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Clean laboratories and clean rooms for analysis of radionuclides and trace elements

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FOREWORD

The need for reliable and accurate measurements of elements at trace and ultra trace concentrations is now well established and has been addressed in numerous textbooks and in a number of publications of the International Atomic Energy Agency. Less well known might be the fact that the reliable analysis of samples is often found to be hampered by insufficient control of the analytical blank. As methods become more and more sensitive, the target elements of major interest in natural matrices become less abundant (e.g. platinum metals in the environment, ultra traces in biomedical research, or semiconductor analysis) and there is increasing demand for speciation analysis, where only fractions of the total trace element content are targeted. Because of the need for stringent control of contamination during sample handling, preparation, separation and enrichment as well as during the determination process, establishment of a clean laboratory environment is mandatory.

Particulate contamination in the laboratory air may be controlled by the use of high efficiency particulate (HEPA) filters, which were developed during World War II for the Manhattan Project and were used to provide containment of radioactive particulates within the laboratory. Today the same application of HEPA filters is used in radioactivity laboratories worldwide. The Class 100 specifications for measurement of particulate air quality apply for clean room facilities as laid down in the regulations, such as Federal Standard 209E or the ISO Guide 14644.

This publication summarizes the requirements of clean laboratory environments, for construction materials as well as for materials used during routine analysis, maintenance, and pitfalls in the analysis of radionuclides and elements at trace- and ultra trace levels. Included are papers contributed by experts from India, the Netherlands, the United States of America and the IAEA Laboratories, Seibersdorf.

The IAEA wishes to thank all the experts who helped in the preparation of this manuscript for their valuable contributions. The IAEA officers responsible for this publication were A.V.R. Reddy and M. Rossbach of the Division of Physical and Chemical Sciences.

EDITORIAL NOTE

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CONTENTS

CONTRIBUTED PAPERS

1. BACKGROUND

Reliable and accurate measurements of elements and radionuclides at trace and ultra trace concentrations are often needed in a variety of analytical activities related to environmental, nutritional, biological tissues and fluids, and high tech materials. Key components determining the quality of the analytical results include the selection of an appropriate analytical methodology, laboratory environment, reagents, samples and standards and, above all, the experience of the analyst and his approach to the problem. Rigid management of each parameter with proper quality control (QC) is of paramount importance for achieving overall reliable results. The accuracy with which an element or isotope can be measured at ultra trace levels is limited by systematic errors associated with all the listed parameters, e.g. the very process of sampling and sample preparation which carries the risk of changing the trace element composition of the sample. It is a fact that accurate analysis of a sample is linked to the control of the analytical blank. One of the main sources of contamination of samples is through particulate matter from the laboratory environment. The laboratory determination of elements at trace and ultra trace levels can be jeopardized by the introduction of particulate matter from the laboratory environment. Uncontrolled air usually contains inorganic and organic matter in the form of aerosols of natural and anthropogenic origin. The composition of air particulate matter differs widely depending on the geographical location, extent of industrialization, vehicular traffic, etc. The development of clean rooms/laboratories for controlled environment is the answer to meet the demands of the ultratrace and trace analysis. This includes providing clean room/laboratory facilities for the various steps in sample preparation, storing the samples if needed, purification of chemicals, and finally for carrying out analysis. A Class 100[1] laboratory is ideal though expensive. Often a judicious use of a Class 1000 or even Class 10000 facilities with a provision for Class 100 boxes, may meet the requirements of many analyses.

The state of the art in the development of clean room/laboratories probably reached a plateau around the mid 1980s. With the increasing demand for ultra-trace element determinations and quality assurance and quality control (QA/QC) in the studies of life sciences, environment and new materials the need for controlled laboratory environment is ever growing. Application of clean techniques to biomedical work has added many improvements in air control technologies. Clean laboratories are required for radionuclide analysis in safeguards and environmental samples and also for trace element measurement where nuclear and related analytical techniques like neutron activation analysis (NAA) and X ray fluorescence (XRF) are involved. Spectrometric techniques like ICP-AES, ICP-MS and AMS require utmost care in sample treatment (digestion, separation, dilution, etc.) as these analytical techniques accept only dissolved samples. It is essential to provide harmonized guidelines to the users of clean facilities so that appropriate QA/QC procedures may be implemented.

This report intends to include salient features of the concept and design for clean rooms/laboratories, the materials needed for clean laboratories, describe current methodologies and practices for planning the installation of a clean environment, protocols for maximizing the benefit-to-cost ratio and for achieving QA/QC. The report aims to provide information to the general researcher and user about the clean facilities. Special emphasis is given to the analysis of radionuclides, and measurement of trace, minor and major elements using nuclear and related analytical techniques such as NAA and XRF. A section on literature is provided for ready reference and details.

1.1. Clean facility options

Selection of components for a clean facility can be taken from a large array of equipment options. A series of options from the simple (less expensive) to the complex (more expensive and

[1] Cleanliness is defined based on the number of particles of a defined diameter per unit volume. According to the US Federal Standard 209E[1], a Class 100 clean room must have no more than 100 particles of diameter 0.5 mm per cubic foot of air. Corresponding SI classification and ISO definitions are compared with Federal Standard 209E and given in Section 2.2.

comprehensive) are described in Section 3. The choice of options will be dictated by the operation processes, financial resources and desired life cycle for the facilities. The simplest system can be installed in existing laboratories to provide significant improvements in processing blanks. However, this environment will significantly shorten the life cycle and degrade the ultimate performance level of the components. An attempt is made to provide with guidance concerning both the advantages and limitations of each example discussed. A few simple explanations are also provided to differentiate between clean facilities, clean rooms and clean laboratories. An understanding of these differences is considered essential for developing operational protocols, maintenance protocols and QC protocols.

1.2. Classification of cleanliness

The level of cleanliness in a laboratory is defined by a maximum permissible number of particles of a particular size, per unit volume of air. Several standards of classification are currently in use worldwide, including the US Federal Standard 209E, Guidelines developed by the Verein Deutscher Ingenieure (VDI-Richtlinien) and the new ISO 14644 series developed by the International Organization for Standardization in Switzerland. Table I shows airborne particulate cleanliness classes and maximum number of permissible particles for defined particle sizes according to the new ISO 14644 guidelines.

The contamination control industry currently uses a government specification known as Federal Standard 209E to provide a qualified and standardized method for measuring how clean the air is in a clean room. Six classes have been established to designate clean room cleanliness, ranging from Class 100,000 to Class 1. The class number refers to the maximum number of particles bigger than one-half of a micron that is allowed in one cubic foot of clean room air. A Class 100 clean room, for example, would not contain more than 100 particles bigger than half a micron in a cubic foot of air.

This US Class 100 is similar to the new ISO Class 5 classification. Airborne particulate cleanliness, according to ISO 14644, is designated by a classification number, N. The maximum permissible concentration of particles, C_n, for each considered particle size, D, is determined from the equation:

$$C_n = 10^N (0.1/D)^{2.08} \quad (1)$$

where units of C_n, in [particles/m^3 of air].

1.3. Calculation of particulate concentration

Clean laboratories have been extensively used to reduce analytical blanks in recent years. It is important to understand that this reduction in analytical blank is accomplished by removal of airborne particles from the area where samples are processed. ”Clean” facilities will not reduce blanks which have their origin in the gas phase.

TABLE I. SELECTED AIRBORNE PARTICULATE CLEANLINESS CLASSES FOR CLEAN ROOMS AND CLEAN ZONES

ISO classification number (N)	Maximum concentration limits (particles/m^3 of air) for particles equal to and larger than the considered sizes shown below					
	0.1 μm	0.2 μm	0.3μm	0.5 μm	1 μm	5 μm
ISO Class 4	10000	2370	1020	352	83	
ISO Class 5	100000	23700	10200	3520	832	29
ISO Class 6	1000000	237000	102000	35200	8320	293
ISO Class 7				352000	83200	2930
ISO Class 8				3520000	832000	29300

(Source: ISO 14644-1)

Clean facilities are classified according to the number of particles of a specific size range per unit volume of air as described in section 2.2. The most commonly used cleanliness class for blank reduction is the ISO class 5 (Equivalent to class M3.5 and the US Federal Standard Class 100). This cleanliness class permits 3520 particles of 0.5 μm per cubic meter. Additionally 10,200 particles of 0.3 μm diameter are permitted per cubic meter for this cleanliness class. The number of atoms or molecules (A) of substance contained in a single particle can be calculated by equation 2.

$$A = (4/3)\,\Pi\,(r \times 10^{-4})^3 \rho A^0/M \qquad (2)$$

where

r is the radius of the particle in microns,
ρ is the density of the substance in gm/cm^3,
M is the molecular weight of the substance,
A^0 is Avogadro's number.

An example is worked out here.

A particle of 0.5 μm diameter and a density of 2 will have a mass of $(4/3)(.00025)^3 \times 2$ or 1.211×10^{12}g. If the molecular weight of the substance is 100, the particle will contain: 7.29×10^{10} molecules.

The total number of particles (N) of a specific size that can impact the work surface of a clean bench can be calculated by equation 3.

$$N=AV \qquad (3)$$

where A is equal to the number of particles of a specific size range per m^3 permitted by the cleanliness class of the bench and V is the volume of clean air flowing over the work surface per unit time.

Another example is described below.

For a class ISO 5 clean bench with a laminar flow of 31 m per minute and a work surface area of 1 m^2, the number of 0.5 μm particles impacting the work per minute will be $31 \times 1 \times 3530$ or 1.943×10^5 particles. The mass of these particles will be 1.331×10^{-6} grams of substance. The surface area of the work surface is 10^4 cm^2. The mass impacting the work surface is therefore 1.321×10^{-11} g per cm^2 per minute. For an exposure cross-section of 10 cm^2 and an exposure time of 10 minutes, the sample could be exposed to 1.321×10^{-9} grams of substance just from the 0.5 μm particles permitted by the ISO Class 5 clean bench. Thus it becomes important to consider methods to control the composition of particles and trajectory of particles as well as the total number of particles. If the elemental composition of the ambient air particles can be determined, one can then calculate the elemental blank to be expected due to particles from the laminar airflow.

1.4. Origin and control of particulate matter

Clean facilities control ambient particulate matter by two mechanisms. The first mechanism is by providing the laminar airflow. Laminar flow prevails at an air velocity of about 32 linear meters per minute. At this velocity, air will sweep particles from the work area with high efficiency as long as the airflow remains laminar. If obstructions cause turbulence, the efficiency of particle removal will be severely reduced. The second method of particle control in clean rooms is by maintenance of the pressure differentials between rooms. The cleanest zone is maintained at a higher pressure than the less clean zone. A pressure differential of 10 pascals is considered adequate to prevent diffusion of particles between clean zones.

Particles that are external to clean room facilities have a variety of origins in nature including volcanic releases and windblown dust. Most particles originate from such sources and can be efficiently controlled by filtration systems.

Particles can also originate from the facility itself. By careful selection of construction materials both quantity and composition of particles can be controlled. Choosing the right materials is one essential step in providing the best environment for the highly specialized analytical work carried out in a clean room facility.

The third source of particles is one that comes from the sample processed within the facility. In addition to controlling the number of particles by managing the air quality, number and type of particles are also controlled to some extent by sample analysis protocols, personnel gowning protocols and material quantity limitations for facility entrance.

To calculate the number of particles that can contribute to an analytical blank, it is necessary to know the exposure cross-section area of an open sample and the exposure time during which a sample is exposed to the clean airflow. These parameters, which are developed for each process, are beyond the scope of this document. However, it is clear that a reduction of either of these parameters by a factor of 2 will result in an approximate factor of 2 in that portion of analytical blank that is due to particulate contamination. Reagent contamination and process ware contamination will not be reduced.

2. TYPES OF CLEAN FACILITIES

The required class of clean environment depends on the type of analysis to be carried out. There is no general rule on the requirement of the cleanliness class. However, one can decide about an optimum cleanliness depending on whether a system is an open or closed one. There are various types of clean facilities from simple inflatable glove bag to a clean laboratory. In this section principle of operation, basic units, performance capabilities and limitations and operational limitations of a few systems are described. They are inflatable glove bags, clean bench, clean glove boxes, clean exhaust fume hood, recirculating clean bench and the clean laboratory concept. Although clean environment may reduce the blank levels in most of the analytical problems, it may be noted that in some cases like dealing with air-borne materials, clean environment alone is not the solution.

2.1. Inflatable glove bags[2]

The inflatable glove bag is not a primary particle control system. Instead, it functions only as a barrier to prevent particles of sample origin from contaminating the clean work area and prevents particles of non-sample origin from contaminating critical samples. The glove bag is a flexible plastic film bag with full length gloves moulded into one surface of the bag. A long, narrow tube of the same material extends from the rear surface of the bag. This tube provides an entry point for inserting samples and/or processing equipment into the bag. After insertion of all items required for the process the end of the tube may be sealed either with mechanical clamps or with a heat-sealing device. If the insertion is done in a laminar flow of clean air then the exposure window can be minimized when performing which require lengthy exposure of the sample. A smaller tube is provided which permits inflation of the glove bag with an inert gas selected. The system remains pressurized during all manipulations required by the process being performed. When all operations have been performed the

[2] Inflatable glove bags can be purchased in a variety of sizes from companies such as Cole Parmer or other local suppliers. An 11” × 20” × 20” glove bag costs approximately US $20.

products are sealed in pressure sealed plastic bags and placed in the entry tube. A double heat seal is applied between the bag and the items to be removed. A cut is made between the two heat seals and the products removed for transport to the area where the product is to be further processed. The sealed bag may then be depressurized and folded for storage if the contents have a future use, or discarded otherwise.

Glove bag, sealing clamps, heat sealer, inflation gas supply and pressure regulator are the components of inflatable glove bag.

Inflatable glove bags may be used to handle, inspect and subdivide samples of relatively high concentrations of materials that are incompatible with other operations within a clean zone. The only function provided by the bag is as a barrier to prevent transport of material from one processing area to another. The integrity of this barrier is a function of thickness and strength of the material used to fabricate the bag.

The use of inflatable glove bag will result in contamination of all inner surfaces with the material being processed and by particles from the non-clean facility. Thus these are not re-usable.

These bags should not be used for processing samples containing hazardous quantities of penetrating radiation, as prescribed by the local regulatory bodies. Thus these can be used for processing permitted quantities of alpha, beta and gamma activity.

2.2. Simple clean bench

A clean air bench provides a curtain of clean air within which simple manipulations such as ion exchange column operations, precipitation, sample transfers and equipment cleaning may be performed in a particle protected environment. The cleanliness class of the clean bench air is limited by the efficiency of the installed filter module, e.g. class of high efficiency particulate air (HEPA) filter.

The primary components required for installation of a clean bench include the filtered air recirculating module, a clean non-particle generating work surface (table or bench) and an isolation curtain to separate the operator from the clean air environment. The air recirculating unit may be purchased from a large number of manufacturers worldwide[3]. Materials of construction include steel, aluminium and plastic. Automatic flow controllers, epoxy coated impeller fans and low noise baffels are useful mechanical options. The device may be hung from the ceiling of the room where required.

The isolation curtain can be a simple 0.3–0.5 mm thick piece of clear plastic sheeting. High optical clarity and good flexibility are desirable features. An antistatic coating of the material is also desirable though not essential. This curtain should be installed from the air recirculating module to within 15–20 cm of the work surface. The clean, non-particle working surface is easily achieved by the use of a self adhesive Teflon film such as Bytac. This product usually consists of vinyl layer (necessary to be adhesive) and a Teflon overlay which are thermally bonded. The use of this film on the surface of the workbench and other exposed surfaces within the clean airflow provides a particle free surface, which may be installed in a few minutes.

The simple clean air bench provides a curtain of clean air over the entire work surface. When items are introduced into this clean air many of the particles on the item are dislodged from the surface and transported into the room air. Laminar flow prevents these particles and other room

[3] Modular units with a size of approximately 6 m × 12 m are most commonly used and can be purchased at a cost of approximately USD 800 to 900.

particulate matter from entering the clean air curtain. By using clean swipes (wet or dry), surfaces of the items in the clean air can be cleaned of nearly all particulate matter. When working in the clean zone the operator must wear laboratory coat and gloves that do not generate particles. The junction between lab coat and gloves must be sealed in a manner that contains the particles which are constantly generated by the skin of the operator. When operated with appropriate attention to clothing, gloving and cleaning techniques Class 100 conditions can be maintained for a wide variety of operations.

The principal performance limitations of a simple clean air bench are, as follows:

- A significant waiting period must accompany each introduction of a new item into the clean air. This permits the airflow to remove and flush the particles that are on the surface of the item.

- If there are any hazardous particles on the surface of the items being processed, they will be redistributed to the air within the room.

- Because no preconditioning of the air entering the filters is possible, life of both pre-filters and HEPA filters is significantly shorter than can be expected when the same equipment is operated as part of a recirculating clean room.

- The clean bench is the only line of protection of items in the room. Therefore, cleanliness of the room will directly affect the ultimate capabilities of the bench.

The clean bench can only be utilized for processes that do not emit hazardous components. Acid fuming, volatile organic extractions and other hazardous operations are not practical in these facilities.

2.3. Clean glove boxes

The clean glove box, when properly designed, also provides a flow of laminar air to a clean workplace. The principal difference between a clean air bench and a clean air box is that all particles are contained within the system and are not mixed with room air. Clean air glove box can be operated in more hostile environments by personnel who do not wear special garments.

A clean glove box should have, at a minimum, an air re-circulating system, a HEPA filter of special cleanliness class, a containment box, a perforated secondary floor to assure laminar flow over the work surface, an air lock for introduction of items to be processed into the glove box and glove ports fitted with sealed gloves of non-particle generating material. A cross-sectional view of the glove box is given in Fig. 1. Desirable accessories include illumination system, separate filtered supply to air lock, electrical distribution system within containment box, waste removal port, attachment port for particle monitor and a flushing system to periodically refresh the internal atmosphere.

A clean glove box will supply Class 100 laminar flow air over a clean work surface. All the operations that can be conducted on a clean air workbench can also be conducted in a clean air glove box. In addition, the enclosed system permits handling of materials, which might be considered too hazardous for clean bench operations. An additional advantage with the clean air glove box is that the operator may work without special clothing. Also, toxic materials can be handled safely.

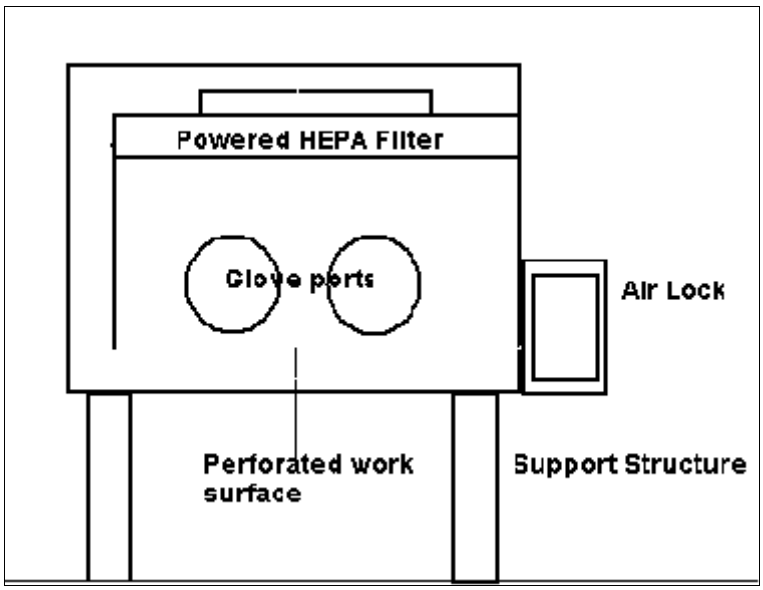

FIG. 1. Glove box.

The operational flexibility of a glove box is much less than that of a clean bench. All equipment, reagents and processing items must be introduced through the air lock. Manipulations are more difficult due to the necessity of working through gloves. This problem can be minimized by operating at a slightly negative pressure to simplify the glove entry process. However, this possesses a significant cost penalty for the added complexity.

The clean air glove box is unsuitable for handling flammable liquids and strong acids. Special air purification systems may be added but are usually so expensive that the use of clean air fume hood would be cost competitive and provide greater flexibility.

2.4. Recirculating clean bench

The recirculating clean bench also provides a flow of Class 100 air over a work surface. There are two main differences between the recirculating bench and simple bench. First, the work surface of the recirculating clean bench is perforated to permit the laminar airflow to pass through the work surface into a plenum where it is redirected to the input of the air recirculating unit. Second, an airtight plenum connects the lower exhaust chamber with the air intake chamber. This effectively permits continuous cleaning of the laminar airflow. Two types of benches are available. In one of them the airflow is horizontal from the back to the front of the bench. The second type of bench utilizes a vertical airflow from the top to bottom in the system. The first design is not efficient for most of the operations involving processing of low-level samples.

The basic components of the recirculating clean bench include a powered fan and HEPA filter, a cabinet enclosed on three sides to confine the airflow, a perforated work surface, an air collection plenum beneath, a return air plenum which conducts airflow from the collection plenum back to the intake of the air recirculation unit, a movable clear plastic curtain and a support structure for the bench system. A recirculating clean bench is depicted in Fig. 2.

These units are available in steel**,** aluminium, fibreglass vacuum formed plastic and welded plastic forms[4]. Accessories can include water taps, gas taps, electrical connections, special integrated support structures and sealed internal light fixtures.

[4] Cost for such devices can range from US $2,000 to $15,000 depending upon sizes and accessories.

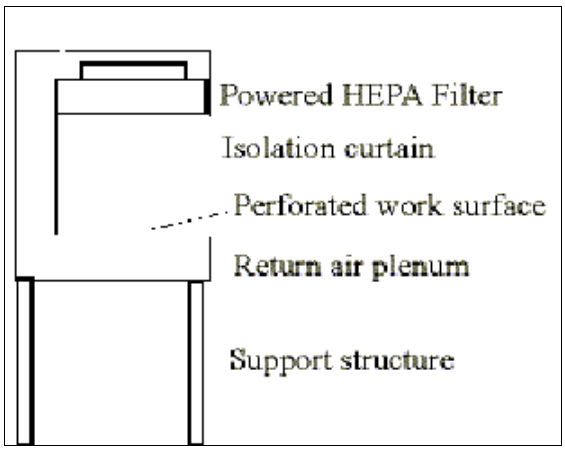

FIG. 2. Recirculating clean bench.

These units are capable of supplying air cleanliness of Class 10 quality. Because the access openings can be completely closed the contents can be well protected in the event of a power failure. This is not true of the simple clean bench. Access to material placed in the clean zone is excellent and good isolation of different processes can be accomplished by installation of vertical dividers. Operators must use appropriate gowning and glove protocols if good cleanliness is to be maintained. Because the air is recirculated through the clean bench the filter life is significantly longer than the units that use room air while operating in a non-clean room.

These units are primarily limited by the environment in which they are installed. If the room is dirty the personnel entry protocols become more critical. The units do not provide humidity and/or temperature control. These units are not to be used for processing toxic materials and flammable liquids in significant quantities. Additionally, they may not be used for fuming operation.

2.5. Clean exhausted hoods

The clean air hood provides a source of laminar air to a perforated work surface in much the same way as the recirculating clean bench. There are two main differences. First, the air recirculating unit removes air from the room and circulates it through the HEPA filter in a single pass cycle. Second, the air from the collection plenum is removed through an externally mounted duct that is connected either to an acid scrubber system or to an approved laboratory exhaust system capable of handling corrosive fumes. The system requires a means to balance the input capabilities of the HEPA air recirculating system and the exhaust capabilities of the exhaust system. Materials of construction must be compatible with the corrosive nature of the fumes being processed. Special units of aluminium or steel coated with Teflon are available for use with perchloric acid. These units require a separate exhaust system equipped with appropriate scrubbers.

All the basic components of the recirculating clean bench are required except the return air plenum. In addition an external exhaust duct and fan system is required. Additional supply air is required in the room where the unit is installed to make up for the air exhausted by the hood. This is approximately 1280 m^3/h for a 0.6m × 1.2 m unit. Materials of construction are usually PVC, polyethylene, Teflon coated aluminium, or fibreglass-epoxy laminates. It is recommended that multiple 0.6 × 1.2 m units be considered rather than single larger units[5]. Flexibility of operations is the primary reason for this recommendation. A cross-sectional view of a clean exhaust fume hood is given in Fig. 3.

[5] The cost of the fume hood with required accessories may range from US $2,500 to $15,000 depending on the size. Cover units will be decided based on the requirements of local regulatory bodies; they could be very expensive.

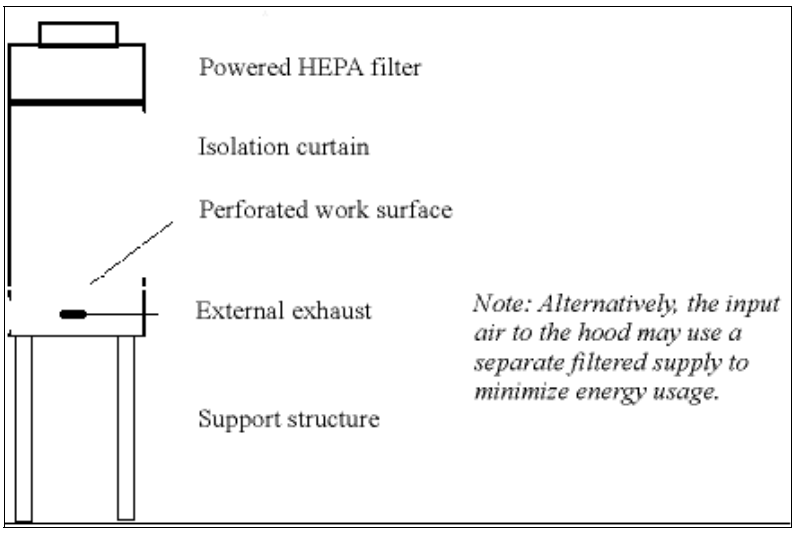

FIG. 3. Clean fume hood.

The modern clean air fume hood is capable of handling any fuming operation conducted in a conventional fume hood. However, the quantities of the acid processed are significantly smaller. The use of perchloric acid requires special material choices, exhaust air handling and HEPA filter media. All are very expensive. The total heat load and fume load within the fume hood must be matched with the exhaust capabilities of the laminar airflow input system. Exceeding these limits can result in both losses of laminar flow and uncontrolled fume releases into the room. If this occurs, particle control is lost.

These fume hoods cannot be used for simultaneous acid fuming and flammable liquid evaporation. Separate exhaust systems should be supplied for hoods used for evaporation of flammable liquids. This is a safety limitation that is common to all fume hoods.

In general terms a single 0.6 m × 1.2 m exhausted fume hood should be limited to contain no more than three 25 cm × 25 cm hot plates. Larger units will interrupt the laminar flow of the air and degrade system cleanliness.

2.6. Air shower enhancement

When clean benches and fume hoods must be operated in a non-clean environment, use of air showers enhances the overall performance of these components. The air shower is placed in front of the bench or hood. This configuration, when implemented with strict clothing protocols, permits short term cleanliness performance which is nearly equivalent to the performance of the same components in a clean room**.** The air enhancement shower serves the same purpose as room pressurization in a clean room. It minimizes the back streaming of particles into the clean work area.

The components required to install an air shower are only a powered HEPA filter unit and a plastic isolation container. Fig. 4 is a conceptual representation of an air shower to enhance performance of a simple clean bench.

When installed in a non-clean room, air shower provides a curtain of clean air which will flush particles from the exterior surfaces of the operator and minimize re-suspension of these particles in the clean work area. The quality of air within the air shower will be nearly equal to the cleanliness quality of the HEPA filter installed in the powered HEPA unit. This will enhance the air quality achievable in the clean bench or clean fume hood by minimizing the quantity of particles available to diffuse into these work zones.

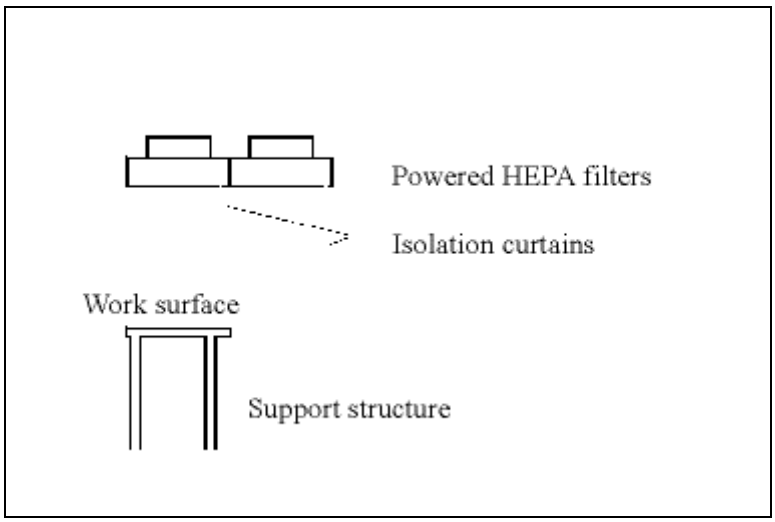

FIG. 4. Air shower enhancement.

As with all filtration systems, operation of the air shower within a non-clean room will degrade the service life of the HEPA filter as compared to the operation of the same unit with in a clean environment. The time required to flush particles from the exposed surfaces of the operator is about 10 minutes. Therefore, the effectiveness of this approach is greater if a protocol requiring an appropriate waiting time is always followed. For this concept, the minimum acceptable clean room attire will be hair covering, laboratory coat and clean gloves with the junction between lab coat sleeves and gloves sealed either by elastic bands or tapes.

2.7. Recirculating clean room

A clean room makes use of some or all of the devices described up to this point. In addition, a clean room uses a dedicated air supply system. This air supply system includes air dehumidifiers, air pre-filter systems, air pressure control systems, air heaters and coolers, air humidifier controls and fire detection sensors to precisely control the air quality within the room. This preconditioned air is supplied to the room via pressurized ducts or via a pressurized plenum above the sealed ceiling of the clean room. This conditioned air is then introduced into the clean room envelop through HEPA filters. These can be either passive terminal HEPA filters or connected with individual pressure controlled ducts or individually powered HEPA filters. The total volume of the air introduced into the clean room envelope is calculated to pressurize the rooms to the specified pressure and replace all leakage air from the room.

The capability to provide preconditioned particle controlled air to an entire room is the primary distinction between a clean room and a room which only contains a clean work station such as those discussed in Sections 3.1 to 3.6. An additional requisite for a clean room is the provision of a dedicated, particle controlled area for replacing or covering street clothing with clean clothing that must be worn in the clean room. This area may provide only for donning this clothing over the street clothing, or facilities may be provided for a complete change of clothing. Clear and well thought-out protocols for clean room entry are absolutely mandatory if the integrity of the clean room is to be maintained.

The air circulation system may be designed to permit continuous recirculation of room air through the HEPA filters. This recirculation capability provides two benefits. First, the air is continuously re-cleaned. Second, quantity of makeup air required is reduced in direct proportion to the percentage of air that is recirculated. This minimizes heating, cooling, humidification and air volume requirements. The quantity of air that must be circulated through the room is a function of the cleanliness class of the room. The higher the room cleanliness class is, the higher the volume of

recirculated air. For blank reduction operations the use of Class M2.5 is only necessary in work areas where sample processing is carried out. General room cleanliness requirements are usually Class M4.5 or Class M5.5**,** depending upon the processes to be performed. Gowning areas should be at least M5.5 **c**lass.

A clean room may make use of all of the system components discussed in the previous paragraphs.[6] There are additional systems that must be provided for a clean room. These are: rough air intake filter system, dehumidifier system, air circulation fan system, 85% filter system, air transport ducts, heating coils, cooling coils, hot water supply, cold water supply, humidifier system, heat, pressure and humidity sensors, control processor, exhaust air ducts, fume hood exhaust (if required), recirculation ducts and gowning room. Fig. 5 shows a conceptual diagram of one type of clean room with the various air paths, and a typical floor plan showing the orientation of the clothing change area is given in Fig. 6.

The primary limitation of a clean room as compared to a clean laboratory is in the flexibility of the system to accept a variety of processes. Because a specified cleanliness class must be a design criterion, all processes must be conducted by protocols designed to maintain such cleanliness level. This will cause extra effort for operations that do not require such strict protocols and has to use special accessory systems when a more restrictive cleanliness class is needed.

The principal operational limitation for a clean room as compared to a clean laboratory is to limit the types of processes that may be conducted simultaneously. This imposes a requirement for intelligent scheduling of the non-compatible processes which must be conducted sequentially. This in turn imposes longer time delays upon completion of the various processes. This limitation is usually unacceptable for continuous sample load of competing sample types but may be acceptable for many research programmes where such scheduling constraints are not a limitation. If only two or three competing processes are involved it may be economically practical to construct additional clean rooms. For larger numbers of processes a clean laboratory should be considered because increasing the capacity of the air supply and control systems is significantly less than the cost of constructing several stand-alone clean rooms.

2.8. Clean laboratory concepts

A clean laboratory utilizes all of the components which may be useful to minimize particle contamination. The defining difference between a “clean laboratory” and a “clean room” is that the clean laboratory combines clean components and clean rooms in a systematic way to provide support to a wide variety of processes. In this way the facility to support each process can be optimized to provide the appropriate cleanliness class, services, and space required. The net result is a laboratory, which is very flexible in scheduling the processes conducted and more efficient in the overall use of resources. The basic components of a clean laboratory and a clean room are not conceptually different. The clean laboratory simply incorporates several clean rooms, transport corridors, personnel support facilities (toilets, clothing change areas and data processing areas) and special purpose rooms (waste management, equipment cleaning, reagent preparation, and weighing rooms) into an integrated facility. The clean laboratory concept facilitates the use of different room pressures to further direct the flow of particles away from the most critical rooms towards rooms with less critical cleanliness requirements.

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[6] The cost of providing a clean room instead of utilizing the basic clean air components is quite high. The minimum incremental cost for a very simple clean room is estimated to be more than US $35,000. This cost will increase by as much as a factor of 10 for a complex room.

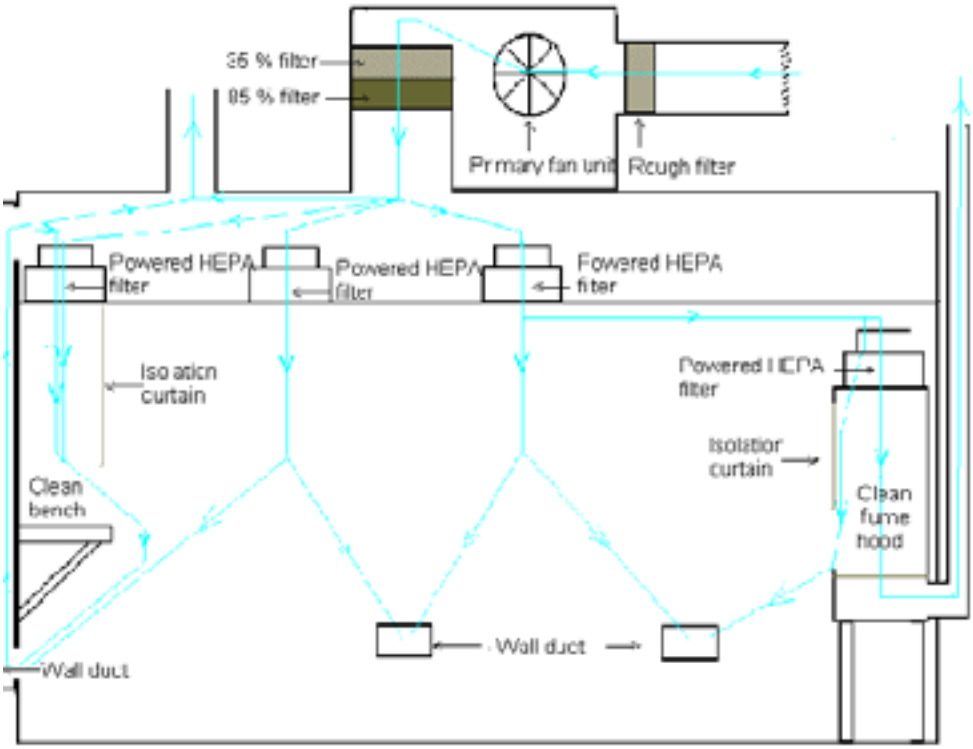

FIG. 5. Basic clean room.

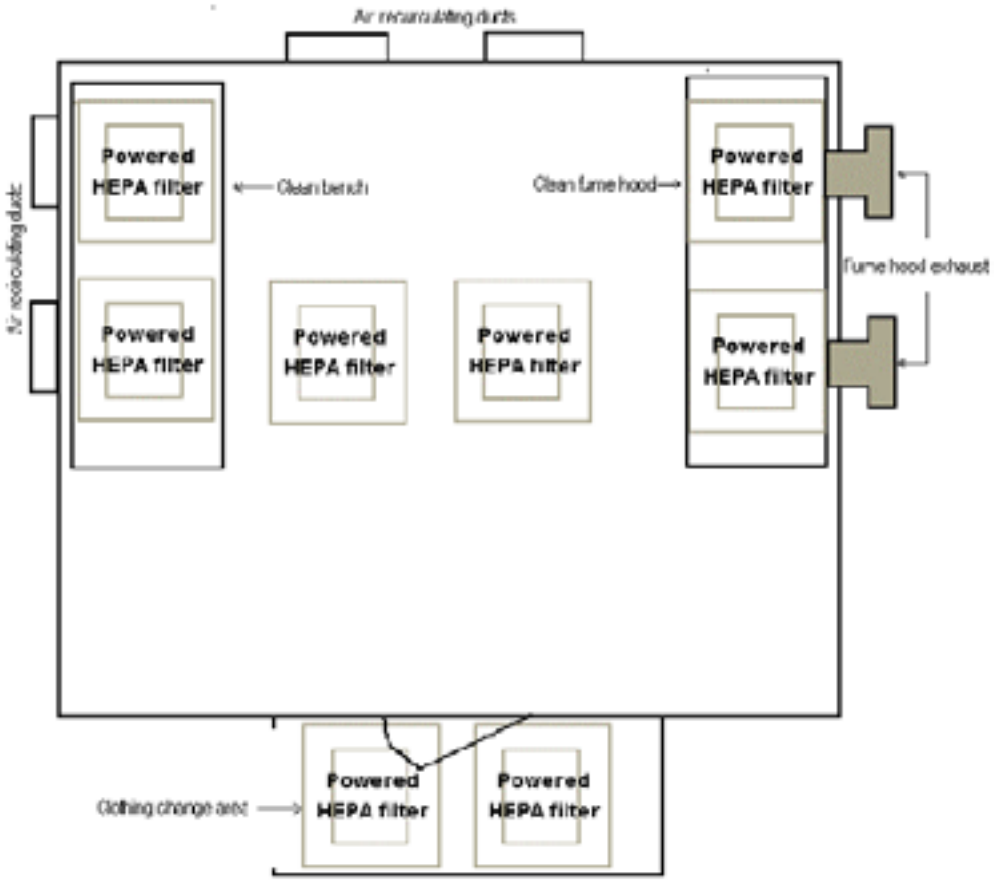

FIG. 6. Clean room floor plan.

The clean laboratory concept places greater emphasis on automated control systems. This in turn requires more accurate testing strategies to monitor the overall system performance. It also places a much greater burden on both facility maintenance and spare component inventory management. Unless these processes are well documented and managed the facility performance and operational availability will be both degraded. Fig. 7 is a floor plan presentation for the IAEA clean laboratory. This plan represents a relatively versatile laboratory capable of processing a rather wide variety of sample types.

Clean laboratory provides the capability to simultaneously support many processes which would require sequential scheduling in single clean room. The clean laboratory environment is optimized to achieve the desired cleanliness class more quickly and reliably than the facilities discussed thus far.

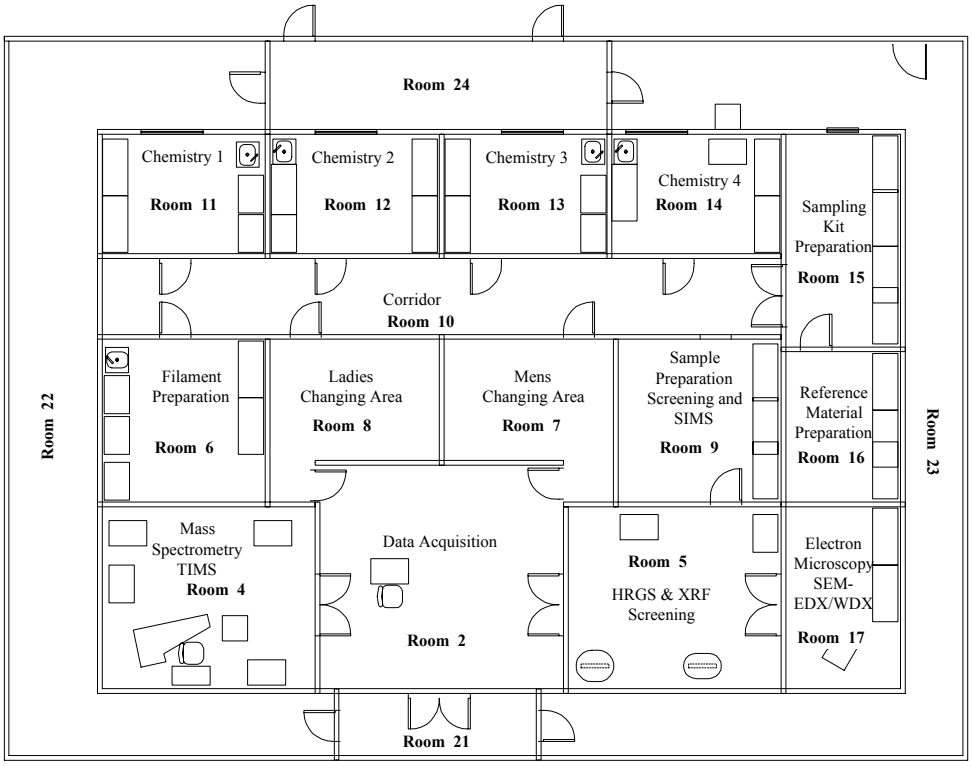

FIG. 7. Floor plan of the IAEA's clean laboratory, a unit of the Safeguards Analytical Laboratory.

Limitations of the clean laboratory are primarily the limits imposed by particle sizes present in the room of specified cleanliness class. The measured blanks are most often limited by reagent purity, material purity and instrumental sensitivity than by particle contamination. With the improvements in technology, sensitive limits in many instrumental analytical techniques are rapidly improving. This demands lowering of the blank levels by either improving cleanliness class or devising alternate cleaner sample processing strategies. Experience has shown that cleanliness can be more quickly improved as compared to improvements in material science and reagent purification. This pattern is likely to continue and even be more pronounced as lower detection limits become possible.

There are a few operational limitations for a properly designed clean laboratory beyond those imposed by the design limitations of the individual components. However, because of the system's complexity, more resources must be devoted to system maintenance. A more in-depth knowledge of the design parameters is required for the rapid and efficient trouble shooting of functional problems. To achieve the state of the art in clean lab environment is clearly not inexpensive. This is the price of having operations and facilities that is nearly state of the art.

3. CLEAN ROOM ATTIRE

All clean facilities require the use of clean room attire to minimize the release of particles generated by the operating personnel. Normal street clothing fabrics generate very large number of particles. In addition, particles adhering to street clothing may introduce contamination into the laboratory environment for trace element analysis. Use of hair cover, lab coat and gloves may be required for operation of simple clean room systems. For operation of clean rooms the minimum attire includes hair covering, lab coats, gloves and foot covers. A separate area is recommended for putting on the required attire. This area should be just outside the entrance door to the clean room. In a clean laboratory, a clothing change area is recommended. This area should provide separate cabinets for storage of street clothing and clean room attire. The two types of clothing should not be stored in a common cabinet.

3.1. Clothing

Clothing protocols can vary from the simple procedure of putting on a minimum clean room attire in a gowning area to complete removal of street clothing, showering with soap and water, and dressing in dedicated clothing of clean room garments stored in cabinets that are reserved specifically for such garments. Each step in strict attire protocols will improve the operator's cleanliness performance at the cost of some increase in complexity of entrance protocols.

3.2. Basic components

Selection of the type of clean room attire will depend primarily on the infrastructure in the proximity of the clean facility. Unless a certified clean room laundry is commercially available, use of disposable clean garments is mandatory if operational costs are to be minimized. Disposable clean room attire is available in a variety of fabrics such as TYVEC**.** This is a Teflon based fabric with very low particle content, good chemical resistance, good flexibility and low cost. Other fabrics may also be used with equal success. A comparison of chemical resistance, chemical composition, heat dissipation characteristics, local availability and cost is advised when making purchasing decisions.

Hair coverings may be loosely classified as partial and full head coverings. The bouffant cap is an example of a partial head cover. A hood type, which covers not only the hair but also the ears, neck and sides of the face is an example of a full head covering. For most sensitive work, full head covering is recommended. Partial head covering is more easily worn for a wide variety of hairstyles. However, because more skin is left exposed while using partial head covering, the cleanliness achievable is significantly less. For operations, which require close inspection of the process being conducted, addition of a full face covering that covers the nose and mouth is recommended. This face covering minimizes expulsion of particles through breathing by the operator.

Body coverings can be either partial as in the case of laboratory coats or full coverage as in the case of coveralls. Laboratory coats permit the release of particles from the operators' lower legs. For rooms where the clean areas are benches and hoods, these particles are usually excluded by laminar flow air and do not significantly contribute to analytical blanks. For areas to be maintained at Class 1000 or 10000, use of coveralls is usually a safer choice. All body covering garments should fit snugly at the neck and wrist to simplify sealing the interface between head, hand and body coverings.

Foot covering can be either dedicated shoes which are changed when entering or leaving a clean zone; shoe covers which cover only the shoes but not the interface between foot and ankle, or shoe covers which seal the junction between foot and leg. It is recommended for the most sensitive work. For areas where a particular tracking hazard exists, use of shoe covers over full coverage boots is often recommended. Foot covers are removed when exiting the zone.

3.3. Gloves

A very large selection of glove styles and materials of fabrication is available to the clean room practitioners. Each has a practical application. Gloves that require a lubricant such as talc should never be considered for clean room applications. Nitrile, vinyl and polyethylene gloves have useful applications within a clean facility. The length of the glove should be adequate to permit easy sealing of the junction between hand and forearm. In general gloves that fit the hand snugly are desirable for clean facility operations. However, use of thin film polyethylene gloves that have been fabricated by heat sealing two films together is a good option for multiple glove operation requiring rapid glove changes between operational steps. These thin gloves must be used only in addition to more robust gloves and never as a substitute for the primary protection.

4. MATERIALS

4.1. Materials for construction of the clean room

Sufficient care has to be taken in choosing material for construction of the clean bench and room. As the primary goal of the clean room is to control analytical blank, it is important to ensure that construction materials neither contain the analytes of interest and nor pose any contamination problem. The conventional galvanized iron ducting tends to get corroded fast and hence should not be used. If the analysis of aluminium is not envisaged, aluminium with a protective coat of epoxy paint could be used for making ductings, door, glass panels, etc., as it does not corrode fast and is fire retardant. But if the clean room is meant for ultra trace analysis of metals including aluminium, it is advisable to go for non-metallic construction, as the metallic parts will start corroding fast with the use of acids and these particles would cause a far more serious problem than the air particulates. But if metallic parts have to be used inevitably, then they could be covered properly with silicone cement or epoxy paint. Teflon coated metallic ducts are available at a higher cost. These will meet fire codes that are too strict for polymeric duct materials. The integrity of such protective coatings shall be periodically examined as the coating may give way with constant use of acids in the bench. The work stations can be made of wood, laminate covered wood, or rigid polymers such as polypropylene or PVC. The HEPA filters with aluminium separators are to be avoided for trace metal applications. Although these filters are acceptable in nuclear and isotopic labs, HEPA filters with plastic separators, or the variety without separators, is preferred in a clean environment. The filters and prefilters are to be mounted on a wooden or plastic frame. Care must be taken to ensure that the blower is made of plastic (fibreglass reinforced plastic or polypropylene) and the motor is epoxy painted. The ductings are to be constructed with HDPE or similar material. The water taps and other fixtures are to be made of PVC or HDPE and only deionized water supply is to be used in the taps. Lights should be covered in non-metallic sockets. Thus, all possible precautions must be taken to prevent generation of metallic particles from the construction materials.

4.2. Laboratory materials for ultra trace analysis

The pipettes and burettes made of borosilicate glass, which find extensive use in conventional laboratories are not suitable for use in trace analysis. Glass has been found to release trace elements (Na, Al and B) especially at both extremes of pH. High pH results in dissolution of the glass surface itself, whereas low pH leads to metal dissolution and desorption from the glass surface. In addition, the clean glass surface is highly reactive, primarily because of the presence of highly reactive Si-OH groups. These groups are acidic in nature and act as effective ion exchange material. Cationic analytes are, therefore, effectively scavenged from solution onto the glass surface [1], as shown below:

$$GlassSiOH + M^{+} \rightarrow GlassSiOM + H^{+} \quad (4)$$

A better but somewhat expensive option is vitreous silica prepared from the vapour phase hydrolysis of silicon tetrachloride or tetrafluoride. Vitreous silica is almost a pure material that can be rigorously cleaned further with acids to remove any surface contamination. It could be used at elevated temperatures as it is stable at high temperatures with nearly zero co-efficient of expansion. Alternatively, silica ware prepared from the rejected lots of silicon from the electronic industry could be used. Silica ware is supplied by several US companies; it is found to be suitable for ultra trace work and is cost effective.

4.3. Plastics

A wide variety of plastics have now been used in the construction of laboratory apparatus and careful selection of materials for trace analysis applications is necessary. The following factors to be considered for assessing the suitability for trace analysis: adsorption properties of polymers for the analytes and contamination arising from residual catalysts and additives used in the manufacture of these plastics [1].

For most applications, a low permeability material is desirable. Permeability is derived from the migration of molecules through microscopic voids between the polymer chains [1]. Absorption of material by polymers is in part due to its migration through the polymer structure. Discolouration of a plastic container is often associated with the migration of material into the polymer matrix. Movement of materials out of the container by permeation takes two main forms. Long term storage of solutions in unsuitable containers is known to result in loss of water through permeation. The resulting change in the concentration of the analyte may not be noticed unless precaution has been taken to weigh the sample before storage. The second complication may arise with analytes that are in equilibrium with some volatile species such as ammonium and sulphide salts. Although only a small proportion of ammonia or hydrogen sulphide may leak out of the container, wall equilibrium will be re-established generating these gaseous products to permeate out of the container [2]**.** Other considerations are of a physical nature, e.g. whether these containers can be dried at elevated temperatures. Considering all these factors, vitreous silica and PTFE ware are recommended for ultra trace analytical applications. HDPE ware is suitable for room temperature applications.

4.4. Purification of reagents

Water should be purified using a suitable purification system (Millipore or equivalent) to get a resistivity of 18 mega ohms cm found to be suitable for ultra trace work. Deionized water is fed to the purification system for further purification. The analytical reagent (AR) grade acids available in the market should be further purified by sub-boiling or isothermal distillation in vitreous silica or Teflon apparatus [3]**.** *A priori* validation of organic reagents is mandatory. The organic solvents and other reagents that may be used for preconcentration/separation work should be further purified using suitable methods [2]**.** Reagents purified by sub-boiling distillation from several sources are available with certificates of analysis. These are provided with trace element analyses for each lot of reagent**.**

5. OPERATIONAL PROTOCOLS

All operations carried out in clean facilities with controlled environments must be properly designed and executed to avoid contamination of the work areas, samples, and possibly the analyst. Clean laboratories require maintenance and certification procedures to ensure uninterrupted cleanliness of the working areas. Consequently, a set of protocols is required to run and maintain a clean room facility effectively. Instructions must ascertain continuous operational functionality and cleanliness of the facility. Operations carried out in the clean room facility need to be in accordance with the initial specifications to which the facility was built. Any change to this set of specifications should be reviewed by the facility's management and endorsed by appropriate clean room support, such as engineering, maintenance and staff. In addition, changes to the initial specifications and/or

uses of the facility should be recorded for future reference. Changes to the existing engineering should be made with caution, as they might render the facility or part of it unusable. An example could be the alteration of humidity control within a facility, negatively impacting on the life expectancy of instrumentation.

The level of operational protocols are determined largely by the *size of the clean facility*, which ranges from the most basic laminar flow/workbench in a conventional laboratory (Sections 3.2–3.4) to a state of the art clean laboratory comprising multiple dedicated rooms (Section 3.8). Operational protocols are also dictated by the extent of *automation and control of the facility environment*. The specific area of activity, i.e. *use of the facility,* will require specific adaptations reflected in the protocols. Research, training, service, and production facilities require operations with stringent protocols in certain areas such as safety. Instrumentation rooms will require different protocols than those used for digesting sample matrices and performing chemical separations. *Handling of toxic, flammable, and radioactive materials* in a facility require additional protocols. *Multi-room facilities* have the option of dedicating rooms for specific purposes. Single room facilities require very strict protocols as the clean area will be used for different analytical tasks, possibly interfering with each other. *Multi-user* facilities require a high level of managerial responsibility to the facility and establishing protocols for specific applications in collaboration with the end user.

5.1. Monitoring of facility parameters

Continuous monitoring of critical facility parameters is the best way to ensure that the level of cleanliness inside the facility/room complies with its specifications. Moreover, differential records can simplify detection of malfunctioning components, signaling that additional maintenance is required or that certain parts, especially filter media, have reached their effective life expectancy.

(a) Monitoring clean room parameters can be as simple as determining the *particle counts* that measure the number and size of particles present in the air. These measurements are compared to the expected number for a given class of cleanliness as provided in the US Standard 209E or the new ISO 14644-1. More sophisticated clean rooms have a central ventilation system for air handling in the facility, which is partly or fully automated. Ventilation systems must be calibrated, maintained, and periodically certified.

(b) *Temperature, humidity, and lighting* are critical parameters in the laboratory environment. Their measurement can be readily automated via sensors, or using inexpensive hand held thermometers and hygrometers. Adequate systems need to be installed, such as dehumidifiers, humidifiers, or entire air-conditioning systems. For example, rooms used to house expensive and sensitive instrumentation, require humidity control to prolong life expectancy and temperature control in order to ensure continuous quality in the measurement process.

(c) *Testing and accepting of a facility*: upon completion of the clean room or facility an acceptance test should be performed and documented for future reference. This test can be as simple as recording particle counts in a laminar flow work area. Complex facilities must be tested and validated by trained specialists following strict protocols according to the ISO standards ISO 14644-1.

(d) *Engineering change orders and work orders*: clean rooms run using sophisticated and complex engineering. Changing the airflow path by obstruction or purposely changing initial settings can adversely affect the facility's performance. Any change to the operational parameters of the facility should be recorded for future reference to ease debugging of the system should it become necessary.

(e) *Records*: any changes, enhancements, and maintenance need to be recorded. Records must be stored for as long as possible. Again, differential records will be helpful in identifying possible problems in the future.

5.2. Use of the facility

(a) *User training*: adequate education and training of people working in the laboratory environment are prerequisites for sustaining cleanliness in and smooth operation of the laboratory. Complexity of work in and maintenance of clean rooms requires that even highly educated personnel need from time to time additional training due to advances in technology, to sustain awareness towards the sensitive environment, and to the introduction of new measurement techniques. Informal education and training of personnel are important. Attendance of meetings and visits to other laboratories afford opportunities for exchange of ideas and specific information.

(b) *Entering clean rooms and facilities*: to maintain integrity of the clean laboratory environment it is mandatory that personnel undergo basic training on how to enter the laboratory. Visitors should not be permitted into the facility unless their purpose is to work on a project in the facility. In such cases visitors must complete basic and advanced training sessions appropriate for their involvement in the laboratory activities. Maintenance personnel would require special training. All the work to be performed in the laboratory must be discussed prior to entering the facility. Procedures for addressing unanticipated situations via reporting or emergency actions should also be covered. All personnel entering the facilities must be aware of most current procedures.

(c) *Clothing procedures*: clean room garments are required in clean rooms and laboratories. Clean workbench and laminar flow restrictions should be set at the discretion of the facility owner. Wearing special garments is mandatory and serves the main purpose of preventing the release of particles from normal clothing or skin into a clean room. Wearing clean room shoes and shoe covers, hair and beard covers and face masks are other preventive measures to reduce particulate matter given off by personnel. It is always a good protocol to encourage personnel to enter the laboratory "clean" in the morning, after having showered and wearing clean street clothes. Clothing procedures may include taking off the street clothes completely before gowning up. Double gowning and gloving procedures are appropriate if personnel have to move between different rooms in the facility to avoid cross-contamination. Activities to be performed in the clean lab should be planned well ahead of time to enable development of the clothing protocol.

(d) *Cleaning procedures:* thorough cleaning of laboratories is mandatory to maintain cleanliness of the laboratory environment. Different techniques and equipment are available for this purpose. Detergents and decontamination solutions should be used only in emergency. High purity water and water iso-propanol mixtures are recommended for wet cleaning of surfaces. Quality of wipers used should be in accordance with the classification of laboratory environments, i.e. Class 100 wipers should be used in areas where this level of cleanliness is required. Potential cleaning materials must be pre-analysed for their inherent content of contaminants. A cleaning schedule should be established, documented, tracked, and followed strictly.

(e) *Introducing equipment and supplies into a clean laboratory environment:* special care must be taken as to (1) the type of materials (e.g. equipment, supplies, and samples) permitted into a clean laboratory environment, and (2) establishing maximum permissible quantities for particular elements and nuclides. The means of introducing such materials is equally important. Vacuuming, wet-wiping, and double bagging techniques are commonly used to avoid contamination of the laboratory environment.

(f) *Practices for interlaboratory transfer:* transfer of materials in between laboratories and work of personnel on different projects in multiple areas should be considered carefully. Bagging and even double bagging techniques for materials, wearing multiple coveralls, and double gloving are common techniques.

(g) *Tracking and management system:* since even extremely small quantities of contaminants will compromise analyses and can possibly render parts of a facility unusable for certain activities, a tracking system must be established and implemented. Each item, from the basic disposable pipette to the spike solution, requires an entry into an appropriate database. The tracking system must comprise such information on vendors and lot number of a reagent.

(h) *Cross-referencing of materials:* it is imperative to cross-reference each reagent and solution prepared from it, as well as samples where certain equipment and reagents used are recorded. This system is the only means of tracing poor analytical performance and uniquely relating it to a particular batch of reagents.

6. EVALUATION OF PERFORMANCE

Quantitative measurements are always estimates of the value of the measure and involve some level of uncertainty. Measurement must be made so that the limits of uncertainty can be assigned within a stated probability. To achieve this, measurements must be made in such a way as to provide statistical predictability. This can be achieved by a well designed and consistently implemented QA programme that includes requirements for low and reproducible analytical blanks. QC should be an integral part of every clean facility.

The main objective of QC is to fit and maintain analytical processes including measurement process in a desired state of stability and reproducibility. In a clean laboratory environment special emphasis must be given to the particulate concentration in the air, its qualitative and quantitative composition, and its time-dependent variability. If trace element analysis is performed for elements that readily form volatile compounds or with a predominant gaseous phase chemistry additional measures of control need to be taken. To ensure continuous and adequate operation of the facility, a QC programme covering the following processes should be maintained: (1) particle counts, (2) blanking processes, (3) repeat analysis of control (blind) samples, and (4) establishing and maintaining control charts for all QC measurements.

6.1. Particle counts

Airborne particulate matter contributes to contamination of samples and blanks at undesirably high levels in ultra trace element analysis. The main purpose of using expensive filtration systems in clean laboratory environments is to control and maintain low particle concentrations in the ambient air. ISO 14644-1 protocol provides a detailed account of classification of air cleanliness and statistical measures associated with it. Particle counts can be performed readily and accurately with modern, multi channel laser particle counters. Many of them have built-in software that performs all necessary calculations. Laser based particle counters are very sensitive instruments and a particle counter built for measurements in a Class 100 environment cannot be used in “dirty” areas.

6.2. Blanking

Measuring the number of particles per unit volume of air does not necessarily correlate with the elemental or nuclide concentrations in the ambient air. For example, a 1μm particle of uranium oxide contains approximately 10^{10} atoms of uranium. Inadvertent contamination of a sample with just one such particle can adversely affect the trace and ultra trace analysis of uranium, e.g. pre-concentration NAA.

To account for such unwanted particles of specific composition, whatever may be the probability of finding such particles, environmental blanks, sometimes referred to as room blanks, need to be prepared. This can be done by exposing an acidified solution of isotopically altered spike, and measuring by means of isotope dilution analysis.

(a) *Blanking of crucial equipment and reagents* for leachable and inherent contamination that is used in the analysis need to be performed for each new batch (identified by "lot number" of a particular vendor).

(b) *Processing blanks* finally need to be run simultaneously with unknowns to account for the overall analytical uncertainty of a particular batch process. Processing blanks play an important role in trace and ultra trace elemental analysis as it is this "blank" value that will ultimately be subtracted from the unknown as "background". Keeping processing blanks as low as possible and as reproducible as possible is imperative and a fundamental objective of all blanking efforts.

(c) *Trackability* is a crucial concept of good laboratory practices (GLP) in ultra trace elemental analysis. Trackability requires identification of each piece of equipment and relating it to a particular batch that has been received from a vendor and keeping records of such. The same concept applies to solutions and reagents.

6.3. Control samples

An additional measure to check on the overall analytical performance (accuracy, precision, and repeatability) of a laboratory involves control samples. A control sample must have a high degree of similarity to the actual samples analysed. Control samples must be sufficiently homogeneous and stable so that individual aliquots measured at different times will have less variability than that of the measurement process. If the value of the measured property is known with sufficient accuracy, both precision of measurements and systematic errors in the measurement can be estimated. Control samples can be well characterized standard reference materials (SRM), but often less characterized (facility internal) materials that have been cross-checked with SRMs might suffice.

6.4. Control charts

Control charts are basic tools for QA. They provide graphical means to demonstrate statistical control, diagnose measurement problems, document measurement uncertainty, and generally aid in methodology development. They are also often used to monitor and document critical aspects of samples and sampling operations [4].

7. TECHNIQUES

The requirements of a clean lab may vary from one technique to the other. They also depend on a specific element or radionuclide and its levels being measured, as well as on the location of the lab where measurements are made.

7.1. Radionuclide measurement

Radioisotopes are characterized by measuring energies and intensities of radiations emitted by them, e.g. α, β and γ, and the half-lives. As the phenomenon of radioactivity is independent of physical and chemical state, radiation measurement is not hindered by the presence of non-radioactive contaminants, particularly γ ray measurements. This is true where the concentrations are above the trace levels. However, in the sub-trace concentration region, radiation measurements become difficult and often not so reliable. In this concentration domain, to ensure reliability, samples must be prepared in clean environment to avoid extraneous contamination. As long as the measurements are passive, clean room requirements are not demanding. A clean bench or a clean fume hood (Sections 3.1–3.3) would meet the requirements. When measuring alpha emitters in the sub-trace level using solid state nuclear track detectors, it has to be ensured that the sample is not contaminated with other alpha emitters. This is true particularly for measuring environmental samples. In the case of measurement of long lived radionuclides in the environment using techniques like ICP-MS and SIMS, sample

preparation and introduction should be carried out in the clean environment (see Section 8.3). Similarly measurements of trace quantities of different elements using PNAA techniques need moderate clean environments.

7.2. Neutron activation analysis

NAA techniques can be broadly classified under two categories depending on whether chemical separations are employed. If an element can be determined without the need for physical destruction of the sample by chemical treatments, the process is called non-destructive NAA or instrumental NAA (INAA). If chemical separations are employed in conjunction with NAA, the process is referred to as destructive NAA. This type of NAA can be further classified into two categories. If irradiation is followed by a chemical separation the technique is known as radiochemical NAA (RNAA). If, on the other hand, the element is chemically separated prior to irradiation, the technique can be further sub-classified into pre-concentration NAA (PNAA or CNAA) and derivative NAA (DNAA). In DNAA, the element of interest that has a poor sensitivity for NAA is either replaced or complexed with another element that can be determined by NAA with higher sensitivity. All other pre-irradiation chemical separations are included in PNAA.

The advantages of RNAA over other forms of NAA and other analytical techniques include freedom from reagent blanks, improvement of detection limits, precision and accuracy, and no requirement of a clean room. In RNAA, however, competing nuclear reactions cannot be eliminated, large volumes of sample cannot be easily irradiated in most of the reactor facilities, and short-lived nuclides cannot be conveniently measured. RNAA methods are generally time consuming and there exists a potential for radiation hazards. Alternatively PNAA methods are employed. There are several advantages of using PNAA [1,5,6,7]. It is important to keep the reagent blanks should be kept to a minimum when pre-concentration methods are employed. It is necessary to select ultra pure reagents and non-contaminating apparatus and to maintain an ultra clean environment for obtaining reliable results. With the increasing availability of highly pure reagents and clean rooms, pre-concentration of trace elements is becoming popular among analytical chemists.

A clean facility is absolutely essential for obtaining reliable values for elements like Al. Al is ubiquitous and has a crustal abundance of 7.83%. The main source of Al in air is soil re-entrainment. High levels of Al are found in air in countries with dry terrain (e.g. African countries), with bauxite deposits (e.g. Jamaica), and with aluminum industries. Long range transport Al has been reported. Some researchers observed an increase in Al levels in indoor air from air-conditioners. It is also present in chemical reagents.

Class 10000 clean lab can give reliable values for elements such as the rare earth elements (REE). High concentrations of REE in air are not that common except at locations or countries with bauxite deposits (e.g. Jamaica) and monazite sands (e.g. India and Thailand). They are present in cosmetics. They are not commonly found in chemical reagents. Due to these reasons, a clean facility is not essential for the determination of REE.

Some elements such as Cd are generally found at very low levels in normal indoor air. These levels do not usually interfere with their determinations in biological and other materials. Levels of Cd, for example, can be higher in some chemical reagents and plastic containers. Good laboratory practices and good methods can do the job.

In conclusion, a clean facility is absolutely essential for reliable measurements of most elements at ultra trace (ppb or less) levels as well as for low blanks, high accuracy, and high sensitivity works and when the sample has to be used for other techniques followed by INAA. In some cases, it may be possible to use less expensive means such as clean benches, boxes, fume hoods, and/or closed systems for obtaining equally reliable results.

7.3. Mass spectrometric techniques

7.3.1. Inductively coupled plasma mass spectrometry

Inductively coupled plasma mass spectrometry (ICP-MS) enjoys excellent sensitivities and promising detection limits in the sub-picogram/mL for many elements. It is recommended that samples be processed in Class 100 work benches located in a clean room of at least a Class 10,000 type. This will minimize and control the blanks so that the best detection limits of the technique are realized in practice. The sample introduction port, including the plasma torch of the instrument, can be arranged to have the class 100 clean air conditions by having a suitable clean air module around.

ICP-MS has proved to be a valuable technique for the determination of long lived radionuclides like Pu, U, Np**,** Th, Tc and Ra in environmental samples [8,9]. As there are isobaric interferences, in the presence of high uranium, these nuclides have to be separated by a suitable chromatographic method before these could be analysed by ICP-MS [10]. The separation procedures are better done under class 100 conditions to minimize the blanks with respect to the naturally occurring nuclides like uranium and thorium. Use of high purity spikes as internal calibration for multi-isotopic elements will significantly improve accuracy of these elements.

7.3.2. Thermal ionization mass spectrometry

Thermal ionization mass spectrometry (TIMS) is a well established and recognized technique for high precision and accurate measurements of isotope ratios for a wide variety of elements throughout the Periodic Table [11]. Combined with isotope dilution mass spectrometry (IDMS) and using spikes with isotopic compositions significantly different from the natural ones, this instrumental technique is also used for elemental assays in a variety of different matrices. As a result of sensitivity, selectivity, and high precision and accuracy this technique has found widespread use such as in geo- and cosmochronolgy, nuclear technology, and life sciences. Because many applications of TIMS rely on measurements of very small changes in the isotope ratio a clean laboratory environment is mandatory. Most laboratories maintain costly clean work areas or clean rooms. Chemical and physical sample preparations and instrumental measurements are performed preferably inside of a clean room facility.

TIMS is used routinely in safeguards oriented applications to assay for uranium and plutonium, and measure the isotopic composition in inspection samples to verify the correctness and completeness of declared values. However, most of the applications are in the geological sciences measuring isotope ratios of element pairs, such as the U-Pb couple, for dating purposes.

7.3.3. Secondary ionization mass spectrometry

Secondary ionization mass spectrometry (SIMS) is widely used for trace element measurements in solid materials, especially semiconductors and thin films [12]. The SIMS ion source produces ions directly from solids without prior vaporization by a process called “sputtering”. Accelerator mass spectrometry (AMS), see Section 8.3.4, also uses this type of ionization process. SIMS primary ion beam can be focused to less than 1 micrometer in diameter. Microanalysis can be facilitated by precisely focusing the primary beam onto the sample surface. During SIMS analysis the sample surface is slowly sputtered away. Continuous analysis while sputtering produces information as a function of depth.

SIMS can also be used as an ion imaging tool and to measure isotope ratios. Ion images show secondary ion intensities as a function of location on the sample surface. Isotope ratio measurements are operational, similar to depth profiling, except that precision and accuracy requirements are higher. Significantly, in SIMS each individual particle becomes a sample on its own. This makes SIMS studies vulnerable to contamination with particles chemically and isotopically foreign to the sample. Sample preparation, sample loading and instrument should be kept in a clean environment, depending on the application, to preclude contamination.

7.3.4. Accelerator mass spectrometry (AMS)

Accelerator mass spectrometry is the analytical technique of choice for the detection of long lived radionuclides, which cannot be analysed with conventional radiometric or mass spectrometric techniques. AMS allows an isotopic sensitivity as low as one part in 10^{15} and detection limits of 10^6 atoms for ^{14}C (5.73 ka), ^{10}Be (1.51Ma), ^{26}Al (720 ka), ^{36}Cl (301 ka), ^{41}Ca (104 ka), ^{129}I (15.7 Ma) and other natural and anthropogenic, long-lived radionuclides. AMS has been recently used in the analysis of ^{236}U and other actinide nuclides and fission products with important applications in environmental monitoring for nuclear safeguards and nuclear waste management [13].

The main *transport mechanism of AMS radioisotopes* in the environment is by particulate matter, such as aerosols and in the gaseous phases, e.g. CO_2. An immediate consequence is that AMS sample preparation is best performed in a clean environment. Samples in the sub-milligram range and samples with very low specific activities can be seriously affected by airborne contamination and a controlled clean laboratory environment is very desirable [14]. Prime examples are analysis of rare actinide nuclides, such as ^{236}U in microgram samples of uranium oxide, and processing volatile long lived fission products (e.g. ^{129}I), or products of neutron irradiation (e.g. ^{36}Cl) in the vicinity of nuclear reactors [15].

Isobaric interferences adversely affect AMS detection limits. Some isobars, e.g. ^{36}S, ^{10}B, or ^{41}K, are ubiquitous in the environment and can contaminate the target materials used in the ion source or may be inherent to ion source materials. As for elemental contamination, isobaric interferences are introduced into the sample by airborne transport mechanisms. Clean environments are desirable and mandatory for state of the art analyses. In addition to sample preparation areas, sample loading environments need to be free of those constituents, a fact that has been often neglected in AMS facilities.

8. MAINTENANCE OF THE FACILITY

Proper maintenance of clean facilities is required to achieve continued satisfactory performance of the facility. The more complex the system is, more effort is required for maintenance. Table II is a partial listing of maintenance activities that must be documented and scheduled. The clean room shares many of these requirements. The complexity of each item will however be greater than for a clean room. Clearly the maintenance of the components is much less complex for maintaining a clean room than for a clean laboratory.

9. STAFF AND TRAINING

It is essential for the analysts to be provided with suitable training in clean room practices in ultra trace measurements. The training course must consist of lectures by experts in the field to cover the following topics: (i) Trace and ultra trace measurements, (ii) concept of clean facilities like laboratories and clean rooms, (iii) materials for clean facilities, (iv) protocols for clean room practices, (v) QA/QC in ultra trace measurements, (vi) maintenance of clean facility and (vii) man in the clean environment.

It is also mandatory to provide training to the existing staff on the latest developments periodically, both in the maintenance and awareness of measurement techniques.

TABLE II. FACILITY MAINTENANCE REQUIREMENTS

S. No	Requirement	Clean facility component	Clean room	Clean lab.
1	Maintenance schedule	X	X	X
2	Filter changes	X	X	X
3	Spares inventory	X	X	X
4	Gowning requirements	X	X	X
5	Particle count test	X	X	X
6	System lubrication	X	X	X
7	Shut down protocol	X	X	X
8	Restart protocol	X	X	X
9	Cleaning protocol	X	X	X
10	De-ionized water system		X	X
11	Chilled water system			X
12	Hot water system			X
13	Air supply system		X	X
14	Humidity control		X	X
15	Airflow control		X	X
16	Air pressure control		X	X
17	Sensor calibration		X	X
18	Primary filter system		X	X
19	Intermediate filter system		X	X
20	HEPA filter system	X	X	X
21	Exhaust air system		X	X
22	Fume Exhaust system		X	X
23	Drawing controls	X	X	X
24	Change order controls		X	X
25	Prepare log		X	X
26	Work order control		X	X
27	Electrical connection maintenance	X	X	X
28	Required performance tests	X	X	X

10. CONCLUSIONS

(1) A clean facility is essential to meet the analytical challenges of today and tomorrow. Steps must be taken to establish such facilities for trace element and radionuclide monitoring and research.

(2) Clean facilities are needed more so in countries with dry terrain where the suspended particulate matter load of air is high due to the re-entrainment of soil particles to atmosphere. As the clean facilities are rather expensive to build, possible locations may be identified (RCA, AFRA, and/or ARCAL countries), in establishing and maintaining one or two multi-user, regional “Class 100” clean facilities to start with.

(3) All measurements do not necessarily require elaborate clean facilities. The alternative systems such as closed clean sample processing systems, which are neither as expensive and nor as difficult to maintain, suffice for such purposes.

(4) Maintenance of clean facilities is expensive and needs an appropriate infrastructure, and sometimes a change in the thought process.

(5) As the attitude of man and awareness about latest developments are essential in maintaining and using a clean facility, continued education and training activities of laboratory staff to stimulate further advancement are very important.

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CONTRIBUTED PAPERS

METAL FREE CLEAN ROOM FOR ULTRA TRACE ANAYSIS

T.R. MAHALINGAM
Materials Chemistry Division,
Indira Gandhi Centre for Atomic Research,
Kalpakkam, India

Abstract

Clean laboratory environment is a prerequisite for all kinds of analytical tasks, particularly if trace or ultra-trace concentrations in natural matrix materials are targeted. A pragmatic approach to install a clean-room facility is described, and advice is given on how to circumvent potential sources of element contamination in laboratory air. Following such advice it should be possible to operate an ICP-MS for U, Th and trace element analysis in biological tissues and fluids.

1. INTRODUCTION

The need for accurate measurements of trace element concentrations has been felt long back by the semiconductor industry. But of late the importance of several essential and toxic trace elements in our health has been recognized so much so that biological trace element research has evolved as a major field of research [1]. Trace measurements are thus required in a wide range of disciplines, from medical diagnosis through nuclear science and the semiconductor industry, to the environment. Our understanding of the chemical composition of the environment has changed radically with the development of new sensitive analytical techniques and appropriate methodologies. For example, over the past two decades, concentrations of many trace constituents in the deep ocean have appeared to drop by orders of magnitude. Needless to say this has little to do with a real drop in concentration, but reflects improvements in sample handling and analytical techniques. Graphite furnace AAS, NAA and the more recent TXRF and ICP-MS are some of the techniques which have capabilities of ultra trace detection. Isotope dilution mass spectrometric (IDMS) techniques guarantee very high accuracy.

As ours is a nuclear research centre, the job of our section is mainly to cater to the chemical characterization including the trace analysis of reactor materials like uranium, plutonium; sodium used as a coolant in our fast reactor; stainless steels and other structural materials; and water and organic solvents used in fuel reprocessing research. Accordingly we had developed new sensitive analytical methods for the trace metal characterization of sodium and uranium [2]. A novel on-line solvent extraction method has been developed for the analysis of uranium with levels of detection in the ppb regime [3]. Similarly a novel laser vaporization technique has been demonstrated for the introduction of very small sample volumes in ICP-MS [4]. In addition we also cater to the analytical needs of research on superconductors and sensors. In collaboration with the Geological Survey of India we have developed and standardized a nickel sulphide fire assay method for the determination of platinum group elements in geological minerals and ores [5]. In addition we have some interests in biological and environmental trace elements research [6–9]. We have developed the necessary analytical methods for the determination of several trace metals in blood plasma and red blood cells and generated the reference values for these trace elements in the blood of the Kalpakkam population and got some interesting correlation between trace metal profile and coronary risk index [6–8].

It has been our experience that if the analytes are present at parts per million levels, the measurements do not pose any serious problems and are handled routinely taking some simple precautions to avoid contamination. The determinations at lower concentrations viz. parts per billion and below are termed ultra trace analysis. In ultra trace analysis the effects of contamination from laboratory air or furnishings, apparatus, containers, reagents and even humans have become increasingly important, as the high sensitivity of several instrumental methods has lowered the detection limits to the nano, pico and even femto gram regime. So, development of clean sampling,

handling and analytical procedures, which in themselves can involve a substantial investment of time and resources, has become a necessary step towards obtaining accurate analytical measurements. The importance of controlling the analytical blank with the help of a clean room to produce a particulate poor environment has been discussed by several researchers [10–12]. Such particulate contamination may be controlled by the high efficiency particulate air (HEPA) filters. The design of a clean laboratory making use of HEPA filters, as used at NIST, has been discussed by Moody [13]. We based the design of our clean room based on the available information in the literature. Especially we took very good care to avoid any metallic components in our clean room. Wherever it is unavoidable, these parts have been covered with a lining of HDPE or painted with a good coat of epoxy painting.

2. HEPA AND CLASS 100 CLEANLINESS

It is now known that the environmentally induced blanks originate largely from the particulate matter in the laboratory air. Both the elemental composition and numbers of particles may be expected to vary with the location. The particulate counts above 0.5 μm in the atmosphere could range generally from 10^6–10^7 counts per cubic foot. The Federal Standard 209D defines a number of air qualities, with the class 100 standard being widely adopted for clean rooms. Although the requirement is frequently stated as a measurement of no more than 100 particles per cubic foot, which are 0.5μm or larger, it is actually a bit more complicated. The important analytical consideration is the total mass of particles, regardless of their size or number. As can be seen from Table I, the standard is considerably stricter on larger particles. Fortunately, the efficiency of HEPA filters is at a minimum at 0.3 μm rather than between 1–10 μm where the particles that contribute most to the analytical blanks are found. Hence air filtration with HEPA effectively controls the particle counts and air borne contamination. HEPA filters are composed of thin porous sheets of ultrafine glass fibres. The sheets are pleated with aluminium or plastic separators. The analyst must select the *filters with plastic separators* or the *separatorless* variety that are available nowadays. As the aluminum has been found to corrode and contribute to large blanks, it must be avoided. Both the prefilters and the HEPA filters should be housed in wooden or some non-metallic frames.

TABLE I. FEDERAL STANDARD 209D AIR CLEANLINESS CLASS LEVELS

	Particle size (μm)				
Class	0.1	0.2	0.3	0.5	5.0
1	35	7.5	3	1	0
10	350	75	30	10	0
100	na	750	300	100	0
1000	na	na	na	1000	7
10000	na	na	na	10000	70
100000	na	na	na	100000	7000

- The numbers shown are the maximum number of particles greater than the specified size (in μm) permissible per cubic foot (0.028 m^3) of air. Note that the numbers of particles shown do not make any assumptions about the actual size distributions of particles to be found in particular situations [10].
- na — not applicable.

3. LAMINAR VERSUS TURBULENT AIR FLOW

Two types of airflow systems have been designed to remove particulate matter. The first is the conventional clean room that contains several HEPA filters spaced at intervals at the ceiling. The air is removed by return air grills located at the side walls at floor level. Because of the turbulent flow conditions prevailing within such a facility, an airborne particle can pass a critical work area several

times before leaving the work environment. These rooms generally meet the Federal Standard Classes 10,000–100,000.

The second type is the laminar flow clean room making use of laminar patterns of unidirectional flow. Laminar air moves in one pass from a bank of HEPA filters to the work area and exits from the clean room without any turbulence. When one wall of a room provided laminar flow, Class 100 conditions are maintained in the area close to the HEPA filters. When a HEPA filter bank is mounted in the ceiling with suitable perforations in the floor for the return air, then the entire room can be maintained at a more uniform level of cleanliness that could be close to Class 100.

4. METAL FREE CLEAN ROOM

Since maintaining a high class of air cleanliness in a large environment is quite expensive, one can have task-*specific* air cleanliness: a clean workbench in a clean room, where the air cleanliness at the bench or work station is one or two orders of magnitude higher than the surrounding environment. We have adopted this approach in our design of the clean room. We have installed four numbers of class 100 vertical laminar flow chemical workstations in a compact clean room of Class 10,000.

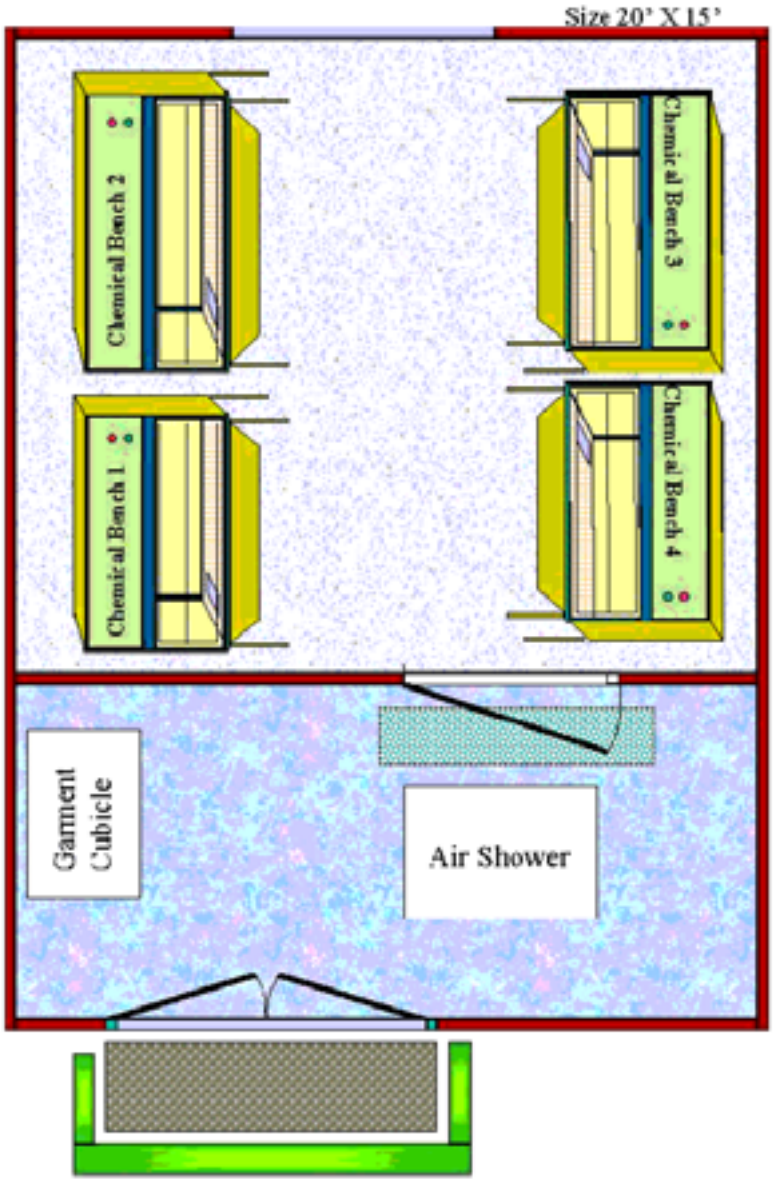

FIG. 1. Clean room facility.

The air supply line and exhaust are separate for the workstations and the room. For the room, it is purified and cooled air that is recirculated with 10% of fresh air added each time. For the workstations, the air is separately supplied through prefilters and HEPA filters and exhausted by separate blowers continuously. So it is a once through system without any recirculation to facilitate rapid exhaust of the acid fumes in the chemical workstations. Also it is uncooled air that is supplied in three of the workstations to minimize the cost. This is adequate, as the operations will involve sampleroom, where the air cleanliness at the bench or work station is one or two orders of magnitude higher than the surrounding environment. We have adopted this approach in our design of the clean room. We have installed four numbers of class 100 vertical laminar flow chemical workstations in a compact clean room of class 10,000. The air supply line and exhaust are separate for the workstations and the room. For the room, it is purified and cooled air that is recirculated with 10% of fresh air added each time. For the workstations, the air is separately supplied through prefilters and HEPA filters and exhausted by separate blowers continuously. So it is a once through system without any recirculation to facilitate rapid exhaust of the acid fumes in the chemical workstations. Also it is uncooled air that is supplied in three of the workstations to minimize the cost. This is adequate, as the operations will involve sample dissolution with acids and other pre concentration steps. Cooled air provision (once through) has been given in one of the workstations to carry out any solvent extraction with volatile solvents. So in the *workstations where the samples are to be processed*, *Class 100 cleanliness will always be maintained*.

We have taken all precautions to see that the materials used in the construction of the room and the work stations are *essentially metal free,* as the metallic parts will start corroding fast with the use of acids and these particles would pose a far more serious problem than the air particulates. The workstations are made of wood with melamine laminations. The HEPA (patented Gradvel) [14] separatorless Minipleat variety prefilter both mounted in non-metallic frame. The motor is epoxy painted and the blower is in polypropylene housing with polypropylene impeller. It has polypropylene perforated table, lights covered and held in non-metallic sockets. The water taps and sinks provided are also made of polypropylene. The bolts and nuts are made of HDPE and wherever metallic bolts are required they were coated with epoxy paint. The door is made of wood and the door handle of plastic. The false ceiling is made of wood and the entire room is painted with antifungal epoxy paint. The sharp corners have been made into smooth curves (coving) so that continuous smooth clean airflow is facilitated and segregation of dust in these corners is avoided. The main blower supplying air to the room and the split air conditioners are located at a distance of about 20 feet in a separate building to avoid both the vibrations and the noise. The main blower is epoxy painted and the casing is made of FRP. The ducting to the clean room is made of HDPE, suitably insulated. So all possible precautions have been taken to prevent generation of metallic particles from the materials of construction of the room. As horizontal laminar flow could produce cross contamination problems, vertical laminar flow is preferred in the workstations where acid digestion of samples is planned. *The sample solutions thus prepared will be transported in an airtight Teflon box to the instrument lab. Sample introduction into the instrument will be carried out under class 100 conditions.* A compact Class 100 workbench has been designed to house the sample introduction port of the ICP-MS and graphite furnace AAS the two principal techniques, which would be used for ultra trace analysis.

The air pattern in the laminar flow hood or in the laboratory can be conveniently examined by placing some liquid nitrogen in a polythene beaker and observing the “smoke” flow at any location in the laboratory. Commercial “smoke sticks” based on titanium tetrachloride contaminate the laboratory with titanium and HCl and hence to be avoided. Drawing the beaker of liquid nitrogen along the face of the HEPA filter will immediately show the airflow during actual working conditions. This practice also will help to find out the extent to which bulky equipment in the hood causes turbulence. The position of the equipment could be adjusted to have a minimum of turbulence. The liquid nitrogen should be placed frequently at the bottom of the laboratory entrance door to verify that the clean room is indeed under a slight positive pressure.

Vertical flow units require a sliding polycarbonate front panel to keep it virtually closed when fuming operations are on. If the exhaust of the hood is too powerful then some room air will be

sucked in. Smoke patterns with liquid nitrogen define the exhaust capability needed to remove the toxic fumes. A simple test is to fasten a strip of plastic with cellophane tape to the front of the clean hood. If the strip is drawn into the hood, then the airflow is not balanced. The exhaust louver must then be positioned so that the plastic strip is forced gently toward the open laboratory. The shutter is to be opened only for intermittent operations and to be quickly closed to minimize ingress of fumes in the room. If the fumes are known to be quite toxic then it is advisable to wait till the complete fuming is over before the hood is opened or the fume hood could be kept at a slight negative pressure to ensure operator's safety.

The vertical flow also directs the chemical fumes downward that is on to the surface of the hot plate. Hence the *traditional metallic hot plates cannot be used*. Hot plates with *ceramic tops* should be used. Even the metallic body of the hotplate should be painted with epoxy paint and covered with Perspex sheets. This is very important as the hot plate will be continuously showered with hot corrosive acid fumes and with time, will be generating lot of metallic particles and giving rise to severe contamination problems.

5. AIR VELOCITY

In a turbulence study [11] with titanium tetrachloride smoke tests in vertical flow velocities ranging from 3.05 to 19.8 m/min, no smoke particles were detected 0.6 m upstream at velocities of 6.1 to 19.8 m/min. At 3.05 m/min counts averaged 18–25 $\times 10^6$ smoke particles per cubic foot of air. 4.6 m/min appear to be a marginally slow flow rate, which dramatically removed air borne bacteria from an operating table during surgical procedures. Speeds below 4 m/min lower the efficiency of the HEPA filter. Fewer particles impinge on the filter medium; more particles can find their way through the maze of the filter. In addition air currents from the general laboratory area encounter less resistance in gaining entry into the hood. Generally in the laminar flow installations, an airflow of 20 m/min is maintained. Speeds higher than 30.5 m/min tend to produce electrostatic charges on polythene and Teflon containers[11]. The air flow has been kept at 20 m/min and the number of air changes per hour has been kept at forty per hour in our clean room.

6. MAINTAINING THE CLEAN AIR QUALITY

The air quality in the room and in the workbench should be periodically checked with particle counters which work on light scattering principle. The general clean room area is class 10,000 and the workbench is of class 100. Monitoring of the air in our room shows about 2000 counts/cft and in the work bench 20 counts/cft. These levels can be maintained by daily maintenance of floors with a vacuum cleaner or by wet mopping with pure water with a sponge mop. By way of maintenance, periodic replacement of HEPA and prefilters is essential, and the need for replacement can usually be determined by the pressure drop across the filter as it becomes progressively blocked by particles. If this operation involves the replacement being carried out within the clean area, then the facility will need careful cleaning of surfaces, and key equipments if any, before any sample processing is taken up.

7. CLEANING PROCEDURE FOR SURFACES

In between two operations, it is a good practice to clean the floor of the workstation thoroughly to avoid cross contamination. Once the particles are deposited at a surface, they are strongly held by gravitational and Van-der-Waals forces and if the particles are moist the surface tension forces also aid to keep the particles on to the surface [11]. To release the particles back into atmosphere, the adhesion forces operating on it must be overcome by the air flow. When air flows over a surface, there is a static layer of air at the surface which is termed the boundary layer, the thickness of which depends primarily on the air velocity. In clean rooms or unidirectional flow hoods, the boundary layer of static air at a surface is typically several millimeters thick at normal air velocities; thus as the small particles do not extend out to this layer, they cannot be remobilized into the atmosphere by this flow. Hence they are not readily removed by the ambient airflows in a clean hood or clean room.

Hence appropriate surface cleaning methods are important. Libermann [11] has investigated different cleaning methods, which included sophisticated high pressure washing, ultrasonic agitation, and use of hydrophobic solvents such as Freon, the most effective method for removing particles was the simple procedure of wiping with a lens tissue using ethanol. In many cases high purity water can also be an effective wash agent. Wiping with ethanol periodically also helps in eliminating the static electricity that tends to build over the plastic surfaces.

8. THE HUMAN FACTOR

Human tissues and secretions are likely to come inadvertently into contact with the sample. Problems, which arise from this source range from the deposition of finger lipids to the introduction of dandruff. Cosmetics can be a significant source of contamination, because of metal oxides and other materials that are added for colour and texture. Coughing and sneezing can produce a large number of particles (about 6×10^5 of 0.5μm dia.). The human skin continuously sheds dead cells and cell fragments typically 20–40μm in dia. and 2–4μm in width. The number of particles emitted by individuals rapidly increases with increasing activity. Thus, an individual in normal clothing sitting or standing motionless can generate on the order of 10^5 particles (>0.3μm) per minute, where as a person working normally can generate about 10^7 particles [10]. Hence, the analysts should cover their body with special garments.

TABLE II. ELEMENTS PRESENT IN COMMONLY USED COSMETICS [15]

Cosmetic	Particles 05 μm or larger Per application	Elements present
Lipstick	1.1×10^9	Bi
Blusher	6.0×10^8	Mg, Si, Fe, Ti
Powder	2.7×10^8	Si, Mg
Eye shadow	8.2×10^9	Bi, Si, Mg
Mascara	3.0×10^9	Fe

A pro-active attitude of the analysts who work in the clean environment is essential. There should be a continual awareness of problems, which may be developing, e.g. corrosion of a component or a build up-of unused materials and equipment in the laboratory, which may lead to subsequent contamination problems. It is much better to deal with them at an early stage than to find high and variable blanks later, which invalidate a series of analyses that have involved time and resources to undertake. Appropriate training must be imparted to the analysts on contamination control measures.

9. CLEAN ROOM PRACTICES

The entry of both men and materials in the clean room should be carefully controlled. Only the things absolutely essential should be taken in. The bottles and other apparatus should be wiped clean using a lint free tissue with suitable solvent to minimize particulate loading in the room. A compact vacuum cleaner should be kept in the airlock room for cleaning the pieces of equipment before they are taken inside the clean room. To minimize particulate contamination from humans, the analysts working in clean rooms should wear special laboratory coats, head masks and gloves. These garments should be:

(1) made from materials that do not shed fibers
(2) made without any metallic components (buttons, zips) that can corrode and contaminate
(3) able to retain particles
(4) comfortable to wear, and if possible resistant to acids and reagents.

Materials used for clean room garments include polyester fabrics, spun bonded nylons and polypropylene composites [10]. Thin clear disposable polythene gloves are recommended for use in the clean room. These can be generally used fresh off the pack and comfort can be improved by wearing glove liners made of non-particle generating fabric.

Footwear can transport significant amounts of debris into a clean environment. This is generally controlled effectively by adopting the following practice: the analysts should remove their shoes outside the clean room and enter the anti-room through a tacky mat, which effectively removes the footprint. Special footwear with shoe covers is kept in the garment cubicle in the anti room, which serves as an air lock between the outside area and the clean room. The analysts should wear these special garments, shoe covers and gloves before entering the clean room. This anti room also is Class 10,000 and is at a positive pressure (0.25 inch water) as compared to outside atmosphere, and the clean room itself will be at a positive pressure as compared to this anti room. The garment cubicle is provided with UV illumination to kill all bacteria.

There are many other sources of contamination in an apparently clean trace analysis laboratory. The injudicious use of some paper products can cause significant contamination. One must especially guard against the practice of wiping the sample or the spillages with tissue papers. It is advisable to use a damp clean lint free cloth for wiping the spillages. There is a range of other materials commonly used in the laboratory that contain significant amounts of potential contaminants (see Table III).

TABLE III. EXAMPLES OF TRACE ELEMENT CONTENT OF MATERIALS COMMONLY USED IN LABORATORIES

Material	Zn	Fe	Sb	Others
Paper tissues	49	1		Cr 0.5
White plastic tape	3000	67		
Adhesive tape	1.5	5		
PVC tubing	7	270	2.5	Cu 0.6
Rubber tubing	40000		0.36	Cr 420, Co 7.5 Co 3.1, Sc 3.1

NOTE: Concentration in μg/g except for Fe, which is given in mg/g [16].

10. LABORATORY MATERIALS FOR ULTRA TRACE ANALYSIS

The pipettes and burettes made of borosilicate glass, which find extensive use in conventional laboratories, are not suitable for use in trace analysis. The major chemical constituents of glass are given in Table 4. Glass has been found to release trace elements (Na, Al, B) especially at both extremes of pH. High pH results in the dissolution of the glass surface itself, whereas low pH favors metal dissolution and desorption from the glass surface. In addition the clean glass surface is highly reactive, primarily because of the presence of the highly reactive Si–OH groups. These groups are acidic in nature and are an effective ion exchange material. Cationic analytes are therefore effectively scavenged from solution onto the glass surface [10].

$$GlassSiOH + M^{+} \rightarrow GlassSiOM + H^{+}$$

A much better but more expensive option is vitreous silica prepared from the vapour phase hydrolysis of silicon tetrachloride or tetrafluride. Vitreous silica ware could be satisfactorily cleaned with acids. It could also be used at elevated temperatures if necessary. The trace metal contents of different kinds of silica are given in the Table IV.

TABLE IV. THE MAJOR CHEMICAL CONSTITUENTS OF GLASS [17]

	Typical concentration (wt %)									
Type	SiO_2	Al_2O_3	ZrO_2	Na_2O	K_2O	Li_2O	B_2O_3	CaO	MgO	BaO
Soda A	73	1		17	0.5		5	4		
Soda B	74	2		13	0.5	3	11	0.5		
Borosilicate A	81	2		4	0.5	13				
Borosilicate B	73	6		7	0.5	10	1		2	
Alkali-resistant	71	1	15	11	0.5	1				
High silica	96	0.5								
Vitreous silica	100									

TABLE V. TRACE IMPURITIES (PPM) IN VITREOUS SILICA [11]

	Silica		
Element	Transparent from quartz	Transparent from SiX_4	Translucent from silica sand
Al	74	<0.25	500
B	4	0.1	9
Ca	16	<0.1	200
Cr	0.1	0.03	nd
Cu	1	<1	nd
Fe	7	<0.2	77
K	6	0.1	37
Li	7	nd	3
Mg	4	nd	150
Na	9	<0.1	60
P	0.01	<0.001	nd
Ti	3	nd	120

nd = not detected.

11. PLASTICS

A wide variety of plastics has now been used in the construction of laboratory apparatus and careful selection of materials for trace analysis applications is necessary if apparatus is not to interfere in the procedure. The following factors to be considered for assessing the suitability for trace analysis: adsorption properties of polymers for the analytes and contamination arising from residual catalysts and additives used in manufacture. Other considerations could be more of a physical nature such as whether the containers can be dried at elevated temperatures. The trace element content of some plastics is summarized in Table VI.

Another important factor in the choice of polymer is its permeability (Table VII). For most applications, a low permeability is desirable. Permeability is derived from the migration of molecules through microscopic voids between the polymer chains. The absorption of material by polymers is in part due to its migration through the polymer structure. Discolouration of a plastic container is often

associated with the migration of material into the polymer matrix, and the removal of such material is quite difficult. The movement of materials out of the container by permeation takes two main forms. The long term storage of solutions in unsuitable containers is known to result in loss of water through permeation. The resulting change in the concentration of the analyte may go unnoticed unless precaution is taken to weigh the sample before storage. The second complication may arise with analytes, which are in equilibrium with some volatile species such as ammonium and sulphide salts. Although only a small proportion of ammonia or hydrogen sulphide may leak out of the container wall, there will be re-establishment of the equilibrium generating further gas to permeate out of the container [10].

Based on the above considerations, vitreous silica and PTFE seem to fit for ultra trace analysis. HDPE could be used at room temperatures.

12. CLEANING OF THE LABWARE

The fresh silica or plastic ware should be thoroughly cleaned as follows: degreased by cleaning with a non ionic detergent like Triton X-100, followed by 8 hours soaking in 1:1 nitric acid and 1:1 hydrochloric acid. Final washing and soaking in high purity water should last for eight hours before they are taken for use.

13. STORAGE OF SAMPLES

As a general practice, every effort should be made to carry out the analysis as soon as possible after obtaining the sample or preparing a solution of it for analysis. But many a time it may not be possible to avoid storage completely. Two factors that are of concern to a trace analyst are the following: (1) there should not be any contamination from the container, (2) there should not be any analyte loss by adsorption to the container walls. Both of these factors are taken care to a large degree if vitreous silica ware or PTFE containers are used at the low acidic pH.

TABLE VI. TRACE ELEMENT CONTENT OF SOME PLASTICS (NG/G) [10]

	LDPE	HDPE	PP	PS	PC	PVC	PTFE	FEP	ETFE
Na	1300	15000	4800	2200	2700	20000	160	400	600
Al	500	30000	55000	500	3000		230	200	
Cl	7000	30000	180000		50000	major		800	10^6
K	>5000	>600						90000	1100
Ca		800000							
Ti		5000	60000	1000					
Mn		10	20	20				60	
Co			40		6				
Zn		520000							
Br	>20	800	>5	>1	29000	>6	>2		240
Sn						2.4×10^6			
Sb	5	200	600						
La				0.3			0.6		1
W								700	
Au			0.1	0.04	0.03		0.4		0.4

FEP: Fluorinated ethylene propylene; HDPE: High density polyethylene; LDPE: Low density polyethylene; PC: Poly carbonate; PP: Polypropylene; PS: Polystyrene; PVC: Poly vinyl chloride; PTFE: Poly tetra fluro ethylene; ETFE: Ethylene-tetrafluroethylene.

TABLE VII. METAL IMPURITIES LEACHED FROM 500 ML OR 1 LITRE PLASTIC CONTAINERS IN ONE WEEK BY NITRIC ACID (1+1).

Element	Teflon FEP	HDPE	LDPE	PC
Pb	2	2	0.7	0.3
Ti	≤1	≤1	1	≤0.8
Ba	4	≤0.2	2	0.3
Te	0.6	0.2	≤0.5	0.3
Sn	1	1	≤0.8	0.2
Cd	0.4	0.2	0.2	0.3
Ag	≤8	0.2	nd	nd
Sr	0.2	1	0.2	≤0.2
Se	0.2	0.4	3	0.5
Zn	4	8	2	0.8
Cu	2	0.4	2	0.8
Ni	2	1.6	0.5	0.7
Fe	20	3	3	3
Cr	0.8	0.2	0.8	0.3
Ca	80	0.6	10	3
K	2	2	2	2
Mg	8	0.6	10	3
Al	6	1	1	5
Na	6	10	8	3
TOTAL	148	50	38	23

- FEP: Fluorinated ethylene propylene; HDPE: High density polyethylene; LDPE: Low density polyethylene; PC: Poly carbonate
- Concentrations in ng per square centimetre of surface. Leaching experiments were carried out at room temperature except for the Teflon bottles, which were leached at 80°C nd = not detected [10]

TABLE VIII. PERMEABILITIES OF POLYMERS AT 20–30°C [9]

Polymer	Gases			Water
	N_2	O_2	CO_2	
FEP	21.5	59	17	500
PTFE	0.09–1.3	0.25–5.4	0.48–12.5	3–360
PP	4.4	23	92	700
LDPE	20	59	280	2100
HDPE	3.3	11	43	120
PVC	0.4–1.7	1.2–6	10.2–37	2600–6300
PC	3	20	85	7000
Silicone rubber	1000–6000	6000–30000	106000	
Polyamide	0.1–0.7	0.38	1.6	700–17000

- Units; cm3(STP)/cm2/mm/sec/(cmHg) × 1010 [10].
- FEP: Fluorinated ethylene propylene; PTFE: Poly tetra fluro ethylene; PP:Polypropylene; LDPE: Low density polyethylene; HDPE: High density polyethylene; PVC :Poly vinyl chloride; PC: Poly carbonate.

14. SOLUTION STANDARDS

The standard solutions for ultra trace multielement techniques such as ICP-MS should be prepared from spec pure metals or salts of high purity six nines or five nines. The concentrated stock solution standards of concentration 1g/L for multi elements can be kept in polythene (HDPE) containers at 1N nitric or suitable acid medium. Under these conditions these standards have been found to be stable for about 3 months. The working standards in the ppb-ppt levels should be prepared freshly each time by suitable dilution of the stock standard just before the analysis. These are best prepared in small volumes of 5–10 ml in clean polypropylene vials with screw cap. Micro pipettes (Finn pipettes or equivalent) with disposable polypropylene tips are used for making these dilutions. Of course the vials and other apparatus used should be thoroughly cleaned by soaking them for eight hours in 1:1 nitric acid, 1:1 hydrochloric acid , followed by cleaning and soaking in high purity water (18 MΩcm resistivity) for eight hours. These vials are tested for blanks by ICP-MS before they are used for the preparation of working standards. The high purity water is prepared by purifying the distilled water further through the Millipore water purification system based on ion exchange. The acids are purified by sub boiling distillation in a vitreous silica apparatus. Right now the levels of trace metals in the acid are found to be in the sub-nanogram/ml levels for the different elements and our process blanks for e.g. in blood analysis has been found to be in the range 3–18 ng/ml for the various analytes, the maximum being for zinc and copper. . These purification facilities are now being installed in the class 100 workstations in the clean room. With this arrangement, our reagent blanks from acids and our process blanks in our experiments are expected to go down by two or three orders of magnitude to improve our ultra trace analysis in future.

15. DETERMINATION OF LONG LIVED RADIONUCLIDES BY ICP-MS

ICP-MS has proved to be a valuable technique for the determination of long lived radionuclides like Pu, U, Np**,** Th, Tc and Ra in environmental samples. Excellent detection limits in the femto gram regime have been reported in the literature [18,19]. As there are isobaric interferences, in the presence of high uranium these nuclides have to be separated by a suitable chromatographic method before these could be analysed by ICP-MS. Two types of chromatographic resins, Dowex IX8 and TEVA, have been examined for separating plutonium from environmental matrix elements. Sufficient decontamination factors 10^4–10^5 have been obtained for many of the matrix elements including uranium, which interferes with Pu estimation in ICP-MS. The detection limit for Pu was found to be 0.1 pg/g for the sediment samples. Many certified reference materials from the IAEA have been analysed [20]. Similarly, use of high efficiency nebulizers such as direct injection nebulizer (DIHEN) has found to yield excellent detections limits in the pg/L levels for the actinides [21]. Clean room practices are essential for controlling the blanks with respect to the naturally occurring nuclides like U and Th especially so in a radiochemistry laboratory like ours where U, Th are handled routinely. The sample handling involving dissolution and pre-concentration/separation are to be done in a clean room. Because of the highly toxic nature of these elements, even though at environmental levels, it is essential to observe special precautions while carrying out the pre-concentration stage. After pre-concentration, if the concentration levels of these nuclides demand safe handling, all the precautions associated with handling radioactive substances are to be followed. The chemical workstations shall have good exhaust and preferably under slight *negative pressure* as compared to the clean room. Most of the operations are to be carried out with the front panels closed. Once prepared, the sample solutions can be sealed in an airtight Teflon container and transported to the instrument laboratory. *The instrument should also be adapted for the glove box operation if necessary.* Details of glove box adaptation of ICP-MS have been reported in the literature [22,23]. We too plan to house the plasma torch and the sliding interface of our ICP-MS in a glove box to facilitate handling radioactive samples. *This not only will serve as a safe barrier for the operator to handle radioactivity, it will also serve as a clean Class 100 enclosure (by equipping with HEPA for air filtration) around the plasma torch and the sample introduction port of ICP-MS that are exposed to the atmosphere*. The sample solutions prepared in the clean room can be satisfactorily analysed both for *radionuclides and other heavy metals*. But the latest commercial ICP-MS instruments claim very high sensitivities with

specially equipped interfaces and nebulizers. Probably some of the nuclides present at environmental levels could be directly measured without any pre-concentration. In that case there is no need for any glove box. It is sufficient if we have class 100 clean air modules around the ICP torch and the sample introduction port of the ICP-MS.

16. CONCLUSION

We intend to use the clean room for our future work involving study of long lived radionuclides and toxic element speciation in the environmental samples. Use of Certified Reference Materials or independent alternate techniques are essential for validating the analytical measurements. With the commissioning of the KAMINI (Kalpakkam Mini reactor) reactor at Kalpakkam, the technique of neutron activation analysis also could be used in future in addition to ICP-MS and GFAAS. The extent to which the clean room practices are needed will ultimately depend upon the elements of interest and the level and reproducibility of the blanks. In all cases, if the precautions are to operate effectively, there must be continual awareness of the potential problems by the operating analyst, and regular and effective protocols must be maintained. Future trends in ultra trace analysis probably will involve the increasing isolation of the analyst from the analytical operations through the use of robots in controlled closed environments.

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QUALITY CONTROL OF THE LABORATORY ENVIRONMENT

S. VOGT
International Atomic Energy Agency,
Clean Laboratory, Agency's Laboratories, Seibersdorf,
Seibersdorf, Austria

Abstract

A laboratory performing U and Pu analysis for safeguarding of legal limit compliance has to demonstrate the utmost care to avoid any sources of cross-contamination and source deterioration during sample handling to satisfy the customers' requirements. An example is given, from the IAEA Safeguards Laboratory in Seibersdorf, how a clean room is designed to accommodate the TIMS facilities for U and Pu analysis in the femto- or attomole range. Continuous blank monitoring and particle counts are mandatory to ensure absence of any accidental sources of contamination.

1. INTRODUCTION

The objectives of quality control in the laboratory environment are to assist in maintaining and fine tuning a measurement process in a desired state of stability and reproducibility. In its pursuit, the demand for analytical data is steadily increasing. Decisions need to be made such as to the health of individuals, quality of the environment, or for verification purposes in nuclear safeguards that nuclear materials are not diverted from declared nuclear activities. Modern, highly sophisticated instrumentation allows rapid measurements as low as in the femto- to attomole range. The quality of analytical data is normally evaluated on the basis of its uncertainty when compared with requirements of their final use. If data are consistent and the uncertainty is small when compared to the requirements, the data are considered of adequate quality. Data with highly variable uncertainties are normally viewed of low or inadequate quality.

Achieving quality control objectives require a thorough QC programme of the laboratory environment. Safety in the laboratory, efficiency in performing a requested task, preventive maintenance to avoid expensive repair works, and customer satisfaction are important goals of such a QC programme. In trace and ultra-trace analytical work, whenever blank corrections are significant, special emphasis of such a programme must be given to blank control. The extent of control over the analytical blank, reflecting the contamination from all sources external to the sample, can seriously affect the accuracy of low level trace determinations. Most sources contributing to the blank are variable, and it is this variability that determines the uncertainty of the blank correction and often the limit of quantification that can be determined reliably. To improve both accuracy and limit of quantification, it is imperative to control the variability of the analytical blank. The principal sources of contamination are the environment where the analysis is performed in, equipment used in the analysis, reagents used for the analysis, and analyst(s) performing the analysis.

2. CONTROLLED ENVIRONMENTS AND THE IAEA'S CLEAN LABORATORY

Controlled clean environments can be as simple as a laminar flow hood used to perform certain steps during sample preparation or as sophisticated as a clean room facility. Inflatable glove bags are cheap solutions for carrying out single steps during sample handling usually to avoid contamination of the sample or the environment with sample material. They do not, however, provide a controlled environment.

The IAEA's clean laboratory performs three major tasks in support of nuclear safeguards administered by the Department of Safeguards [1]. All sampling kits are prepared and assembled

under strict clean room conditions. Only materials identified to be devoid of the elements of interest to the safeguards efforts are used. All returned sampling kits are screened using radiometric and other techniques such as electron-microscopy (SEM-EDX/WDX), and finally archived. Sampling kits returned from inspections are delivered to the Clean Laboratory carrying only a bar code label, so as to ensure the sample's confidentiality. All returned materials are archived and distributed by the Clean Laboratory Unit. Control samples are maintained and administered by the Clean Laboratory, and are prepared under strict cleanliness requirements. Finally, the Clean Laboratory participates as a Network Analytical Laboratory for the Department of Safeguards in the analysis of inspection samples. Analytical requests are mainly for uranium and plutonium isotopes and elemental ratios in bulk samples using thermal ionization mass spectrometry (TIMS), and uranium isotopes on individual particles using secondary ionization mass spectrometry (SIMS).

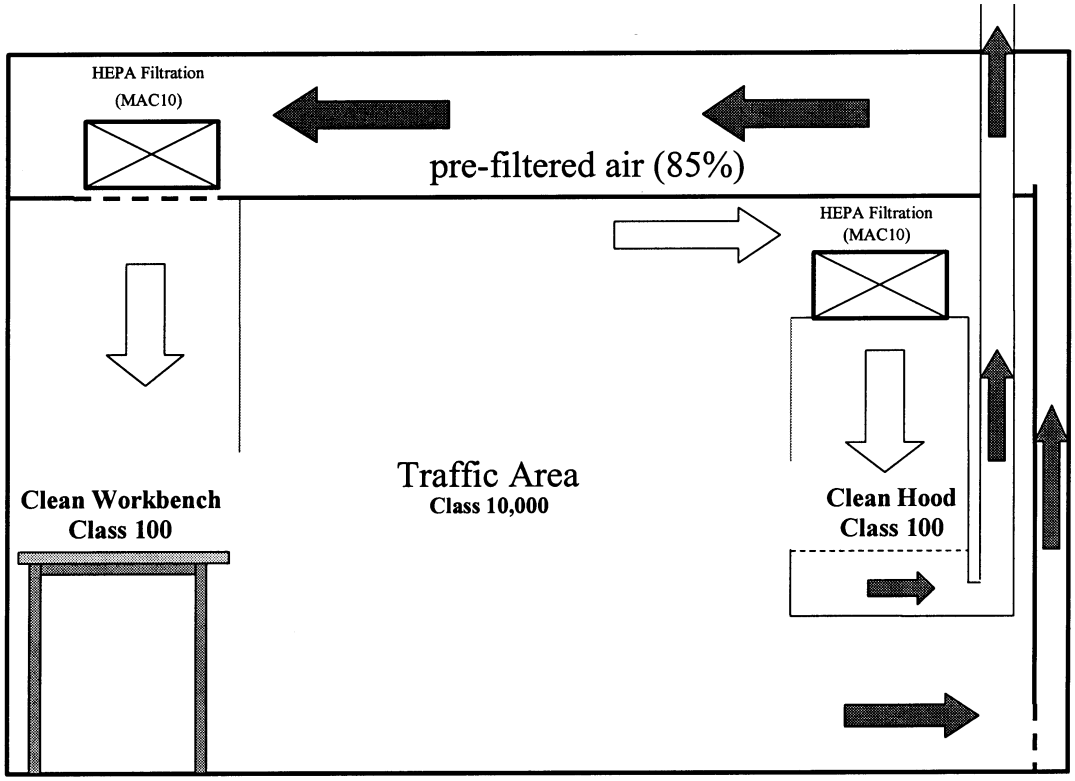

FIG. 1. Air circulation in the clean rooms [2].

The IAEA's Clean Laboratory uses dust filtered air at 22°C and 40–60% relative humidity. The pressure is higher than atmospheric pressure and is set up to 30 Pa in each chemistry room of the clean laboratory. The laboratory inclusive traffic areas and chemistry rooms, has a pressure gradient of 30 to 10 Pa [2]. In general, the 85% pre-filtered air comes from the plenum through the High Efficiency Particulate (HEPA) filter down to the bench and into the room. These filters supply air of class 100 (defined as ≤ 3500 particles of ≥ 0.5 μm per m^3 air) or better to the work areas, such as work benches and clean fume hoods, where open samples are handled. Traffic areas are at class 100000 without adding any additional filter units in these areas. A cross-sectional view of the air circulation in a typical chemistry room is shown in Fig. 1.

Because many of the measurements to be performed require determinations of multi-elemental isotopic composition it is important that potential sample contamination by metals be minimized. Consequently, all interior walls are from epoxy covered aluminum walls. Cabinets are constructed from wood and hoods from welded polypropylene. To minimize the possibility of introducing dirt generated by the deterioration of floor material into the air stream, the flooring is from seamless PVC.

In order to maintain controlled environments such as a clean room facility, it is necessary while in routine operating mode to develop and follow a formal set of procedures and work instructions. These encompass regulations such as access to all or certain parts of the facility, cleaning and gowning requirements for entering and working inside of the facility, equipment cleaning procedures as well as access restrictions for equipment. Appropriate training of the personnel working in the facility, including cleaning schedules and practices, is mandatory. To maintain blank levels at the desired levels, restrictions in the type and amount of materials, including chemicals, must be imposed. Table I presents some activity limits for open radioactive sources in the IAEA's Clean Laboratory. Preventive maintenance, such as changing the filter media and performing calibrations of sensors at fixed time intervals, will assist in the efforts to maintain the laboratory in continuous operating mode. Depending on the regulations and work performed in the controlled environment, re-certification to the original specifications at the inception of operation might be required. The IAEA's Clean Laboratory is re-certified annually by an independent clean room engineering contractor.

TABLE I. ACTIVITY LIMITS FOR OPEN RADIOACTIVE SOURCES IN THE CLEAN LAB

Isotope	Activity limit [Bq]	Specific activity [Bq/g]	Mass limit [ng]
^{234}U	3.7×10^3	2.29×10^8	1.62×10^3
^{235}U	3.7×10^4	7.77×10^4	0.48×10^9
^{236}U	3.7×10^4	2.35×10^6	1.58×10^7
^{238}U	3.7×10^4	1.23×10^4	3.0×10^9
^{238}Pu	3.7×10^3	6.44×10^{11}	5.75
^{239}Pu	3.7×10^3	2.26×10^9	1.63×10^3
^{240}Pu	3.7×10^3	8.36×10^9	4.40×10^2
^{241}Pu	3.7×10^3	4.14×10^9	0.89
^{242}Pu	3.7×10^3	1.44×10^8	2.56×10^3

3. RESULTS

3.1. Particle counts

To verify that a controlled environment is operating within specifications, certain measurements have to be performed routinely at fixed time intervals. The maximum permissible concentration of particulate matter in the ambient air is defined in the US Federal Standard 209E and similar standards, such as the new ISO 14644 series or the German standard as given in the VDI series 2083.

In the IAEA's Clean Laboratory five individual particle counts for each designated clean area per room are taken once every month using laser based particle counters. Table II shows data from a measurement campaign in the sampling kit preparation room of the Clean Laboratory. Presented are averaged counts for each area, workbench where the kits are physically assembled and traffic area of the room, before and after a six hour working day. As expected the particle counts increased sharply during the assembly of sampling kits. However, the higher particle counts after completion of the sampling kits are well within specifications of the US Federal Standard 209E for a Class 100 area ($\leq$10500 particles of $\geq$ 0.3 μm per m^3 air and$\leq$ 3500 particles of $\geq$ 0.5 μm per m^3 air).

TABLE II. PARTICLE COUNTS TAKEN IN ROOM 15 OF THE CLEAN LAB IN THE TRAFFIC AND WORK AREA BEFORE AND AFTER ASSEMBLY OF SAMPLING KITS

Particle size [µm]	Particle counts Workbench (Class 100) (before)	(after)	Particle counts Traffic area (Class 10000) (before)	(after)
0.3	0 ± 0(0)	3990 ± 4095 (5705)	1085 ± 525(1295)	104440 ± 14525(110530)
0.5	0 ± 0(0)	420 ± 525(630)	12 ± 10(17)	3815 ± 595(4095)
1	0 ± 0(0)	35 ± 35(35)	245 ± 140(280)	174 ± 35(175)
5	0 ± 0(0)	0 ± 0(0)	0 ± 0(0)	0 ± 0(0)

Particle counts are given in number of particles per m^3 with standard deviation (n=5), and the 95% UCL (in parenthesis).

3.2. Blanks

It is not sufficient for the tasks of the IAEA's Clean Laboratory to rely on particle counts to prove that the ambient environment is free of contamination. While the particle counts can be considered as a more qualitative measure, the routine measurement verifying the absence of uranium in the ambient air is mandatory. This type of blanking is called *room blank*. The detection limit for uranium using TIMS is about 10^7 atoms (5×10^{-15} g), and just one particle of UO_2 with a diameter of 1µm contains 10^{10} atoms (10^{-11}g) of uranium [3]. This shows the importance of performing room blanks routinely, to verify that no contamination of sampling kits occurred during their assembly and that no elevated uranium concentration in the ambient environment might alter the analytical efforts, i.e. give rise to episodic or continuous high blank levels.

Verification of the absence of uranium or establishing its very low concentration in reagents (*reagent blank*) and equipment (*equipment blank*) used during the chemical sample preparation for bulk analysis by TIMS is also mandatory. A typical example is the *loading blank* to verify that the solutions used during the filament preparation, the filament, and the ion source of the mass spectrometer, are free of contamination. Fig. 2 shows a control chart for the loading blanks of uranium using the ^{233}U enriched spike used during routine sample preparation.

In addition to particle counts and the elaborate blanking efforts, routine analysis of **control samples** has been added recently to evaluate the robustness, i.e. accuracy, precision, and reproducibility, of the overall analytical procedures. **Control charts**, as the one presented in Fig. 2., showing the accuracy, precision, and the variables contributing to the background of the analyses assist in obtaining statistical quality control.

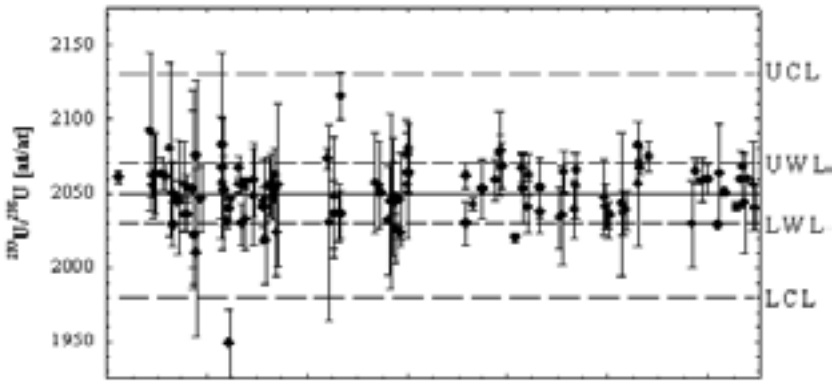

FIG. 2. Results of loading blank measurements of ^{233}U enriched spike IRMM 04/01 on the Finnigan MA T262.

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ISOTOPE SPECIFIC ANALYSIS AND CLEAN REGIME

J.R.W. WOITTIEZ, J.E. SLOOF
Netherlands Energy Research Foundation,
Petten, Netherlands

Abstract

Isotope specific clean regimes for accurate isotope specific analysis are based on the awareness of the conflict that arises when a sample is introduced in the clean area. Sample matrix, spikes and standards will inevitably contaminate the clean-lab and facilities. The conflict can be controlled by acknowledging that each analytical problem demands its own specific clean facilities. The use of a "sub-unit", being a small, stand alone, temporarily dedicated collection of clean facilities, is suggested as a practical approach. The validity of the concept is demonstrated by successfully solving two Isotope Specific Analytical problems using dedicated sub-units. One is the determination of trace amounts of uranium, cadmium and chromium in municipal wastewater. The other is the determination of uranium and its isotopic composition in nuclear waste material.

1. PRINCIPLES

Each analysis is subjected to systematic errors. These errors can arise in all phases of the analysis. During sampling, transfer, storage, preparation of the sample and actual detection, sample-specific changes or analyte-specific changes will occur. Contamination is an important cause of analyte-specific changes. Contamination could be defined as an unintentional addition of the analyte of interest to the sample through exogenous sources. Addition of the analyte has to be done intentionally in case of spiking such as in isotopic dilution measurements, radiochemical separations or tracer experiments, using either stable or radioactive tracers. Strictly speaking, the definition should thus be extended to an unintentional and unknown change of concentration and isotopic composition of the analyte of interest by exogenous sources. Sometimes the analysis is focused on the chemical form of the analyte. Now a rather complete definition of contamination could be an unintentional, unknown change of concentration, isotopic composition or chemical form of the analyte of interest by exogenous sources.

To minimize this unintentional influence of exogenous sources, many technical solutions have been developed during the past decades, leading to the well established and documented concept of clean laboratories equipped with clean facilities. A comprehensive review is given by Iyengar, Subramanian and Woittiez [1]. A summary on exogenous sources of contamination and their impact is given in this contribution.

The principle of the concept "clean lab and facilities" is to protect any sample against an environment that potentially contains one, some or a large number of contaminants. To maintain the validity of this concept, it is necessary to apply the same "philosophy of protection" to the clean lab and facilities themselves, i.e. to consider the clean lab and facilities just as vulnerable to contamination as the sample. Introduction of any sample into the clean lab and facilities for execution of analytical procedures inevitably means introduction of contaminants via the sample matrix. Introduction of contaminants also occurs with standard addition and spiking procedures. Processing the sample outside the clean lab and facilities violates the protection of the sample. It is clear that application of the "philosophy of protection" to both a sample and clean lab and facilities will lead to a protection conflict. The importance of the conflict may vary.

In this paper, a more refined set of principles is proposed to meet the contradictory demands of isotope specific analysis and clean labs and facilities. The practical use of these specific principles is demonstrated in applications on the determination of chromium, cadmium and uranium by neutron activation analysis (NAA) and uranium assay and uranium isotopic ratio determination by thermal ionization mass spectrometry (TIMS) and isotopic dilution mass spectrometry (IDMS).

2. EXOGENOUS SOURCES OF CONTAMINATION AND THEIR IMPACT

Well known exogenous sources of contamination are air particulate matter, the analytical laboratory, analytical personal, chemicals and equipment (including inappropriate cleaning procedures).

A clean air environment is defined by the maximum number of particulates per m^3 of a certain size. The US standard for permissible particle size distribution defines different classes, Class 100 being the most stringent. Class 100 environments should have no particles larger then 5 mm, not more then 10 particles per ft^3 (350 per m^3) larger then 2 mm and not more then 100 particles per ft^3 (3500 per m^3) larger than 0.5 mm. For a Class 1000 environment these numbers are 10, 100 and 1000 respectively. This division in classes not only refers to particle sizes, but intrinsically also to the total mass of airborne particulates; it is clear, that 10 mm particulates have bigger masses than 0.5 mm particulates. Class 1000 labs are acceptable as non-laminar-flow clean labs, while class 100 is the requirement for laminar flow boxes. Not only the number and size of particles are importance with respect to contamination, but the composition of the particles is just as vital.

All constructive material inside the analytical laboratory (walls, floors, ceiling, doors) and all furniture should be made from polymers or coated with epoxy resin. Concrete, paint, stainless steel, etc. will eventually become sources of contamination by corrosion.

The analysts' skin, hair, clothes and shoes are always a source of contamination. The entrance of the clean lab is usually a chamber where special clean lab clothing and shoes, have to be put on. To minimize transfer of contamination from the entrance to the clean lab, the analyst has to step on cleanable sticky mats on the clean lab floor, which adsorb dust particulates. Entering the clean lab is quite an operation. In practise, the elaborate protocol in the entrance enhances the cleanliness of the clean lab; it serves as a real and a psychological barrier.

A strict regime on allowed activities has to be maintained, which determines what equipment and chemicals are needed inside the clean lab.

A refrigerator may be needed in the clean area, but its heat exchange unit is a notorious source of dust. The same is true for the vacuum unit of a freeze-dryer. Here also, oil vapour from the pump is the unavoidable consequence. A balance is necessary and should be placed in a laminar flow box. Preferably, open dry or wet ashing should be done in a clean lab to prevent contamination with particles from ordinary laboratory air, thus a hot plate (ceramic instead of stainless steel) or oven is obligatory. To do so, a laminar flow hood has to be installed. Stainless steel cods of Teflon-lined pressure vessels are eventually corroding and should not be used or stored in the clean-lab.

No chemicals should be allowed but high purity water and acids used for wet ashing. Standard solutions, stabile or radioactive spike solutions, pure elemental compounds, their oxides or salts are prohibited. Also, ordinary cleaning products like detergents, soap, abrasives, etc. should not be used. Cleaning is to be performed by water and non-fraying textile.

3. "PHILOSOPHY OF PROTECTION" CONFLICT

Analytical procedures for the determination of trace amounts of isotopes (either for isotopic ratio determination, element determination, or activity determination) demand clean facilities. Maintenance of clean facilities asks for a protection philosophy similar to that for samples. A "philosophy of protection" conflict arises with the introduction of the sample in the clean lab and facilities.

Three illustrations of such conflicting situations are given here:

- One example is the determination of elemental impurities (Cr, Zn, Cu, Mn, Ni, Tl, U etc.) on the lower ng/g level in isotopically enriched pure CdO. Determination of the impurities asks for sample preparation using clean facilities; introduction of hundreds of milligrams of the CdO sample in the clean facilities results in a contamination with Cd with deviating isotopic composition. This is a disaster for any future trace determination of Cd using isotope specific techniques.
- A second example is the determination of a few ng/g of Pt in road dust. The Pt determination on this level asks for clean facilities, while the road dust matrix contaminates these facilities with many transition elements to an extent which exceeds many times the contamination by air-particulate matter from an ordinary chemical laboratory.
- A third example is the determination of less than 1 Bq amounts of Cs-137 as an impurity in a solution containing MBq's of some "pure" radionuclide. The determination of Cs-137 asks for clean facilities. Processing of MBq of the "pure" radionuclide needs radiochemical facilities that have been designed to face radioactive samples of variable nature, amongst which are spent fuel samples containing MBq's of Cs-137. Cross-contamination is inevitably present here.

A practical approach in solving the "protection" conflict for the analyst is to acknowledge the specific nature of each analytical problem and attribute selected clean facilities to that particular problem. Therefore, clean facilities are subjected to permanent re-design. Preferably, a collection of small, temporary, problem devoted, stand alone clean facilities — so-called sub-units — have to be designed in the clean lab to protect the sample against the environment, as much as to protect the clean environment against the sample. This means that digestion and chemical separation take place in devices that have no contact to the outside world, i.e. closed systems. When necessary each sub-unit is provided with its own balance for weighing. Once all necessary analyses have been done, the unit is dismantled, decontaminated and, as far as applicable, disposed. In order to minimize the risk for cross-contamination, as many disposable clean facilities as possible would be required, and a rigid, specified decontamination regime when non-disposable clean facilities have to be re-used. A consequence will be an increased production of solid and liquid (radio)chemical waste. The sub-unit approach, when applied in a clean lab, may consequently enhance the exploitation costs of the clean lab. However, when the approach is tried in a normal chemical lab equipped with selected, appropriate clean facilities, it will be cost effective compared to the construction of a complete clean-lab. Future developments in clean facilities and waste management may even further lower the cost-effect ratio.

4. APPLICATIONS

Two examples are presented which show application of clean sub-units with closed systems. One refers to the determination of selected trace elements in municipal wastewater, the other to the determination of uranium and its isotopic composition in nuclear waste material.

In both examples emphasis is laid on the description of the closed systems.

4.1. Neutron activation analysis of trace elements in aqueous samples

The chemical principles of the technique are based on the work of Greenberg and Kingston [2]. They developed a method to concentrate transition- and rare earth elements from natural waters on a chelating agent and determined the elemental contents by neutron activation analysis of the agent. At the Isotope Specific Analytical Laboratories of NRG-Petten, the design of the method was modified as to make it a permanent clean sub-unit. This sub-unit was recently applied for the certification analyses of ng/g amounts of uranium, cadmium and chromium in municipal waste water.

The heart of the sub-unit is a twelve channel liquid chromatograph, based on a twelve channel peristaltic pump. Each channel is completely isolated from every other channel, to avoid cross-contamination. All parts of the chromatograph, including the frame, are made of polymeric materials. Samples only contact Teflon (TFA), Tefzel and Polyethylene (PE). A picture is shown in Fig. 1 and a simplified scheme in Fig. 2.

The first and second channels of the chromatograph are always used for multi-element standards, processed as samples. The results are used to check chemical recoveries. Aliquots of the same standards are pipetted directly on the chelator, which is irradiated without further processing, to serve as the reference for the specific sample batch. The third channel is always for a certified reference material (CRM) and the fourth (and if necessary the twelfth) channel, for blanks. Channels 5–11 are used for samples.

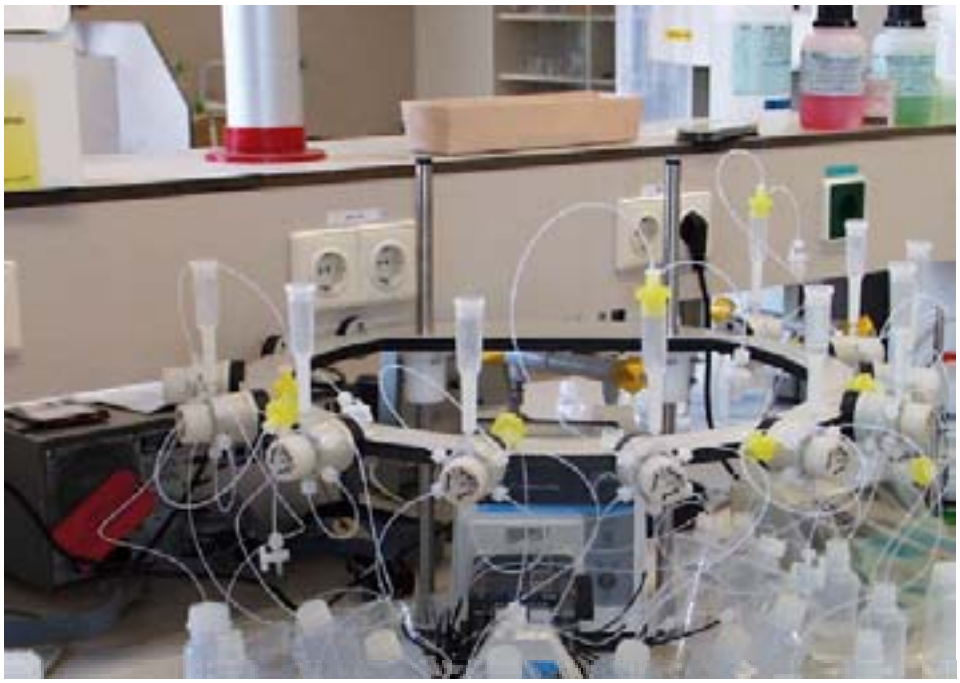

FIG. 1. Image of the twelve channel chromatograph.

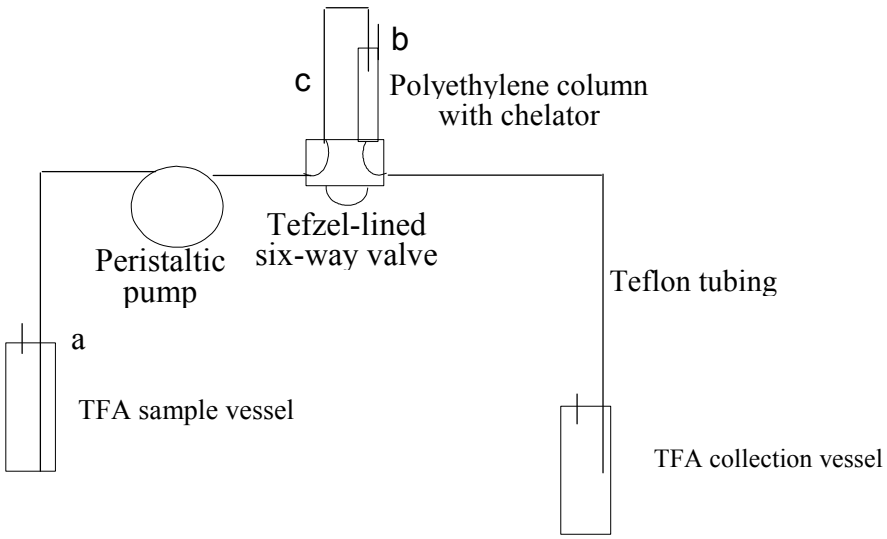

FIG. 2. Scheme of one of twelve identical channels of a chromatograph for pre-concentration of trace elements.

Each sample to be processed is transferred to the appropriate TFA sample vessel and weighted. This happens in a dedicated laminar flow box. The TFA sample vessel is closed with a screw cap which contains Teflon tubing already connected to the tubing of the peristaltic pump (cf. Fig. 2, item a). From this moment on the sample is isolated from the outside world.

Subsequently, the PE column is connected directly to the six way valve. The column is filled with the chelator and closed with a PE snap cap. From this moment on the chelator is isolated from the outside world. The cap contains a connection for both the Teflon tubing and PE syringe with PE cap (cf. Fig. 2, item b). The chelator is cleaned and equilibrated using a syringe.

Next, the column is connected to the Teflon tubing from the six-way valve (cf. Fig. 2, item c) and the pump is started. In this way, the sample passes the column without experiencing the outer world.

After sample loading the chelator is washed to remove interfering elements like Na, K, Ca, Ba, Cl and P. The chelator now only contains transition, rare earth, and actinide elements from the sample.

Finally, the PE column with chelator is provided with a new PE cap, removed from the chromatograph and provided with a PE foot closure. Without transferring the chelator, the closed PE column is irradiated, i.e. the PE column serves as the irradiation containment of the chelator. Only after the irradiation, the chelator is removed from the column to a suitable counting vessel and analysed by low energy, low background gamma ray spectrometry.

By operating as described, both sample and chelator do not experience any outer world between the moment they are transferred to their respective containment and the end of irradiation. Likewise, the outer world does not experience anything of the samples and standards processed in the chromatograph.

Table I shows some results recently obtained with this system for the analysis of uranium, cadmium and chromium in municipal wastewater. Also values for blanks and the Canadian CRM NASS-2 sea water are given.

TABLE I. RESULTS OBTAINED WITH THE 12-CHANNEL CHROMATOGRAPH

Sample	U-content (µg/kg)	Cd-content (µg/kg)	Cr-content (µg/kg)
Blank	0.010 ± 0.003	0.002 ± 0.002	1.0 ± 0.2
NASS-2 sea water	3.28 ± 0.10	<0.1	<1
Certified value	3.00 ± 0.15	0.029 ± 0.004	0.175 ± 0.010
Municipal waste	0.68	5.24	19.6
ibid.	0.63	4.90	16.3
ibid.	0.68	4.62	19.1
ibid.	0.65	4.59	17.8
ibid.	0.71	4.50	20.1
Average ± st. dev	0.67 ± 0.03 (N = 5)	4.77 ± 0.30 (N = 5)	18.6 ± 1.5 (N = 5)
Intercomparison average	not known	5.39 ± 0.77	20.2 ± 3.5

It is clear from the results that the sub-unit approach works properly. It only generates procedural blank values on (Cr) or far below (U and Cd) the µg/kg level. It is also shown on Table I that the analytical results for uranium, cadmium and chromium on the lower µg/kg level in aqueous samples are satisfactory when compared to the certified values or inter-comparison averages.

4.2. Determination of uranium and its isotopic composition in nuclear waste material by TIMS/IDMS/NAA

Nuclear waste material that is high in Cs-137 and Sb-125 activity had to be analysed for uranium and its isotopic composition. The uranium content was expected to be a few mg/kg. The high radioactivity of the sample, unknown uranium isotopic composition and low uranium content ask for a "clean sub-unit approach".

The sub-unit was constructed from a dedicated laminar-flow box, a closed quartz Bethge-type digestion system in a dedicated fume-hood and a 1 channel/multi-eluent chromatograph (BioRad Biologic-LP). After mineralization, uranium is separated from the sample by a solid-liquid extraction column chromatographic procedure using a diamyl-amylphosphonate column. Detection is done by TIMS and NAA.

Samples are mineralized in a quartz Bethge device. Its use is extensively discussed elsewhere [1]. The relevant feature here is the complete isolation of the sample from the outside world during operation.

For the solid-liquid extraction column chromatographic procedure, use is made of a one channel/multi-eluent chromatograph (BioRad Biologic-LP).

A schematic view of the chromatograph is given in Fig. 3.

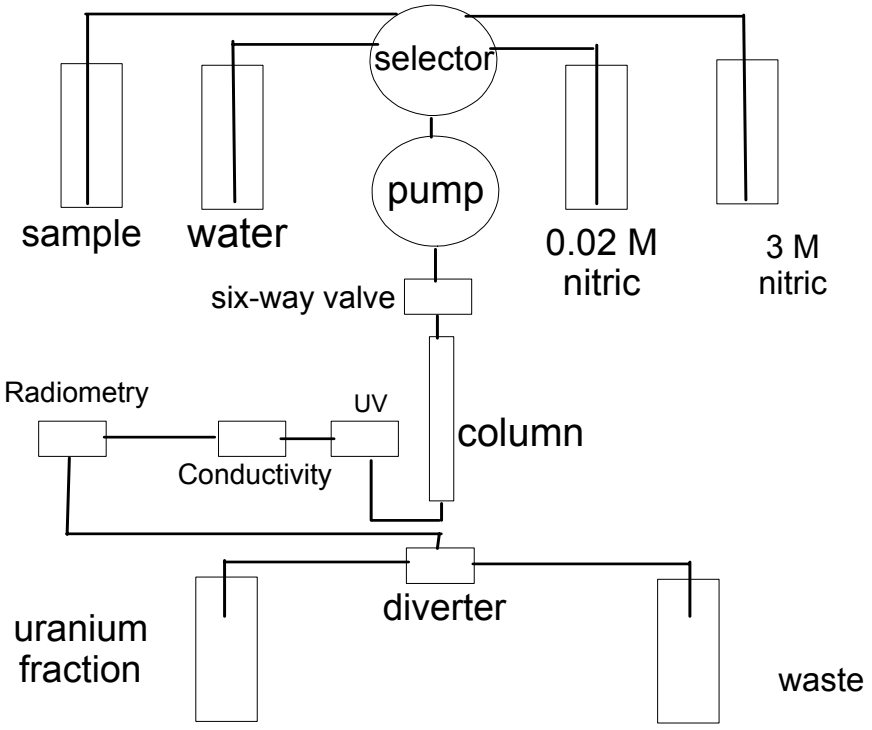

FIG. 3. One-channel/multi-eluent BioRad Biologic LP chromatograph for the separation of uranium from spent fuel.

The Biologic LP functions as a stand-alone apparatus. By remote control several different eluents, amongst which is the sample, can be pumped through the column. The chromatograph runs a sequence of loading, washing end elution steps fully automatically. The performance of the separation is followed on-line by UV- and conductivity monitoring, as well as by on-line radiometry. All eluent vessels are made from TFA, all tubing from Teflon, the column container is made of PE and the selector, six-way valve and diverter are all Tefzel lined.

In the laminar flow box, a sample is weighted in a newly prepared quartz vessel, which is part of the Bethge device. When samples are meant to be for the uranium determination with IDMS, spiking is also done here. The quartz vessel is closed with a stopper and transported to the Bethge device for mineralization.

After mineralization, the sample is evaporated to near dryness and diluted in the Bethge device. The quartz vessel is removed from the Bethge device, closed with a stopper, transported to the laminar flow box and its content transferred to a TFA sample vessel. The TFA sample vessel is closed with a PE screw cap, already containing Teflon tubing. It is placed inside the chromatograph and the tubing is connected, which isolates the sample from the outside world.

The PE column container is filled with the phosphonate material and connected to the six way valve. From this moment on, the phosphonate no longer experiences the outside world. The column is washed and equilibrated using water, 0.02 M nitric acid and 3 M nitric acid.

The sample is passed through the column. Fission-and activation products and most elemental impurities are removed by washing with 3 M nitric acid. Uranium is recovered by eluting the column with 0.02 M nitric acid. The column is treated as nuclear waste.

After each run, both the Bethge device and chromatograph are decontaminated by extensive washing procedures. Each sample has its own quartz vessel, TFA sample vessel and column.

Aliquots of the uranium fraction are used for uranium isotopic analysis by TIMS or NAA and for uranium assay by NAA. In case of a spiked sample, the fraction is used for uranium assay by IDMS. Table II shows selected results.

TABLE II. SELECTED RESULTS OBTAINED WITH THE BIOLOGIC LP SINGLE CHANNEL CHROMATOGRAPH FOR THE DETERMINATION OF URANIUM CONTENTS AND ISOTOPIC COMPOSITION IN PARTIALLY PROCESSED SPENT-FUEL SAMPLES

Sample number	Isotopic abundance of main U isotope by TIMS (at%)	Isotopic abundance of main U isotope by NAA (at%)	Uranium content by IDMS (mg/kg)	Uranium content by NAA (mg/kg)
1	90.735	90.8	0.812	0.86
Duplicate	90.719	91.4	0.813	0.80
2	91.324	92.6	0.894	0.86
Duplicate	91.314	91.4	0.895	0.85
3	91.291	93.1	1.043	1.06
Duplicate	91.338	92.8	1.046	1.05
4	91.425	92.8	0.975	0.96
Duplicate	91.422	93.4	0.975	0.96
5*	ND	ND	ND	0.010
Duplicate	ND	ND	ND	0.010
Blank	ND	ND	ND	0.006
Duplicate	ND	ND	ND	0.006

* = chromatographic waste fraction re-analysed.

It is obvious from the Table, that the "clean sub-unit" approach generates reproducible and accurate results. Results obtained by NAA, though less precise, support the results obtained by TIMS/IDMS.

The major pitfall in using a chromatograph for chromatographic separation lies in memory effects (cross-contamination) between samples. Valves, selectors, tubing and on-line detectors all may absorb uranium from one sample and deliver it to the next one.

A similar reasoning is valid for the quartz Bethge device. The first sign of (cross-) contamination would be non-reproducibility in the isotopic composition of duplicates.

It is demonstrated here, that a conscientious use of digestion equipment and a single-channel analyser, with thorough cleaning and decontamination procedures between samples, results in reliable determinations of µg amounts of uranium and its isotopic composition and thus that memory effects can be controlled.

5. CONCLUSIONS

It is clear from the results that a sub-unit approach can be used successfully for accurate isotope specific analysis, both in the field of elemental analysis on the lower µg/kg level and in the field of isotopic composition analysis. The concept of small, dedicated, temporary sub-units may generate increased amounts of (radio)chemical waste, but it might prove to be a cost-effective practical alternative to a complete clean lab.

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PROJECT PLANNING FOR AN ENVIRONMENTAL CLEAN LABORATORY: A SUMMARY OF REQUIREMENTS AND APPROACH

R.E. PERRIN[1]
Edgerton, Germeshausen and Grier (EG&G), Inc.,
Littleton, Colorado,
United States of America

Abstract

Planning for the design, construction and operation of an environmental clean laboratory is a complex operation. Key elements of the project are process engineering, laboratory and facilities, management and infrastructure, and demonstration of operability. This paper describes in summary detail the overall approach and work processes that have been used to develop the systems requirements for a facility now under construction.

1. INTRODUCTION

The purpose of this paper is to describe the requirements and activities associated with planning for a highly sensitive environmental clean laboratory that can handle and analyse micron-sized samples of nuclear material collected from the environment. This paper describes in summary detail the overall approach and work processes in following a systems engineering requirements concept. It is designed to assure that all elements of project planning are reflected in the design and operations objectives for the facility. For illustration purposes, work performed under a co-operative bilateral agreement between the US Department Of Energy (US DOE)/Los Alamos National Laboratory (LANL) and the Japan Atomic Energy Research Institute (JAERI) is referenced to illustrate the complex planning requirements necessary to establish a functioning clean laboratory that will perform with the required sensitivity and accuracy. This example, though specific to the JAERI objectives, represents those requirements, regardless of application, that must be addressed from a planning basis to assure that an operable laboratory exists.

The USA is assisting Japan to design, construct and operate a clean laboratory. The laboratory is to provide the following capabilities:

(1) Capability for low-level nuclear isotopic measurements specifically aimed at analysis of nuclear safeguards environmental samples

(2) Capability to support nuclear research by internal JAERI staff and visiting researchers

(3) Future expansion capability into analysis of particles and noble gases collected at monitoring stations in the Japanese territory.

This assistance is being provided through action sheets or bilateral agreements written jointly by JAERI and the US DOE. Los Alamos National Laboratory has the prime responsibility for the US effort. Radian International and now EG&G have provided much of the engineering and technical support to the project.

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[1] Mailing address: P.O. Box 6925, Woodland Park, Colorado 80866-6925, USA.

2. PROJECT MODEL

Fig. 1 shows the model that was developed for defining the systems requirements for the project. The key elements of the project are process engineering, laboratory and facilities, management and infrastructure and demonstration of operability. The progress of the project is shown from top to bottom. Co-operative two way interactions between elements are shown horizontally in the diagram. The process engineering phase precedes the "laboratory and facilities " and "management and Infrastructure" elements and is interactive through quality assurance in all phases of the project. A similar project model will be helpful for any entity wishing to plan, construct and operate a similar facility.

To support any such facility, specific activity based processes are required. These processes combine laboratory and facility resources, protocols and procedures, and trained staff to produce a desired output. Process engineering develops the process flow diagrams that guide laboratory and facilities design, specifies instrumentation, materials and equipment, and guide personnel recruitment, training and procedure development.

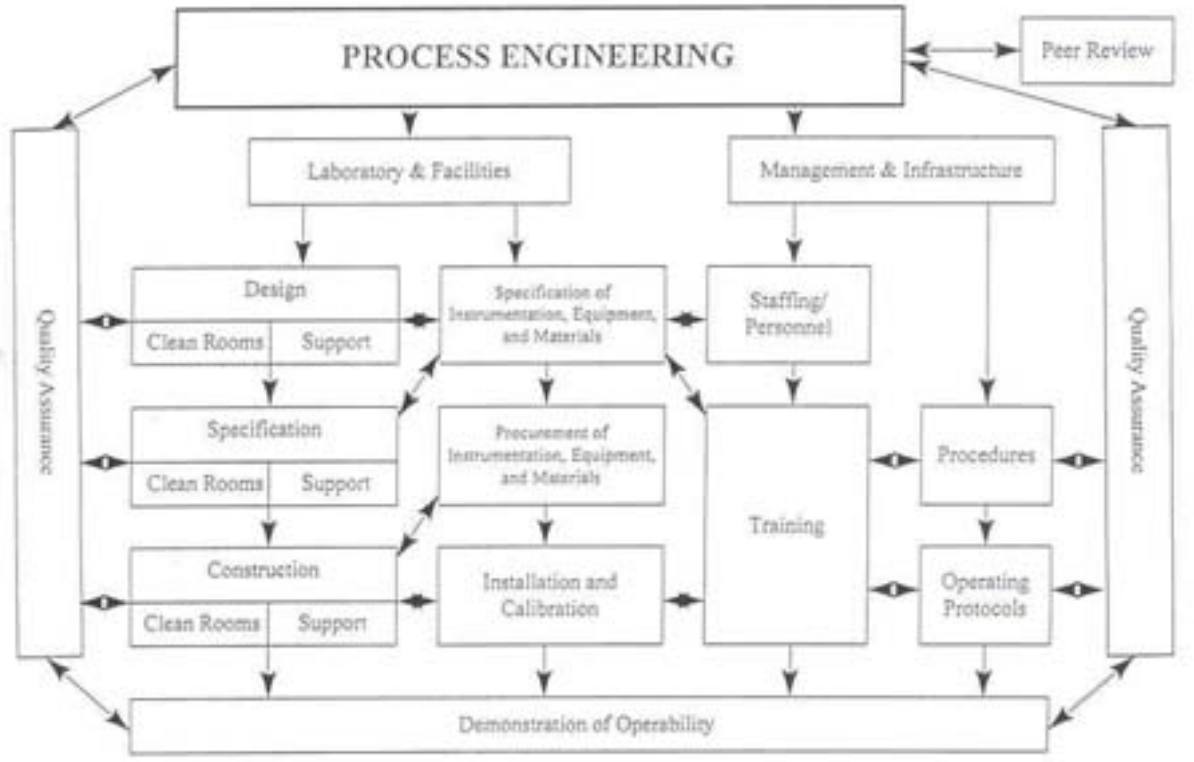
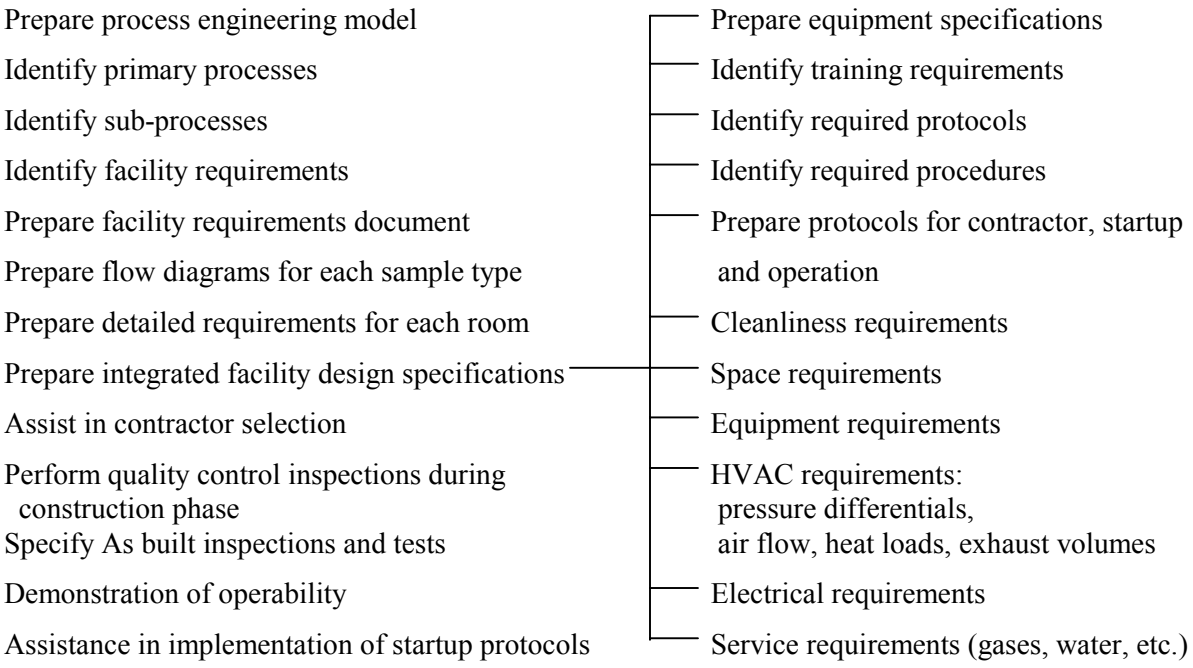


FIG. 1. Project model.

3. ASSISTANCE ACTIVITIES

What follows diagrams the activities that have developed from application of the project model. It will be helpful to discuss a number of these activities in detail to demonstrate the scope of the effort required to provide adequate assistance.

Example: assistance activities for clean room planning



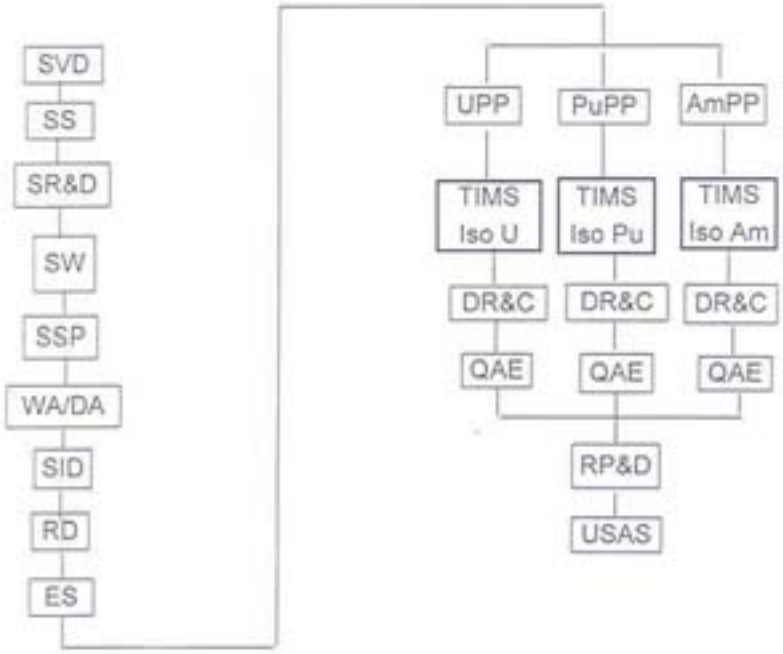
We have already discussed the development and application of the project model. Identification of the primary processes required several interactive meetings between the JAERI staff and the assistance team. Extensive written reviews of proposals preceded each meeting. The primary processes required to meeting the goals of the laboratory are.

- personnel support
- sample receipt and management
- bulk analysis
- particle analysis
- material receipt and control
- reagent management
- spike preparation and calibration
- reference material and management
- sample kit preparation, validation and distribution
- TIMS analysis
- SIMS analysis
- SEM/X ray analysis
- ICP-MS analysis
- high resolution gamma spectrometry analysis
- liquid scintillation alpha and beta analysis
- alpha spectrometry analysis
- demonstration of facility operability.

Each of these primary processes requires detailed specifications, operating protocols and personnel training plans for successful completion of the project. Each primary process includes a number of sub-processes that must be identified and evaluated. Table I illustrates the sub-processes required for one primary process. Each of the 17 primary processes required evaluation similar detail. In addition to identifying the sub-process, their effect on facility design, equipment specifications, staffing and training, the requirements for procedures and protocol preparation were estimated. This process identified over 150 separate sub-processes. Many of these were common to several primary processes.

TABLE I. PROCESS REQUIREMENTS SUMMARY ILLUSTRATION

Sub-processes	Project	Generic	Clean Lab.	Support	Proc.	Staffing and Training	Protocol
Data validation	X		X			X	X
Gross activity	X		X		X	X	X
Coding & analysis requests	X		X			X	X
Alpha screening	X		X		X	X	X
Beta gamma screening	X		X		X	X	X
Passive neutron screening	X			X	X	X	
Cleaning and repacking	X		X		X	X	X
Interim storage	X		X		X	X	X
Archive storage	X		X			X	X
Final disposal		X		X	X	X	



SVR	=	Sample receiving validation and packing
SS	=	Sample screening analysis
SR&D	=	Sample request preparation evaluation
SW	=	Sample weighing
SSP	=	Sample spike addition
WA/DA	=	Wet ash/dry ash
SID	=	Silicon destruction
RD	=	Residue dissolution
ES	=	Element separation
PP	=	Product purification
TIMS	=	Thermal ionization mass spectrometry
DR&C	=	Data reduction and calculation
QAE	=	Quality assurance
RP&D	=	Report preparation and distribution
USAS	=	Unused sample to archive storage.

FIG. 2. Flow sample diagram of aerosol sample.

Flow diagrams were then prepared for each of the sample types expected to be received in this laboratory. The most simple of these diagrams is shown in Fig. 2.

These diagrams were found to be very useful in identifying sub-processes and in tracing the flow of the sample through the facility. In some cases rooms were relocated to optimize the sample flows. A more complex sample flow diagram follows is shown in Fig. 3.

For this sample type, several treatment options are available. Each can yield different information from the same sample. The resources for each treatment option must be provided for in the facility design. Complete flow diagrams can assure that this is done.

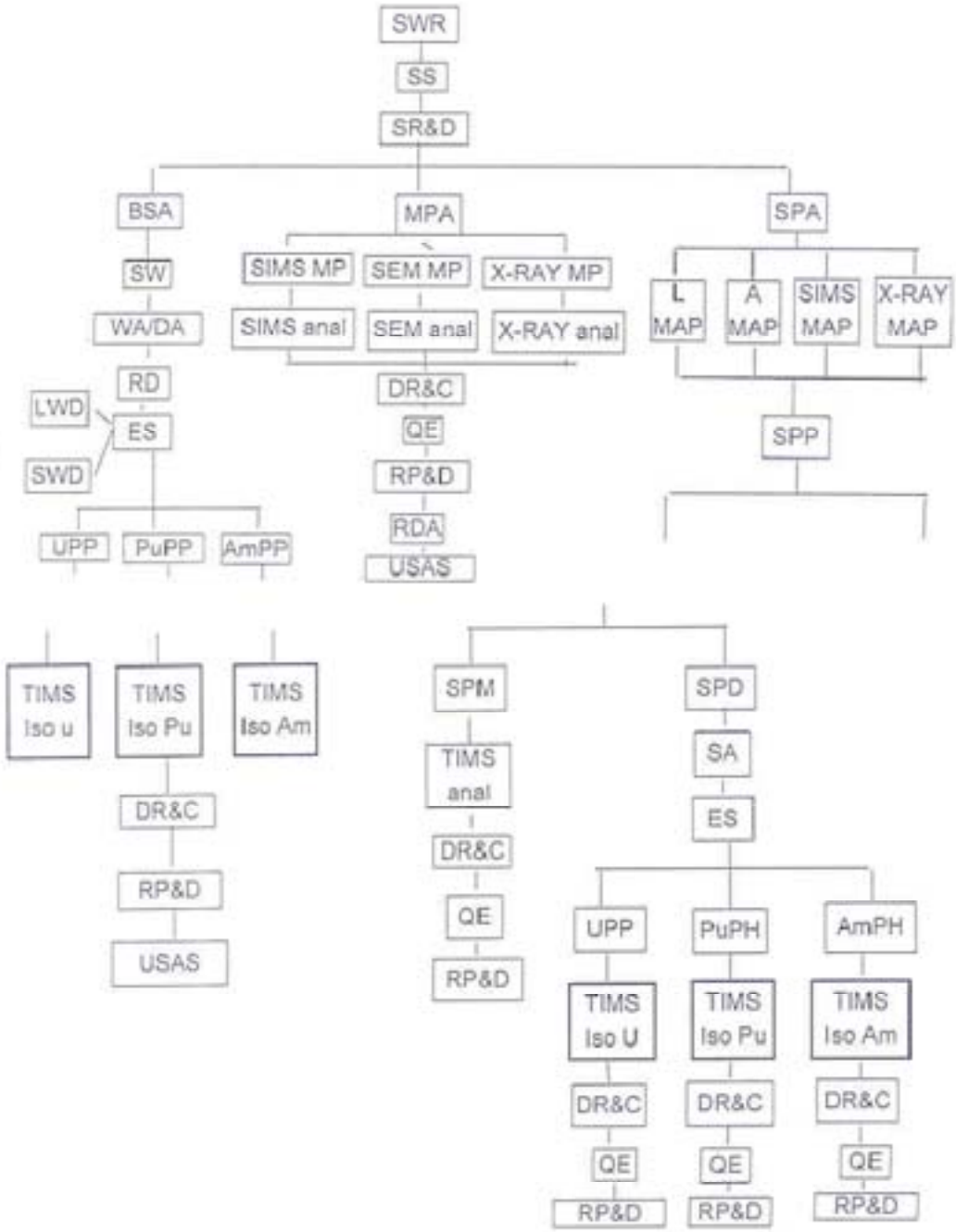


FIG. 3. Flow diagram for smear samples.

4. DESIGN ACTIVITIES

Based on the information gathered during the development of the primary process lists, sub-process lists, and flow diagrams, a listing of facility requirements was prepared. Table II illustrates one page of this list. A total of 47 separate facility requirements were identified for this project.

TABLE II. FACILITY REQUIREMENTS ILLUSTRATION

Facility	Location			Processes served
	Clean room	Attached support	Other support	
1. Sample receiving and repacking room	X			6,7,8,14,15,17
2. Sample storage room	X			10–17
3. Sample			X	7–17
4.Particle recovering and mounting room	X			3,4,8,10–13,16,17
5. Particle picking room	X			3,4,8,10–13,16,17
6. Men's change area	X			1
7. Women's change area	X			1
8. Toilet area	X			1
9. Shower area		X		1
10. Materials cleaning and repacking	X			3–17
11. Chemical separation room (3 required)	X			3–10, 13,17
12. Wet and dry ashing room	X			3–13, 15,17
13. Balance room	X			1,3, 6–12, 15,17
14. Spike and reagent preparation room	X			3,4, 6–10, 16, 17
15. Radiometric measurements room	X			1, 3–9, 12, 14–17

From this list, a "Systems Requirements Document" was prepared. This document contains details concerning all the items outlined below. This document forms the basis for all agreements required for US support and assistance to Japan on this project, and for design of the facility.

Standard clean laboratory systems requirements document:
Table of contents

2.0 PROCESS ENGINEERING

- 2.1 Process engineering design
- 2.2 Sample handling and management
- 2.3 Material handling and management
- 2.4 Personnel support
- 2.5 Laboratory and facility maintenance
- 2.6 Analytical production

3.0 LABORATORY AND FACILITIES

- 3.1 Design requirements and considerations
  - 3.1.1 Facility layout
  - 3.1.2 Laboratory facility requirements
  - 3.1.3 Interfaces with the JAERI Tokai facility
  - 3.1.4 Permits and approvals
- 3.2 Design
  - 3.2.1 Site work
  - 3.2.2 Arcitectural
  - 3.2.3 Air recirculation system
  - 3.2.4 Required utilities
  - 3.2.5 Mechanical
  - 3.2.6 Communications system
  - 3.2.7 Electrical
  - 3.2.8 Landscaping
- 3.3 Specifications
  - 3.3.1 General requirements
  - 3.3.2 Site work
  - 3.3.3 Architectural
  - 3.3.4 Construction materials
  - 3.3.5 Specialties
  - 3.3.6 Furnishings
  - 3.3.7 Special construction
  - 3.3.8 Conveying systems
  - 3.3.9 Mechanical
  - 3.3.10 Electrical
  - 3.3.11 Communication systems
  - 3.3.12 Landscaping
- 3.4 Construction
  - 3.4.1 Contractor approval
  - 3.4.2 Requirements verification
  - 3.4.3 Change control
  - 3.4.4 Schedule
  - 3.4.5 Interface issues
  - 3.4.6 30 clean construction protocol
  - 3.4.7 Construction testing and certification
- 3.5 Instrumentation, equipment and materials
  - 3.5.1 Instrumentation
  - 3.5.2 Equipment
  - 3.5.3 Materials

4.0 MANAGEMENT AND INFRASTRUCTUR

- 4.1 Staffing and personnel
- 4.2 Training
- 4.3 Procedures and operating protocols

5.0 DEMONSTRATION OF OPERABILITY

5.1 Systems performance tests
5.2 Instrument acceptance tests
5.3 Work area blanks and standards tests.

The design of the clean rooms reflects several personal preferences developed over the years. The preferred criteria for environmental clean rooms are, as follows:

- All work surfaces to be Class 100 or better
- All sample treatment rooms to be Class 1000 or better
- All corridors to be Class 10,000 or better
- All instrument rooms to be Class 100,000 or better
- All spike, reference material and reagent processing rooms to be Class 1000 or better
- All spikes, reference materials and reagents to be handled only in Class 100 work areas
- Construction materials for exposed clean room surfaces limited to aluminium, plastics and wood
- Only high purity deionised water supplied to sinks
- All supply air and return air ducts fabricated from aluminium or PVC.

It is emphasized that these are preferences resulting from personal experience in clean laboratories. Other preferences may work equally well if data exists to support the performance of preference selection. Our experience has shown that a facility designed to accommodate these preferences will permit the analysis of low-level uranium samples with a total process blank below 5×10^7 atoms. A 0.2 micron diameter spherical particle of U_3O_8, with a density of 8, contains 7×10^7 atoms of ^{238}U. This process blank is adequate for all of the analyses required to meet the objectives of this project.

5. SUMMARY

In summary, these illustrations demonstrate the complexity of planning an environmental clean laboratory. One aspect of this planning process may require additional emphasis. It is not enough to design a well thought out facility and plan for all necessary operations. If a comprehensive preventive maintenance plan is not included in the planning process, the facility will not meet its long-term objectives. Performing only failure maintenance will compromise both facility availability and analytical quality. Because of the inherent complexity of such facilities, a comprehensive plan for regular maintenance and component replacement is required if high productivity of the facility and staff are to be achieved.

CONTRIBUTORS TO DRAFTING AND REVIEW

Chatt, A.	Dalhousie University, Canada
Horvat, M.	Jozef Stefan Institute, Slovenia
Mahalingam, T.R.	Indira Ghandhi Centre for Atomic Research, India
Perrin, R.	Edgerton, Germeshausen and Grier (EG&G), Inc., United States of America
Reddy, A.V.R.	International Atomic Energy Agency
Rossbach, M.	International Atomic Energy Agency
Tuniz, C.	Permanent Mission of Australia in Vienna, Austria
Vogt, S.	International Atomic Energy Agency
Woittiez, J.R.W.	Netherlands Energy Research Foundation, Netherlands