# IAEA-MEL's AQCS PROGRAMME FOR MARINE RADIOACTIVITY MEASUREMENTS

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**Abstract**

The main objectives of the IAEA-MEL's Analytical Quality Control Services (AQCS) for marine radioactivity measurements are discussed and future plans for the organization of intercomparison exercises and the production of certified reference materials are presented. The new developments should also include implementation of quality assurance programmes in Member States' laboratories, training in quality management and accreditation programmes.

## 1. INTRODUCTION

The IAEA-MEL has been assisting Member State laboratories in Analytical Quality Control Services (AQCS) for marine radioactivity measurements for almost 30 years (Fig. 1). AQCS through world-wide and regional intercomparison exercises and the provision of reference materials has been recognized as an important component of the IAEA's programme. Altogether 41 intercomparison exercises for radionuclides were organized and 35 reference materials produced.

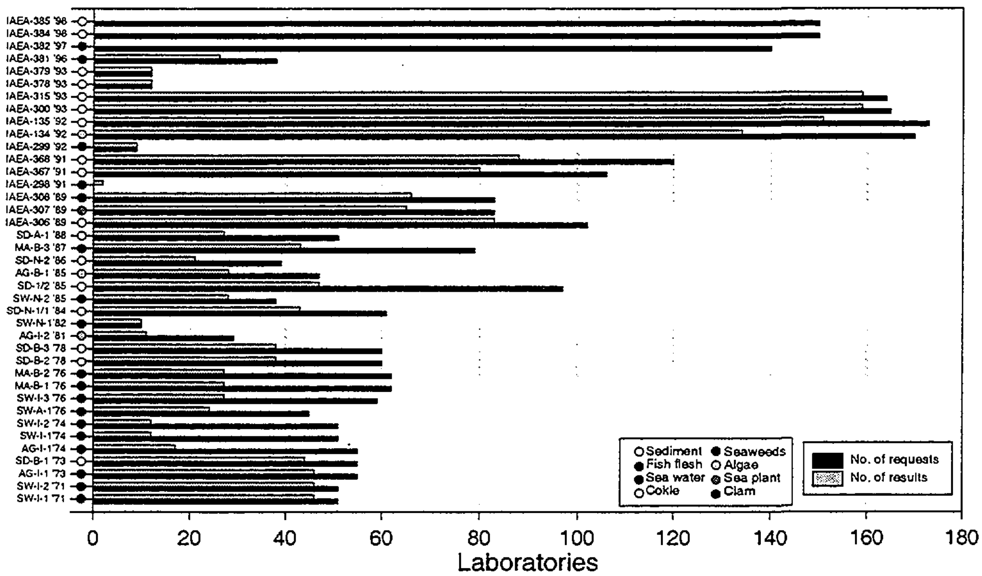


*FIG 1. Intercomparison exercises carried out by IAEA-MEL for radionuclides*

Following many years of experience in the AQCS programme and the recent recognition of the great importance of environmental data in economic, ecological and legal decision-making, the IAEA-MEL has been reviewing whether further steps should be taken to assist Member States in improving data quality. In particular, it has been proposed [1] that the AQCS programme should address in full the complex problem of the total quality management of analytical laboratories, including quality assurance programmes and manuals, regional and world-wide intercomparison exercises with proper feedback, production of certified reference materials according to ISO standards, training in quality assessment and control and accreditation of analytical laboratories.

The objectives of the AQCS programme carried out at IAEA-MEL to assist Member State laboratories in marine radioactivity measurements can be summarized as follows [1] :

i) To assist in quality assurance programmes through the organization of world-wide and regional intercomparison exercises.
ii) To provide feedback on the performance of the laboratories, to organize evaluation meetings, training and AQCS missions to improve their performance.
iii) To assist in the preparation of quality assurance programmes and manuals.
iv) To provide analytical reference methods.
v) To establish analytical competence and methods needed to produce certified reference materials according to ISO requirements.
vi) To provide reference materials and certified reference materials in sufficient types and quantities.
vii) To assist in accreditation programmes.

Further we shall discuss several concepts related to the full implementation of such a complex AQCS programme deriving background information from IAEA-MEL's experience obtained in recent years. All these activities would enable attainment and maintenance of the high standards and quality of the AQCS programme.

## 2. QUALITY ASSURANCE PROGRAMMES

Quality assurance (QA), as a system of activities and actions [2] to be taken to ensure confidence that analytical results will satisfy given quality requirements, has two components :

- quality assessment - a mechanism to verify that the system is operating within acceptable limits,
- quality control - a mechanism to control quality of data (to control errors).

The objectives of the QA programme include maintaining a continuous assessment and control of the quality of data, identifying proper analytical methods, providing permanent records of the performance of instruments, standardising analytical procedures, ensuring sample integrity, improving record-keeping and identifying training needs. The common aim of all these objectives is to provide data of high quality.

The QA functions include the development (or selection) of proper methods for sampling, sample preservation, sample pre-treatment, sample analysis and methods for evaluation and reporting of results. Further, they include intralaboratory and interlaboratory methods of validation and evaluation studies, establishing quality control guidelines and maintaining quality control sample programmes.

A laboratory without a proper QA programme cannot operate successfully. This is especially important when the laboratory produces series of data that are of interest in national or international programmes.

A QA programme (including good laboratory practices [3]) should be described in one of the most important laboratory documents - the QA manual. The document should contain the QA policy and objectives and should document the organisational structure of the laboratory, staff responsibilities, the analytical methods (including sampling, field measurements, sample handling, protocols, data reduction and evaluation), the materials and standards used, the QA procedures, interlaboratory comparisons, the recording system and the database. This comprehensive document should be regularly up-dated with any developments in the laboratory [4].

Therefore there is a need to assist Member States by establishing QA programmes in their laboratories, by providing them with examples of QA manuals, by training people in QA and by conducting AQCS missions.

As a typical example, we present in Fig. 2 the quality control chart (as drawn up in our laboratory) for analysis of $^{239,240}$Pu in Irish Sea sediment (IAEA-135), which is frequently used as the

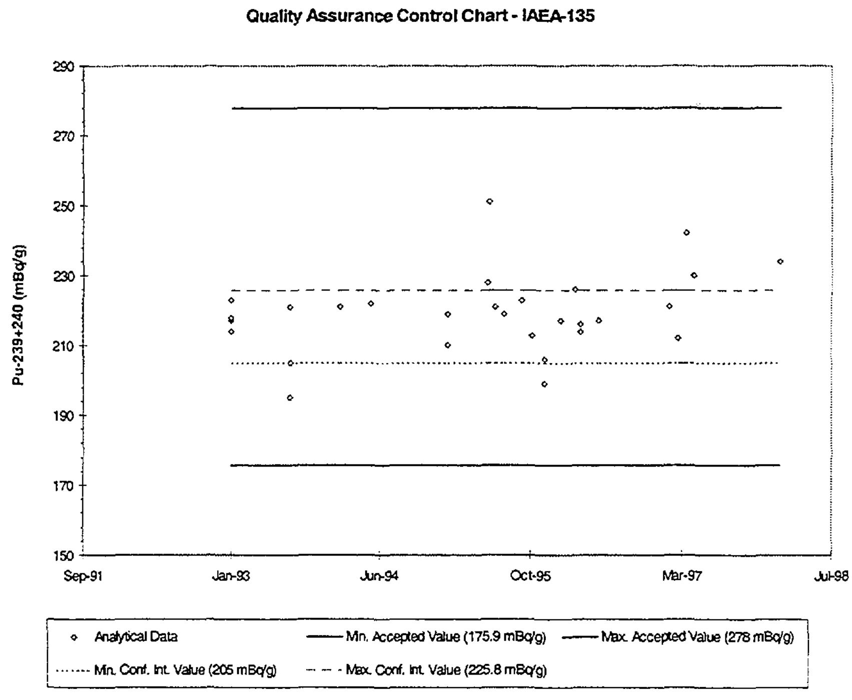


*FIG. 2. Quality control chart of analysis of $^{239,240}Pu$ in Irish Sea sediment (IAEA-135)*

reference material. The obtained data are mainly within a confidence interval of $\alpha = 0.05$, although a few exceptions were observed.

## 3. INTERCOMPARISON EXERCISES

Until now, both world-wide and regional intercomparison exercises have formed the main part of the AQCS programme [5]. They considerably helped Member State laboratories to assess their performance, supplied them with reference materials and enabled them to gather information on their performance. Although there has been remarkable growth both in the number of participating laboratories (from 40 to more than 150, see Fig. 1) and in the quality of data, we cannot yet be satisfied with the performance of all participating laboratories. As an example, Fig. 3 shows the performance of laboratories for $^{239+240}Pu$ analysis in marine sediment from the Irish Sea (IAEA-135). It can be seen that there are about 20 laboratories performing very well in this intercomparison exercise (the total number was 151 and Pu results were reported by 48 laboratories). Usually the best results are for $^{137}Cs$. Several laboratories have problems with analysis of other radionuclides e.g. $^{90}Sr$, $^{241}Am$, $^{210}Po$, $^{210}Pb$ etc. Also, the geographical distribution of participating laboratories is very irregular (Western Europe - 90, Eastern Europe - 33, North America - 17, Central and South America - 13, Asia - 16, Australia - 6, Africa - 3, the Middle East - 2).

In a more recent intercomparison exercise on Arabian Sea sediment (IAEA-315) out of 159 laboratories, 52 reported $^{239,240}Pu$ results (Fig. 4). Although the $^{239,240}Pu$ concentration was lower by about a factor of three, the performance of laboratories was better than in the case of IAEA-135.

Following the ISO recommendation [6] in a recently completed intercomparison run with Irish Sea water (IAEA-381) z-score evaluation was introduced. Fig. 5 shows that for $^{137}Cs$ and $^{239,240}Pu$, all participating laboratories have z-scores below 2, which indicates satisfactory performance (similar results were obtained for other radionuclides as well).

Several new marine samples for the next world-wide intercomparison exercises have been prepared, including Irish Sea fish (IAEA-382) Fangataufa lagoon sediment (IAEA-384), Irish Sea sediment (IAEA-385), Bikini Atoll sediment (IAEA-410) and Pacific Ocean sediment (IAEA-412).

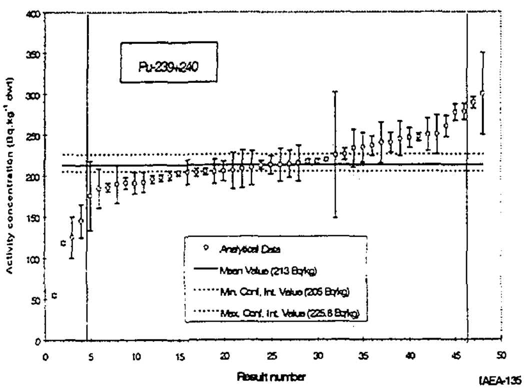


*FIG 3. Data evaluation for $^{239,240}Pu$ in Irish Sea sediment (IAEA-135)*

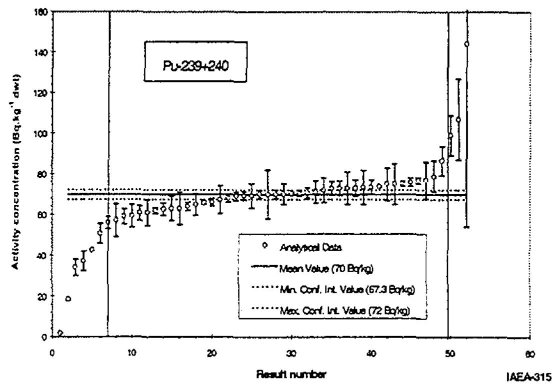


*FIG 4. Data Evaluation for $^{239,240}Pu$ in Arabian Sea sediment (IAEA-315)*

In order to improve the present situation in Member State laboratories it would be necessary to assist them with reference methods and protocols, to improve feedback from the intercomparison exercises by organising evaluation meetings to understand their analytical problems, to assist them with QA programmes and manuals and to organise QA oriented training and AQCS missions.

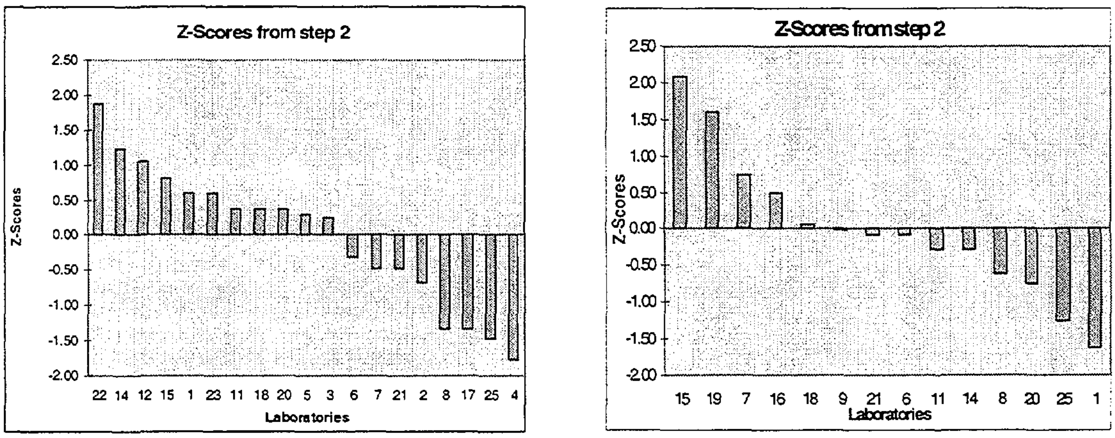


*FIG. 5. Z-scores (left $^{137}Cs$, right $^{239,240}Pu$) of participating laboratories in the intercomparison exercise on Irish Sea water (IAEA-381).*

## 4. REFERENCE METHODS

Reference methods [7, 8] represent a wide-ranging series of methods and guidelines used in marine radioactivity studies. Their format and terminology have recently been prescribed by the ISO [9], so they have a wide application throughout the world. They have considerable influence on the accuracy, precision and general reliability of data and represent a dynamic system based on current methods and new methods still under development. Periodic evaluation and modification of reference methods should keep the list up-dated with recent trends in analytical measurement techniques. Reference methods should be available for different sample matrices and elements. Their provision involves an extensive and time-consuming effort, including global cooperation and testing in expert laboratories.

## 5. REFERENCE MATERIALS

Reference materials (RM) represent samples of well established properties used for the assessment of analytical methods. More rigorous materials - certified reference materials (CRM) or standard reference materials (issued by the National Institute of Standards and Technology - NIST) have property values certified by technically valid procedures (at least with two independent methods) traceable to SI units [10].

RM and CRM have great impact on the development of methods of known accuracy. They represent important benchmarks in QA, identifying weak methodologies, detecting training needs, up-grading the quality of laboratories' performances and assessing the validity of analytical methods. The reference methods (or international standard methods) can only be accepted on the basis of interlaboratory tests performed on selected CRM.

The IAEA-MEL has until now produced RM as the final products of their world-wide intercomparison exercises. Regional exercises normally resulted in intercomparison samples with only recommended values. As RM and CRM represent high quality materials which usually cannot reasonably be used in everyday practice, laboratories produce their own secondary RM or working RM for frequent use in QA programmes.

With the aims of increasing the importance of the quality of data, of providing the required traceability to SI standards and of improving the accuracy and precision of Member State laboratories, the AQCS programme should also concentrate on the production of CRM. The CRM should be available for different environmental matrices e.g. sediment, water and biota.

The required long-term availability of CRM (over 10 years) necessitates their long-term stability and collection and preparation of large volume samples (over 100 kg). The relative precision of all reported data should be better than 5%. This would require highly homogenised samples thoroughly tested for any inhomogeneities of major elements (inhomogenieties should be below 2%). The principal analyses should be accompanied by supporting characterisation e.g. for multielemental composition, mineralogy, particle size distribution, radionuclide speciation studies etc. At least two independent analytical methods should be used for reporting certified values. This may not be a major problem in the case of radionuclides, where different methods are available (e.g. radiometric methods, ICPMS, TIMS, AMS, NAA etc.). Also, traceability to SI units seems to be more easily applicable to radionuclides than to elements and organic compounds. The interlaboratory testing should be based on expert laboratories only (10-20 laboratories).

Specific needs should be addressed for the production of CRM for mass spectrometric methods (e.g. AMS, ICPMS, TIMS) where usually small samples with very low radionuclide concentrations are necessary and require special treatment.

## 6. TRAINING

Training activities represent an important part of the complex AQCS programme. All three forms, i.e. long-term training in the Agency's or expert laboratories, training courses and expert missions to MS laboratories, can contribute significantly to the quality of data. However, more time should be devoted to all kinds of training in QA programmes. For example, the proposed calendar of IAEA-MEL training courses up to the year 2000 foresees, in some cases, up to 40% of time to be spent on QA programmes.

There is a general feeling that the Agency's support to developing Member States by supplying equipment in the framework of Technical Cooperation should be accompanied on a wider scale by the AQCS programme in order to create active laboratories producing reliable data.

## 7. ACCREDITATION

Laboratory accreditation, as a formal recognition by an independent research-based organisation that the laboratory is competent to perform specific analyses, has grown in importance in the last few years with the view to ensuring and maintaining high quality data. The accreditation process is beneficial both to the laboratories and to the users of their data. The objectives include the assurance of the validity of data, the acceptance of data without further tests, the credibility of laboratories, the promotion of good testing practices, the improvement of testing methods, the provision of technical information and the creation of competence.

The criteria for accreditation are very severe in order to maintain a high standard of analytical measurements. They include evaluation of the organisation, its staff, its facilities, the equipment, quality assurance, laboratory performance and protocols, quality of supplies, sample handling and recording system, the testing methods and procedures and the validation of performance and deficiency corrections.

To fully implement a complex AQCS programme in both the Agency's and MS laboratories, accreditation is the surest way forward. The Agency's laboratories should prepare for accreditation in accordance with the relevant ISO documents [5] and assist MS laboratories participating in the Agency's AQCS programme with their accreditation.

## 8. CONCLUSIONS

The Agency's AQCS programme, if taken in its complexity, can significantly improve the implementation of nuclear and non-nuclear analytical methods in MS laboratories. Data credibility is becoming essential in national and international programmes to which laboratories with different practices contribute. The AQCS programme should improve (a) the reliability of results from MS laboratories, (b) the comparability and compatibility of results and (c) their traceability to the SI system. The MS laboratories should benefit from regular participation in world-wide and regional intercomparison exercises, from intercomparison evaluation meetings, assistance in QA programmes and provision of QA manuals, supply of RM and CRM, analytical support from the Agency's laboratories (reference methods, AQCS missions) and long and short-term training. Recommendations to MS laboratories based on corrections to and improvements of their performance, evaluation of new ideas and developments in QA programmes, analytical methods and equipment, and finalisation of reference methods and protocols should considerably improve the quality of data.

The efforts of the AQCS programme should be directed towards international cooperation and coordination with active IAEA participation in standardisation activities.

New intra-Agency activities, like the development of internal QA programmes, reference methods, certified reference materials and development of the accreditation system, well harmonised in the different Agency units taking part in the AQCS programme, would considerably improve the Agency's output to the MS. The full implementation of the complex AQCS programme will, however, require new resources (manpower, budget) and wider cooperation with the Department of Technical Cooperation.

**Acknowledgement**

The IAEA-MEL operates under a bilateral agreement between the IAEA and the Government of the Principality of Monaco.

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