QUALITY ASSURANCE SYSTEM IN GAMMA SPECTROMETRY LABORATORY

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ABSTRACT

On basis of guidelines for development of QUALITY SYSTEM for a testing laboratory (European Standard Series EN 45000) a quality assurance system was implemented in gamma spectroscopy laboratory, where routine measurements of natural (mainly Ra-226, Ra-228, Ra-224, K-40) and artificial (mainly Cs-137 and Cs-134) isotopes are performed. We measure a variety of samples, but mainly coal, vaste rock, ashe, deposits, vegetation and air filters.

Laboratory of gamma spectroscopy in Central Mining Institute has three HPGe detectors. There is one coaxial detector with 45% relative efficiency, one detector for low energy region and one detector with extended range). We have also two Ge(Li) detectors from former Czechoslovakia. Shielding is made mainly of steel (40 cm) with the interior covered with lead and copper. The electronics and software (Genie-PC) was bought at „Canberra" and „Silena".

The paper describes not only the system of quality assurance but also main problems met by its implementation and results of intercomparison measurements. The QAS has been introduced in 1992. In 1993 the Accreditation Certificate of Testing Laboratory for our Laboratory has been obtained from the Polish Bureau of Research and Certification as a fifth laboratory in Poland.

INTRODUCTION

Quality assurance is one of the most important requirements of unification of testing laboratories in different countries, according to EN 45000 standarts. It is especially important for testing and research laboratories, dealing with environmental and workplace monitoring, because there is an increasing demand for assessment of data quality in environmental monitoring of natural radioactivity. The data from these measurements are used not only for assessment of health effects but also for enforcement activities, the establishment of guides or standards. Therefore the precision and accuracy of the data must be assured so that desicions concerning environmental or occupational impact are based on data of known reliability.

Each laboratory providing environmental radiation measurements should have an internal quality assurance program in operation to ensure, that instrumantation is calibrated properly and applied analytical procedures are being carried out properly as well. Such program also includes continuous monitoring of instrumentation, frequent analysis of replicate samples to check precision and regular measurements of samples with known activity, to check the accuracy of the system. A very important role in QAS plays also the procedure of internal and interlaboratory intercomparison runs.

Central Mining Institute (CMI) is located in Katowice, the capital of Upper Silesia, an industrial area with more than 60 coal mines, many coal-fired power plant, metallurgy factories etc. It is also the area with enhanced natural radioactivity, caused mainly by waste water and waste rock from underground coal mines. Laboratory of Radiometry in the Central Mining Institute is making measurements of the natural radioactivity in coal mines and in the natural environment. Our work is aimed mainly to the radiation protection of miners, monitoring of radioactive contamination of the natural environment and remedial actions, if necessary. We are also making measurements and testing of building materials and other samples including foodstoof. We are also making measurements of radon concentration in mines, dwellings, soil etc.

We have started to implement the quality assurance system in the 1990 according to EN 45000 standard and the ISO/IEC Guide No. 25. The formal application for the accreditation have been send in 1992 and the accreditation has been granted in December 1993 by the Polish Bureau of Research and Certification. Our Accreditation Certificate of the Testing Laboratory is No 5 in Poland.

The scope of the accreditation is as follows:

- measurements of concentration of radionuclides Ra-226, Ra-228, Ra-224, Pb-210 in liquid samples, water and aqueous solutions;
- measurements of concentration of gamma-emitting radionuclides in solid samples;
- measurements of gamma doses and gamma dose-rates;
- measurements of potential alpha energy concentration of radon progeny in air;
- calibration of portable instrumentation for measurements of potential alpha energy concentration in the air.

We would like to share our experience and present problems occurring during implementation and maintaining of the quality assurance system. Our laboratory uses mainly three techniques: liquid scintillation spectrometry, gamma spectroscopy and thermoluminescent (TL) dosimetry. We would like to concentrate on some problems only, with special emphasis on the gamma spectroscopy technique.

ORGANISATION

Laboratory of Radiometry (LR) employs fifteen people, divided into four groups. Three groups are doing measurements and testing. They are independent from another group doing expertise.

The main technique used in LR is liquid scintillation counting, which is used by first group for:

- determination of ^{226}Ra, ^{228}Ra and ^{224}Ra in water and aqueous solutions;
- determination of ^{210}Pb in water and aqueous solutions;
- measurements of ^{222}Rn in air;
- measurements of radon daughter products in air (as a reference method for calibration of other instrumentation);

All listed above methods, but measurements of ^{222}Rn in air are accredited.

Another group is making measurements of radon daughters concentration and gamma doses. For these measurements TLD technique is applied. Only calibration of instruments for radon daughters measurements is done by liquid scintillation counting.

The third group is doing measurements of concentration of gamma emitting natural and artificial radionuclides in solid samples such as:

- ashes and slags from power plants
- coal
- rocks and sediments
- soil
- river bottom sediments
- air filters
- building materials
- raw materials and industrial products
- industrial waste products
- foodstaff.

Measurements are done by gamma spectroscopy in energy range from 5 to 2000 keV, using HPGe and Ge(Li) detectors .

Wide range of techniques gives used in our laboratory gives the opportunity to compare results, accuracy and precision obtained by different techniques of measurements.

EQUIPMENT AND INSTRUMENTATION

The general director of the Institute declared to provide all necessary funds to maintain the top, updated level of instrumentation and equipment and to make possible appropriate training of the staff. Therefore our laboratory is very well equiped. The equipment and the system of quality assurance in the LSC part of our laboratory is described elsewhere [1].

In our laboratory of gamma spectroscopy there are following detectors:

- HPGe from PGT - with 45% relative efficiency
- HPGe from Canberra for low energy region
- HPGe from Silena - extended range detector, 28% relative efficiency
- two Ge(Li) detectors from former Czechoslovakia.

Shielding is made mainly of steel (small pieces of stell scrap in boxes - total thickness 40 cm) with lining made of lead and copper. Two HPGe detectors are coupled with electronics and software (**Genie-PC**) from Canberra, another HPGe detector is connected with electronics and software from Silena. Ge(Li) detectors are coupled with multichanel analysers NOKIA-POLON manufactured in Poland and equipped with our own software for gamma spectroscopy analysis.

This instrumentation and software enables for example measurements of Ra-226 concentration as low as 1 Bq/kg.

LABORATORY PROCEDURES

High quality of our test and measurements is assured by strict following laboratory procedures:

- receiving and registration of samples
- preliminary preparatory of samples
- measurements by gamma spectrometers
- reports
- calibration (calibration is made at least once a week)
- internal tests (double measurements are required for at least 10% of samples)
- interlaboratory tests (must be performed at least once in a year)
- preservation of samples and results after measurements (samples are kept at least one year)
- audits
- and other general procedures.

INTERNAL AND INTERLABORATORY TESTS

Internal test are often made and the results are carefully analysed on regular basis. The schedule and requirements of internal tests is given in laboratory procedures.

Even more importance is given to interlaboratory tests [2]. There is a special procedure describing the requirements and responsibility for interlaboratory tests. Each interlaboratory test is concluded by a standard report, containing all information about participants and the test as well, in which the result is evaluated. In case the results are not satisfactory there is an analysis of possible errors and measures to be taken.

Although LR makes internal and interlaboratory tests in all subjects covering the described above activity of Laboratory of Radiometry, we will here give only some examples of determinations, made by gamma spectroscopy technique.

Internal tests

According to the laboratory procedure internal tests have to be done for at least 10% of all samples. It includes replicate measurements of the same sample on the same detector as well as measurements of the aliquots on the different detectors with applying of different software for data processing. Also cross-checking with liquid scintillation counting technique have been provided for radium isotopes in solid and liquid samples.

If one of the results of double samples differs more then the accepted error, this case is analysed for possible reasons and two additional measurements of such sample have to be made.

Results obtained for reference, standard and background samples are also stored and controlled periodically, for monitoring long term stability of the instrumentation. Although standard and background samples give rather stable results, to avoid errors caused by fluctuactions of these parameters, three months average values are used for calculations of radioisotopes concentrations in analysed samples. Results of measurements of a standard sample prepared from standard uranium ore provided by NBL mixed with coal of low natural radioactivity are given in Fig.2. These results cover a two years period. The concentration of ^{226}Ra calculated from a value declared by NBL is 1225 Bq/kg. The average measured value basing on 186 keV line of ^{226}Ra is 1227 Bq/kg with the standard deviation 36 Bq/kg. The average concentration of this isotope measured basing on ^{214}Pb lines was 1255 Bq/kg with the S.D. 39 Bq/kg.

Fig. 1. BACKGROUND

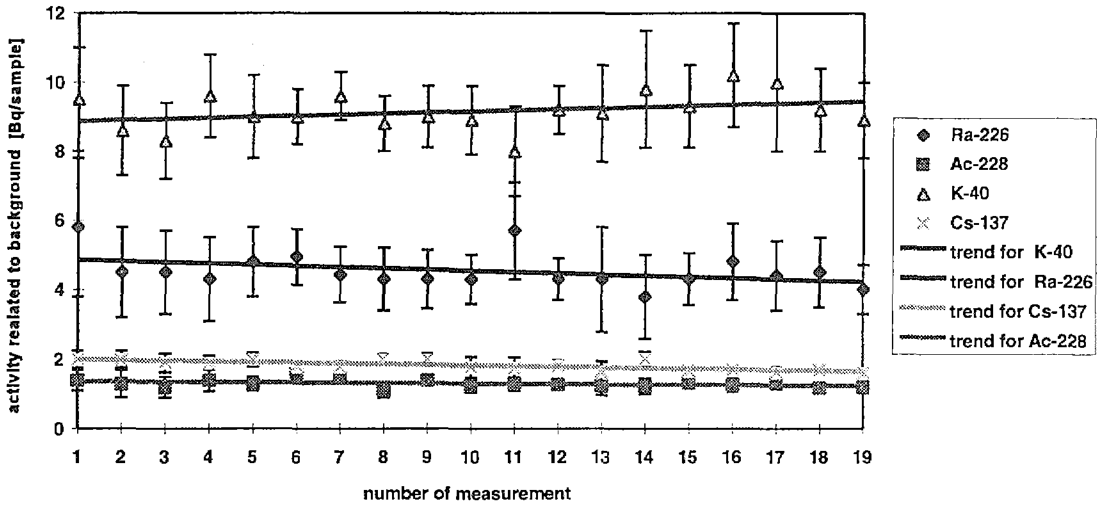
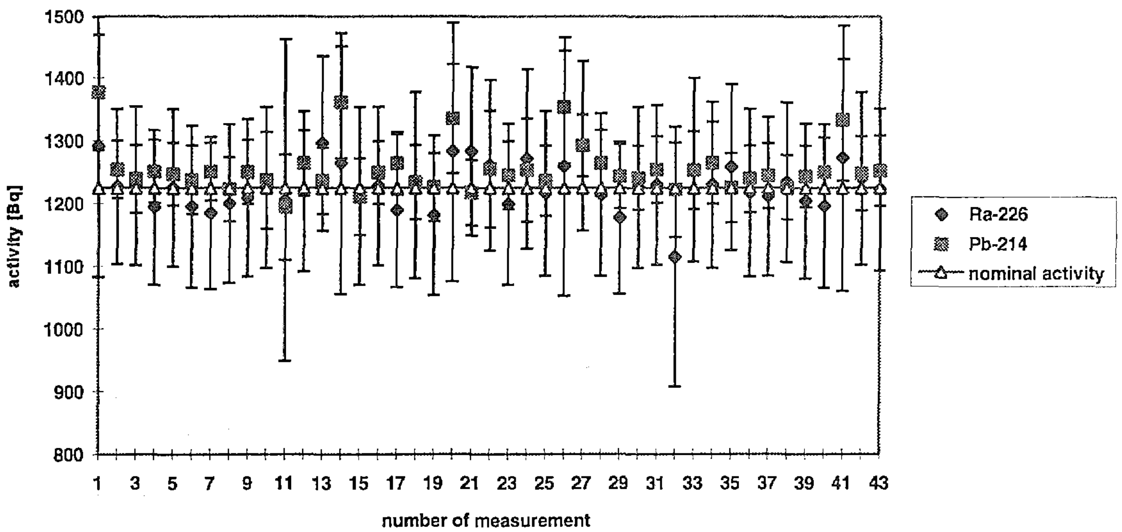


Fig. 2. Uranium standard



Results of measurements of background using as a sample an empty container are given in Fig.1. Results obtained in the area of lines of most often measured radioisotopes are given in this figure. Correlations of results of double measured samples are shown on Fig.3a and 3b.

Fig.3a. Results of measurement of replicate samples Ra-226

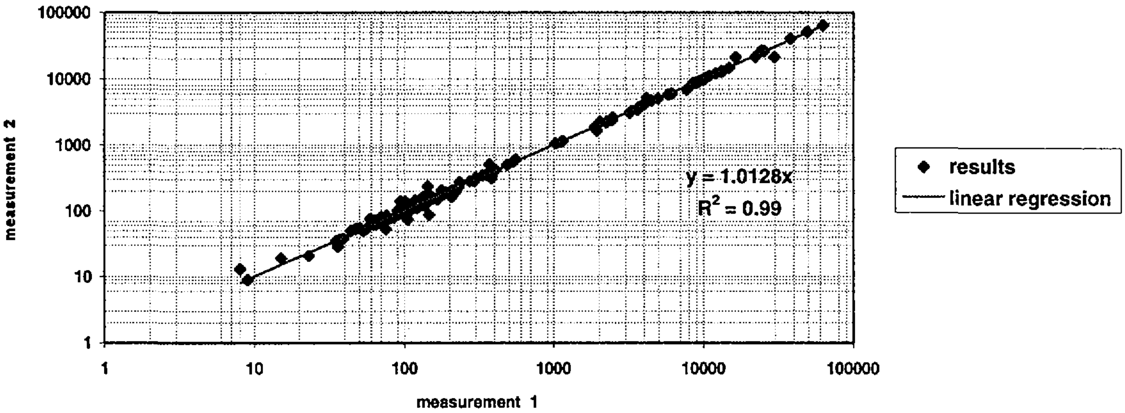
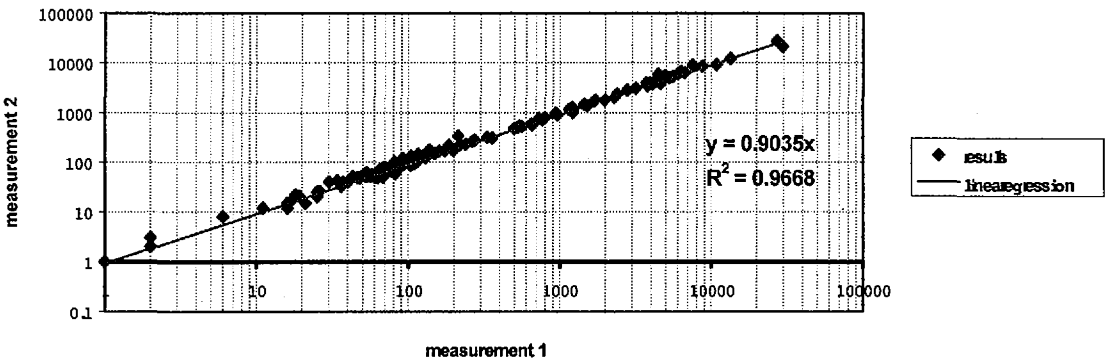


Fig. 3b. Results of measurement of replicate samples Ra-228



Interlaboratory tests

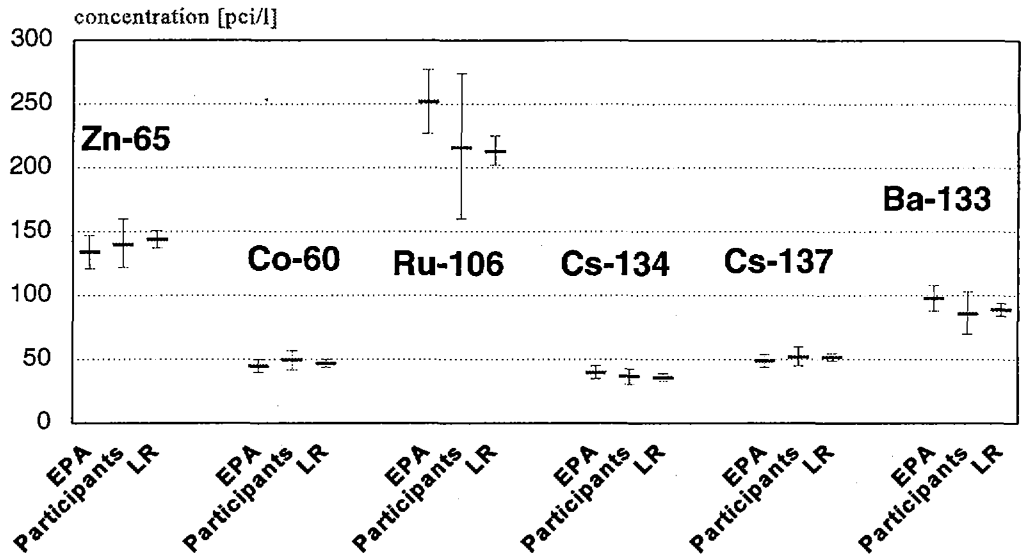
Our laboratory participated on regular term in two international intercomparison programmes:

1. Intercomparison organised by IAEA, which includes determination of different isotopes in diferent materials, as sea sediments, soils, fish, milk etc. In this programme all results are compared with the grand average obtained by all participants, but outlayers [3].
2. Intercomparison organised by US EPA - within this programme samples are distibuted regularly. We participated particularly in determination of gamma emitting isotopes and radium isotopes in water. Results are compared with a known value and with a grand average. In this programme results are given very fast, during the couple of months [4,5,6].

We also participate occasionally in other bilateral interlaboratory test in Poland and with our foreign partners. The results of one of intercomparison runs performed within EPA programme are shown on Fig.4.

According to our Quality Manual interlaboratory tests must be done at least once a year for each certified determination. The only exception is measurements of radon daughters in air, because organisation of such intercomparison is difficult.

Fig.4. Results of intercomparison measurement
EPA "Gamma in Water" exercise 10.06.94



AUDITS

Complete internal audits are carried out at least once a year. In any circumstances, when changing any parts of the procedures or introducing new members of staff or even during holiday replacements we are making additional internal audits. Our special concern is a friendly and open relationship and trust between the head of the laboratory and scientific and technical staff as well as among the entire group. To maintain a close contact every day get together with a briefing and coffee are held. On this briefings all current problems are discussed and main decision are given.

Once a year a general audit by a scientific director of the institute is done. At this occasion the plan of work and general trends of our activity as well as basic needs are discussed. Also results of interlaboratory test are presented. Each year an audit by auditors from Polish Bureau of Research and Certification has to be made. All certified activities are controlled during the external audit, with a special emphasis on one specific type of measurements. Last time the gamma spectroscopy laboratory was checked.

MAIN PROBLEMS IN IMPLEMENTATION AND MAINTENANCE OF QAS

The first problem, which we faced during implementation of quality assurance system in our laboratory was connected with psychological aspects of this system. We had to change our minds in aspects concerning sample preparation, data acquisition, sample storage and reporting of the results.

Another problem concerns reference materials and intercomparison runs. The best intercomparison run (in our opinion) - the EPA programme - has been temporary ceased for foreign participants since 1994. The IAEA programme is good due to very wide variety of different samples but sometimes not very convenient - reporting of results one year or more after measurements is not very helpful for participants. The lack of domestic intercomparison programme forced us to make some intercomparison measurements based on private contacts. We feel an urgent need of such programme in Poland.

We think, that many problems which we found during implementation of QAS in our laboratory would be avoided if we could learn from someone elses experience in this subject. Therefore it would be very helpful to establish a close contact between laboratories with implemented QAS system.

Now the main problem consists in overloading with work, because there is difficult to predict the need for tests and provide appropriate training of new staff and sufficient space in the laboratory.

CONCLUSIONS

European norms EN 45000 and the ISO guide are very helpful in introducing of quality assurance in a testing laboratory. However, because of the specific field of activity and conditions, most arising problems must be solved by the laboratory head and personnel. From our experience there is clear that only very close and good co-operation of the all personnel allows introducing and maintaining quality assurance.

We found that international intercomparison runs and exchange of reference materials is especially helpful in constant proving and maintaining the quality of tests and measurements. Additionally, these tests give the opportunity to get new scientific contacts and co-operation.

One of the main difficulties consists in keeping a rhythmic work of the laboratory. This is caused by changing and unpredictable needs for research and test from the industry and authorities. On the other hand it is more difficult for an accredited laboratory to be flexible, because the selection and the training of the staff must be more careful and the laboratory must fulfil a certain standard.

Up to now we have not a contact with other accredited gamma spectroscopy laboratories, so all arising problems must be solved ourselves.

Concluding we can say that after two years of experience all the personnel of the laboratory is very satisfied of achieved level of work, even those who were rather reluctant at the beginning of the process.

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