BARC/2003/E/013

BARC/2003/E/013

A CLEAN LABORATORY FOR ULTRATRACE ANALYSIS THE ULTRATRACE ANALYTICAL FACILITY (UTAF)

by

S.G. Jadhav, Suvarna Sounderajan, Sanjukta A. Kumar, A.C. Udas, M. Ramanamurthi, M.M. Patrecha

and

M. Sudersanan
Analytical Chemistry Division

भारत सरकार
Government of India
भाभा परमाणु अनुसधान केंद्र
Bhabha Atomic Research Centre
मुबई Mumbai - 400 085, भारत India
2003

BARC/2003/E/013

BARC/2003/E/013

GOVERNMENT OF INDIA
ATOMIC ENERGY COMMISSION

A CLEAN LABORATORY FOR ULTRATRACE ANALYSIS THE ULTRATRACE ANALYTICAL FACILITY (UTAF)

by
S. G. Jadhav, Suvarna Sounderajan, Sanjukta A. Kumar,
A. C. Udas, M Ramanamurthi, M. M. Palrecha
and
M.Sudersanan
Analytical Chemistry Division

BHABHA ATOMIC RESEARCH CENTRE
MUMBAI, INDIA
2003

BIBLIOGRAPHIC DESCRIPTION SHEET FOR TECHNICAL REPORT
(as per IS : 9400 - 1980)

01	*Security classification :*	Unclassified
02	*Distribution :*	External
03	*Report status :*	New
04	*Series :*	BARC External
05	*Report type :*	Technical Report
06	*Report No. :*	BARC/2003/E/013
07	*Part No. or Volume No. :*	
08	*Contract No. :*	
10	*Title and subtitle :*	A clean laboratory for ultratrace analysis: the ultratrace analytical facility (UTAF)
11	*Collation :*	27 p., 10 figs., 4 tabs., 1 ill.
13	*Project No. :*	
20	*Personal author(s) :*	S.G. Jadhav; Suvarna Sounderajan; Sanjukta A. Kumar; A.C. Udas; M. Ramanamurthi; M.M. Palrecha; M. Sudersanan
21	*Affiliation of author(s) :*	Analytical Chemistry Division, Bhabha Atomic Research Centre, Mumbai
22	*Corporate author(s) :*	Bhabha Atomic Research Centre, Mumbai-400 085
23	*Originating unit :*	Analytical Chemistry Division, BARC, Mumbai
24	*Sponsor(s) Name :*	Department of Atomic Energy
	Type :	Government

30	*Date of submission :*	May 2003
31	*Publication/Issue date :*	June 2003
40	*Publisher/Distributor :*	Head, Library and Information Services Division, Bhabha Atomic Research Centre, Mumbai
42	*Form of distribution :*	Hard copy
50	*Language of text :*	English
51	*Language of summary :*	English, Hindi
52	*No. of references :*	10 refs.
53	*Gives data on :*	

60 *Abstract :*

There has been an increasing demand for the quantification of various elements at extremely low concentrations in a variety of samples such as high purity materials, environmental and biological samples. The need for a controlled environment to obtain reliable and reproducible data necessitates the use of strategies and practices to minimize contamination during the analytical procedure. This report describes the protocol observed in our clean laboratory to eliminate contamination and ensure low laboratory blanks and some of the methodologies developed to carry out the analysis. The analysis is carried out by Graphite Furnace Atomic Absorption Spectrometry and electrochemical techniques such as Anodic/Cathodic/Adsorptive Stripping Voltammetry. Characterisation of 5N (total impurities 10ppm) arsenic is routinely carried out. Al in serum of patients suffering from end stage renal failure are also analyzed. Pine leaves, spinach, carrot puree and milk powder have been characterized for Al and Hg content and bovine serum has been characterized for Cu, Zn, Na, K ... samples as part of intercomparison exercises.

70	*Keywords/Descriptors :*	ABSORPTION SPECTROSCOPY; VOLTAMETRY; ARSENIC; QUANTITATIVE CHEMICAL ANALYSIS;; ENVIRONMENT; PARTICULATES; BLOOD SERUM; BISMUTH; CADMIUM; IMPURITIES; SENSITIVITY; TRACE AMOUNTS; NUCLEAR FUELS
71	*INIS Subject Category No :*	S37
99	*Supplementary elements :*	

A CLEAN LABORATORY FOR ULTRATRACE ANALYSIS
THE ULTRATRACE ANALYTICAL FACILITY (UTAF)

S.G.Jadhav, Suvarna Sounderajan, Sanjukta A.Kumar, A.C.Udas,
M.Ramanamurthi, M.M.Palrecha & M.Sudersanan
Analytical Chemistry Division
BARC, Mumbai –400085.

ABSTRACT

There has been an increasing demand for the quantification of various elements at extremely low concentrations in a variety of samples such as high purity materials, environmental and biological samples. The need for a controlled environment to obtain reliable and reproducible data necessitates the use of strategies and practices to minimize contamination during the analytical procedure. This report describes the protocol observed in our clean laboratory to eliminate contamination and ensure low laboratory blanks and some of the methodologies developed to carry out the analysis. The analysis is carried out by Graphite Furnace Atomic Absorption Spectrometry and electrochemical techniques such as Anodic/ Cathodic / Adsorptive Stripping Voltammetry. Characterisation of 5N (total impurities 10ppm) arsenic is routinely carried out. Al in serum of patients suffering from end stage renal failure are also analyzed. Pine leaves, spinach, carrot puree and milk powder have been characterized for Al and Hg content and bovine serum has been characterized for Cu, Zn, Na, K in samples as part of intercomparison exercises.

परा इनूरकख विश्लेषण हेतु एक स्वच्छ प्रयोगशाला
परा अनुरेख विश्लेषणात्मक सुविधा (UTAF)

एस.जी.जाधव, सुवर्णा सौंदरराजन, संजुक्ता ए. कुमार, ए.सी.उदास,
एम. राममूर्ति, एम.एम.पलरेचा एवं एम. सुदर्शनन
विश्लेषणात्मक रसायनिकी प्रभाग
भापअ केंद्रए मुंबई - 400 085

सारांश

उच्च परिशुद्ध पदार्थों, पर्यावरणीय तथा जैविक नमूनों जैसे अनेक प्रकार के नमूनों में चरम निम्न सांद्रता पर विभिन्न तत्वों के प्रभावीकरण हेतु मांग में वृद्धि हुई है । विश्वसनीय एवं पुनःप्राप्य डाटा प्राप्त करने हेतु आवश्यक नियंत्रित वातावरण के लिए विश्लेषणात्मक क्रियाविधि के दौरान होने वाले विसंदूषण को कम करने के लिए कुशलता एवं अभ्यास की आवश्यकता है । रिपोर्ट में विसंदूषण को दूर करने के लिए स्वच्छ प्रयोगशाला में पालन किये जाने वाले प्रोटोकोल एवं निम्न प्रयोगशाला रिक्तता सुनिश्चित करने तथा विश्लेषण हेतु विकसित पद्धतियों पर चर्चा की गयी है । यह विश्लेषण ग्रैफाइट भट्टी परमाणु अधिशूषण वर्णक्रममापी और एनोडीक/कैथोडीक अधिशूषण विपट्टन वोल्ट धारा मिति जैसे विद्युतरासायनिक तकनीकों द्वारा किया जाता है । 5 N (10 ppm कुल अशुद्धता) अर्सेनिक का नेमी अभिलक्षणन किया जाता है । तीव्र वृक्क पात के अंतिम चरण में ग्रस्त रोगियों के सीरम में Al का भी विश्लेषण किया जाता है । अंतर्तुलनात्मक अभ्यास के रूप में देवदार के पत्ते, पालक, गाजर का शोरबा एवं दूध के पाउडर को भी Al एवं Hg के लिए अभिलक्षित किया गया और गोजातीय सीरम को Cu, Zn, Na, K को नमूनों में अभिलक्षित किया गया है ।

A CLEAN LABORATORY FOR ULTRATRACE ANALYSIS
THE ULTRATRACE ANALYTICAL FACILITY (UTAF)

S.G.Jadhav, Suvarna Sounderajan, Sanjukta A.Kumar, A.C.Udas,
M.Ramanamurthi, M.M.Palrecha & M.Sudersanan
Analytical Chemistry Division
BARC, Mumbai –400085.

INTRODUCTION

The term trace analysis is widely used to describe the application of analytical techniques under circumstances where the amount of analyte is very small. It has a special significance in nuclear technology. Materials such as fuels, moderators, cladding materials, control and other structural materials have very specific purity requirements. There is also a widespread need for trace analysis in a variety of other applications such as characterization of ultrapure materials for technological advances, establishment of meaningful tolerance limits for pollutants, analysis of geological and celestial samples, determining the distribution and abundance of trace constituents to effectively use the ocean resources, scientific data for forensic investigations, studying the physics and chemistry of semiconductors, elucidating the role of trace metals in biological functions and determining mechanisms by which heavy metals induce toxicity [1,2].

The purity of a material is specified on the "number of nines" concept. A specification of 5N indicates a purity of 99.999% and 4N5 indicates a purity of 99.995% The N value is assigned after totaling all the impurity concentrations expressed as a percentage and subtracting from 100. Characterisation of elements at microgram to nanogram levels constitutes a major task in trace analysis. This requires the development of new methods and techniques for highly sensitive and accurate analysis. This also requires methods for preventing and controlling contamination in the laboratory. Contamination is the most insidious problem affecting accurate trace analysis. It is caused by particulates in the air, handling of sample, reagents, trace elements in containers used during the analysis and from the analyst. The magnitude of the contamination is measured by the process blank. Thus it is imperative to minimize the magnitude and variability of the blank to obtain the best possible detection limits. This objective is achieved by carrying out the analysis in a clean room. The Ultra Trace Analytical Facility (UTAF) was set up with the above objectives in mind.

CLASSIFICATION OF CLEAN LABORATORIES

A clean room is any room or area where an attempt is made to limit, control, and eliminate the amount of airborne contamination. The word "attempt" is important, because there is no such thing as a totally clean room, i.e., a room with absolutely no contamination. There are only degrees of cleanliness, but more often than not, these less than perfect conditions will suffice for the purpose at hand. Clean laboratories are classified according to the international airborne particulate cleanliness classification by ISO [3](Table 1).

In June 1999 the International Standards Organisation (ISO) published a standard ISO 14644-1 [3]. The classes are based on the formula

$$C_n = 10^N \times (0.1/D)^{2.08}$$

where C_n is the maximum permitted number of particles per cubic meter equal to or greater than the specified particle size. It is rounded to a whole number, N is the ISO class number, which must be a multiple of 0.1, and be 9 or less, D is the particle size in micrometers.

Cleanliness class designations and quantity have changed from FS209E. Along with the obvious change to metric measure of air volume, ISO 14644-1 adds three additional classes , two cleaner than Class 1 and one less clean than Class 100,000.

ULTRA TRACE ANALYTICAL FACILITY (UTAF)

A. Non Laminar Flow Positive Pressure Clean Laboratory

A schematic layout of UTAF is given in figure 1. The laboratory was constructed by suitably modifying the existing laboratory and is designed to have different areas of air cleanliness. It has a total area of 100 m^2. The laboratory is isolated from the external air. The outside air is cooled and blown into the clean room after filtering through prefilters and a bank of HEPA filters using two blowers (4). This clean room is maintained at a positive pressure to allow clean air to diffuse into the other parts as well as to prevent air from the other parts of the laboratory to enter into the clean room. Double door entry system is used for personnel to step in and out of the clean room. Five laminar flow work benches designed to provide class 100 or better work surfaces are located inside the clean room. Besides the laminar flow benches, two other plain benches for accommodating the Atomic Absorption unit and the Electro

benches for carrying out the various cleaning and sample processing operations. The clean benches and other fixtures are made of wood and/or polypropylene. The wooden surfaces are laminated. The walls are coated with epoxy paint to prevent any dust arising from them. The laboratory is equipped with several polypropylene sinks and storage shelves and cabinets made of laminated wood. The flooring is made of 2mm thick sheets of PVC. The laboratory is divided into four zones as indicated in the figure 1. The innermost zone (Zone A) housing the LAF benches is the cleanest zone with respect to particulate matter as air filtered through the HEPA filters directly enters this zone before diffusing to other zones. The laminar air flow benches are housed in this zone. Zone B (Electroanalytical Room) is the area where filtered air mixes with air diffusing out of Zone A and is therefore less clean than Zone A. Zone C (General Laboratory) and Zone D (AAS Room) receive a mixture of filtered air and air diffusing from Zone A and Zone B and are therefore less clean with respect to particle counts than the other two zones. Particle counts are taken in four different zones of UTAF daily using a LASAIR II particle counter (PMS Inc., USA). It is a 1CFM particle counter using a 50 mW infrared diode laser lasing at 780 nm. It accumulates counts in 6 channels, the channel thresholds being 0.5, 0.7, 1.0, 2.0, 3.0, 5.0 and 10.0 μm. It has a countings efficiency of >50% and can count a maximum concentration of 900,000/ft^3.

The minimum number of sampling locations to be measured for particulate counts is given by the formula

"$N_1=\sqrt{A}$" where

N_1 = the minimum number of sampling locations, rounded to a whole number and A = the area of the clean room or clean air controlled space in m^2.

Data was collected in both "at rest " condition and under "operational" condition. Sampling locations were evenly distributed throughout the area of the clean room and positioned at the height of the work activity. The laminar benches meet the ISO Class 4 (FS Class 10) criteria, Zone A meets ISO Class 6 (FS Class 1000), Zone B meets ISO Class 7 (FS Class 10000) and Zone C and D meet ISO Class 8 (FS Class 100000) criteria.

b. Laminar Air Flow Benches

There are four laminar air flow benches located in Zone A, two of which are vertical type (7.5 ft^2) and two horizontal type (6.0 ft^2). These benches are equipped with HEPA filters with PVC frames and separators working at an efficiency of 99.7 % for 0.5μ particles. They are made of polypropylene and laminated particle board. The

horizontal type work benches are used for storage of clean vessels. The vertical flow benches are used for carrying out chemical work. One of the benches is equipped with an exhaust system to enable handling of acids.

c. Clean Room Apparel

The analyst is one of the principle sources of contamination and all care is taken to prevent contamination from clothing, hair and hands. The analyst wears clean room garments which produce little or no particle emission by itself. Synthetic fibers woven into fabrics best meet these requirement. All clean room apparel used in the laboratory is made of Dacron. Dacron is emission free, crisp and soft and drapes smoothly. It develops low static charge, retains it's whiteness and is one of the most wrinkle resistant fabrics known. It is quick drying, retaining less than 1% of the moisture on washing. All personnel working in the clean laboratory wear full length two piece coveralls with plastic buttons(knee length) and pajamas. They are also equipped with hand gloves, overshoes with PVC soles, surgeon type caps covering the head and ears.

Tacky Mats (sticky mats) are placed at the entrances of all zones to trap any dust on the footwear onto these mats.

d. High Purity Water

The most important aspect in ultra trace analysis is the availability of ultra pure water and ultra pure reagents. The most important class of reagents are the mineral acids. Large amounts of ultra pure acids and water are required all through the various steps of ultra trace analysis, starting from cleaning of vessels to sample dissolution. Ultra pure water is obtained starting from tap water using a two stage purification procedure supplied by Barnstead Thermolyne. In the first stage, a high capacity cartridge D 0803 purifies tap water to provide feed water with a resistivity of 2 MΩ. This feed water is then fed into a Type D 4700 Nanopure analytical deionisation system to provide water with a resistivity of 18.3 MΩ required for ultratrace analysis. The D 4700 system consists of one pretreatment cartridge, two ultra pure cartridges and one organic free cartridge. The water is collected in 15 litres polythene carboys. Some of the important trace impurities in this water which have been measured by GFAAS.are given in Table 2.

e. Mineral Acids

HNO_3 is used extensively in the laboratory for cleaning of labware and during the analytical procedures itself. For preparation of acid baths and for cleaning purposes, acid purified from analar grade acid by sub-boiling distillation is used (fig 4). The purity levels of this acid with respect to common trace impurities are given in Table 2. Double distilled acid has also been prepared in this apparatus and surpasses the quality of Suprapure Nitric Acid. For purposes of sample preparations and dilution of standards, Suprapure Nitric Acid from E.Merck, Germany is used. The acid is accompanied with a purity certificate and the maximum concentrations of common trace impurities does not exceed 5 ng/ml

f. Labware and Cleaning Procedures

Contamination and losses of trace elements by leaching or adsorption from the laboratory ware lead to inaccuracies in trace elemental estimations. Glassware is especially known to cause errors due to it's adsorption and leaching properties and it's use is best avoided or minimised to the extent possible. Labware made of polythene, teflon PFA or high purity quartz are used for most applications. The various acid baths are made of seamless, rotational moulded polyethylene tanks which are lightweight and easy to handle. These tanks are resistant to most acids and alkalis. They are made with wide top flanges and include covers.(Fig 5)

The labware to be used is subjected to a rigourous cleaning procedure prior to use. They are first washed with tap water to remove surface dirt and dust and then rinsed with acetone to remove greasy material. This is followed by thorough rinsing with ultrapure water several times. They are then immersed in three successive baths of 10% , 1% and 0.1% of distilled nitric acid in polyethylene tanks for one week each. The labware is left in the last bath till further use. They are then once again rinsed several times with high purity water before the actual use.

The high purity water is stored in HDPE dispensing jugs of 20 liters capacity. Samples after dissolution are stored in pre cleaned Teflon PFA vials with screw caps.

g. Sample Dissolution

Sample dissolution carried out by the traditional open beaker digestion is an antiquated method susceptible to errors primarily from contamination and losses of analytes from the sample. Sample dissolutions in UTAF are carried out using a closed system Microwave Sample Dissolution equipment. The system ensures a

clean, closed and controlled reaction environment. The amount of acid used is diminished to stoichiometric quantities of the reagent, further reducing the possibility of contamination from the acid. The closed vessel decomposition is carried out in specially constructed PFA vessels capable of withstanding pressures up to 600 PSI (Fig 6). This leads to superheating of the acid mixtures, resulting in a dramatic acceleration of the digestion reaction — up to 100 times faster than a hotplate! The system ensures high throughput and rapid sample turnaround, improved recoveries of volatile analyte , eliminates cross contamination and reduces acid trace impurities thus leading to lower detection limits and higher sensitivity. The microwave power output is managed through direct feedback from temperature and pressure probes, providing control of the chemical reactions. The system is constructed of solid steel, encased in a protective polymer shell, and has a fluoro polymer coated cavity and an impact resistant door for safety and durability.

h. Instrumental Facilities

(i) Graphite Furnace Atomic Absorption Spectrometry (GFAAS)

Graphite furnace atomic absorption spectroscopy (GFAAS) is a highly sensitive spectroscopic technique that provides excellent detection limits for measuring concentrations of metals in solid and liquid samples. The detection limits range from the ppb to ppm levels for the most common contaminants. In this technique , a beam of electromagnetic radiation of specific wavelength (resonance wavelength) corresponding to the element of interest is passed through the atomic vapor of the element, and measurement is made of the attenuation of the intensity of the beam as a result of absorption. Since atomic transitions are sharp and intense, very low concentrations also give rise to measurable absorbance. The relationship between change in intensity and concentration is given by the Beer Lambert's relationship

Absorbance (A) = Log $I_0/I_t = \epsilon$ c l where I_0 = Incident beam intensity

I_t = Transmitted beam intensity

ϵ = extinction coeffecient

c= concentration and

l=path length

Quantitative analysis by AA depends on: (1) accurate measurement of the intensities of the incident and transmitted beam and (2) the condition that the radiation absorbed is proportional to atomic concentration as per the above relationship. A calibration curve is constructed by plotting concentrations of standards vs. the absorbance

(fig 7). The samples can be tested and measured against this curve. Samples to be analyzed by AA must be vaporized or atomized, typically by using a flame or graphite furnace.(Fig 8) The graphite furnace is an electrothermal atomizer system that can produce temperatures as high as 3,000°C. The heated graphite furnace provides the thermal energy to break chemical bonds within the sample and produce free ground state atoms. Ground state atoms then are capable of absorbing energy, in the form of light, and are elevated to an excited state. The amount of light energy absorbed increases as the concentration of the selected element increases. The absorbance of the analyte in the sample is measured and the concentration determined from the calibration curve.

GFAAS instruments have the following basic features (fig. 9)

1. a source of light that emits the resonance radiation
2. an atomization chamber (graphite tube) in which the sample is vaporised
3. a monochromator for selecting only one of the characteristic wavelengths (visible or ultraviolet) of the element of interest
4. a detector, generally a photomultiplier tube suitable for low intensity applications, that measures the amount of absorption
5. a signal processor and computer system

Working standards in the range of expected analyte concentration are prepared just prior to analysis by diluting a suitable stock solution of the analyte standard. Dilutions are carried out with variable volume micropipettes using disposable precleaned polypropylene tips. Transfer of sample and samples from clean room after processing for analytical measurements is carried out through pass through transfer boxes which open in a specific direction to allow clean air to pass from the inner room to the outside. (fig.1)

(ii) Stripping Voltammetry

Stripping voltammetry is one of the most sensitive, convenient, and cost effective analytical method for detection and quantitation of metal contaminants in solution samples. The method preconcentrates the analyte at the working electrode by a bulk electrolysis step. This preconcentration step does not require any sample handling thus reducing the incumbent risk of contamination. As a result of the insitu preconcentration step, stripping voltammetry techniques yield extremely low detection limits, detection limits of 10^{-10} M being common for many metals. Several

metals such as Sb, Bi, Cu, Pb and Cd can be analyzed simultaneously on account of their differing half wave potentials by this method giving it a multielement capability. Following the electrodeposition step, the material is stripped from the electrode by electrolysis using a potential sweep. Current peaks appear at potentials corresponding to the oxidation of metals as they are oxidized (stripped) from the electrode back into the solution. The amount of the metal deposited at the cathode during the preconcentration is a function of the deposition time, the stirring rate and the electrode surface area. The peak height or area can be correlated with the concentration of the metal ions in the solution. Prior calibration with standard solutions containing known quantities of the metal ions is carried out and from the peak location and height on the sample, the identity and concentration of the analyte can be determined.

The stripping step may consist of a positive or a negative potential scan, creating either an anodic or cathodic current respectively. Hence, Anodic Stripping Voltammetry (ASV) and Cathodic Stripping Voltammetry (CSV) are two specific stripping techniques.

In anodic stripping voltammetry, the cell is made up of a solution of a supporting electrolyte (eg. KCl) and three electrodes: a working electrode-hanging Hg drop electrode or a mercury film deposited onto glassy carbon, a reference electrode of silver/silver chloride and an auxillary electrode of platinum wire. The mercuric ion is added directly to the supporting electrolyte and during the preconcentration step it codeposits with the metal of interest.

Two of the techniques that can be used to reoxidize the metal from the electrode surface are linear sweep voltammetry and differential pulse voltammetry. In linear sweep voltammetry, the potential is scanned linearly as a function of time. In differential pulse voltammetry, a rectangular pulse is applied to a potential ramp. The output for differential pulse voltammetry is in the form of a peak whose height is proportional to the concentration of the analyte. Differential pulse voltammetry is a current measurement technique that enhances the analytical (faradaic) current and avoids the background (charging) current, resulting in a higher signal to noise ratio for a better detection limit.

Cathodic Stripping Voltammetry is used to determine those analytes that form insoluble compounds with mercury. In CSV, the mercury working electrode is not inert, but takes an active part in the formation of the deposit. Stripping in CSV consists of a cathodic scan to reduce the surface deposited mercuric compound

back into the solution. This technique finds use in the quantification of anions such as Cl^-, Br^-, I^-, S^{2-}, SeO_3^{2-} etc.

Adsorptive stripping methods are used for the quantification of Ni, Co and Cr as some of their complexes have a tendency to get adsorbed from aqueous solutions on to the mercury surface. This is followed by stripping the complex back into the solution by applying a cathodic potential scan.

A typical anodic stripping voltammetry scan of high purity arsenic sample taken for 5 trace elements viz. Cd, Pb, Cu, Sb and Bi is shown below (fig 10). After the procedure has been standardized, analysis of a large number of samples can be carried out routinely and rapidly with a high degree of precision and accuracy. Analysis can be carried out in the sub ng to μg levels for the above elements.

I. Role of Blank in Analytical Determinations

An important prerequisite in ultra trace analytical measurements is the reduction and control of the blank. Accurate and detailed measurements of the blank are necessary for any trace and ultra trace measurement. The quality of the data will be highly dependant upon these blank determinations. The blank values in UTAF for some of the common elements are given in table 3 . The values for the same elements in an ordinary laboratory by an identical procedure and measurement protocol are juxtapositioned to highlight the improvement observed in the clean room.

J. Sample Analysis

Table 4 lists some of the samples analysed at UTAF. The list of samples encompasses a range of material such as high purity arsenic, biological, environmental and agricultural samples. The range of concentrations measured fall in the ppb to ppm range. The methodologies used in these analyses have been developed in this laboratory and published.

CONCLUSIONS

The Ultra Trace Analytical Facility is ideally suited to carry out analysis of samples in which the levels of impurities are in the low ppm or ppb range. This has relevance in diverse fields such as clinical, biological, agricultural, metallurgical and environmental samples. A large number of such samples are being routinely analysed in this laboratory and with the experience gained and with the upgradation of the existing facilities, possibilities of analysis at even lower levels can be opened up.

REFERENCES

1. *Ultrapurity in Trace Analysis* , J.W.Mitchell, Analytical Chemistry, 45, 492A(1973).
2. Federal Standard 209E, Federal Standard Airborne Cleanliness Classes in Cleanrooms and Clean Zones, Sep 1992.
3. A Clean Laboratory for Ultralow Concentration Heavy Metal Analysis, C.F. Boutron, Fresnius J Anal Chem 337(1990) ,482.
4. Nuclear and other *Instrumental Methods of Analysis at Trace and Ultratrace* Levels of Concentrations, Jayaram Arunachalam, Ph.D. Thesis, Bombay University, October 1991
5. Insitu Matrix Volatilisation Studies and Characterisation of some Trace Elements in High Purity Arsenic by GFAAS, A.C.Udas, M.B.Sanglikar, Sanjukta A.Kumar, M.Ramanamurthi, M.Sudersanan and P.K.Mathur, *Atomic Spectroscopy*, 21(2000), 71.
6. Adsorptive Stripping Voltammetric Determination of Chromium in Gallium, M.M.Palrecha and P.K.Mathur, Talanta, 45 (1997), 433.
7. Square Wave Adsorptive Stripping Voltammetric Determination of Chromium in High Purity Arsenic Metal, Suvarna Sounderajan and M.M.Palrecha, presented in the Seventh International Symposium on Electrochemical Science and Technology (SAEST-VII), Nov 27-29, 2002.
8. *Determination Of Trace Amounts of Selenium in Arsenic Metal by Differential* Pulse Cathodic Stripping Voltammetry, Suvarna S.Shelke, S.S.Sawant, M.M.Parecha, A.V.Kulkarni and P.K.Mathur, Bulletin of Electrochemistry, 18 (2002), 13.
9. Determination of Zinc in High Purity Gallium by Differential Pulse Anodic Stripping Voltammetry, Suvarna Sounderajan, Avanti Singh and M.M.Palrecha, Indian Journal of Chemistry, 41A (2002), 1433
10. Simultaneous Determination of Cd(II), Cu(II), Pb (II), Bi(III) and Sb(III) in High Purity Arsenious Oxide by Differential Pulse Anodic Stripping Voltammetry, Suvarna Sounderajan, M.M.Palrecha and P.K.Mathur, presented at national seminar on Trends in Chemical Research, GND University, Amritsar, March 1999.

TABLE 1 WHOLE NUMBER ISO/ FEDERAL STANDARD AIRBORNE PARTICULATE CLEANLINESS CLASSES

Airborne Particulate Cleanliness Classes						
CLASS	**Number of Particles per Cubic Meter by Micrometer Size equal to or greater than the specified size**					
ISO 14644-1 / FS 209E	0.1 µm	0.2 µm	0.3 µm	0.5 µm	1 µm	5 µm
ISO 1/*	10	2				
ISO 2/*	100	24	10	4		
ISO 3 / 1	1,000	237	102	35	8	
ISO 4 / 10	10,000	2,370	1,020	352	83	
ISO 5 / 100	100,000	23,700	10,200	3,520	832	29
ISO 6 / 1000	1,000,000	237,000	102,000	35,200	8,320	293
ISO 7 / 10000				352,000	83,200	2,930
ISO 8 / 100000				3,520,000	832,000	29,300
ISO 9				35,200,000	8,320,000	293,000

* No equivalent FS Classification

TABLE 2 METTALIC IMPURITIES PRESENT IN SINGLE DISTILLED NITRIC ACID, SUPRAPURE NITRIC ACID AND NANOPURE WATER

Elemental	*Nitric acid (10%) (ng/ml)*	*Nanopure Water (ng/ml)*	*Suprapure Nitric Acid (ng/ml)*
Al	65	1	14
Cd	0.8	0.7	n.d.
Cu	2.5	2	n.d.
Fe	18.8	0.8	13
Ni	8.5	1.5	n.d.
Pb	3.4	0.5	n.d.
Sb	n.d.	n.d.	n.d.

n.d. : Not Detected

TABLE 3 COMPARISON OF BLANK VALUES IN CLEAN ROOM vis. a vis. ORDINARY LAB

ELEMENT	CLEAN	ORDINARY
Al	26	126
Cd	0.2	1.0
Cu	1.0	5.0
Co	1.8	33
Cr	0.2	16
Fe	17	110
Mn	3	3
Pb	5	16
Zn	5	15

TABLE 4 TYPICAL SAMPLES ANALYSED AT UTAF

SAMPLES	ELEMENTS	RANGE OF CONCENTRATIONS MEASURED
		GFAAS
		ppm
Arsenic Samples	Al, Ag, Bi, Ca, Cd,	0.05-1
High Purity (5N)	Co, Cr, Cu, Fe,	
	Mn, Mo, Se, Te,	
	Sb, Ni, Pb, Mg,	
	Zn, Sn	
Blood Serum	Al	0.01
Whole Blood	Se	0.01
Spinach	Al	1.9 mg/g
	Hg	<0.2
Carrot Puree	Al	1.0
	Hg	<0.2
Milk Puree	Al	5.0
	Hg	<0.2
Cauliflower	Al	19.9
	Hg	<0.2
Bovine Serum	Cu	0.04 - 1.5
	Fe	0.2 – 2.0
	Zn	0.04 – 0.1

Table 4 (Contd...)

SAMPLES	ELEMENTS	RANGE OF CONCENTRATIONS MEASURED ELECTROCHEMICAL METHOD
Arsenic samples high purity (5N) Water	Cd, Pb, Cu, Sb, Bi, Se, Ni and Co	0.05-0.5 ppm
	In	0.03-2.0 $\mu g\ ml^{-1}$
	As	0.1-4.0 $\mu g\ ml^{-1}$
Waste water	Cr(III) & Cr(VI)	10-50 $ng\ ml^{-1}$
Sea water	Ni	0.01-0.5$\mu g\ ml^{-1}$
Gallium metal	Pb, Zn	10-50 ppm
Pd-Charcoal	Pd	0.5 $mg\ g^{-1}$
Plant samples	Se	< 0.1 ppm

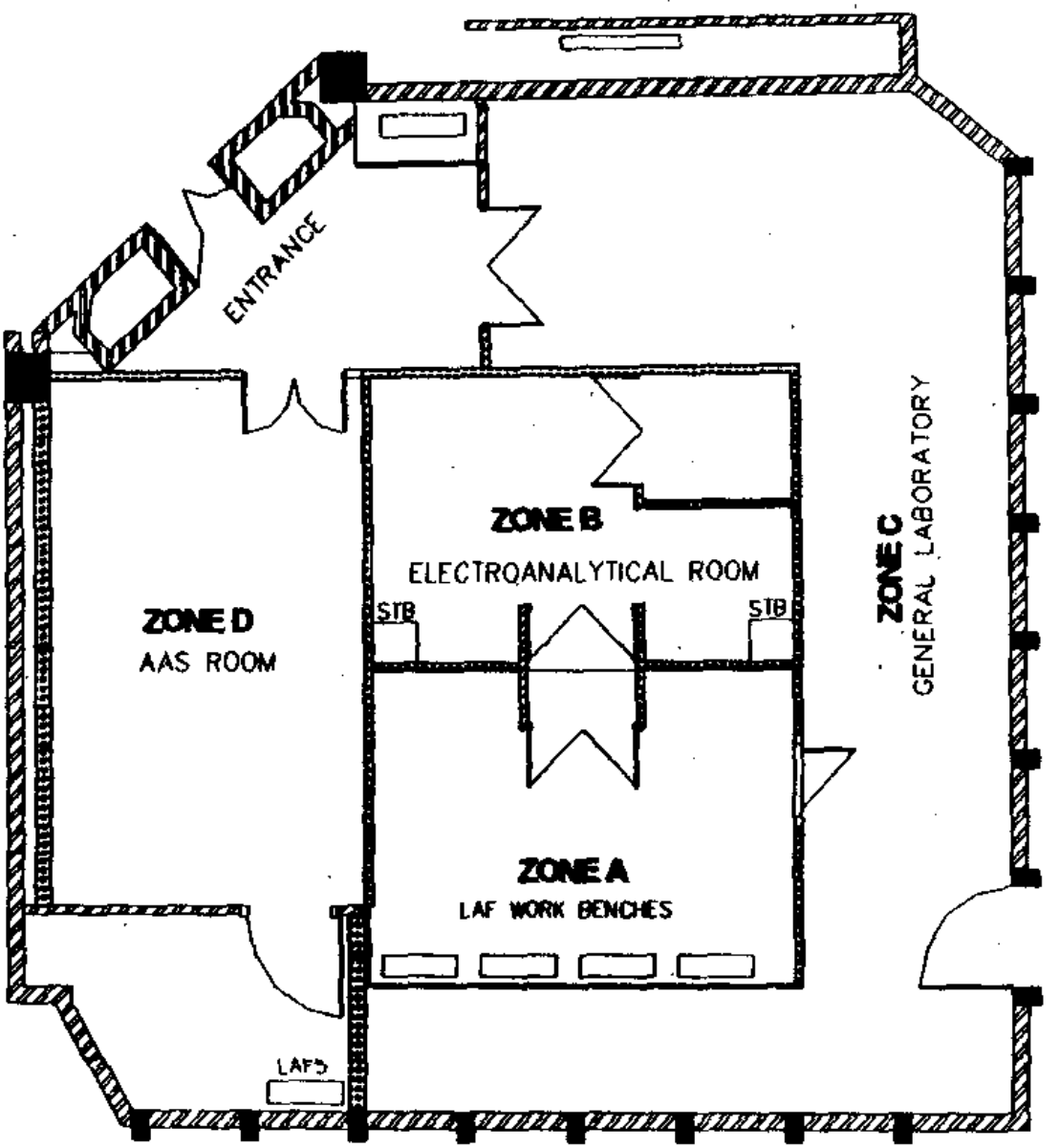

FIG. 1 ULTRA TRACE ANALYTICAL FACILITY
ANALYTICAL CHEMISTRY DIVISION

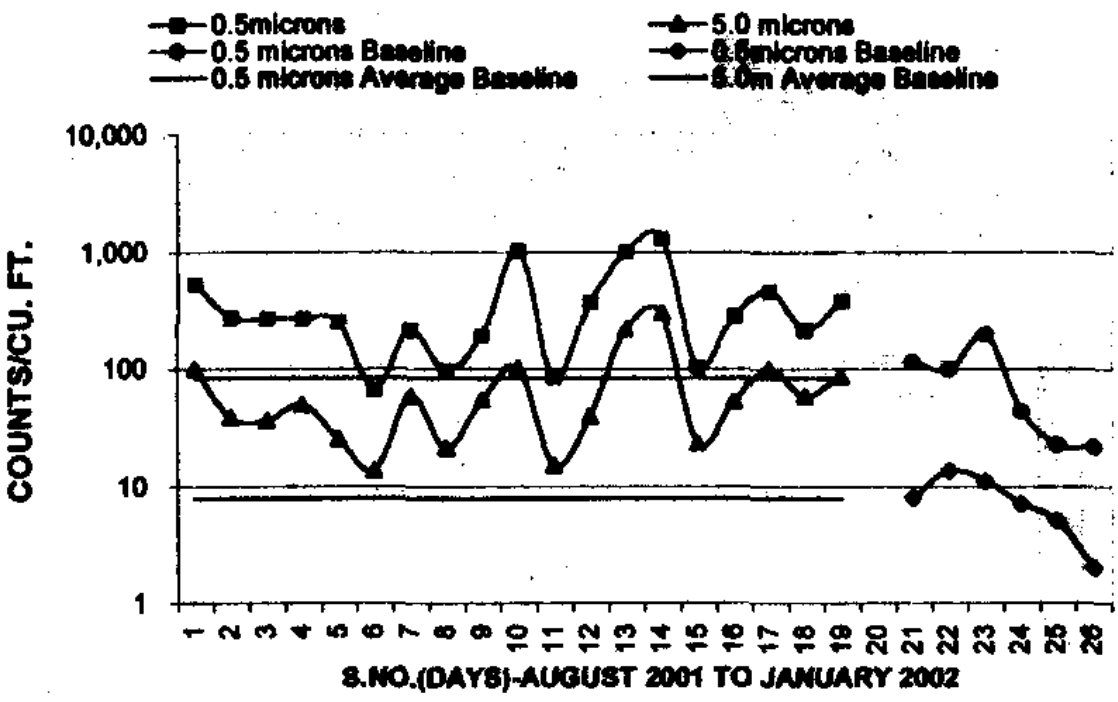

FIG. 2 PARTICLE COUNTS IN LAF ROOM-TYPICAL DATA

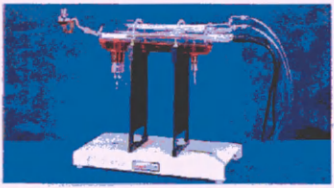

FIG. 4 SUB BOILING DISTILLATION APPARATUS

FIG. 5 POLYTHENE TANKS USED FOR ACID BATHS

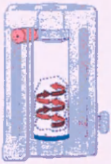

FIG. 6 HIGH PRESSURE VESSEL

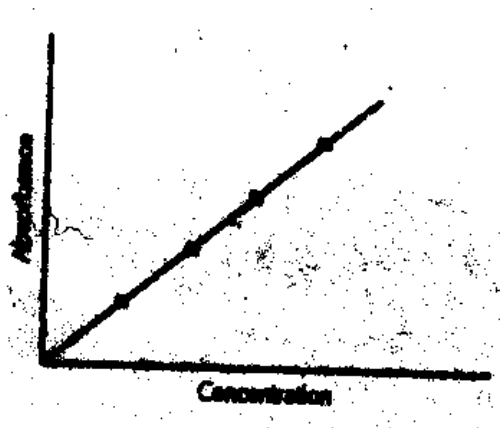

FIG 7 CALIBRATION GRAPH IN GFAAS

FIG. 8 GRAPHITE CUVETTE

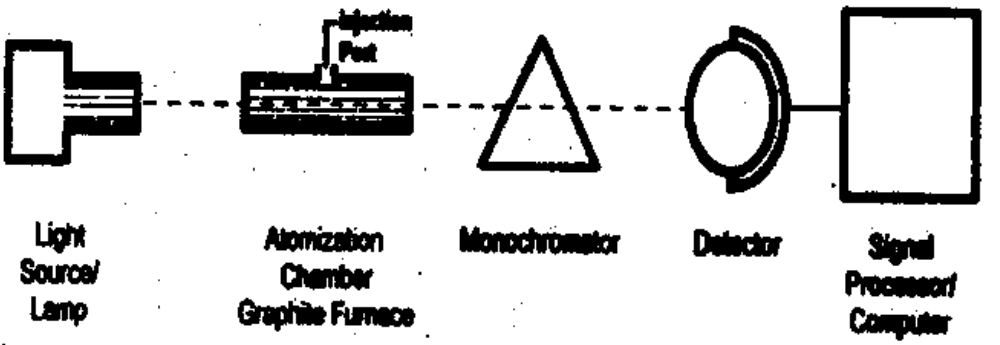

FIG. 9 DIAGRAM OF GFAAS COMPONENTS

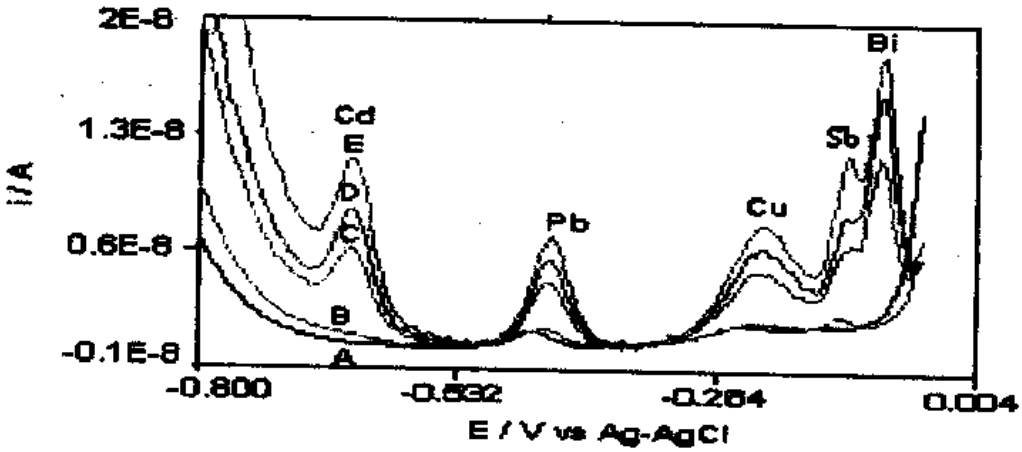

Figure 10 Typical Voltammetric scan of high purity arsenic by DPASV
A : Blank B : Sample (4 mg/ml) C,D,E : Standard Additions
Cd : 0.4,0.8, 1.2 ng/ml : Cu : 1,2,3 ng/ml : Pb. Sb., Bi : 2,4,6 ng/ml

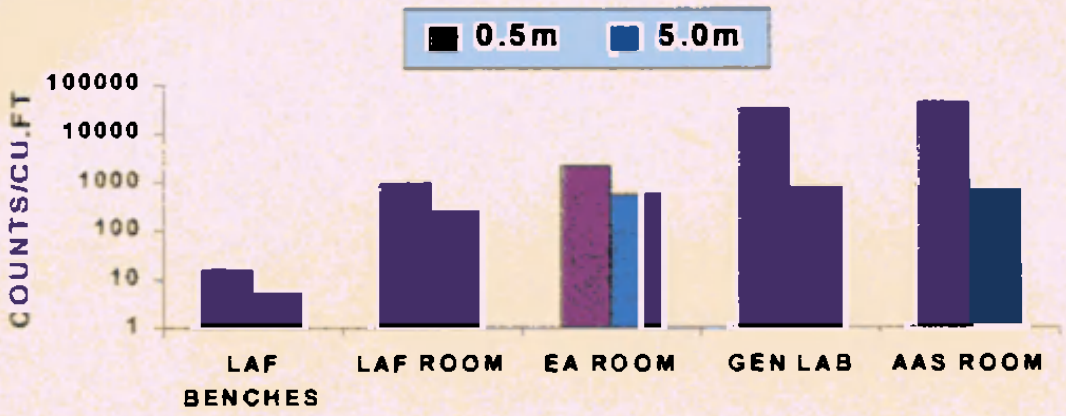

FIG. 3 PARTICLE COUNTS DISTRIBUTION AT UTAF