to the sample digested from Step 2 and cover with a watch glass or vapor recovery device. Place the sample on/into the heating source and reflux at 95 ˚C ±5 ˚C for 15 min. Filter the digestate through Whatman™ No. 41 filter paper (or equivalent) and collect the filtrate in a 100 mL volumetric flask. Make to volume and analyze by FLAA or ICP-OES.

Samples that are digested using the 3050B[6] digestion method can also be analyzed using U.S. EPA SW-846 Method 6010D and Method 6020B.

Microwave digestion:

a. General procedure
This procedure is a suitable digestion method for the preparation of soil, sediment, and solid waste samples for ICP-OES and ICP-MS analysis. If samples are being prepared for ICP-MS analysis, it would be recommended to dilute them further due to the acid concentration in the final solution.

Weigh 0.20 ±0.001 g of sample into a microwave digestion vessel and add 9 mL of concentrated HNO_3 and 3 mL of concentrated HCl. Digest the sample according to the manufacturer's guidelines or method for the sample. Once cooled, transfer the sample to a 50 mL volumetric flask and make up to volume with ultrapure water.

b. U.S. EPA 3051A[7]

This microwave extraction method is designed to mimic extraction using conventional heating with HNO_3, or alternatively, HNO_3 and HCl, according to U.S. EPA Method 200.2[2] and Method 3050[6].

Step 1:
Weigh a well-mixed sample to the nearest 0.001 g into an appropriate vessel equipped with a controlled pressure relief mechanism. For soils, sediments, and sludges, use not more than 0.50 g. For oil or oil contaminated soils, initially use not more than 0.25 g.

Step 2:
Add 10 ±0.1 mL concentrated nitric acid (HNO_3) or, alternatively, 9 ±0.1 mL concentrated HNO_3 and 3 ±0.1 mL concentrated hydrochloric acid HCl to the vessel in a fume hood (or fume exhausted enclosure).

Step 3:
Seal the vessel according to the manufacturer's directions. Properly place the vessel in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure sensors to vessels according to manufacturer's specifications.

Step 4:
The temperature of each sample should rise to 175 ±5 ˚C in approximately 5.5 ±0.25 min and remain at 175 ±5 ˚C for 4 min 30 s. When using temperature feedback control, the number of samples to be simultaneously digested may vary, from one sample up to the maximum number of vessels that can be heated by the magnetron of the microwave unit according to the heating profile specified previously in this section. The number will depend on the power of the unit, the number of vessels, and the heat loss characteristics of the vessels.

Step 5:
At the end of the microwave program, allow the vessels to cool for a minimum of 5 min before removing them from the microwave system. When the vessels are cooled to near room temperature, determine if the microwave vessels have maintained their seal throughout the digestion.

Step 6:
Complete the preparation of the sample by venting the microwave containers in a fume hood before uncapping to avoid a rush of acid vapor that may still be in the headspace. When sufficiently cool to handle, carefully uncap the vessels using the procedure recommended by the vessel manufacturer. Quantitatively transfer the sample to an acid-cleaned bottle. If the digested sample contains particulates that may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered (Whatman™ quantitative filter paper, ashless, Grade 41).

Step 7:
Transfer or decant the sample into volumetric ware and dilute the digest to a known volume. The digest is now ready for analysis of the elements of interest using appropriate elemental analysis techniques.

Samples that are digested using the 3051A[7] digestion method can also be analyzed using U.S. EPA SW-846 Method 6010D and Method 6020B.

c. HJ 832-2017[11]

Soil and sediment – Digestion of total metal elements – Microwave assisted acid digestion method. Also refer to GB 17378.3[8] and HJ/T 166[12].

Step 1:
Weigh a well-mixed sample 0.25–0.50 g to the nearest 0.001 g into an appropriate vessel equipped with a controlled pressure relief mechanism. Add 6 ±0.1 mL concentrated HNO_3, 3 ±0.1 mL concentrated HCl and 2 ±0.1 mL HF to the vessel in a fume hood.

Step 2:
The temperature of each sample should rise to 160–190 ˚C in approximately 12 min and remain at 160–190 ˚C for 5 min. At the end of the microwave program, allow the vessels to cool to room temperature.

Step 3:
Rinse the inner surface walls of the vessel with a small amount of dilute HNO_3, then transfer the sample to a 25 mL volumetric flask. Continue to rinse the inner walls with nitric acid, transfer the sample solution to the volumetric flask, and then dilute with ultrapure water to the mark.

Part 2 Food and beverage samples

2-1 Food samples:

a. Rice flour: (refer to Thermo Scientific™ AN 43326[18])
Weigh 0.5 g of sample into a microwave digestion vessel and add a mixture of 5 mL HNO_3 and 1 mL HCl. Digest the sample according to the manufacturer's guidelines. Ensure the samples are heated to at least a temperature of 200 ˚C for 15 min.

After digestion, transfer the sample to a 50 mL volumetric flask and make up to volume with ultrapure water.

b. Milk powder:
Method 1. This procedure is for analysis by AAS: (refer to Thermo Scientific AN 44371[22])
Weight 1 g of the sample into a 15 mL centrifuge tube and add 5 mL of Thermo Scientific™ Triton™ X-100 solution (0.2% m/v). Seal the tube and mix by vortex oscillation. Place the tube in an ultrasonic bath and sonicate for 1 hr. Afterwards, add Triton X-100 solution to bring sample to a volume of 10 mL. The samples are then mixed a final time by vortex oscillation prior to analysis.

Triton™ X-100: CAS9002-93-1, Sigma Aldrich

Method 2. This procedure is for analysis by ICP-OES: (refer to Thermo Scientific AN 44392[24])
Weigh 0.5 g of sample into a PTFE high pressure microwave vessel and add 7 mL of concentrated HNO_3. Carefully wash down material adhering to the walls of the vessel with the nitric acid. Add 1 mL of concentrated H_2O_2; this will increase the oxidation potential for decomposition of the organic matrix. Digest the sample in the microwave as per the manufacturer's guidelines.

After digestion and cool down to room temperature, transfer the sample into a 50 mL volumetric flask. Rinse the digestion vessels with ultrapure water and transfer the resulting liquid to the flask. Finally, make up to volume with ultrapure water.

c. Fruits (freeze-dried sample): (refer to Thermo Scientific AN 44474[31])
Weigh an aliquot of 0.3–0.4 g of sample into a microwave digestion vessel. Add a mixture of 5 mL HNO_3 and 1 mL HCl (35% Optima™ grade, Fisher Chemicals) to the vessel and place in the microwave digestion system (Milestone ETHOS 1 used in this study/application note). After digestion, transfer the sample into a 50 mL volumetric flask and bring up to volume with ultrapure water. The amount of total dissolved solids is around 0.6% in the sample solution. No further dilution is applied prior to analysis by ICP-MS.

d. Meat: (refer to Thermo Scientific AN 44459[28])

Step 1:
Weigh 0.5 g of homogenized sample into a pre-cleaned, dry 75 mL microwave digestion vessel.

Step 2:
Add gold, to stabilize mercury, to the sample so that the final concentration is 200 $\mu g \cdot L^{-1}$ in the sample solution. Add 2 mL of HNO_3, 1 mL H_2O_2, and 0.2 mL HCl and keep the sample in a fume hood for 60 min to allow for pre-digestion.

Step 3:
Add 1 mL of ultrapure water and digest the samples using a microwave digestion system (CEM Mars 6 used in this study/application note). Ensure the samples are ramped over 40 minues to a temperature of 200 ˚C and then held at this temperature for 30 min.

Step 4:
After digestion is complete, allow the vessels to cool to room temperature. Quantitatively transfer the sample to a pre-cleaned 50 mL volumetric flask and add ultrapure water to volume. Mix thoroughly with a vortex mixer prior to analysis by ICP-MS.

e. U.S. FDA method[33] – Elemental Analysis Manual for Food and Related Products:

Step 1:
Transfer an analytical portion of the sample with a pipette or spatula or by pouring into a tared, clean digestion vessel liner. Measure the weight of the sample to an accuracy of 0.001 g. For safety purposes, limit the sample weight to 0.5 g if the composition is unknown.

Step 2:
Pipette 8 mL or weigh 11.3 g of high purity HNO_3 into the vessel liner and wash down any material on the vessel walls. Add 1 mL of high purity 30% H_2O_2. Seal the vessels, tighten pressure relief nuts, and run the microwave digestion program as prescribed by the analytical method.

Step 3:
After digestion, allow the vessels to cool to below 50 ˚C and then transfer them to an exhaust hood and vent excess pressure slowly. Quantitatively transfer and dilute the digestion solution with deionized water as prescribed by the analytical method. This analytical solution should be transferred to a plastic bottle or a capped polypropylene centrifuge tube for storage.

A typical microwave digestion program is given in the Table below.

Digestion	Peroxide oxidation
Maximum power (W)	1200
Control pressure (psi)	800
Ramp time (min)	25
Hold time (min)	15
Control temperature (˚C)	200

2-2 Beverage samples:

a. Juice: (refer to Thermo Scientific AN 43151[15])
For determination of trace elements, weigh 20 g of sample into a 100 mL volumetric flask. For determination of major elements, weigh 2 g of sample into a 100 mL volumetric flask. Make all samples to 100 mL volume with ultrapure water.

b. Carbonated non-alcoholic beverages: (refer to Thermo Scientific AN 44421[25])
Prior to analysis by ICP-OES, reduce the influence of dissolved CO_2 gas on nebulization and transport by degassing the samples in an ultrasonic bath. Weigh 10 g of each sample into a 50 mL volumetric flask. Make the samples up to volume with ultrapure water.

Part 3 Industrial samples

3-1 Metal samples:

a. Steels and alloys (refer to Thermo Scientific AN 43146[14])
The samples are digested in a microwave digester.

Weigh 0.5 g of sample into a microwave digestion vessel and add 10 mL of concentrated HCl and 2.0 mL of concentrated HNO_3. Seal the vessel and digest at 180 ˚C for 20 min.

When the microwave program is finished, allow the vessels to cool, transfer the sample into a 100 mL volumetric flask and bring to volume with ultrapure water.

b. Titanium alloys (refer to Thermo Scientific AN 40988[13])
Alloy samples can be dissolved and diluted (1 g to 50 mL) according to ASTM Method E2371-13[1]. This includes the digestion of the sample in 15 mL HCl followed by 2 mL HF and 2 mL HNO_3. Clear solutions on completion of the digestion step indicates complete dissolution.

3-2 Oil samples:

Lubricating oil (refer to Thermo Scientific AN 44426[26])
Homogenize the sample by heating to 60 ˚C and sonicating. Weigh a portion of the sample and dilute by weight with a suitable solvent, such as kerosene or xylene, so that the diluted sample contains 10% of the original sample by weight.

3-3 Refinery products:

(refer to Thermo Scientific AN 44465[29])
A major challenge when analyzing different types of petrochemical samples is the difference in vapor pressure and viscosity, which ultimately requires a dedicated configuration of the sample introduction system. Whereas samples with high viscosity, such as crude oil or fuel oil, require heating of the sample introduction system components to remain fluid, samples with a lower boiling point, such as naphtha, need cooling of the spray chamber to avoid overloading the plasma and sudden extinction.

To homogenize the properties of samples, and with the objective to accommodate different sample types in a single batch, samples are prepared differently in each case.

Fuel oil/crude oil sample preparation: Heat the oil sample in a water bath to 40–60 ˚C and mix thoroughly to ensure the homogeneity. Weigh 1 g of homogenized oil sample into a 50 mL volumetric flask. Dilute the sample by 300-fold using xylene as the diluent using serial dilutions.

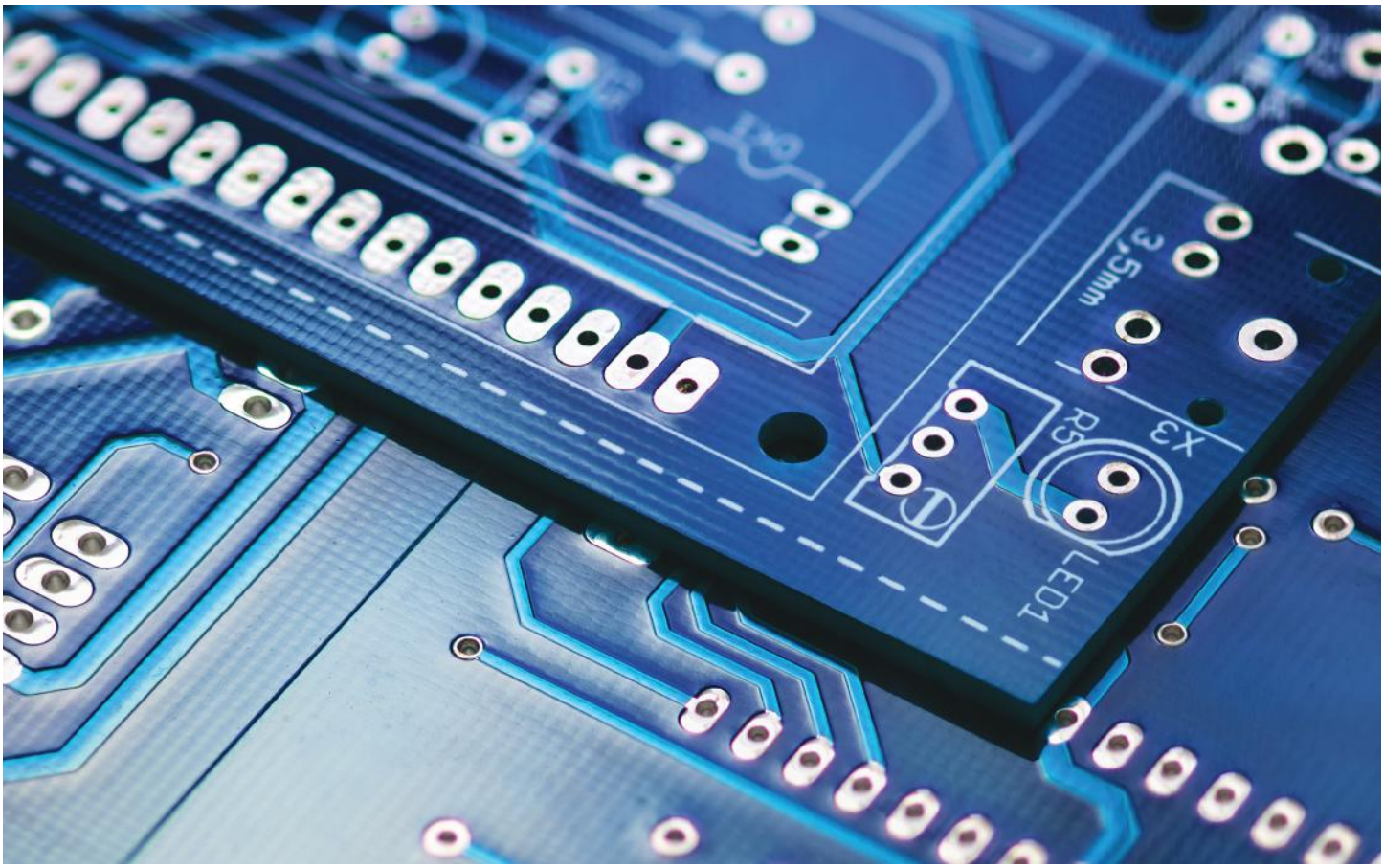
Heavy naphtha: Directly aspirate into the ICP-MS after addition of the required amount of internal standards stock solution.

Light naphtha: Dilute 10-fold using xylene followed by addition of the required amount of internal standards stock solution before aspiration into the ICP-MS.

3-4 Electronic waste:

(refer to Thermo Scientific AN44466[30])
Sample powders of electronic waste samples like mobile phone screens, printed circuit boards, etc. are prepared using a grinder. Small (<5 cm) metal components like magnets may be left whole.

Weigh 1 to 2 g of sample material into a beaker and digest overnight in a concentrated acid mixture ($HCl:HNO_3$ = 1:1). If the sample is a magnet, digest with aqua regia. Filter the samples and dilute so that the final solution contains 10% (v/v) acid.



Part 4 Clinical research and pharmaceutical samples

4-1 Blood:

(refer to Thermo Scientific AN 44453[27])
To prepare blood samples for analysis by ICP-MS, pre-treat with chelating agents such as tetramethylammonium hydroxide (TMAH) or ethylenediamine-tetra acetic acid (EDTA) to avoid coagulation of the blood. Alternatively, a closed vessel microwave digestion can be used to reduce the impact of the matrix.

Blood samples can be diluted manually to a final acid concentration of 0.5%. In brief, a 0.1 mL aliquot of whole blood is transferred into pre-cleaned sample tubes followed by the addition of 4 mL of ultrapure water. After addition of 25 µL of concentrated HNO_3 (Optima™ grade, Fisher Scientific), the sample is made up to a total volume of 5 mL with deionized water, mixed thoroughly using a vortex shaker, and analyzed.

4-2 Serum:

(refer to Thermo Scientific AN 43283[16])
The sample can be gravimetrically diluted by a factor of 10 in pre-cleaned (72 hr in 2% HNO_3, washed in ultrapure water) polypropylene bottles with HNO_3 (0.5% m/m, Fisher Scientific) and tetramethylammonium hydroxide (TMAH, 2% m/m, Sigma-Aldrich) in ultrapure water.

4-3 Urine:

(refer to Thermo Scientific TN 43357[19])
A 2 mL aliquot of urine sample is filtered through a 0.45 µm PTFE membrane filter (Sartorius, Göttingen, Germany) and diluted with 0.5% (v/v) HNO_3.

4-4 Pharmaceutical:

a. (refer to Thermo Scientific AN 44385[23])
In this study/application note, a cough medicine in the form of an effervescent tablet is diluted with a few mL of ultrapure water to degas the CO_2. After the reaction subsides, the aliquot is acidified to a final concentration of 5% HCl, spiked accordingly for the various tests of the validation procedure, and filled up with ultrapure water to a final volume of 50 mL.

b. (refer to Thermo Scientific AN 43325[17])
Pharmaceutical products are brought into solution via a microwave digestion system (Milestone Inc., Shelton, CT, USA). Different microwave procedures are available to address specific sample matrices.

Samples of each drug (0.5 g) are weighed into 15 mL disposable glass vials. 3 mL of HNO_3 is added to each tube. In compliance with the repeatability requirements defined in USP <233>[32], six separate preparations of each material are prepared.

Sample vials are transferred into the microwave digestion system, which is then closed, pressurized with nitrogen at 40 bar, and maintained at a temperature of 200 °C for 15 min. High pressure digestions are recommended due to the use of lower temperatures to minimize the loss of volatile elements.

When sufficiently cooled, the clear, colorless digested material is transferred to polypropylene vials and made up to 50 mL with ultrapure water.

Part 5 Plant and vegetation samples

5-1 Plants:

a. (refer to Thermo Scientific AN 43446[20])
Approximately 0.3 g of sample is digested using a mixture of 5 mL HNO_3 and 1 mL HCl in a closed vessel microwave digestion system. The samples are heated to and maintained at a temperature of 200 ˚C for 15 min. After digestion, the samples are made up to a volume of 10 mL using ultrapure water.

b. (refer to Thermo Scientific AN 44366[21])
Prior to digestion each sample is weighed (0.5 to 0.8 g) into a PTFE high pressure vessel, HNO_3 (6 mL, concentrated, Fisher Scientific) and H_2O_2 (2 mL, concentrated, Fisher Scientific) are added. If material is adhered to the walls of the vessel, it is washed down carefully with the acid. Hydrogen peroxide is added to aid digestion of the organic matrix.

A microwave digestion system equipped with a segmented rotor and a temperature sensor is used for the digestion. The samples are heated to a temperature of 200 ˚C for 15 min.

After digestion, each sample is transferred to a volumetric flask (50 mL). The digestion vessel is washed with ultrapure water and the wash solution is transferred to the flasks. The flask is then made up to volume with ultrapure water prior to analysis by ICP-OES.

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Part 6 References

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Find out more at **thermofisher.com/TEA**

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