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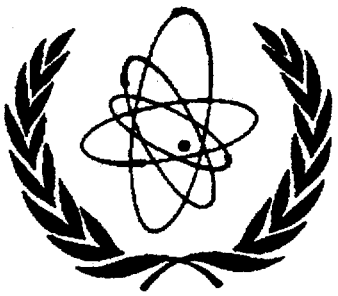

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XA0056181

Final Report of the IAEA Advisory Group Meeting on Accelerator-based Nuclear Analytical Techniques for Characterization and Source Identification of Aerosol Particles

held at the Jozef Stefan Institute, Ljubljana, Slovenia

4-7 July 1995

IAEA Advisory Group Meeting on
Accelerator based Nuclear Analytical Techniques for Characterization and Source Identification of Aerosol Particles

Ljubljana, Slovenia
4-7 July 1995

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Preface

The field of aerosol characterization and source identification covers a wide range of scientific and technical activities in many institutions, in both developed and developing countries. This field includes research and applications on urban air pollution, source apportionment of suspended particulate matter, radioactive aerosol particles, organic compounds carried on particulate matter, elemental characterization of particles, and other areas.
The subject of this AGM focused on the use of accelerator-based nuclear analytical techniques for determination of elemental composition of particles (by either bulk or single particle analysis) and the use of accumulated knowledge for source identification.

☞ Note: See Appendix I for Glossary

Emergence of megacities as a result of rapid urbanization all over the world and increase in the industrialization to provide services to such cities have given rise to severe air pollution problems in urban areas of the world. Sanitary engineering practices have improved over the past few decades, but, because of rapid urban population growth, especially in developing countries, severe air and water pollution problems remain. These demand the very best of scientific effort to solve the problem.

Among other things, indoor as well as ambient air pollution causes or aggravates diseases like asthma, chronic bronchitis, emphysema, and lung cancer. In many areas polluted air in the workplace and severe episodes of air pollution which are manifested by eye irritation, reduced visibility, etc. are frequently encountered. This happens when the capacity of self purification of the local atmosphere through dilution and dispersion is exceeded. Aerosol contamination of the atmosphere also causes large scale effects on the Earth's radiation balance and climate. Thus, a broad range of applications, from occupational exposures to global environmental change, of aerosol chemical characterization can be made.

The situation calls for studies and surveillance to monitor the situation so that action can be taken to correct it. The pollutants can be either gaseous or particulate matter. Accelerator based nuclear analytical methods are specially well suited for the elemental composition characterization of suspended particulate matter.

At the same time that environmental problems have grown to prominence in most countries, there has been a shift of emphasis in the use of low energy ion accelerators from fundamental research in nuclear physics to applications of nuclear science and technology to other fields. Especially interesting are applications of low energy ion accelerators to environmental problems.

In this report the Advisory Group presents terms of reference for aerosol characterization, the techniques that have been applied, and some recommendations for future opportunities and developments that the IAEA may wish to encourage.

Aerosol interactions and mechanisms are increasingly becoming recognized as controlling environmental processes on spatial scales ranging from local to global. Changes in aerosol particle concentration and chemical composition are leading to changes in impacts of particles on humans directly, on living organisms generally, and on the Earth's radiation balance and climate. These changes may be unsustainable in the long run, and corrective actions may soon need to be taken to avoid the changes becoming irreversible. Aerosol particles remain many days suspended in air before deposition and are transported between countries and even continents. Therefore, this issue must be addressed at the international level.

Such corrective actions need to be based on accurate characterization of aerosols, especially for anthropogenic particles, and identification of their sources. Analysis of samples of particles collected from ambient air for their elemental composition can provide especially useful information about their environmental impacts.

Aerosols affect human health, welfare, and the environment on scales from local to global. Some ways are:

- Inhalation of aerosol particles is the principal route of some pollutants from the environment to the human body. The efficiency of uptake is especially high for submicrometer diameter particles, those much finer than the common dust particles that the upper respiratory tract was evolved to block. Fuel combustion and industrial activities often produce these unnaturally fine particles in profusion. Lead and some other metals thus released to the atmosphere cause especially dangerous exposures to humans because they are deposited deep in the lungs and enter the blood stream.
- Light scattering by aerosol particles is the principal cause of haze and impaired visibility. Pollution particles are especially at fault since they tend to be very fine, of diameters comparable to the wavelength of visible light and thus efficient at scattering light. Their chemical composition affects their size under varying relative humidity conditions and thus their light scattering characteristics.
- Aerosol particles serve as nuclei for cloud droplet formation and other condensation processes. The role of clouds in climate change is one of the largest uncertainties in predicting the extent of global warming by greenhouse gases, and the role of aerosols in cloud formation requires detailed characterization of aerosol chemical composition.
- Cloud droplets, nucleated by aerosol particles, serve as chemical reactors for the conversion of sulfur dioxide pollution gases to strongly acidic sulfuric acid, a major contributor to acid deposition by rain or dry fallout.
- Suspended particulate matter in the stratosphere controls the concentration of ozone and its effectiveness in blocking harmful solar ultraviolet radiation from reaching the earth surface.
- The historical record documents many instances when volcanic eruptions and other natural calamities have caused sudden changes in climate, often the direct result of large scale injection of aerosol particles high into the atmosphere. Now, with increasing fluxes of anthropogenic particles into the atmosphere, not only from the steady releases from energy and industrial activities but possibly sudden releases from accidents, we may experience equally serious disruptions in climate as the result of human activity. Characterization of

these aerosols now, before serious large scale effects are experienced, may enable us to avoid their most serious consequences.

ACCELERATOR BASED TECHNIQUES FOR ANALYSIS OF AEROSOL PARTICLES

Since their original development in the 1930s, particle accelerators have become a major tool for research in nuclear physics. When the requirements for higher particle energies seemed to make many accelerators obsolete, applications of ion accelerators that provide beams of energies between 0.1 and 10 MeV grew rapidly. Thus, ion beam analysis (IBA) techniques, such as Rutherford Backscattering Spectrometry (RBS), Nuclear Reaction Analysis (NRA), Particle Induced X-Ray Emission (PIXE), Particle Induced Gamma-Ray Emission (PIGE), Elastic Recoil Detection Analysis (ERDA) and Proton Elastic Scattering Analysis (PESA) were soon developed.

A common feature of these techniques is that they are based on the irradiation of a sample of interest with a positive ion beam. The subsequent emission of a different kind of radiation defines each technique. Other characteristics shared by these techniques are that they provide information on the concentration of the elements present in the sample and that they are in general non-destructive. All these techniques have found wide applicability in many fields, such as materials science, archaeology, medicine, biology, and environmental science.

PIXE, which uses characteristic X-rays emitted by the sample, has been extensively applied for aerosol characterization studies, and in recent years this application continues to grow because of the increasing recognition of the importance of aerosols. PIXE owes its success in this area of application to its well-known characteristics, which include the small sample size requirement, its non-destructive character, the multi-elemental capability (sensitive to all elements from Na to U), the low absolute detection limits (down to picograms), the speed of the analysis (typically less than 10 min per sample) and the fact that the analyses are easily automated. In fact, thin layers of material on a thin backing film, such as the aerosol samples obtained with filter samplers or cascade impactors, are ideal study objects for multi-elemental analysis by PIXE. Furthermore, for a sensitive multi-elemental analysis of the small sample deposits that are obtained by compact size-fractionating cascade impactors there is no real alternative to PIXE. More important, PIXE is able to provide concentration data for many key elements of various natural and anthropogenic aerosol types. The major naturally produced aerosols (with key elements detectable by PIXE given in parentheses) are sea salt (Na, Cl), mineral dust (e.g., Al, Si, Ti), and biogenic aerosols that are emitted by plants on land (K, Ca, Zn, Rb) or are the result of gas-to-particle conversions from oceanic emissions (S). The anthropogenic aerosol types include sulfate from fossil burning (S), particulate emissions from coal burning (As, Se, ultrafine Si), particles from residual oil burning (V, Ni), leaded gasoline automotive emissions (Br, Pb), incinerator emissions (K, Zn, Pb), particulate effluents from smelting activities and metal production (Mn, Ni, Cu, Zn, Pb), and biomass burning products (K, Zn, Rb). The inclusion of many of these key elements in multi-element aerosol data sets is essential for source identification work and for the successful application of receptor models for source apportionment.

The PIXE technique has very favorable features for compositional studies on bulk and size-segregated aerosols, but by itself it does not provide all the desired information. It does not give the total particulate mass and provides no data for the very important carbonaceous fraction of the aerosol and the nitrogen compounds. However, some of these limitations can be overcome by the

use of complementary IBA techniques, such as PIGE and PESA, but it is also very appropriate to complement the accelerator-based techniques with other techniques. Very useful techniques in this respect are gravimetry or beta-gauge techniques for measuring the particulate mass, optical and thermal-optical techniques for the determination of black carbon and organic carbon, and ion chromatography for measuring major anionic and cationic species. With regard to the elemental analyses, one can benefit from complementary analyses by INAA, which provides better results than PIXE for several key elements, including Na, Al, V, As, Se, Sb and I.

When PIXE and the other IBA techniques are carried out at the millimeter scale, which is typically the case, average compositions of many particles are observed. However, it is possible to focus the incident ion beam down to micrometer dimensions, as is done in a nuclear microprobe, and this offers the possibility to obtain quantitative and sensitive (down to the μg/g) elemental information on individual aerosol particles, and to examine whether the elements are present in the bulk or on the surface of the particles. This information provides clues to the sources (and source processes) of the elements, to the various processes to which the particles may have been subjected during their transport from source to receptor, to their role in heterogeneous atmospheric chemistry, and to their possible effects on human health.

PROCEDURES FOR AEROSOL SAMPLING AND ANALYSIS

AEROSOL COLLECTION DEVICES

Various types of collection devices are being employed for aerosol characterization studies by PIXE and related accelerator-based nuclear analytical techniques. Although one could use open-face total filter holders, it is preferable that the sampling device is equipped with a PM10 (particulate matter 10 μm) inlet and that the aerosol particles are separated into a coarse and a fine size fraction, with the division between the two fractions around 2 μm equivalent aerodynamic diameter (EAD). Excellent devices for such collections are the stacked filter unit (SFU) sampler, the dichotomous sampler (or virtual impactor), and the two-stage circular streaker. The latter offers very good time resolution, and takes advantage of the fact that only a limited sample area (less than 0.1 cm^2) is needed in a PIXE analysis, whereas the former employ 47-mm (or 37-mm diameter) filters which can also be analyzed by a variety of other nuclear-related techniques (for example INAA). As collection substrate, polycarbonate filter is recommended.

Because PIXE requires only a small sample size, it is useful for detailed studies on elemental mass size distributions, which are becoming increasingly important. This can be done by using cascade impactors, which separate the aerosol particles in up to 10 size fractions, and whereby the particles are collected on each stage in a small area that can be fully enveloped by the PIXE beam, such as in the Battelle-type cascade impactor or the small deposit area low pressure impactor (SDI). Another possibility is to resort to a device whereby the particles are homogeneously deposited on each stage, such as in the rotating version of the micro-orifice uniform deposit impactor (MOUDI).

Some of the non-IBA techniques (and incidentally, also some of the complementary IBA techniques) require collection substrates that are not so suitable for PIXE. An elegant solution is to resort to multi-channel collection devices, such as the IMPROVE sampler, in which aerosols are

collected concurrently on up to 4 different filter materials for subsequent analyses by different techniques.

AEROSOL ELEMENTAL ANALYSIS

The analysis of aerosol samples by PIXE and complementary IBA techniques is generally straightforward. Often the samples are already collected on holders that can be directly irradiated, or otherwise the filter or the collection substrate (or a section thereof) simply has to be glued to a suitable target holder. For PIXE, a proton beam of 2 to 3 MeV is recommended. The bombardments are normally done in vacuum, so that elements down to Na can be detected. The size of the beam at the specimen (target) depends somewhat on the sample type, but for aerosol filter samples it is advisable to use a beam diameter of up to 10 mm. The beam current density at the target is typically of the order of a few hundred nA per cm^2 and the data collection is done for a beam charge (proton dose) up to 100 μC.

The calibration of PIXE systems can be performed using thin film standards that are commercially available or made in-house. The quantification for aerosol samples is straightforward, and matrix corrections are usually negligible or small, although particle size effects may be substantial for the light elements (Na, Mg, Al). An accuracy and precision of better than 10% are normally achieved. In a typical aerosol sample, about 20 elements are generally observed. The list of elements includes Na, Mg, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Br, Rb, Sr, and Pb. The detection limits are in the range of 0.1 to 5 ng per cm^2 (or per cubic meter of air), depending on the element, sampling volume and PIXE experimental conditions.

QUALITY ASSURANCE AND QUALITY CONTROL

In order to produce reliable results, a well designed QA/QC programme is essential for any laboratory involved in air pollution studies. This can be accomplished through:

1. Written internal protocols in sample collection, sample handling, irradiation and data analysis.
2. Correct representation of uncertainties of measurement.
3. In-house intercomparison with different analytical techniques applied to the same aerosol sample.
4. Interlaboratory comparisons by analyzing the same aerosol sample in different PIXE laboratories, taking advantage of the non-destructive character of the PIXE analysis.
5. Participation in international intercomparison exercises.
6. Aviability of suitable aerosol reference materials.

The speed and sensitivity of PIXE analysis makes it possible to acquire larger data sets than by using many other analytical methods with equivalent time and effort. Data sets of hundreds of samples for concentrations of 20 or more elements are not uncommon. Sets of this size are suitable for data processing by computer assisted statistical methods, making it possible to examine correlation and other relationships that can lead to resolution of components in mixtures, time series relationships, environmental transformation and transport processes, and other information of value in assessing the causes and consequences of environmental contamination by human activities or natural events.

The analysis of aerosol samples by PIXE and other IBA techniques typically provides elemental concentrations, expressed in ng per cubic meter of air, often in specific particle size ranges. Such results may be evaluated in several ways. The first step is the calculation of average concentrations and associated standard deviations (or of medians and interquartile ranges) for the series of samples or for a subset of that series. Such information is, for example, required to assess the exposure of people to the aerosols and the potentially toxic elements, both in indoor and outdoor environments.

Subsequently, depending on the sampling strategy and of the objective of the study, one may examine the variation of the elemental concentrations as a function of time, and determine the diurnal, weekly or seasonal variability. Such data may then be employed to identify times or episodes with a high risk of elevated concentrations and to define local, national and international regulations.

Major objectives in several urban and regional aerosol studies are to identify the dominant aerosol types (or their source regions and transport pathways), to apportion the particulate mass (PM), the major aerosol constituents, acidic species and heavy metals to these aerosol types (or source regions), and to determine the relative contributions from natural and anthropogenic sources. To aid in meeting these objectives extensive use is made of receptor modeling approaches, including (1) chemical mass balance (CMB) methods and (2) multivariate techniques, such as absolute principal component analysis (APCA) and multiple linear regression analysis (MLR). All these models require multi-elemental (or multi-species) data sets and assume a linear mixing of the contributions from the different sources. In CMB the concentrations of the elements in the sample are regressed against so-called source profiles (the compositions of the particles emitted by the various sources, as seen at the receptor site). It has the advantage that it works on a single sample basis, but it requires the knowledge of the number of (major) sources and of their elemental profiles. APCA requires no a priori knowledge on the number and compositions of the contributing sources. It makes use of the variance in a multi-sample, multi-species data set that is introduced in it as a result of different relative contributions from the various sources over the various samples (because of for example changes in wind direction or in emissions). Most of the variance in the data set is explained by a limited number of principal components, which can be identified as sources types (e.g., oil combustion products, automotive emissions, crustal material). For these components elemental profiles (source profiles) are then evaluated and, finally, the contribution from each source type to the concentration of each element (or to the particulate mass) in each sample is determined.

The concentrations of the aerosol types, as obtained by APCA or CMB for each individual sample, or of good indicators for these aerosol types can be related to (1) standard meteorological parameters (pressure, temperature, relative humidity, wind direction, wind speed) or to (2) the origin of the air mass, as deduced from air mass back trajectories. The first approach is particularly

useful in urban air pollution studies in order to assess the effect of meteorology on the atmospheric concentrations or to identify the location of specific pollution sources. Relation to air mass trajectories allows one to identify the source regions and transport pathways of the major aerosol types, and one may also obtain clues about the possible atmospheric processes to which these aerosols may have been subjected during that transport. When large data sets are available (of at least 100 samples collected over time spans of preferentially one year or longer), one can also employ statistical approaches to relate the atmospheric concentrations of individual aerosol species (or of aerosol types) to the trajectory data and pinpoint the source areas in finer detail.

From cascade impactor measurements raw elemental size distribution data are obtained, with mass of the element per m^3 and per stage (or per logarithmic size interval). It is useful to convert these raw size data into smooth uni or multimodal size distributions by means of an inversion code, thereby taking into account that the collection efficiency curves of the impaction stages are S-shaped instead of step functions. Lognormal curves can then be fitted to the inverted size distributions, so that the elemental mass concentration, geometric mean aerodynamic diameter and standard deviation of the different contributing modes are obtained. This allows one to reduce the information from the cascade impactors into a form which makes it much easier to investigate variations in size distributions of specific elements from sample to sample or to compare size distributions from different elements. The detailed size distribution data provide clues to the source processes of the elements, they are needed to calculate the penetration (and deposition) of elements into the lungs, or for the calculation of dry deposition velocities, and they are required for relating the chemical properties of the aerosol to their optical (radiative) properties.

Individual particle analyses by the nuclear microprobe on a large collection of aerosol particles have the potential to provide a finer resolution of the contributing sources than is possible with regular broad beam PIXE and receptor modeling. Furthermore, the source identification in nuclear microprobe analysis can be done on a single aerosol sample. Very useful tools for this purpose are hierarchical and non-hierarchical cluster analysis. In these methods, the elemental compositions of the different individual particles are used to form particle groups (clusters) of similar composition, which can than be related to specific sources or source processes.

RECOMMENDATIONS

Taking into consideration the above-mentioned facts and recognizing the importance of aerosol pollution problems and their impacts on human populations and social development, this Advisory Group (AG) makes the following recommendations:

- The AG strongly recommends that attention be given to air pollution problems, both in developed and particularly in developing countries.
- The Agency should continue to give high priority to environmental and aerosol studies in its programme.
- Since this field is especially important for developing countries, the AG recommends that the Agency encourage aerosol characterization projects in its Technical Co-operation programme.
- Many aerosol projects or studies need a multi-disciplinary approach. Accordingly, the AG recommends that a multi-organization approach be taken whenever a project of considerable magnitude is to be undertaken, especially at the international level. In particular, the AG recommends that IAEA work on environmental problems closely together with other UN organizations, such as WMO, WHO, FAO, UNEP, UNDP, IOC, and other international bodies or programs, such as IGBP and its core project IGAC, WCRP, ICSU, IUGG.
- The AG considers that Regional aerosol characterization projects in Latin America and West Asia are especially timely at present. Regional projects could optimize the use of accelerator capabilities available in the region for the common use of the countries, so that well organized regional projects on environmental studies could be carried out with modest funds and could strengthen scientific cooperation between countries in these regions.
- The AG recognizes Africa as a region in need of environmental science programs. It is therefore recommended that the IAEA give high priority to education and international cooperation in environmental studies in the region of Africa.
- The AG recommends that laboratories starting aerosol environmental programmes use accelerator-based nuclear analytical techniques, especially PIXE, as an efficient way for that research. 2-3 MV particle accelerators are particularly well suited.
- The AG notes a lack of specialized aerosol standard reference materials and strongly recommends that the Agency include development of reference materials suitable for accelerator-based aerosol analysis in its AQCS programme.
- Based on satisfactory results of previous intercomparisons and CRPs, the AG recommends continuing world-wide blind intercomparisons for laboratories involved in aerosol analysis. Such intercomparisons may help to identify where accelerator facility upgrades may be needed to assure high quality aerosol characterization results.
- An interregional workshop on accelerator-based nuclear analytical techniques for characterization and source identification of aerosol particles. This workshop should emphasize experimental work which includes all important aspects of measurement, from sample collection and preparation through elemental analysis of samples to data analysis and interpretation.

APPENDIX I: GLOSSARY

IAEA	International Atomic Energy Agency
AGM	Advisory Group Meeting
AG	Advisory Group
IBA	Ion Beam Analysis
RBS	Rutherford Backscattering Spectrometry
NRA	Nuclear Reaction Analysis
PIXE	Particle Induced X-ray Emission
PIGE	Particle Induced Gamma-ray Emission
ERDA	Elastic Recoil Detection Analysis
PESA	Proton Elastic Scattering Analysis
INAA	Instrumental Neutron Activation Analysis
PM	Particulate Matter
EAD	Equivalent Aerodynamic Diameter
SFU	Stacked Filter Unit
SDI	Small Deposit Impactor
MOUDI	Microorifice Uniform Deposit Impactor
QA/QC	Quality Assurance/Quality Control
CMB	Chemical Mass Balance
APCA	Absolute Principal Component Analysis
MLR	Multiple Linear Regression
AQCS	Analytical Quality Control Services
CRP	Coordinated Research Programme
WMO	World Meteorological Organization
WHO	World Health Organization
FAO	Food and Agriculture Organization
UNEP	United Nations Environment Programme
UNDP	United Nations Development Programme
IOC	Intergovernmental Oceanographic Commission
IGBP	International Geosphere Biosphere Program
IGAC	International Global Atmospheric Chemistry project of IGBP
WCRP	World Climate Research Programme
ICSU	International Council of Scientific Unions
IUGG	International Union of Geodesy and Geophysics

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APPENDIX III: AGENDA OF THE AGM AND TITLES OF SCIENTIFIC PRESENTATIONS

Preliminary Agenda for the Advisory Group Meeting on Accelerator-based Nuclear Analytical Techniques for Characterization and Source Identification of Aerosol Particles (F1.AG-904). Institute Josez Stefan, Ljubljana, SLOVENIA. 4 - 7 July 1995.

	Tuesday, 4	Wednesday, 5	Thursday, 6	Friday, 7
9:00 - 10:20	9:00-9:20 Registration **9:30 Opening** ***Dr. R. Martincic*, Head Dept. of Low and Medium Energy Physics, J. Stefan Institute.** ***Dr. V. Valkovic*, Head PCI IAEA Seibersdorf Labs.**	Paper Presentations	Discussions	Conclusions and Recommendations
10:20 - 10:40	**Coffee Break**			
10:40 - 12:50	Introductory and organization Remarks (V. Osorio, IAEA Physics Section) Paper Presentations	Paper Presentations	Discussions	Conclusions and Recommendations
13:00 - 14:00	**Lunch**			
14:00 - 15:20	Paper Presentations	Discussions	Conclusions and Recommendations	Conclusions and Recommendations
15:20 - 15:40	**Coffee Break**			
15:40 - 18:00	Paper Presentations 19:30 Welcome Party	14:00 Visit to the Reactor Experimental Facilities Discussions	Conclusions and Recommendations 19:30 Dinner	Closing and Final Remarks

Paper Presentations (limited to 20 min.)

- **"Particle-induced X-ray emission (PIXE) analysis of atmospheric aerosols at the University of Gent. Collection devices for size-fractionated samplings. Methods for source identification and apportionment. Examples of applications to local, regional and global problems".** *Dr. W. Maenhaut University of Gent*
- **"PIXE Qualification of Aerosols collected in Hungarian cities".** *Dr. E. Koltay ATOMKI, Debrecen*
- **"Studies of volcanic, rural and urban aerosols in Chile by PIXE and proton elastic scattering".** *Dr. R. Morales. Univ. de Chile.*
- **""AS-Pb interference in the PIXE analysis of airparticulates".** *Dr. M. Khaliquzzaman, Dhaka Atomic Energy Center.*
- **"On the need to set standard aerosol sampling procedures in urban areas".** *Dr. J. Miranda UNAM Mexico*
- **"Accelerator facilities at Teheran Research Center". Dr. Afaredih**
- **""The use of nuclear analytical techniques in aerosol characterization in the Amazon basin."** *Dr. P. Artaxo, Univ. Sao Paulo.*
- **"Monitoring and Assessment of Airborne Lead Contamination, an Application of PIXE Analysis".** *Dr. J. Winchester Florida State University.*

Topics for Discussions

- Present status of accelerator-based nuclear analytical techniques for air pollution analyses: Advantages and weakness
- Uniqueness of accelerator-based nuclear analytical techniques for aerosol analysis (bulk and single-particulate analysis)
- Present status of sampling procedures, techniques and equipment.
- Needs for standardization procedures and standard reference materials
- Needs and problems on data analysis and available nuclear data.
- Needs and recommendations on establishment of accelerator-based nuclear analytical techniques in developing countries
- Role of the accelerator-based nuclear analytical techniques for aerosol analyses in the IAEA Technical Co-operation programme.