LITERATURE SURVEY OF METHOD OF DETERMINATION OF ARSENIC IN ENVIRONMENT

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CERTIFICATE

This is to certify that the dissertation entitled "***Determination of Arsenic in Environment***" is bonafide work carried out by Mr. Chandresh Deepak Agarwadekar under the supervision and guidance of Dr. Kiran .T. Dhavskar in the partial fulfilment of the requirement for the award of the degree of Master of Science in Chemistry at the School of Chemical Sciences, Goa University.

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DECLARATION

I hereby declare that the work embodied in this report entitled "Determination **of Arsenic in environment**" was carried out by me during the year 2021-2022 under the guidance of Dr.Kiran.T.Dhavskar. In keeping the general practice of reporting scientific observations, due acknowledgements have been made wherever the work described is based on the findings of other investigators.

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Introduction

Arsenic, As, belong to the group of elements that are called metalloids. A metalloid is a chemical element that has properties of both metals and non-metals. Arsenic is from all its features mostly recognized as a poison. Arsenic has a complex chemical behaviour since it exists in four different oxidation form. Depending on oxidation state And presence in environment, arsenic species exhibit different toxicity .Arsenic species can be present in environment, arsenic species exhibit different toxicity .Arsenic species can be present in all types of environment and can originate from the natural and anthropogenic sources. Natural sources of Arsenic are: rocks with incorporated arsenic compounds, activity of volcanoes and some biological processes. Anthropogenic sources are numerous, from mining to different types of production .When the arsenic compounds reach groundwater, it is hard to distinguish the origin, both natural and anthropogenic arsenic species are released.(Rajakovic & Rajakovic-Ognjanovic, 2018)

In the past, a number of methods have been developed to determine arsenic content and accumulation effects in environment. Various analytical commonly used are based on hydride generation coupled with atomic absorption spectroscopy or inductive coupled plasma – atomic emission spectroscopy (ICP-AES) However, these methods are often complex, lengthy, and /or expensive.

Arsenic can accumulative in soil through the use of arsenic pesticides, leaching

Of wood preservatives, application of fertilizers, irrigation and dust from the burning of fossil fuels, and the disposal of industrial and animal wastes. Anthropogenic influence on arsenic levels in soils depends on the intensity of human activity, distance from pollution sources, and pollutant dispersion pattern.

DETERMINATION OF ARSENIC IN WATER

There are a variety of chemical methods from classical to contemporary analytical techniques that are used for the determination of arsenic and arsenic species in water.

Historically ,colorimetric spectrophotometric methods have been used to determine total arsenic concentration .several commercial field kits have been on Marsh and Gutzeit reaction .All as species in a sample reduce ton As or arsine ,AsH_3, these tests are obvious ,visible proofs for Arsenic detection , and they are popular and useful in the field of forensic toxicology.

The colorimetric methods are easy to use and inexpensive in terms of equipment and operator cost. They are useful for the semi quantitative determination of high concentrations of arsenic in water. Spectrophotometric methods are based on conversion of arsenic to the coloured compound such as molybdenum blue, or silver diethyldithiocarbamate [15, 16].

Electrochemical methods, particularly voltammetry methods, are affordable, sensitive and ease of fabrication, and they are noteworthy for arsenic determination. Much work has been done in this area [12]. The ASV methods using platinum and gold electrodes, and CSV method using a glassy-carbon electrode have very low detection limit for arsenic determination. Determination of total As is performed by reducing As (V) to As (III) using various chemicals) using various chemicals, and the limits of detection achieved were in vicinity of 0.02 $\mu g\ L^{-1}$. Also, arsenic in drinking water can be measured with Cu(II) by differential pulse cathodic stripping voltammetry (DPCSV) using hanging mercury drop electrode (HMDE) as working electrode and Ag/AgCl as reference electrode [12, 17, 18].

At present, for total As concentration determination, laboratories is the extraction of arsenic, which has to be both mild and effective, at the same time. The second step is separation of various forms of arsenic species. The final step is the measuring step which gives the answer to the quantification of each present arsenic compound.often prefer more sensitive methods such as AAS, AES, MS or AFS. Usually, the total concentration of arsenic needs to be determined, then to perform speciation analysis properly, the best option is coupling of two analytical techniques. One technique is used for the separation of all chemical forms of arsenic that are present in water, and the other is used for the detection of these species. Besides coupling analytical techniques, there are necessary steps for complete analysis of arsenic.

Sophisticated coupling technique

Analytical methods for determining different arsenic species have become increasingly important due to different toxicity and chemical behaviour of various arsenic forms. Methods that involve the coupling of separation techniques, such as IC and HPLC with a sensitive detection system, such as ICP-MS, HG-AFS, HG-AAS and GF-AAS [3, 11, 13, 14, 19]. speciation analysis follows. HPLC has been a preferred technique used for separation of arsenic compounds. Coupled with ICP-MS for determination, as HPLC-ICP-MS system it is a method of choice for separation and measurements all arsenic species in water. In addition, applying IC coupled with ICP-MS, it is possible to separate and estimate arsenic species in water: As(III), As(V), DMA, MMA, AsBet. A representative result is presented in Figure 3 [19].

The evaluation of analytical method is based on defining: selectivity, repeatability, accuracy, specific features of the method and the limits of detection and quantification (LoD ining

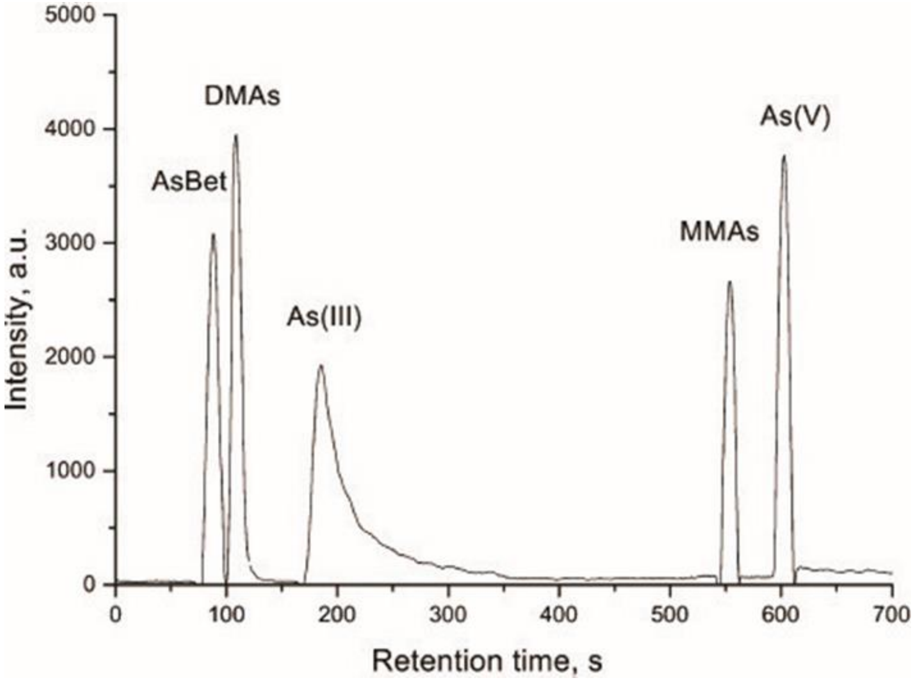

Figure 3. Determination of five arsenic species by IC-ICP-MS. Mobile phase: NaOH

And LoQ). These limits, these numbers give the information on the smallest concentration that can be detected and quantified with certain accuracy that has been defined [10]. The LoD was discussed and determined for the induced coupled plasma-mass spectrometry (ICP-MS) measurements of arsenic [11]. Thorough analysis has shown that the best option for

LoD would be experiments, which would include the repetition many times. If experiments would be repeated 100 times, it is expected that only five measurements would be inadequate. Although this is ideal, the time consumption for the repetitive measurements is not acceptable. The most important conclusions were that LoD is not permanent and constant value, and it has to be verified and adopted for each new case. LoD is a basic parameter for estimation of the LoQ. It was concluded in [11] that the traditional (IUPAC) method is the one that could be applied.

In this work, methods for arsenic and arsenic speciation separation, determination and removal were reviewed. There are numerous methods for separation and determination of arsenic species in water. It is very important to recognize easy, simple and inexpensive methods to estimate the very low concentrations of arsenic.

The total concentration of arsenic in drinking water can be detected by simple Gutzeit method, and some similar colorimetric methods of comparing stains produced on treated paper strips. Although its minimum detectable concentration is 1.0•µ L−1, these tests should be used when only a qualitative or semi qualitative detection is needed.

For precise, and reliable determination of arsenic in water, only sophisticated analytical techniques as ICP-MS, GF-AAS and HG-AAS can be applied. These methods are approved by US EPA. The features of these methods are high sensitivity, high accuracy, minimal sample volume; no sample pre-treatment and short measurement

For As speciation analysis, well-established methods that involve the coupling of separation techniques, such as HPLC with a sensitive detection system, that is, ICP-MS, are recommended, and they are mostly used. Through the limits, it is possible to define the smallest concentration of analyte that can be reliably detected and quantified. Limit of detection for the HPLC-ICP-MS system is 0.001 µ L−1. This system is also expensive and needs lot of knowledge for operating and interpretation of data.

The Pearce method with its modification

The Pearce method consists of a fusion with an alkaline carbonate and nitrate to from the arsenate which is precipitated as silver arsenate. The arsenic is determined indirectly through the silver, either by cupellation or titration with ammonium sulphocyanate .The criticisms of this method have been principally on the fact that the solubility of the silver arsenate varies with the condition of the solution and can never be counted on as constant.

Determination by use of Magnesium Mixture

The determination with Magnesium mixture is perhaps the best method known. It consists in bringing all the arsenic which is precipitated as ammonium magnesium arsenate by means of magnesium mixture in alkaline solution. This is burned and weighed as the magnesium pyro arsenate. (Putaraporn et al., n.d.) 29155469(pdf)

Spectrophotometric method

From the batch method of extraction 300mg of sorbents matrix were taken in each 100 ml flask with 20 ml of 0.1 mg/L of As(iii) and 1 ml of DMF was added in each sets and the pH of the solutions mixture was adjusted to 3.5 by using dil. HCI and dil.NaOH solutions . The mixture of the sorbent matrix and As (iii) solutions were shaken by mechanical shaker at about 400-500 rpm at room temperature for half hour. A reagent blank solution was prepared in the same procedure as the mixture solution in absence of Arsenic and the Absorbance of the sample solution was measured against the blank at maximum wavelength of 281 nm. The amount of As (iii) absorbed on sorbent materials were calculated by standard calibrated curve.

Determination of Arsenic in Soil

Neutron Activation Analysis

Nuclear reactor facility

The renewed triga mark III nuclear research reactor which was operated by the office of Atomic Energy for peace, Bangkok , Thailand .At the irradiation position , thermal neutron fluxes 8.7 x 1011ncm-2s-1and an epithermal neutron flux 1.0 x 1010ncm-2 s-1was used.

Gamma spectroscopy

The system consisted of a Tennelec coaxial type HPGE detector and relevant electronic .Recorded spectra was evaluated with the program Assayer from the software package Oxford Win of 16KMCA 8192 channel

Sample material

About 10 kg of five natural agricultural soil samples were collected from both unpollutant, non-residential areas and presumably polluted mining industrial areas in Ronpibul district. The samples were dried in air, quartered, ground and powered in an agatemorrtar, pass through 325 mesh sieves and stored in polyethylene bags

Standard and soil preparation

Each of 5 candidates' homogenized soils sample aliquots weighing about 350-400 mg were placed in polyethylene vial and heated sealed. For standard, SRM -2710 was re homogenized by shaking for a few minutes and aliquots of 100 mg of standard were prepared in a similar way with the samples**.**

Quantitative analysis

Optimization condition

Standard and soil sample no 5 were used to optimized conditions .Optimum conditions were determined by varying irradiated and cooling times in both a thermal neutron flux position and an epithermal neutron flux position. The irradiated standard and sample were allowed to decay for a predetermined time, transferred to clean counting and then measured by HPGE detector

Validation method

5 standard vials and 5 copper wires were introduced simultaneously in a rabbit. The Arsenic concentrations in SRM-2710 were determined by irradiated for 30 min, 1 day for cooling and 300s for counting and detected 559.1 KeV the photo peak of 76As by HPGE detector.

Quantitative analysis of soil sample

The sample were positioned in the rabbit with 5 vials which is comprised of 3 sandwich together with 2 standards. The Rabbit was irradiated for 30 min in both thermal and epithermal neutron irradiated positioned, cooled 1 day and counted of 559KeV the photo peak of 76As for 300s.(Putaraporn et al., n.d.)

Method 2

Instrumentation: AAS

All HR-CS GFAAS experiments were performed on an Analytic Jena Model contrAA-600 atomic absorption spectrometer (Analytic Jena AG, Jena, Germany) equipped with a transversally heated graphite tube atomizer and an MPE-60 (Analytic Jena) auto sampler. The primary radiation source used in this equipment is a xenon short-arc lamp, which emits a spectral continuum between 190 and 900 nm. The spectral line of arsenic 193.696 nm and the integrated absorbance of three pixels (CP±1) were selected for the analyses. The method of interactive background correction (IBC) was chosen for signal evaluation and the Aspect CS 2.1.2.0 Software® (Analytic Jena AG, Germany) was used for the assignment of atomic lines and molecular bands**.**

Pyrolytic graphite coated tubes with PIN-graphite platform (Analytic Jena, Part No.: 407-A81.025) were applied for the optimizations with aqueous standards. Solid sampling (SS) graphite tubes without a dosing whole (Analytic Jena, Part no. 407A81.303) and SS graphite platforms (Analytic Jena, Part no. 407-152.023) were used for all analyses using certified reference materials (CRM) and soil samples. An M2P microbalance (Sartorius, Göttingen, Germany) was used for weighing the samples directly onto the SS platforms, which was transferred to the atomizer with the aid of a pre-adjusted pair of tweezers. High-purity (99.996%) argon (White Martins, São Paulo, Brazil) was used as graphite furnace purge gas.

Reagents and solutions

All reagents used were of analytical grade or higher purity. Ultrapure water with a resistivity of 18.3 MΩ cm was obtained from a model Milli-Q Integral ultrapure water system (Bedford, MA, USA). Nitric and hydrochloric acid in ultrapure grade were obtained from Sigma-Aldrich (St. Louis, MO, USA). The both acids were used for microwave-assisted digestion and the nitric acid was used to decontaminate all containers and glassware.

A stock solution of Arsenic of 1.0 g L^{-1} was used to prepare the calibration curve and aqueous and aqueous standard solutions. Zirconium, iridium and tungsten platform surface by injecting ten successive volumes of 40 uL of a solution containing 1.0gL^{-1} and applying the temperature program in table1 after each injection. A mixture of Pd/Mg nitrate solution was tested as chemical modifiers in solution 10 microliter applied over the samples in combination with permanent modifier and Tungsten solutions /magnesium nitrate Solutions as a modifier in solution was also tested.

Certified reference materials and samples

To verify the accuracy of the method, two CRM were used: PACS-2-Marine sediment (National Research Council Canada, Ottawa, Canada) and BCR 142-Soil (Community Bureau of Reference, Brussels, Belgium). Eleven soil samples were collected around Jorge Lacerda–Tractebel Suezin power plant, in Santa Catarina, southern part of Brazil. This power plant is the largest TPC in Brazil and has an installed capacity of 857 MW. The coal used by the TPC is a blend of different suppliers, mainly from Santa Catarina, mixed with imported coal. The blend is basically composed of bituminous coal with high sulphur content (>2%) and 40% ash [18]. The power plant is surrounded by small farms, which produce, mainly rice and corn. The farms cultivate rice in paddy soils, using as water source the Tubarão River, which also supplies the coal-fired power plant. The soil samples were collected in three rice farms, according to the Figure 1, where the closest sampling point was 2.5 kilometres from the power plant, and the farthest one was 4.2 kilometres far from the power plant. The samples were collected from approximately 10 cm with a Teflon spatula and stored in sterile polyethylene bags. After collected, roots and leaves were manually removed, the samples were left to dry at room temperature for 48 hours, then were mashed in a grail and sieved in a 200 µm mesh.

Procedure of direct solid sample analysis and microwave-assisted digested samples analysis.

About 0.2 mg of soil CRM or soil sample was weighed directly onto the SS graphite platform, which was inserted into the graphite tube and submitted to the temperature program shown in Table 2. Calibration curve have been established using aqueous standard solution (50 µg L−1 As) manually injected onto the SS graphite platform pre-treated with Zr as permanent chemical modifier.

For the accuracy evaluation, the samples collected in Capivari de Baixo were submitted to a microwave-assisted digestion. As described by Schneider et al. [21], about 0.15 g of the sample was directly weighed onto Teflon vessel, added 10 mL of aqua-regia and submitted to a temperature and pressure program described at Table 2. After this procedure, the samples were transferred to a falcon flask, fi filled up to 15 mL with ultrapure water and determined by HR-CS GF AAS with the developed method. (Schneider et al., 2018)

Determination of Arsenic in Air

Analytical techniques in total As determination in PM range

Colorimetry

As early as 1969, a tentative method method for As content in PM was proposed by tabor et al. based on the As reduction to arsine with Sncl2 and Zn which reacts with a pyridine solution containing silver diethyldithiocarbamate .the resulting red complex was measured by photometry at 536 nm. As the minimum As that could be measured was 0.1mircogram m-3, the applicability of this method was not effective to real air samples, with typical concentrations of As in the ng m-3 range.

Hydride generation atomic absorption spectroscopy

Braman indicated in the 1970 that hydride generation with sodium borohydride for Arsine evolution was faster and less prone to interferences than the classical approaches using Zinc or magnesium. The Arsine detection by flame or flame less atomic absorption spectroscopy

(AAS) gave better detection limits than colorimetry by a factor of two. In this work, Braman already indicated the suitability of HG in combination with atomic absorption or emission spectroscopy for the detection of total As and its species atmospheric samples in Atmospheric samples.

In HG-AAS the sample is acidified with and a NaBH4 or KBH4 solution is added. The Arsine is transported by an inert gas carrier to a atomizer, A T- shaped quartz tube. The atomization of the arsine is achieved by heating the quartz tube either electrically or by an air/acetylene flame.

Electro thermal –atomic absorption spectroscopy

ET-AAS, also referred as graphite furnace atomic absorption spectroscopy (GF-AAS), is an alternative to HG-AAS for total As determination in PM. Its use is more extended than HGAAS, as it is included as one of the detection techniques recommended in the European Standard for As in PM10 (EN 14902:2005) [102]. This technique has been employed by several authors: Chakraborty et al measured concentrations of As ranging 9-512 ng m-3 in Delhi (India). Acevedo-Figueroa et al. [63] and Gioda et al. [85] found low As concentrations below 0.5 ng m-3 in PM2.5 and PM10 samples from Puerto Rico. Tsai et al. [87] analysed different PM fractions in Taiwan (PM2.5-PM10), finding As concentrations between 0.18 and 12.4 ng m-3. Higher As concentrations (maximum of 95.4 - 149 ng m-3) were reported by Šerbula et al. [75] and Kovaćević et al. [76] for PM10 samples taken at Bor (Serbia), nearby a copper smelter. Also, Thomaidis et al. [62] found 29.3 ng m-3 in Athens

(Greece), whereas Onat and Sahin [55] found 171.8 ng m-3 in industrial areas of Istanbul (Turkey). (Shahlaei & Pourhossein, 2014)

When employing ET-AAS for As determination, molecular interferences from the PM matrix should be taken into account. The common procedure is to work with Zeeman background correction and to add Pd (NO3)2 [55] or a mixture of Pd (NO3)2 and Mg (NO3)2 [62, 104] as matrix modifier. More recently, Pasias et al. [67] has proposed a zirconium-iridium permanent modifier for simultaneous determination of As, Pb, Cd and Ni. Some authors follow an established method for As determination by ET-AAS, corresponding to US EPA 7060A [64, 73]. There are some discrepancies between the LOD described in the literature, as they ranged from as low as 0.05 ng As m^{-3} [55] to 2 ng As m^{-3} [75]. In this later case, no indication is given if a matrix modifier is employed for analysis, and the amount of sample employed for the digestion is not indicated, parameters that can affect the LOD.

Inductively coupled plasma mass spectrometry (ICP-MS)

ICP-MS has the capability of multielemental analysis, providing at the same time low detection limits (e.g. LOD 0.05 ng m-3 [68]). It is nowadays the most employed technique for total As determination in PM. As it happens with ET-AAS, ICP-MS is also a recommended analytical technique, as indicated in the European Standard for As in PM10 (EN 14902:2005) [102], and therefore its use is more extended than other techniques. The As content in atmospheric samples has been evaluated in several countries around the world by ICP-MS analysis: Gidhagen et al. [61], Richter et al. [57] and Hedberg et al. [83] analysed PM10 samples in several areas of Chile, some of them near copper mines and smelters, with maximum As concentrations between 30.7 and 190 ng m-3.

Inductively coupled plasma- optical emission spectroscopy (ICP-OES)

In addition to ICP-MS, the literature cites few other plasma applications, such as ICP-OES Halek et al. [70] applied this technique for As determination in atmospheric samples of Teheran (Iran), where low As concentrations were found, with average means of 0.20-0.27 ng m-3, with a rather high LOD referred to the analysed solution (50 µg l-1). The poor sensitivity of ICP-OES makes this technique only suitable for samples with extremely high As concentration, like the ones analysed by Querol et. al. [77] in the Guadiamar Valley (Spain) after a severe spill of heavy metals waste, in which concentrations up to 2681 ng m^{-3} of As were found in TSP. Also, Morales-García et al. [88] found mean concentrations of 424.4 ng m^{-3} in Puebla City (Mexico), with a maximum of 4877 ng m^{-3}, due to the proximity of the active volcano Popocatépel. When HG is employed in combination with ICP-OES, the LOD is improved (e.g. 1 µg l^{-1}), as reported by Pandey at al. [64], who found a mean As concentration of 7.85 ng m^{-3} in Bhilai (India) with HG-ICP-OES.

Direct techniques

The term "direct techniques" is used in this context for those one that do not precise a chemical treatment, and analysis is performed directly on the PM retained onto the filters. In this sense, the literature describes As determination by PIXE, INAA and XRF.

PIXE has been one of the first techniques that was reported for analysis of As in PM. Already in the 1980's it was employed by Winchester and Bi [109] for PM analysis in samples of Beijing (China), finding As concentration between <0.28-10.3 ng m^{-3}. Also, Romo-Kröger et al. [50] found high As concentration (23-241 ng m^{-3} in fine particles) with PIXE, near a copper smelter in Chile. However, its use is scarce due to the fact that it is a technique n to analyse samples on an hourly basis, using a "streaker" sampler, thus providing more detailed information in comparison to most of the studies in which the samples correspond to a 24 hour sampling of available for most researchers. In spite of it, PIXE presents as an advantage the capacity period, as it has been described by D'Alessandro et al. [110] and FernándezCamacho et al. [111]. In this sense, PIXE analyses with a streaker sampler will help to identify peaks of pollutants in ambient air coming from anthropogenic or natural sources.

There are also few references about As determination in PM by INAA. Freitas et al. [54, 60] measured As in PM10 and PM2.5 of Lisbon and other locations in mainland Portugal, with mean As concentrations of 3.6 and 2.8 ng m^{-3}, resp .Also Šlejkovec et al. [123] determined total As concentrations by INAA in urban samples from Budapest (Hungary), with concentrations ranging from <0.30 to 5.3 ng m^{-3}. XRF has also been proposed for multielemental determination in PM10, although there are few references for As with this technique: Waldman et al. [112] determined As concentrations up to 64 ng m^{-3} in Wuhan (China), Vercauteren et al. [113] found a mean of 3.8 ng m^{-3} in Flanders (Belgium). In spite of that, an interlaboratory comparison exercise of As, Cd, Ni and Pb in PM10 conducted in Europe has demonstrated that XRF can provides similar reliable results as those obtained by

ICP-MS or ET-AAS [78]. Recently Godelitsas et al. [114] has described the use of Synchrotron-radiation µ-XRF for As determination in PM samples from Athens (Greece). (Sánchez-Rodas et al., 2015)

X-Ray Fluorescence Spectroscopy

In X-Ray Fluorescence (XRF) a beam of X-rays irradiates the particulate matter sample. This causes each element in the sample to emit characteristic X-rays that are detected by a solid-state detector or a crystal spectrometer. The characteristic X-ray is used to identify the element and the intensity is used to quantify the concentration of the measured element. X-ray fluorescence spectrometry (including energy dispersive mode and wavelength dispersive mode) can be used for all elements with atomic weights from 11 (Na) to 92 (Uranium) Furthermore, multiple elements can be determined simultaneously.

This method has the advantages of being non-destructive, requiring minimal sample preparation, providing immediate results and having low equipment cost. However, the detection limit is higher than other analysis techniques. In addition, it requires a thin collection deposit (i.e. 10 to 50 µg/cm^2) and it involves complex matrix corrections. Elements lighter than aluminium are often difficult to determine because of their low fluorescent yields and particularly because of the strong absorption of fluorescent X-rays by the substrate on which they are collected (US EPA, 1999a). Because high-volume samplers utilize quartz-filters that cause high background when employing XRF, analysis by XRF usually requires Teflon or Nylon filters used in the dichotomous or the Partisol samplers.

Proton Induced X-Ray Emission Spectroscopy

Some work on trace metal analysis has also been performed using Proton Induced X-Ray Emission (PIXE) Spectroscopy . PIXE analysis is very similar to XRF analysis in that the sample is irradiated by a high-energy source, in this case high energy protons to remove the inner shell electron. Fluorescent X-ray photons are detected employing the same detection methods as XRF and used to identify and quantify different elements in the sample.

PIXE is one of the more commonly used elemental analysis methods because of its relatively

Low cost, non-destructive, multi-element capabilities. It is potentially capable of determining 72 elements with molecular weights between those of sodium and uranium, simultaneously (ARPEL, 1998). The method provides the sensitivity for accurate measurements at the nomogram or less level for many important trace metals in the urban atmosphere. The PIXE method has the ability to analyse a very small sample diameter in addition to evenly distributed wide-area samples, which is advantageous because it permits analysis of individual particle size fractions collected with single orifice type cascade impactors. PIXE is capable of measuring smaller quantities of particulate matter, although it has the same limitations as with XRF concerning light elements. In addition, facilities for this method are expensive and not common and it is less suitable for routine filter analysis than other multi-elemental methods because of more complicated sample preparation (US EPA, 1994). Analysis by PIXE typically involves collecting particulate matter by dichotomous or Partisol samplers.

Alternative Analytical Method

There have been several reports of Energy Dispersive X-Ray (EDX) Spectrometry being used in conjunction with Scanning Electron Microscopy (SEM) . Scanning Electron Microscopy with Energy Dispersive X-Ray (SEM-EDX) Spectrometry uses a computer controlled scanning electron

Microscope equipped with image analysis software to determine the size and shape of a moderate number of particles and EDX to provide qualitative and a moderately sensitive quantitative elemental analysis in a similar manner as XRF analysis. Generally, low loadings are required to employ this technique, therefore, a low-flow device such as dichotomous, Partisol or the MiniVol samplers should be used.

The primary advantage of the SEM-EDX technique is the ability to characterize individual particles both chemically and physically. The Expert Panel on the U.S. Environmental

Protection Agency PM2.5 Chemical Speciation Network has recommended using the SEM-EDX for analysis of air filters. The panel found that microscopic techniques could be used to characterize both the morphology and the chemical composition of individual particles .The disadvantages of the SEM-EDX technique include poor quantitative sensitivity and practical difficulties such as excessive time for a representative analysis and the occurrence of both particle damage and compositional changes during analysis . In addition, the EDX technique often results in potential spectral interferences requiring complex spectral deconvolution procedures. (On et al., n.d.)

Conclusion

As the Arsenic is toxic and poisonous it determination is important for the environment .Arsenic can determined from air, water and soil using neutron activation analysis, X-ray fluorescence , colorimetry, ET- AAS, atomic absorption spectroscopy, PIXE, atomic emission spectroscopy , spectrophotometry and ICP with hyphenated technique. It was observed that the best method for determination are advanced hyphenated techniques as it can give result of even small amount of Arsenic in the sample to analysis.

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