ION BEAM USAGE IN ENVIRONMENTAL CHARACTERIZATION

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Abstract

There are many circumstances we were interested in getting more knowledge on environmental atomic and molecular composition, as monitoring pollution, identifying pollution sources, looking for air, water and soil on surface and underground composition. Accelerator based techniques offer a large palette of measurement capabilities, with high sensibility and accuracy, requiring small samples but they are expensive and time consuming, and need to be organized in such a manner to get a holistic characterization of an array of interest in a single sampling, or repetitive for trend identification. First 1980s sets of measurements were using gamma-spectroscopy associated to geodesic studies for singular events forensics. Samples from environment were taken and measured for elemental and molecular composition using, PIXE, PIGE, CPAA and NAA. Atmospheric pollution, and atmospheric corrosion, became important, and we have used TLA method to characterize the material corrosion in air, and simultaneously measure the acidity in air using special filtration unit in parallel, and taking precipitation samples.

During 1990s we were interested in tracking pollution, and we used the protocols developed by Nagoya Technical Univ., Prof. Susumu Amemyia, which was based on mobile air samplers with 0.4μ and 8μ pores, tuned for industrial pollution, and the IMPROVE protocol, developed with Prof. Thomas Cahill at UC Davis, CA participation, using 2.5 μ and 10 μ filters, simulating better human bucko-pharyngeal cavity, augmented with nearby soil, vegetation and water samples.

Using the lessons learned, in order to get competitive, fast and useful data, there is the need to consolidate the accelerators in clusters application ready, with some process automation, able to process many samples a day under quality assurance standards. Another direction is to develop mobile equipment for on-site measurements that will deliver the primary data collected during the sampling time, covering all aspects of the studied area.

1. INTRODUCTION

Accelerator based techniques especially IBA (Ion Beam Analysis) offers elemental elements high performance measurements, with capability to go deep into details, but environmental measurements address more aspects than simply knowing momentary elemental composition of some samples.

1.1. Environment measurements expertise evolution

Starting in 1980 in Romania, I have started measuring environment as related to singular events, to industrial pollution and general environment research using nuclear technologies. By 1980s we started measuring environmental radioactivity for forensic purposes, but we encountered many complications due to heterogeneous nature of radioisotope distributions and space radioactivity. Chernobyl accident found us so unprepared to reliably characterize the impact in air, on ground and water. Post-accident we developed a combination of measurements, aiming to identify the contamination not only with radioactive material, but with chemicals made of stable elements also. The interest was to correlate various contaminants in air bound to various size aerosols, and we started developing our multi-stage stacked filter unit, that was applied successfully to water, lubricant and wine filtration. Have been developed tangential filtering units to filter large volumes. Atmospheric pollution was another niche where the high sensitive IBA (Ion Beam Analysis) methods were prone to success. Here appeared the need for measuring bio-samples and organic substances with high saturated vapour pressure which are evaporating in vacuum and under beam's energy deposition as heat, led us to the development of methods with beam extracted in air, or controlled atmosphere, generically called bio-PIXE.

During 1990s we were interested in tracking pollution, and we used the protocols developed by Nagoya Technical Univ., Prof. Susumu Amemyia, which was based on mobile air samplers with 0.4μ and 8μ pores, tuned for industrial pollution [1], and the IMPROVE protocol, developed with Prof. Thomas Cahill at UC Davis, CA participation, using 10 μ and 2.5 μ filters, simulating better human bucko-pharyngeal cavity [2], augmented with nearby soil, vegetation and water samplers. Starting from mid 1980s we have added remote sensing capabilities using "Landsat" thermal and multi-spectral vision for characterizing short range transportation, where the data

were correlated with elemental analyses of samples from the area in order to produce signature patterns for image automatic identification. Measurements across US, Europe and Romania were made, mainly measured by PIXE and XRF, while stoichiometry was used to identify substances by educated guess.

After year 2000, at Los Alamos, NM soil and vegetables samples were taken to scale the impact in the area of the so called “nuclear legacy”, on local environment and looked to more evolved AMS methods using RGAs in order to better identify the molecular compositions and develop accelerator based forensic installation, using both the analytical and radiolysis capabilities, in an effort to extract maximum information from the measurements, sometimes needed to be performed in real-time.

These experiments drive us to the conclusion that for best accurate and complete results one has to use a plurality of methods applied quasi-simultaneously on the same sample.

In most of the cases was difficult to say which was the real contribution of a singular event on the composition of elements and molecules in an area, and most often we used in volume sampling to obtain the densities of each component, and from those variation to estimate the single event’s contribution. In this case accelerators have to cooperate doing simultaneously elemental analysis and atomic mass spectroscopy, to identify all molecular species, and to corroborate these results with CPAA, NAA and RBS for solid samples. Radioactivity is also an important feature and makes a more complex image. In some cases, using radiolysis and sequential measurements allows obtaining more complete information about volatile substances, which may be detected with Residual gases analysers on line with the beam, is sample temperature is also considered by using IR imager.

It has been detected an affinity between some substances and elements to associate with certain particulates and aerosols, as in the case of wine clearing by ultrafiltration, where wen removing a class of particulates was changing the wine scent and aromas, and a study in depth was required to better understand these manifestations. For example vinegar molecules were blended on 0.3 PM particulates, but after removing the 0.1 PM particles wine becomes diluted vodka with no taste.

Using the lessons learned, in order to get competitive, fast and useful data, there is the need to consolidate the accelerators in clusters application ready, with some process automation, able to process many samples a day under quality assurance standards. Another direction is to develop mobile equipment for on-site measurements that will deliver the primary data collected during the sampling time, covering all aspects of the studied area. That will represent virtual centres of excellence, assuring accelerators an important role in environmental and micro-particle knowledge progress.

1.2. Holistic view on environment investigation

The assembly of living & non-living organisms and even the space around that community is termed as an ecosystem. There is a specific area for different types of ecosystems, and all the organisms & the environment, where we understand the surroundings or conditions in which a living entity, as a person, animal, or plant lives or operates, interact with each other in that particular area. We distinguish air, aquatic and terrestrial and combinations of them may form an ecosystem.

In order to know if an ecosystem is safe, one needs to know a lot of details about it starting from living beings down to atomic and molecular composition, to which one adds the main events and modifications that may affect it. The changes in this composition it is called pollution when introduction of harmful materials into the environment takes place. Pollutants can be natural, such as volcanic ash, or may be the result of a human activity, such as trash or runoff produced by factories, and usually they damage the quality of air, water, and land [1].

2. ENVIRONMENT MEASUREMENTS

The environment measurements evolved gradually, driven by threat agents, as preparedness initiative in order to develop necessary knowledge and infrastructure for the task. As general characteristic of our expertise, we allocated a lot of time and effort developing measurement and interpretation methods and devices, but after implementing and using them we learned that it was not enough; we analysed the failures and the results took lessons learned and applied corrections and improvements. The hardest level to achieve was to make the resultant knowledge really useful, and applying it to drive to profit for the involved society.

2.1. Radioactivity measurement

During 1980s the close observation of singular effects was done by taking specific actions to identify a possible pollution resulted from that, and in most of the cases that was reduced to terrain measurements in order to identify any changes that possibly occurred in that area due to a reported event. The most common measurements were those of gamma radiation, X, alpha, beta, γ, plus EM, gravitation and weather.

The equipment used as FIG 1 shows was a Canberra 30 multichannel analyser over a 4-chanel Tektronix oscilloscope having nearby some nuclear electronics in NIM rack and some other standard, receiving signal from two NaI spectrometric gages inserted in a lead shield; in conclusion laboratory grade inadequate used in open environment with some adaptations and precautions. Underneath there are two portable GM radiation detectors. On the right side in FIG. 2 there are two images of the radiation measurement system deployed in terrain using an SUV. The result was a spectrum or numbers on a pre-calibrated SCA gates indicating the radiation rate for various energies specific to various isotopes we intended to identify.

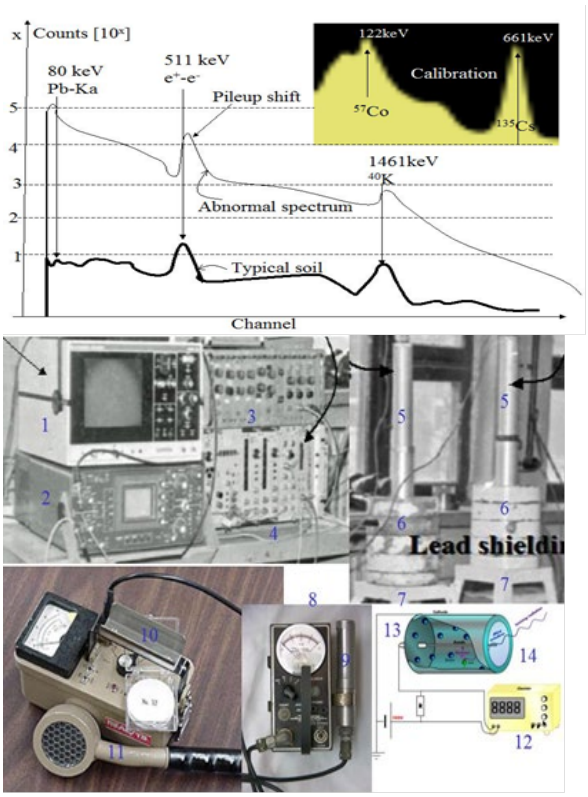

FIG. 1: 1980s radiation measurement devices and soil NaI gamma spectrum

FIG. 2: Mobile NaI gamma spectrometry of ground

The problem was that almost all the time we were missing the initial (before the presumed incident) radiation measurement and had to compare lateral measurements and use reasoning to say that the anomalous measurements are due or not to the previously reported incident. The problem was that a good background measurement that took about 10 min. and the equipment power consumption was high, at about 500+W, difficult to provide from car batteries therefore the measurement sessions were short, of about 2-3h, followed by a recharging break. We learned that connecting 2,3 gages at the same equipment does not increase too much the total required power. Soil samples were taken for analysis at soil specialized laboratory with low gamma background and analytic chemistry capabilities. In most of the cases was difficult to assign the findings to the related event, and measurement results had mainly an anecdotic value, being stored in a database. We also learned about the need of using two spectrometric gages one pointed towards ground and another towards atmosphere to measure and compensate for cosmic background, which may vary during the acquisition time, and may be influenced by atmospheric conditions too.

2.2. Atmospheric corrosion on solid samples measurement

In 1980 air corrosion was a rare, exotic subject, most of the educated people trend to ignore that, and to minimize the effects of pollution too, My mentor Eng. Livovschi P. had a collection of samples metal foils was exposed on a wall in Campulung, AG., Romania and from time to time, about every quarter, I.A.Aro Metrology division, where using a high accuracy calliper, made by the Tessa Co. in Switzerland, with accuracy better than 0.1 micron, was measuring the dimension of the samples. In 1983 we tried to improve this experiment providing radioactive labelling to few samples (3 samples, a brass (RL_ Brass), copper (RL_Cu) and steel (RL_Fe) (RL stands for Radioactive Labelled)) [3] and measure in parallel with the rest, not to perturb too much his experiment, which also contained Chromium (Cr), Aluminium (Al), tin (Zn), lead (Pb), as shown in FIG. 3

After radioactive labelling, the parts were measured by me using a spectrometric gauge, and by Metrology Department with Tessatronic micro-meter and the results were similar inside a margin of error of 15%, and are presented in Fig. 2 in the bottom-right side chart.

These measurements were made for about 10 years, from 1982 to 1992, in Campulung Muscel town, shown in Fig. 2 in aerial view, and between 1984-1986 radioactive samples have been used, both in Campulung and Bucharest due to radiation decay time. In that time, Campulung had a chemical plant producing synthetic fibres, a cement plant and a car factory. Pollution was mainly due to cogeneration and exhausts from these plants. Bucharest had a more complicated pollution picture, with lots of cars and plants.

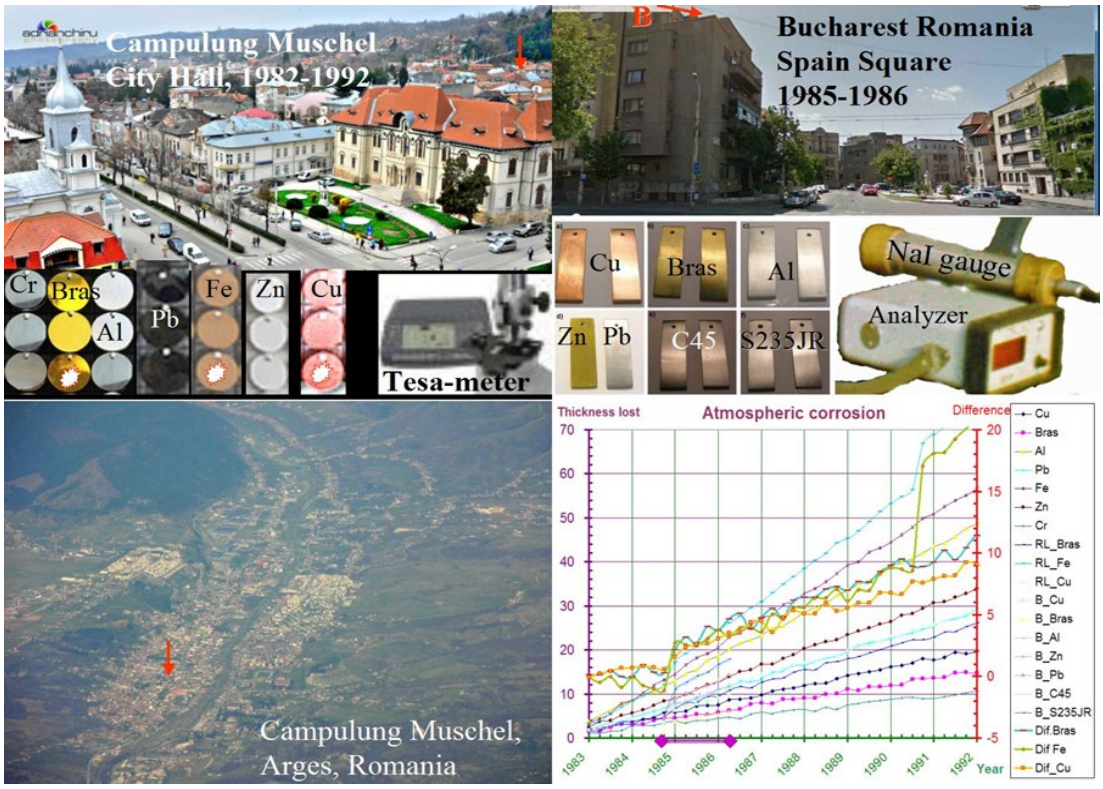

FIG. 3: Atmospheric corrosion locations and results for various materials

By 1988 it was observed that radioactive labelling modifies the properties of the structure and is prone to indicate corrosion by 30-70% faster than usual. In fact on his samples I have applied 1 micro-Amp.*Hour, in a volume of 50 microns and 1 cm^2. The concentration of H atoms was about 25 $*10^{15}$ atoms in about 5 * 10^{20} Fe atoms, result a concentration of 50 ppm of Hydrogen. The concentration of radioactive atoms was about 5 ppb, that is a relatively small trace, but what matters most is the number of dislocations induced into the target lattice and the thermal annealing proceed induced by irradiation, warming up the surface by few hundred Celsius degrees, because a power of about 15 W was applied into 5 mm^3 of material, driving to a power density of about 3 kW/cc, that is at the limit of material resistance, in spite we have used air jet cooling.

That was a very important learning; I had further applied in labelling as a precaution, and studied the effect on material under irradiation, being introduced in the labelling procedure as a quality assurance measure.

2.3. Aerosol particulate measurement by PIXE

At later time, starting from 1994, due to two international collaborations and a new attitude towards environment developed mainly after 1990, we have started measuring the pollution in various places in Romania and abroad. Cooperation was made with Nagoya University, Prof. Susumu Amemyia who developed an industrial pollution mobile aerosol tracking system, based on a staked filter unit using Millipore foil filters of 0.4 microns and 8 microns [4]. A second cooperation was done with U.C. Davis CA. Prof. Thomas Cahill who developed the "IMPROVE" (Interagency Monitoring of Protected Visual Environments) air quality protocol.

Prof. Cahill developed a stacked filtering unit that simulates the buccal- pharyngeal cavity retention, having a front filter of 2.5 microns and a 10 microns second filter.

We were thinking of unifying their two methods, because might have a better significance, as for example:-

particles bigger than 10 μm are due to local transportation, having a Stokes falling speed in mm/s maintaining in atmosphere for less than 1 day, but in windy conditions are airborne;

- particles with 2.5 μm< PM < 10 μm are retained in the nose filtering system and pharynges, are mainly airborne, and we lose the entire content of smaller particles.
- particles with 0.4 μm < PM < 2.5 μm are retained in the lungs, clogging there and some over time being dissolved or expelled, and with Susumu's filter we lose all smaller particles.
- particles smaller than 0.4 microns are passing through lung's walls into blood, influencing metabolism and mode, and here is one of the dangers nano-particles are presenting for users and manufacturers, and special protection measures have to be taken. We were interested in smaller particles and suggested the introduction of supplementary filter, but technologic difficulties made us postpone the idea;
- particles 0.4 μm < PM < 2.5 μm are also due to long range atmospheric transportation, and have a complex range, density, shape, chemical affinity correlation. We encounter these in upper atmosphere during Chernobyl pollution in May 1986, we observed that were mainly connected with water aerosols, mainly behaving like fog, but we were unprepared to perform coherent measurements [5].

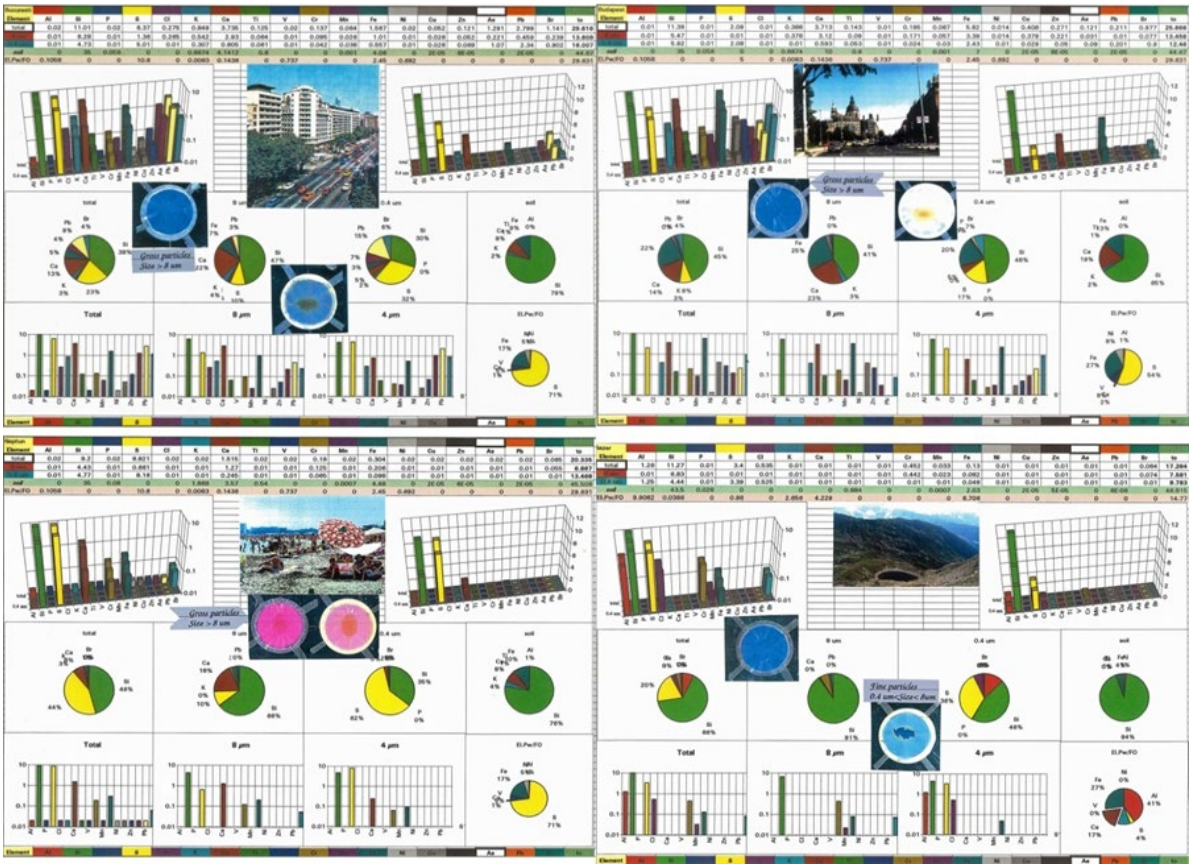

FIG. 4: Measurement of elemental pollution in various places (Bucharest, Budapest, Neptun, Balea Lake)

In spite we had available our membrane filter production at NIPNE-VDG Tandem, of Dr. Isabella Valcov, filters of 0.4 μm that we might like to use, but due to lack of resources in this complex system stacked filter unit, that to end up with a liquid or gel filter total collector, we never made the next development step.

We have used Amemyia's VDG system and measured pollution in various places, and the results are shown in FIG. 4, with the intent to correlate with corrosion information. This did not work out so well, because the bulk of atmospheric information was missing, and measurements remained just some numbers.

In FIG. 4 top-left is shown the results of pollution measurements in Bucharest Romania, on ½ mile N from University square, near Romanian Athenaeum, in the top-right is a measurement in Centre of Budapest, than in bottom left is a measurement on the Black Sea west beach in Neptune resort, and near Balea Lake, in Fagaras mountains at an altitude of 2300 m (about 7300 ft). In each image there are given a table of PIXE measurements, in the top, showing on columns header the detected elements and on lines, the separation between filters where 8 μm is meaning, all the particles with PM (Particle Magnitude) > 8 μm, and a residual amount of smaller particles (unknown for us); 0.4 μm means all the particles with 0.4 μm < PM < 8 μm and a residual amount of smaller particles also remained unknown for us. In the dark squares on blue disk is the filter picture as it was taken out from the sampler, and after being measured in the 4 MeV proton beam, that produced a dark burnup spot due beam power deposition heating and lack of cooling. Tom Cahill used to prevent this used a tiny He/Ar gas flow on the back of the filter, but we did only after observing this effect.

The problem was, the accuracy of the measurement, because due to heat up the vapour pressure of various solid particles present on filter, equals the vacuum pressure and evaporation/sublimation process occurs, and the concentration of that particle is reducing during the measurement [6].

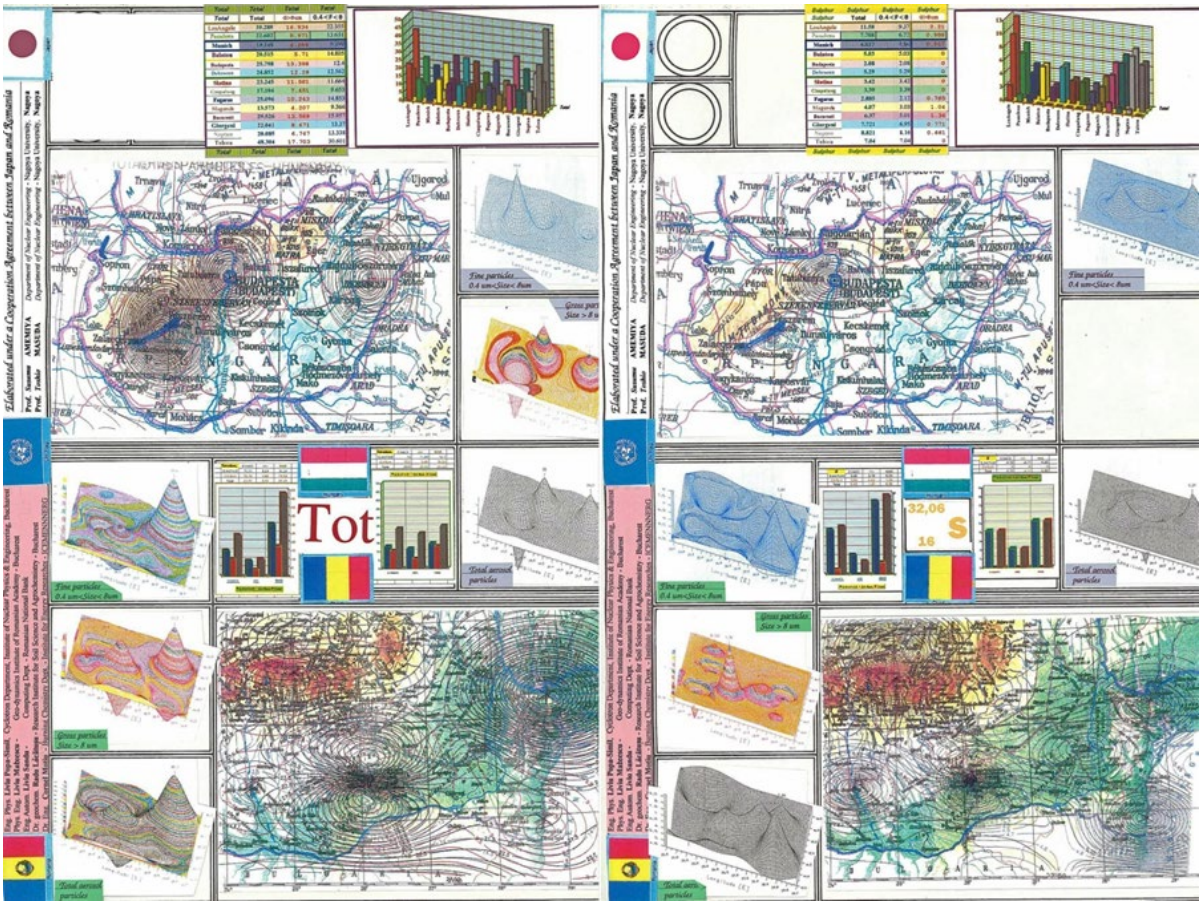

FIG. 5: Maps of pollution and elemental distributions in SE Romania and Hungary

We did two thinks to compensate for this effect:

- we took out the proton beam in air, and consider the X ray attenuation
- we made sequential acquisitions, dividing in 4 or 8 a regular acquisition duration.
- A better solution might have been to use a double stage RGA-300 and monitor the sample vapours from the vacuum application down to target evaporation under beam.

Next year in 1995 we performed another set of measurements with the intent to see the variation for a larger area on each element, and a sample of data is presented in Fig. 4.

In Fig. 4 are given the maps of Romania and the NW neighbour Hungary, as we have taken in cooperation with Susumu Amemyia, in summer of 1995, for the total particulates, on the left side and Sulphur on the right side, as samples, because we have another 19 elements measured, that may make this content too bulky, and is not related to the corrosion subject discussed here.

The images are divided in half, the upper side is about contain 2 maps, one of Hungary, in top and one of Romania underneath, and over each are overlapped the iso-levels of density of concentration in air (mg/m^3) for the 3 particle magnitude categories (Total, Coarse, Fine) and nearby there are given 3D views of the distributions, a list with places and measured concentrations for analysed element and charts showing the magnitudes. By 1997 we have extended the area of study in Europe tracing the pollution along Bucharest to Lisboan, [7] road, and improving the maps over Romania. In this case we tried taking mud and dusting samples in the road vicinity, in order to subtract the weight of short-range transportation, but the discrepancies become even higher[8]. Later we tried to use remote sensing data, and satellite imagery to correlate the spectral signature with field composition, in order to distinguish after applying weather behaviour previous air reaching a sampling point what came from local transport and what is in air from long range transportation, and that helped us using the mobile sampling with a network step of less than 100 km to localize pollution sources with less than 10 km accuracy. Some luck was also needed. In this process we have extended the use of ion beam accelerator, not only for performing PIXE and PIGE for elemental identification, but also applying RBS mm beam over various magnitude filters to better understand the elemental composition and associations inside aerosolized particulates, as in Chernobyl case where ^{131}I and ^{135}Cs associated in water droplets carried by wind and giving pollutant rains.

3. DISCUSSIONS

Based on these measurements, we succeeded to map in some approximation the distribution of each element, using stoichiometry we estimated the amounts of chemical combinations present in air.

In the measurements, we measured only solid particulates with very low vapour pressure, which did not evaporate in vacuum and under beam heating.

We had used remote sensing for local transportation validation, as well the soil and all available pollution data.

We had localized the pollution sources with an accuracy of few miles, using a grid of 100 miles step weather data and some luck.

We have failed to make any correlation between the corrosion wear rate previously measured and the airborne solid particulates, elemental concentrations we have measured and I have understood, why, and developed corrective measures

3.1. New equipment development

The corrosion measurement may be used as a comparative tool for the degree of air chemical cleanness between locations, but it is difficult to calibrate and correlate with weather events. The corrosion speed depends on many factors that makes any correlation attempt very difficult, but is a good reference if one has enough time and long term measurement reproducibility, in order to get the work done.

3.1.1. Aerosol particulate measurement

Based on lessons learned from previous experiments we simply understand that the accelerator based measurements have to be accompanied by collateral data measured with additional instrumentation, in order to corroborate all information available, and develop the right knowledge.

The system presented in FIG. 6 was designed for environmental anti-terrorism applications and top-left is shown a pattern of polymer/explosive radiolysis, in main detectable gases using X ray exposure., that may be considered to be introduced as an RGA in the PIXE irradiation chamber to measure the gases released during elemental analysis due to beam power deposition and associated radiation damage[9]. On the sample filters also aerosols and chemical fractions with high vapour pressure are on that trend to evaporate during vacuum

application, therefore we proposed a more complicated PIXE analysis and using a dual stage RGA and vacuum with moderate heat from laser and IR spectrometer for molecular identification of volatiles, and then a vacuum stage, using PIXE and RGA for elemental composition identification, finally completed by a neutron activation analysis, for light species, designed as a sequential or parallel test.

That will come at a cost of $20-$50 per sample. Improve the aerosol sampler with an ultrasonic concentrator with FX and Particle magnitude measurement capability as seen in top-right picture, and a snifter, as in bottom-left picture, as a multi-gas analyser, to build the device sketched in bottom left side of Fig. 6, able to take samples that are locally stored by the robotic arm, for weekly or monthly delivery, but also transmit real time data for basic air quality monitoring, as an integrated device for safety and security system network [10].

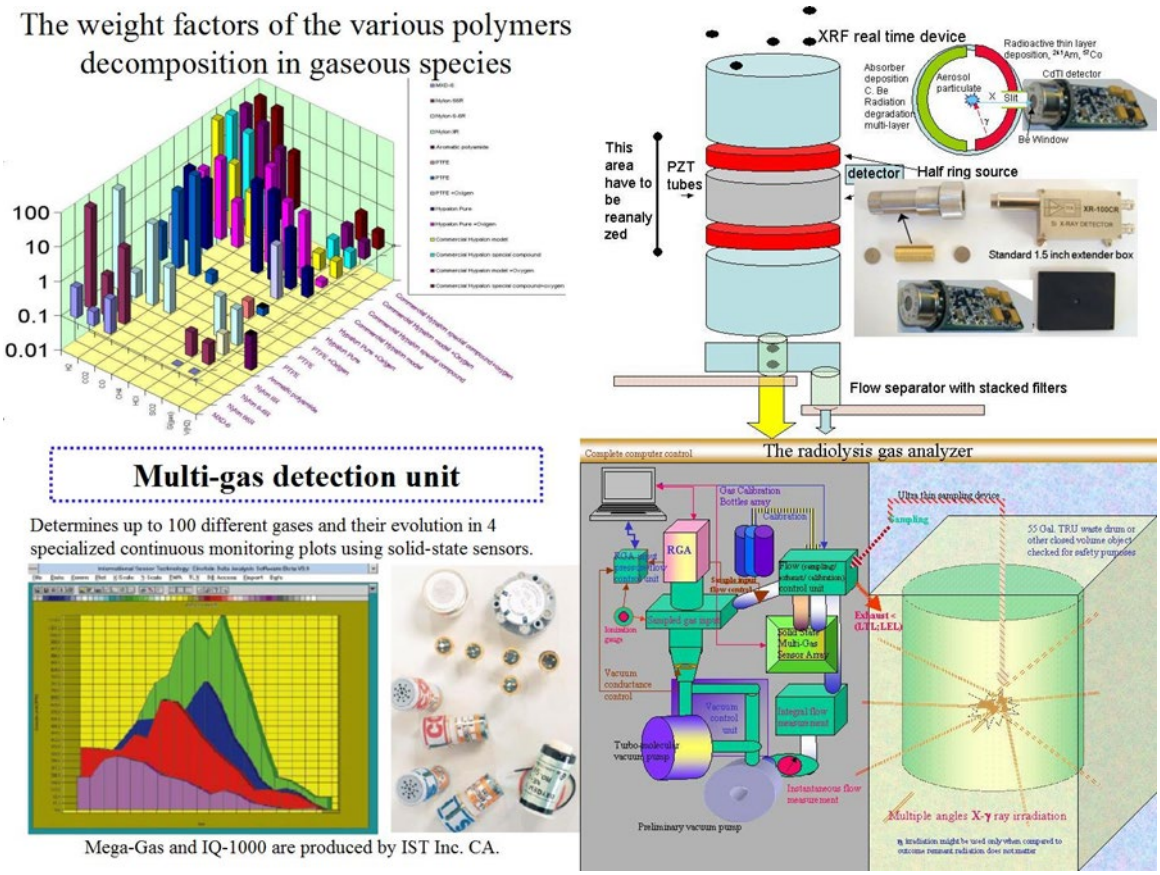

FIG. 6 – New enhancements possible to bring to aerosol sampler

A long-term device has to be similar with Improve system developed by Prof. Tom Cahill, with automatic sample filters loading that include more filtration stages as 20; 10; 5; 2.5; 1; 0.8; 0.5 and 0.2 microns, that to operate together with a particle magnitude meter. In parallel it will need a separate unit for organic fractions and bio-organisms. Before exposing the samples to vacuum, a high resolution image and initial weight might be important. The vacuum application and later the ion beam for PIGE and PIXE have to be done assisted by a RGA in order to analyse the volatiles elemental and molecular composition. It is useful to add in parallel with particulate accumulation an industrial gas analyser and a weather station connected and accumulating data that will be assigned to each sample. Water composition may be similarly analysed, by a filtration unit first and then taking a liquid sample for AMS, that may operate on spot using quadruple separators or laboratory grade accelerator based for high sensitivity trace analysis.

3.1.2. Mobile network for fast environment voxel diagnosis

A main problem we encounter and was difficult to solve was the usefulness of acquired knowledge, meaning by this that we need that knowledge level that to empower us to apply corrections to that environment voxel that to be profitable for all participants and stakeholders, and by this to recover the costs. In order to do this

we need to upgrade the system with a set of airborne measurement devices, and to add all the kind of collateral samples we may take from soil, underground water and water bodies, in a synchronous manner, correlate with local weather as seen in Fig. 7. In this case the procedure becomes as complex as a military operation and costly too, requiring a lot of logistics. The amount of test equipment grows and the number of samples that have to be analysed grows too. Depending on variability the observation time will be fixed.

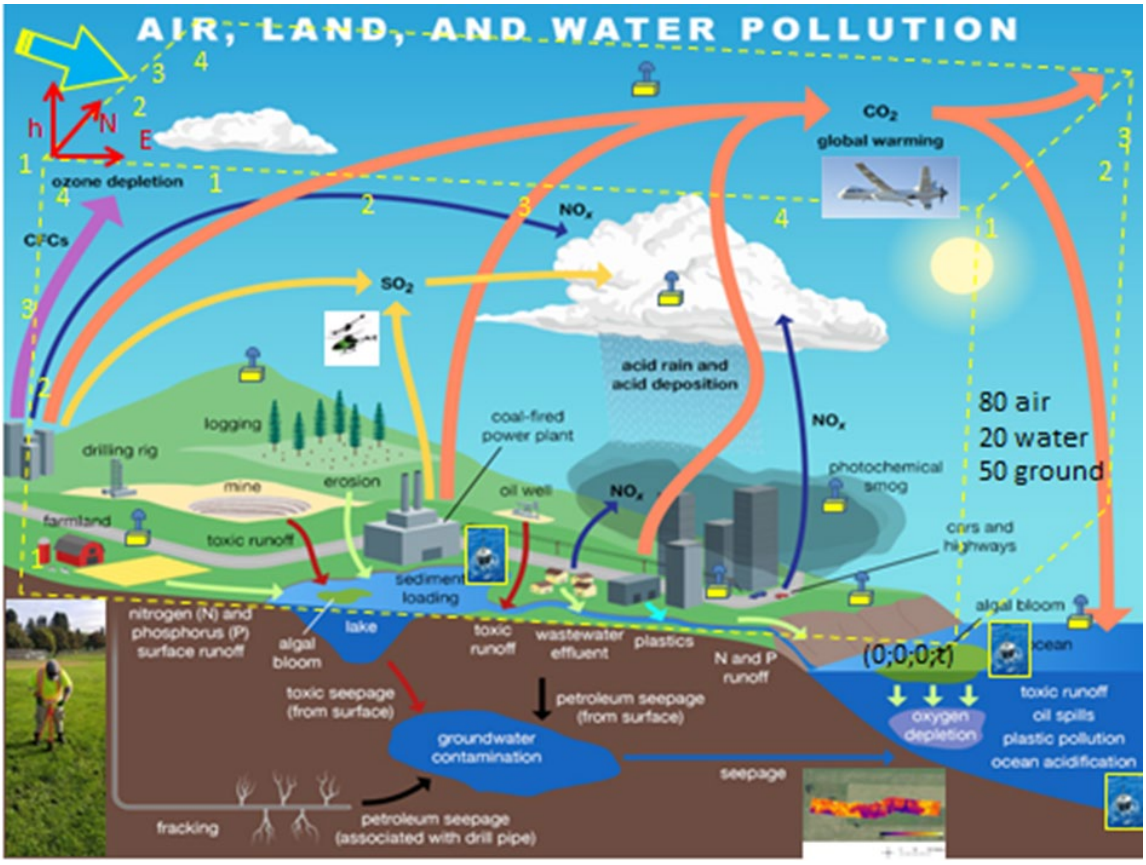

FIG. 6 – Complexity of establishing an effective measurement network

Fig 6 presents an ad-hock network of measurement that is aiming to perform synchronous measurements, and it turns obvious that about 20 helicopters with RF control are needed in addition to other equipment.

3.2. Accelerator usage and suggested improvements

The main task to be performed is elemental analysis, but the measurements have to be monitored by using an RGA with dual stage in order to identify the evaporating particle composition too, together with radiolysis products. In order to study the micro-structure, and isotopes dimensional distribution one may use micro-RBS beam. PIGE may be selectively applied for identifying those elements with prompt γ emission.

Other measurement methods have to be added, with improved equipment that to be able to see also the variability in sample content and create a model for the studied voxel, that to allow the identification of the sensitive points and determine the right corrective actions.

4. CONCLUSIONS

The development of these advanced measurement system must be optimized based on assessment of value of knowledge vs. the capabilities to use the knowledge, improve and get a reasonable ROI (return of investment), except the measurements for the sake of science.

The use of accelerators brings high sensitivity data, contributing to superior understanding.

The cost of a single measurement may be as high as $100k per single shot in an Ev, if the required equipment is already developed and available, that may drive the cost up to $10M or more being recommended

as such device to be developed at government level, requiring a specialized team up to 100 people, with various specializations and a significant accelerator time of minimum 1 week (95% operation time) for a single Ev shot, where Ev will have Dx dimensions usually smaller than a 10 miles cube.

Development of advanced measurement protocols under Quality Assurance norms, and advanced data acquisition, storage, processing and visualization, even using AI processing is a must to be able to lossless handle this huge amount of data.

One needs to understand that the winners are not those who know but those who can, and that is recommended to proactively use knowledge acquired to favourably change the things to improve and steward/protect the Environment, having a reasonable ROI.

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