

A REVIEW ON HIGH PRESSURE ASHER

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ABSTRACT

High pressure asher is an ultimate wet digestion device. The HPA-S acid digestion method is an internationally recognized reference procedure and is in operation as a high-performance routine instrument in numerous laboratories. The High Pressure Asher HPA-S is the reference instrument for wet chemical pressure decomposition. The instrument is TÜV certified.

Complete mineralisation is possible with high pressure asher. The safest way of operation to obtain total mineralisation is to complete any decomposition technique by combustion in a dynamic system, high temperature, dry ashing and possibly quartz lined pressure wet ashing which is possible with this instrument

Keywords: Wet digestion, Asher, Mineralisation.

INTRODUCTION

There are several methods for determination of trace elements in samples such as

- direct determination by spectroscopic methods after dilution with organic solvents,
- determination of the elements in stable emulsion systems and
- the use of conventional digestion methods such as oxygen combustion.

But all these methods have inherent problems. In order to avoid matrix problems, wet digestion methods are a suitable way for sample preparation for the analysis of trace elements in products. There is already an existing ASTM method using the advantage of the HPA-S (high pressure, high temperature, closed vessels, clean vessel materials) for digestion of samples.

In 1973, the introduction of the "VAO", the world's first automatic wet decomposition device, marked the beginning of a development of high-performance instruments for plasma ashing, oxygen combustion, high pressure digestion and for microwave-supported decomposition. The high pressure asher HPA-S unites scientific knowledge of modern trace analysis and the practical aspects of daily routine work in the laboratory. HPA-S represents the ultimate performance in wet chemical high pressure digestion sample preparation for

- AAS
- ICP and
- Voltammetry.[1]

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Conventional dry ashing in muffle furnaces or heated tubes or conventional low pressure wet ashing is cheap and easy to perform, but is not compatible with elemental analysis on the microgram/kilogram level. Acid vapour phase decomposition in closed vessel systems requires considerably smaller volumes of

acids than open systems and the decomposition is performed under high pressure.[3]

This procedure is successfully applied for the digestion of difficult samples, such as silicate materials with conductive heating. The system allowed decomposition of biological samples and high pure silicas. Acid vapour phase decomposition is performed in High Pressure Asher for multi elemental determination in biological materials. HPA-Sis also based on conductive heating. HPA vessels can be pressurised upto 100 atm.[4]

Decomposition of solid samples is an important part of several analytical methods. Indeed, although some analytical methods (XRF and electrothermal atomization) can be applied directly to a solid sample, thus do not always require sample dissolution before measurement, most analytical measurements using highly sensitive methods(AAS,ICPAES etc) are performed on solutions of sample.[5]

Element preconcentration and chemical separation may also be required to improve the quality of the measurements. Of course, when large numbers of samples have to be handled the preparation should be quick and easy, so that automation of the preparation step is becoming increasing importance. Time involved in sample preparation was optimised as 190min followed by cooling time of 60 min. The success of the HPA-S high pressure asher is due to its high quality technology in combination with a high level of service.

PRINCIPLE OF VOLTAMMETRY

Voltammetry is a category of electroanalytical methods used in analytical chemistry and various industrial processes. In voltammetry, information about an analyte is obtained by measuring the current as the potential is varied.[6] Voltammetric methods are among the most sensitive and versatile available to the analytical chemist. They can identify and quantify substances from simple metal ions, through to complex organic molecules. The concentration range spans 9 orders of magnitude and, in many cases, trace level analyses of surface waters and body fluids can be performed with little or no pre-treatment of the sample is required. [7]

PRINCIPLE OF ATOMIC ABSORPTION SPECTROSCOPY

The technique makes use of absorption spectrometry to assess the concentration of an analyte in a sample. It requires standards with known analyte content to establish the relation between the measured absorbance and the analyte concentration and relies therefore on Beer-Lambert Law. In short, the electrons of the atoms in the atomizer can be promoted to higher orbitals (excited state) for a short period of time (nanoseconds) by absorbing a defined quantity of energy (radiation of a given wavelength). This amount of

energy, i.e., wavelength, is specific to a particular electron transition in a particular element.

In general, each wavelength corresponds to only one element, and the width of an absorption line is only of the order of a few picometers (pm), which gives the technique its elemental selectivity. The radiation flux without a sample and with a sample in the atomizer is measured using a detector, and the ratio between the two values is converted to analyte concentration or mass using Beer-Lambert Law.[8]

PRINCIPLE OF ICP-MS

ICP-MS is a type of mass spectrometry that is highly sensitive and capable of the determination of a range of metals and several non-metals at concentrations below one part in 10^{12} (part per trillion). It is based on coupling together an inductively coupled plasma as a method of producing ions (ionization) with a mass spectrometer as a method of separating and detecting the ions.[9]

In trace elemental analysis, the method has advantages of high speed, precision and sensitivity compared to atomic absorption techniques. Analysis of lower concentrations at the same time is more prone to disruption by trace contaminants in labware and reagents used. Specific analytes suffer from interferences exclusive to ICP-MS technique. Verification of analysis results requires additional effort.[10]

CHOICE OF SAMPLE PREPARATION AND ITS EFFECTS

The choice of sample preparation procedure has an impact on the performance of the quantitation technique used, the behaviour of the sought-after element and the amenability of the sample matrix to proper decomposition. Choice of procedure depends on

- nature of organic/inorganic material
- the element to be determined
- the method used for its quantitation

If the method sample preparation is not properly selected it affects the process of analysis and gives inaccurate results leading to false analysis. In some cases improper preparation of sample cause damage to the device.[11,12]

SIGNIFICANCE

This practice is useful for preparation of difficult-to-digest, primarily oils and oily wastes, specimens for trace element determinations of up to 28 elements by atomic absorption or plasma emission techniques. Specimen preparation by high-pressure ashing is primarily applicable to specimens whose preparation by EPA SW-846 protocols is either not applicable or not defined. This sample preparation practice is applicable for the trace element characterization of mixed oily wastes for use by waste treatment facilities such as incinerators or waste stabilization facilities.[13]

SW-846 has two volumes. Volume I contains the preparation and analytical techniques. Volume II contains the field manuals. These procedures and manuals are considered as standards.

SCOPE

This practice covers a high-pressure, high-temperature digestion technique using the high-pressure ashing (HPA) for preparation of oils and oily waste specimens for determination of up to 28 different elements by

- ICP-AES,
- CVAAS,
- ICP-MS, and
- Radiochemical methods.

1. Oily and high-percentage organic waste streams from nuclear and non-nuclear manufacturing processes can be successfully prepared for trace element determinations by ICP-AES, CVAAS, and GFAAS.

2. This practice is applicable to the determination of total trace elements in these mixed wastes. Specimens prepared by this practice can be used to characterize organic mixed waste streams received by hazardous waste treatment incinerators and for total element characterization of the waste streams.
3. This practice has been used successfully to completely digest a large variety of oils and oily mixed waste streams from nuclear processing facilities.
4. This practice is designed to be applicable to samples whose preparation practices are not defined, or not suitable, by other regulatory procedures or requirements, such as the U.S. Environmental Protection Agency (EPA) SW-846 and EPA-600/4-79-020 documents.

This digestion practice is designed to provide a high level of accuracy and precision, but does not replace or override any regulatory requirements for sample preparation[14]

LIMITATIONS

1. This practice is applicable only to organic waste streams that contain radioactivity levels that do not require special personnel or environmental protection from radioactivity or other acute hazards.
2. While the practice has been used to report data on up to 28 trace elements, its success should not be expected for all analytes in every specimen. The overall nature of these oily wastes tends to be heterogeneous that can affect the results. Homogeneity of the prepared sample is critical to the precision and quality of the results.
3. This practice uses hazardous materials, operations, and equipment at high pressure (90–110 bars, 89–108 atm, or 1305–1595 lb/in.²) and high temperatures, up to 320°C, and therefore poses significant hazards if not operated properly.
4. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.[15]

PRINCIPLE

Wet chemical pressure decomposition which has established itself as a versatile, high-performance method for sample preparation for element determination using AAS, ICP-OES, ICP-MS or voltammetry is the basic principle involved in the high pressure asher. It can be used for a variety of applications. The basic principles of successful acid digestion were recognized as early as 1865 by Carius suitable reagents, high temperatures and appropriate vessels.[16]

Wet digestion methods for elemental analysis involve the chemical degradation of sample matrices in solution, usually with a combination of acids to increase solubility. The various acid and flux treatments are carried out at high temperatures in specially designed vessels that help to minimize contamination of the sample with substances in the air, the local environment, and from the vessel walls.[17]

The most commonly used reagents for wet decomposition are mineral and mostly oxidising acids. Wet decomposition has the advantage of being effective on both inorganic and organic materials. It often destroys the sample matrix, thus help reducing some types of interference.[18]

Wet decomposition in open vessels may give rise to systematic errors due to;

- Contamination caused by reagents and container material
- Losses of element caused by adsorption on the surface of the vessel or by reaction with vessel material
- Losses of element by volatilisation.

Losses from the sample may occur due to adsorption onto the vessel walls, volatilization, and coextraction, but these can be reduced by procedural modifications. The use of closed systems, where the digestion reaction is completely isolated from the surroundings, may help to reduce both contamination and sample loss.[19]

ADVANTAGES

The following points summarize the essential advantages of this high pressure, high temperature decomposition technique.

1. High temperatures beyond the boiling point
2. Improved dissolution of inorganic samples
3. Powerful oxidation of organic samples just with pure nitric acid
4. No element losses
5. Low reagent consumption
6. Reduced contamination
7. All biological materials and food stuffs can be completely decomposed with no need for perchloric acid.
8. Materials such as carbon, carbon fibres and mineral oils can be completely mineralised at 300°C using pure HNO_3 .
9. Any technique for trace elemental determination can be employed to analyse the solution.
10. The HPA system is used for digestion of blood samples.[20]

DISADVANTAGES

1. Limited sample weight
2. Complicate vessel construction
3. Higher equipment costs

THE INSTRUMENT

CONSTRUCTION [21]

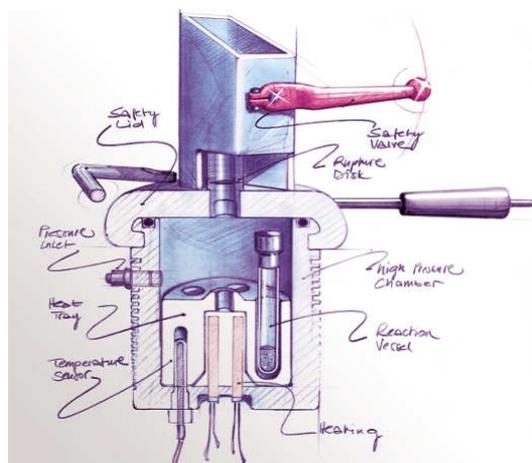


Fig. 1: Diagram showing construction of HPA

RUPTURE DISC

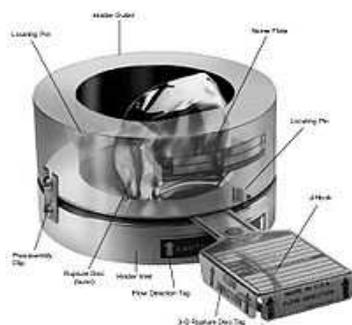


Fig. 2: Construction of rupturedisc

A rupture disc, also known as a bursting disc or burst diaphragm, is a non-reclosing pressure relief device that, in most uses, protects a pressure vessel, equipment or system from over pressurization or potentially damaging vacuum conditions.

A rupture disc is a type of sacrificial part because it has a one-time-use membrane that fails at a predetermined differential pressure, either positive or vacuum. The membrane is usually made out of metal, but nearly any material (or different materials in layers) can be used to suit a particular application. Rupture discs provide instant response (within milliseconds) to an increase or decrease in system pressure, but once the disc has ruptured it will not reseal.

Major advantages of the application of rupture discs compared to using pressure relief valves include leak-tightness and cost.[22]

BAYONET LID

It is electrolytic tinplate, cam screw, cover with inward shaped cams, rolled over edge, inserted sealing washer, staple form, sub-section with the outside shaped cams, rolled over edge. A bayonet lid and valves ensure rapid and easy operation.

PRESSURE VESSEL

1. The pressure vessel is made of high-tensile, corrosion resistant stainless steel and corresponds to international norms. It is delivered with comprehensive test certificates. A built-in fan is used to achieve rapid cooling after decomposition.
2. Pressure vessel is made of High-tensile stainless steel to withstand high constant temperature. The concentric, electric heating heats all the vessels in the heating block uniformly, independent of the size, number and content.
3. The exact temperature regulation up to 320°C is achieved using a program controller and guarantees high reproducible decomposition quality, even for difficult organic samples.
4. Process times can be as long as required.[23]

THE REACTION VESSELS

Hermetically sealed, long-life reaction vessels made of highly pure, resistant materials guarantee perfect decomposition. Conversion kits with different vessel sizes allow the optimal adaptation to different tasks. The vessels are sealed with robust lids using PTFE strips. The nitrogen in the autoclave ensures the complete seal. [24] PTFE is a synthetic fluoropolymer of tetrafluoroethylene that finds numerous applications. The most well known brand name of PTFE is Teflon.

PTFE is a fluorocarbon solid, as it is a high-molecular-weight compound consisting wholly of carbon and fluorine. PTFE is hydrophobic: neither water nor water-containing substances wet PTFE, as fluorocarbons demonstrate mitigated London dispersion forces due to the high electronegativity of fluorine. PTFE has one of the lowest coefficients of friction against any solid.[25]

AUTOCLAVES

1. Autoclaves (steam sterilizers) as horizontal and vertical construction
2. Pass-through autoclaves to be built into a wall for high security areas .
3. Media preparators for fast, controlled preparation and sterilization of liquid media with constant high quality.

TEMPERATURE SENSOR

This is used to record the temperature where the operation has to be carried out. It shows the deflection on any increase or decrease of temperature.

WORKING

The High Pressure Asher (HPA-S) was adapted with a Teflon liner for pressurized wet digestion in open vessels. The autoclave was partly filled with water containing 5% (vol/vol) hydrogen peroxide.

The digestion vessels dipped partly into the water or were arranged on top of the water by means of a special rack made of titanium or PTFE-coated stainless steel. The HPA-S was closed and pressurized with nitrogen up to 100 bars. The maximum digestion temperature was 250 degrees C for PFA vessels and 270 degrees C for quartz vessels. Digestion vessels made of quartz or PFA-Teflon with volumes between 1.5 mL (auto sampler cups) and 50 mL were tested. The maximum sample amount for quartz vessels was 0.5-1.5 g and for PFA vessels 0.2-0.5 g, depending on the material. Higher sample intake may lead to fast reactions with losses of digestion solution. The samples were digested with 5 mL HNO₃ or with 2 mL HNO₃+6 mL H₂O+2 mL H₂O₂.

The total digestion time was 90-120 min and 30 min for cooling down to room temperature. Auto sample cups made of PFA were used as digestion vessels for GFAAS. Sample material (50 mg) was digested with 0.2 mL HNO₃+0.5 mL H₂O+0.2 mL H₂O₂.

The analytical data of nine certified reference materials are also within the confidential intervals for volatile elements like mercury, selenium and arsenic. No cross contamination between the digestion vessels could be observed. Due to the high gas pressure, the diffusion rate of volatile species is low and losses of elements by volatilisation could be observed only with diluted nitric acid and vessels with large cross section. In addition, cocoa, walnuts, nicotinic acid, pumpkin seeds, lubrication oil, straw, polyethylene and coal were digested and the TOC values measured. The residual carbon content came to 0.2-10% depending on the sample matrix and amount.

The samples and acids are placed in closed vessels inside a pressure vessel. This pressure vessel is filled with nitrogen up to 130 bar pressure and heated up to a maximum of 320 °C using a preselected temperature program. The surrounding nitrogen pressure compensates the reaction pressure which arises in the vessels and prevents the vessels opening or bursting. After the reaction, the pressure vessel is cooled down, ventilated and the vessels can be removed without pressure. The decisive parameters are known for many sample materials:

- sample volume
- vessel size
- type and volume of the reagents and
- temperature program. [27-29]

MAXIMUM REACTION CONDITIONS

- HPA-S: 320C / 120bar
- Different microwave systems: 200°C up to 280°C
- PTFE – bomb: up to 200°C

CONDITIONS OF OPERATION

- volume: 1.5 L
- Lid Quick seal with safety lock
- Max. operating temperature 320 °C
- Operating pressure 130 bar / 1900 psi
- Maximum pressure 180 bar / 2600 psi
- Testing pressure 258 bar / 3740 psi
- Power 1700 W at 230V 50 ... 60 Hz
- Dimensions (WxDxH) 460 x 420 x 820 mm
- Weight 45 kg[30]

USE OF HPA-S –AN ECONOMIC OPERATION

1. Due to its simple operation and reliable results, the HPA-S has established itself as an accurate reference and routine instrument in hundreds of laboratories across the world.

- sample throughput,
 - minimum amount of vessel cleaning required and
 - low costs for wearing parts and reagents make the HPA an attractive choice from a financial point of view.
2. Safety without compromise.
 3. The robust construction with multiple pressure relief devices take the danger out of spontaneous reactions.
 4. Secure locks prevent operating errors. The TÜV “GS” certificate documents the high safety level of the HPA-S. There are no compromises when it comes to analytical safety either: Complete decomposition without loss or contamination form the basis for accurate analysis.
 5. The HPA-S system provides maximum safety, comfort and an extensive range of accessories, all helping to meet the requirements of modern laboratories.[31]

APPLICATIONS

1. Food stuffs

The HPA-S is well-established in this field because complete mineralization gives precise results for elements such as Hg, As and Se even with fatty samples. The 90 mL quartz glass vessels allow large sample volumes.

2. Medical analysis

The small 15 mL vessels are ideal for microsamples of a few mg which are decomposed in the lowest volume of acid. This gives low reagent blanks and allows up to 21 samples to be handled at once.

3. Pharmaceutical analysis

The HPA-S is well-established for demanding applications in this field due to its reproducible results and low systematic error. It is renowned for work with low element concentrations, high purity requirements and applications with compounds which are difficult to decompose.

4. Material research

The HPA-S is ideal for total decomposition, leaching or gas phase decomposition of metals, alloys, plastics, construction materials, ceramics, etc. The precise temperature regulation is also useful for special applications such as aging and corrosion tests.

5. Environmental analysis

Here, the HPA-S is used as an alternative or complement to other methods, e.g. microwave decomposition. Its strength lies in ultra-trace analysis. The option of large volumes is advantageous for inhomogeneous samples.

6. Voltammetric analysis

Samples decomposed by the HPA-S have a very low residual carbon content. This makes them ideal for accurate AAS, ICS-OES and ICP-MS analysis and reliable voltammetric tests.

7. Geochemistry

As well as standard applications, there are many publications documenting the determination of platinum group elements and rare-earth elements after high temperature leaching with the HPA-S. Even full decomposition with HF is possible in glassy carbon vessels.

8. Chemical industry

In research and development, production or quality assurance, the flexible use, rapid adaptation to different tasks and a comprehensive collection of methods make the HPA-S the most reliable problem-solver for organic and inorganic matrices.

9. Petrochemistry

Even difficult to decompose raw oils, fuels, lubricants, coal and combustion residues can be decomposed with standard programs at

high temperatures. The safe and economic operation is a recognized benefit of the HPA-S. Years of operating experience with the HPA-S method exists today as a comprehensive collection of applications which is useful to every user.[32]

10. A new method has been developed for the measurement of sulfur in gas oils using a high pressure asher (HPA) for sample digestion. This, combined with a calibrated enriched isotopic reference material, and high precision mass spectrometry, makes it a powerful tool for an accurate and precise determination of the sulfur content in fossil fuels.

The method described here was one of the methods used in the certification of the candidate reference materials BCR-104R, BCR-671 and BCR-672. Approximately 0.2 g of the gas oil samples were spiked with the spike isotopic reference material IRMM-646, which is enriched in ^{34}S , and combusted in an HPA using nitric acid. The isotopes of sulfur were measured as arsenic monosulfides (AsS^+) using a thermal ionization mass spectrometer equipped with a Faraday detector.

The total uncertainty of the sulfur content ($k = 2$) is about 1%. The certified reference materials NIST SRM-2724b and NIST SRM-1619b and BCR-105 were used as part of the method validation work. The mass content of sulfur in BCR-104R was measured to be $1014 \mu\text{g g}^{-1}$, BCR-671 has $450 \mu\text{g g}^{-1}$ sulfur and BCR-672 shows a sulfur mass content of $201 \mu\text{g g}^{-1}$. The sulfur content of the candidate reference materials is homogeneous for the sample size used.[33]

11. The High Pressure Asher HPA-S is the reference instrument for wet-chemical pressure decomposition. It enables safe and accurate decomposition in hermetically sealed reaction vessels made of quartz or glassy carbon.

With various glassware the HPA-S can be adapted for individual requirements ranging from the microanalysis of 21 samples in parallel up to large samples of a few grams. Uniform heating and exact temperature control up to 320°C at a pressure of 130 bar guarantee total mineralization of organic samples at the highest level of reproducibility. The HPA-S is used as a unique trouble shooter for most difficult organic and inorganic samples in routine analysis as well as for R&D applications.

REASONS FOR WIDE APPLICATIONS

- Highest decomposition quality
- Lowest systematic error
- Simple adaptation for different tasks
- Comprehensive application support

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