Another potential application, only superficially explored till now, is the high-level radiation treatment of sewage to render water polluted with pathogens sufficiently safe for re-use in agricultural irrigation. Many technical questions must be answered before this novel idea is proven to be feasible.

In summary, radioisotopes have been a valuable gift to many branches of medicine and biology. Many fundamental questions about the nature of life and human disease are being answered with this tool. The technological advances in computer science and electronics interlaced with newly developed radioactive substances are changing the face of modern medical diagnosis. With the growing number of radiotherapy machines more cancer victims have access to improved management of their disease. There is a basis of hope that further major improvements in radiation therapy may come from research in radiation biology, for instance by increasing radiosensitivity, but a cancer killing "atomic cocktail" is remote and unrealistic at present.

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Fig. 2. from McCready et al. in: Vol. II, Medical Radioisotope Scintigraphy (1973) p. 578. Proceedings of an IAEA Symposium.

Fig. 3. from Rejali et al. in: Dynamic Studies with Radioisotopes in Medicine (1970) p. 116. Proceedings of an IAEA Symposium.

The Growing Need for Analytical Quality Control

by O. Suschny and D.M. Richman

Technological development in a country is directly dependent upon its analytical chemistry or measurement capability, because it is impossible to achieve any level of technological sophistication without the ability to measure. Measurement capability is needed to determine both technological competence and technological consequence. But measurement itself is insufficient. There must be a standard or a reference for comparison. In the complicated world of chemistry the need for reference materials grows with successful technological development.

The International Atomic Energy Agency has been distributing calibrated radioisotope solutions, standard reference materials and intercomparison materials since the early 1960's. The purpose of this activity has been to help laboratories in its Member States to assess and, if necessary, to improve the reliability of their analytical work. The value and continued need of this service has been demonstrated by the results of many intercomparisons which proved that without continuing analytical quality control activities, adequate reliability of analytical data could not be taken for granted.

Analytical chemistry, lacking the glamour of other aspects of the physical sciences, has not attracted the attention it deserves, but in terms of practical importance, it warrants high priority in any developing technological scheme, because without it there is little chance to evaluate technological success or failure or opportunity to identify the reasons for success or failure.

The scope and the size of the future programme of the IAEA in this field has been delineated by recommendations made by several Panels of Experts; all have agreed on the importance of this programme and made detailed recommendations in their areas of expertise.

The Agency's resources are limited and it cannot on its own undertake the preparation and distribution of all the materials needed. It can, however, offer a focal point to bring together different international and national bodies which have an interest in quality control materials. Co-operation is sought with these bodies to co-ordinate efforts, avoid duplication of work and make the best use of available resources.

FUTURE PLANS

To explore the results of programmes over the past decade and to advise the Agency on how to best help laboratories to achieve accurate measurements in the nuclear and radioisotope fields with adequate precision, sufficient speed and at reasonable cost, a Consultants' Meeting on Analytical Quality Control was convened by the International Atomic Energy Agency in May 1973 in Vienna.

A systematic and practicable approach to this problem resulted from the meeting, and plans for reference materials, analytical quality control and related matters were drawn up.

- The future Agency programme will emphasize certification of more standard reference materials with their content of radionuclides and stable elements, and assist in the distribution of national standard reference materials for world-wide intercomparisons, where legal difficulties prevent their direct export from one country to another (i.e. uranium and plutonium).

- Priorities will be established in introducing new standard reference materials and reference materials in the broad fields of analyzing radionuclides and stable elements in nuclear materials, rocks and ores, environmental and medical samples.

- Standard reference materials must be produced with the highest quality, and it is essential that only well-established and recognized procedures be used. While the Agency would like to strengthen its own in-house scientific facilities and orient its efforts towards measurement work on standard reference materials, this would require a staff and facilities increase well beyond what appears practical for the IAEA in the near future.

- Since reliable analytical results are often the basis for critical discussions in assessing nuclear operations, environmental pollution, nutritional deficiencies and mineral resource potential, it is of paramount importance for international organizations and their Member States to have at their disposition the services of analytical laboratories of assured reliability.

- In the Agency's Safeguards programmes, improved availability and intercomparison opportunities involving reference materials for nuclear fuel analysis also will be useful. In the development of Safeguards procedures for evaluating the reliability of analytical data, considerable effort is being put forth to examine the systematics of chemical analysis, to identify the frequency of use of reference materials required, to define biasses introduced by particular methodologies, etc. This kind of activity may be of considerable use outside the Safeguards programme, in monitoring for water or air pollution for example. The Agency plans to exploit the broader applicability of those activities.

• In the field of nuclear fuel analysis one area of particular concern is the analysis of mixed uranium – plutonium oxide fuel materials. These are important to the advanced technological efforts in a number of Member States. Present analytical methods for assay for uranium or plutonium in mixed oxide materials are difficult to master. There is a need for a test material to examine the difficulties involved and it is anticipated that a co-ordinated research programme on some of the analytical problems of mixed uranium – plutonium oxide materials will be undertaken. This would involve obtaining a supply of well characterized (U – Pu) oxide material to be used as a test material. Particular problems to be examined would include dissolution of mixed oxides and analysis for U and Pu in the presence of each other.

PRESENT STATUS OF THE AQC PROGRAMME

The materials presently supplied are classified as follows:

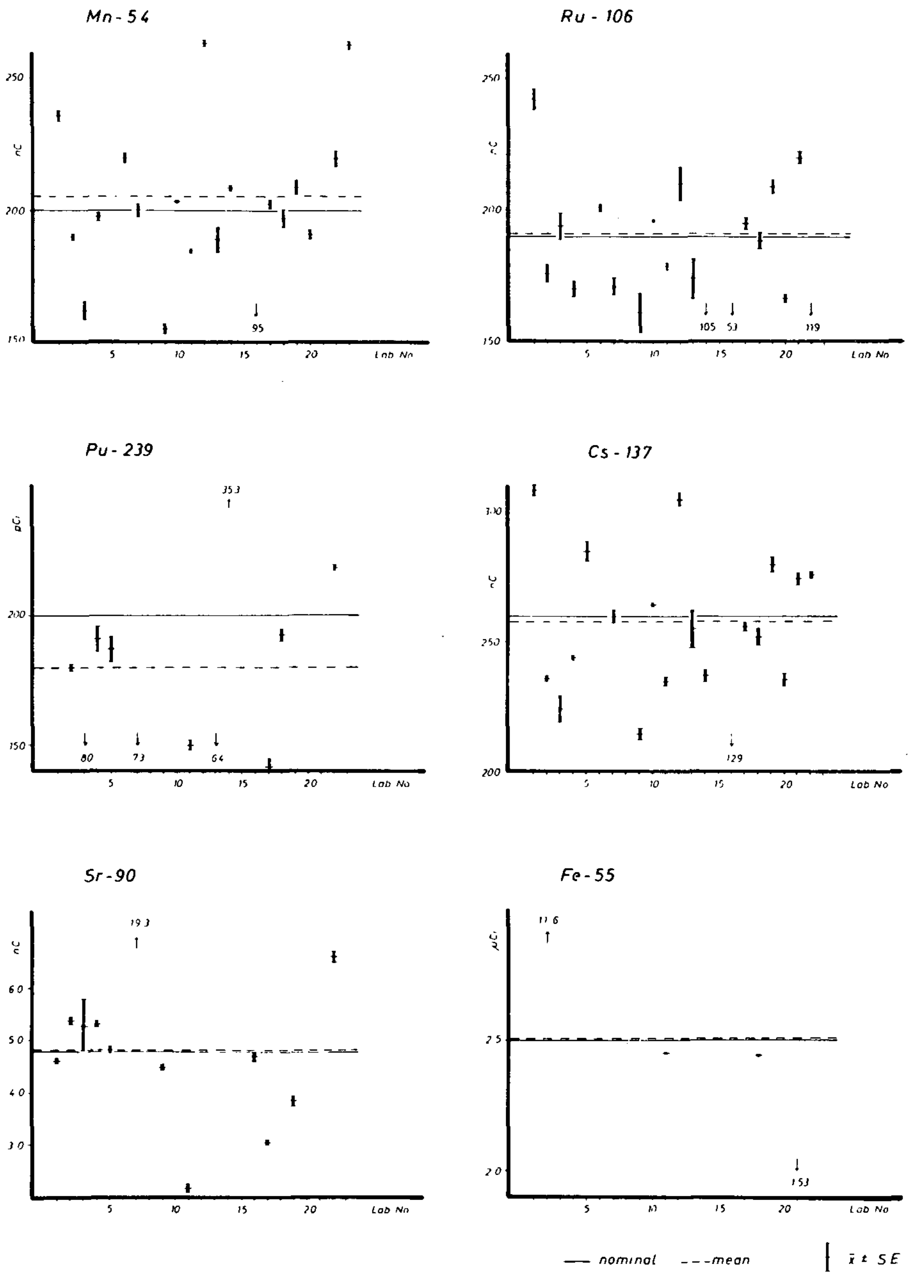
(a) **Standard materials (S):** these have been analyzed at one of the Agency's laboratories or at reliable contractor laboratories. They are supplied with a certificate describing their general composition as well as the content of the element or nuclide of interest and the method(s) by which this value was obtained. The uranium ore standards fall into this category. Occasionally the Agency also supplies standard samples prepared by national laboratories such as the NBS.

(b) **Intercomparison materials (I):** these are materials made available for intercomparison purposes. The homogeneous distribution of elements or nuclides to be analyzed in them is ascertained; their absolute concentration, however, is not always known. They are distributed free of charge (except for postage if its cost is considerable) with the *understanding that the laboratories ordering them report results to the IAEA on a* confidential basis for use by the Agency.

(c) **Reference materials (R):** these are materials which have been previously used for intercomparisons so that their contents have been determined. Generally, the degree of accuracy with which these contents can be named is inferior to that of standard materials.

A list of materials available appeared in Volume 15, no. 3, the June 1973 issue of this Bulletin.

Many of the analytical activities covered by these materials also are of interest to other international, regional and national bodies. The Agency has exchanged ideas with the World Meteorological Organization (WMO) concerning air filter studies (1) and improving accuracy in the determination of air pollution. Samples of medical interest have led to contact with the World Health Organization; stable element materials which are relevant to pesticide studies (2) and other agricultural applications have been discussed with FAO with whom the Agency has a Joint Division. The Agency has also consulted or co-operated on standards and intercomparisons with the BIPM, UNSCEAR, IUPAC, ISO, EURATOM (3) and other international and inter-regional as well as national organizations



AIR FILTER INTERCOMPARISON 1972 · Air-1 · Six radionuclides on air filters and offices. Such co-operation is expected to continue. In addition, an informal network of reference laboratories has been set up by the Agency, selected on the basis of their reputation and continued reliability, to assist in the analysis of reference materials.

The costs of preparing certified reference materials are high and are influenced by the time and effort required. In view of the direct gain through elimination of significant analytical errors, however, costs should not be considered a limiting factor. The efficient use of materials that are expensive demands an awareness of purpose of use, and secondary materials may be adequate for many purposes.

HISTORY OF THE PROGRAMME

In the past ten years the Agency has been active in carrying out analytical intercomparisons of radioactive materials, of materials relevant to atomic energy programmes, and of materials for which analysis by nuclear techniques has seemed preferable to other techniques.

Intercomparisons involve the evaluation of laboratory performance by submitting a sample of the same material to each of several laboratories for analysis. The results of their analyses are compared to indicate ability or inability of the laboratories involved to produce accurate results.

For intercomparison the samples distributed were prepared to closely resemble those materials routinely analyzed by the participating laboratories. They were selected to have reasonable stability with a general composition similar to the materials routinely analyzed and with a comparable concentration of the nuclides or elements to be determined. Homogeneity of the samples was, in all cases, assured down to the required size-range.

More recently, the Monaco laboratory has contributed intercomparison materials for marine radioactivity studies and Seibersdorf has added some uranium and plutonium compounds to be used in safeguards work.

In addition, a number of materials was added to serve the need of scientists working on trace element determinations by nuclear-based methods, mainly neutron activation analysis.

When many countries embarked on prospecting for uranium and other elements, the need arose for ore standards to check on results obtained both in the field and at the supporting laboratories. Three different ore types were used to prepare 4 batches of standards all in the 0.2 - 0.4% range of uranium concentrations. Certification of these was based on analyses carried out at several national laboratories. Recently, to satisfy demand for standards of lower-grade ores, two additional materials in the 0.01 - 0.05% range of uranium concentration were added. The Isotope Hydrology section, meanwhile, prepared two water standards for isotopic ratio analysis. The oxygen and hydrogen isotope ratios were intercompared by 25 laboratories and these standards, together with two older standards distributed in the past by the National Bureau of Standards, but since transferred to the IAEA, are now available from Vienna.

The results of the Agency's efforts in quality control in the nuclear and isotopes fields show that the situation in these fields is qualitatively similar to that in other fields. Quantitatively, however, it may be worse since the number of newcomers and inexperienced laboratories is relatively larger in nuclear techniques than in conventional chemistry. A few examples from the many results of intercomparisons which the Agency has collected, evaluated and sometimes published in the course of the last few years (4-9), will show this.

The first example is from two intercomparisons of the analysis of several radionuclides on air filters carried out on request of the Commission on Instruments and Methods of Observation of the WMO (1). In the first one, organized in 1968-1969, six radionuclides were used, viz. ^{54}Mn, ^{55}Fe, ^{90}Sr, ^{137}Cs, ^{144}Ce, and ^{239}Pu, at levels ranging from 10 pCi to 1 nCi. From the ten participating laboratories, no results were received for ^{55}Fe at the 1 nCi level. The majority of the results received for the other nuclides did not show unusual spread and their overall means did not deviate too much from the true value of the spike added, however, only three out of the ten laboratories reported on ^{239}Pu and only four on ^{144}Ce. Of the 31 averages received for all nuclides taken together, 5 had to be rejected as outliers.

A similar intercomparison was organized in 1972. The two cannot strictly be compared, since in the new intercomparison ^{106}Ru was substituted for ^{144}Ce and the activity level of all nuclides was considerably higher than in the earlier intercomparison (the new one was aimed at analysis in emergency situations). Participation was much better, however, than in the earlier intercomparison (20 laboratories compared to 10) and the number of laboratory averages received was 84 based on 729 individual determinations. Agency investigations include not only radioactive materials but also materials in the analysis of which radiochemical and nuclear methods may be used to advantage (10). Determinations of environmental mercury fall into this category.

Mercury pollution from industrial as well as agricultural uses of the element has caused concern in the last few years and many laboratories have started to analyse for it in environmental materials. The concentrations looked for are between one and one hundred ppb and at this low concentration range the great sensitivity of neutron activation analysis for mercury has made this method one of the methods of choice. To assist laboratories to control their reliability and at the same time to assess the general status of analytical performance in this type of work, the Agency distributed nine different materials containing environmental concentrations of mercury (11, 12). The results of the nine different intercomparisons showed that roughly one third of the 407 individual values (92 laboratory averages) had to be excluded as grossly erroneous. The remaining results still gave standard deviations varying from 23% to 65% of the mean. A separate evaluation of the results according to the analytical methods employed did not show any difference in performance between neutron activation analysis which was used by two thirds of the participating laboratories and atomic absorption which was used by most of the others.

It can be seen from the recommendations of several Consultants' Meetings and Panels organized in the past few years, and from the results of the intercomparisons, that the future plans for the IAEA Analytical Quality Control Programme result from a thorough examination of the needs of the Member States in a number of fields having analytical chemistry as their all-important common denominator. The IAEA will be pleased to receive comments or enquiries regarding this programme. More details are available from:

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