Accreditation of Laboratories for Measurement of Radionuclide Content in Commodities

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Atomic Energy Regulatory Board
Niyamak Bhavan, Anushakti Nagar,
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Foreword

Measurement of radionuclide content in commodities (including foodstuffs and drinking water) has become an important regulatory requirement for international trade. The International Atomic Energy Agency (IAEA) and World Health Organisation (WHO) have specified the radioactivity concentration limits for various commodities and different radionuclides. Currently, such measurements are being carried out in laboratories of various units of the Department of Atomic Energy (DAE). Such measurements can also be carried out in academic institutions, national laboratories and private institutions. All such institutions have to be accredited by an appropriate authority to ensure proper standards and quality assurance in the measurements of radioactivity. The Atomic Energy Regulatory Board (AERB) is entrusted with the responsibility for laying down safety standards and framing rules, regulations and procedures covering regulatory functions envisaged under the Atomic Energy Act, 1962. Therefore AERB has undertaken a programme of accreditation of laboratories for measurements of radionuclide content in commodities.

This booklet describes the operational and technical requirements to be met by laboratories desiring accreditation for measurements of radionuclide content in commodities. The booklet also contains performance testing procedures, post accreditation responsibilities of the laboratories and a format of the application form for seeking accreditation. The booklet has been prepared by a Working Group consisting of AERB staff and other professionals from DAE. In drafting it, extensive use has been made of the information contained in relevant documents of IAEA and the rich experience of DAE in the area for the past five decades. The list of experts who have contributed to the preparation of the booklet is appended in the document.

I hope this booklet will serve the intended purpose.

(Suhas P. Sukhatme)
Chairman, AERB

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Accreditation of Laboratories for Measurement of Radionuclide Content in Commodities

1. Introduction

Measurement of radionuclide content in commodities (including foodstuffs and drinking water) has become an important regulatory requirement for international trade. The International Atomic Energy Agency (IAEA) and World Health Organisation (WHO) have specified the radioactivity concentration limits for various commodities and different radionuclides. Currently such measurements are carried out in laboratories of various units of the Department of Atomic Energy (DAE). Certain academic institutions, national laboratories and private institutions in the country have expressed their readiness to carry out such measurements. Clearly, such institutions have to be accredited by competent national authorities following appropriate procedures to ensure proper standards and quality assurance in the measurement of radionuclide content. It is recognised that the reliability of the results from such laboratories depends to a great extent on the quality of facilities, manpower and procedures followed by the laboratories. It is therefore recommended that such laboratories adopt the procedures laid down in this booklet and participate in interlaboratory comparisons to establish and maintain the validity of their results.

2. Purpose and Scope

The purpose of this booklet is to provide the details of administrative, operational and technical requirements for the accreditation of laboratories for measurement of radionuclide content in commodities. The booklet also gives details of requirements related to infrastructure and qualified staff for a laboratory seeking accreditation. The important radioactivity measurements that are carried out in the context of trade of commodities are for gross alpha, gross beta and ^{137}Cs content. There are often requirements to measure concentrations of naturally occurring radioactive materials (NORM) in commodities. Hence the booklet gives procedures for these measurements. Procedures followed by the accrediting agency for issuance of accreditation are also given in the booklet.

3. Accrediting Agency

Chairman, Atomic Energy Regulatory Board (AERB) is the Competent Authority for radiation surveillance of radiation workers, members of the public and the environment in India. He is the Competent Authority for issuance of accreditation to all laboratories (including DAE laboratories) engaged in the measurement of radionuclide content in commodities. A Standing Committee appointed by the Chairman, AERB recommends all procedures and terms and conditions for accreditation. It reviews all applications from laboratories. Based on the recommendations of the Committee accreditation may be issued by the Chairman, AERB to a particular laboratory for carrying out measurement of radionuclide content in commodities.

4. Infrastructure Requirements

Accreditation will be granted to a laboratory having qualified and trained staff, adequate space and equipment and other requisite facilities, on demonstration of adequate procedures and documentation. The following are the basic requirements to be satisfied by the laboratory.

4.1 Laboratory Layout

Annexe-1 gives a typical layout of a laboratory designed to carry out measurement of radionuclide content in commodities. The laboratory should be designed and built according to the requirements of a normal analytical laboratory with a few additional features such as good ventilation, specially designated areas for receiving and storing samples. Construction materials should be selected so as to have a minimum of background from natural radioactivity and the floors should be capable of carrying a load of at least 2000 kg/m^2 in order to support heavy detector shields.

4.2 Facilities and Equipment

The laboratory shall maintain adequate facilities, equipment, chemicals, and calibration facility with relevant radiation sources so as to accomplish the required function all the time.

4.2.1 Instrumentation Requirements

4.2.1.1 For Alpha Measurement

Alpha counting setup comprising of a ZnS(Ag) counter having an efficiency of about 25% and background 0.002-0.005 cps. Minimum Detectable Activity (MDA) should be 0.01 Bq with 5000 secs counting time.

4.2.1.2 For Beta Measurement

System 1: For measurement of gross beta, the end window GM based counting set up should have an efficiency of more than 10%, maximum background 0.25 cps and a minimum detectable activity of 0.2 Bq with 5000 secs counting time.

System 2: Low background beta counter of background less than 0.05 cps with an efficiency of 30-35%. Minimum detectable activity of 0.03 Bq with 5000 secs counting time should be achieved.

4.2.1.3 For Gamma Measurement

Gamma ray spectrometer system consisting of NaI(Tl) gamma detector of minimum size 7.6 cm x 7.6 cm or HPGe detector of minimum relative efficiency of 20% with respect to 7.6 cm x 7.6 cm NaI(Tl) detector, single channel analyser and multichannel analyser, amplifier, detector bias supply, liquid nitrogen cryostat in case of HPGe. The detector should be housed in shield with adequate lead thickness. The detector is normally housed in a cubicle of internal length, breadth and height of 45 cm each with a shield wall thickness of 7.5 cm Pb with graded lining (2 mm Cd and 1 mm Cu). For very low level counting, it is preferable to have 15 cm Pb thickness (with graded lining) all around the detector including at the top.

4.2.2 Radionuclide Standards for Efficiency and Energy Calibration

Testing of the performance of different counting systems

For alpha counter – ^{239}Pu or ^{241}Am or Natural Uranium

For beta counter – ^{90}Sr, ^{40}K or ^{204}Tl

For gamma counter – point sources of ^{137}Cs and ^{60}Co

For calibration and efficiency of the different counting systems

The standard sources should be certified sources and should be available in geometry similar to the processed sample for counting.

For alpha counter – ^{239}Pu or ^{241}Am or Natural Uranium

For beta counter – ^{90}Sr or ^{40}K made from analar KCl

For gamma counter – ^{137}Cs, ^{22}Na, ^{60}Co, ^{54}Mn and ^{133}Ba.

For efficiency determination of NORM in different matrices - Thorium ore, Uranium ore and ^{40}K as standards.

4.2.3 Laboratory Equipment and Chemicals

The following items should be available in the laboratory undertaking measurement of radionuclide content in commodities. Freezer for storing samples, laboratory fume hood, hot air oven (120°C), muffle furnace which can go upto 500°C for obtaining white ash, crucibles, distilled water unit, silica dish, magnetic and electrical stirrer, distill water, hot plate, filter paper circles, funnel stand, millipore filtering assembly, pressure vacuum pump, balance range 10 g –1000 g with error ± 1 g, single pan analytical balance (0.1 mg), infrared lamp, centrifuge, stainless steel and aluminum planchets of 25 mm diameter, lanthanum and bismuth carriers, AR grade chemicals like ammonium phospho molybdate (AMP), HCl, HNO_3, H_3PO_4, acetone, colladion, glassware like beakers, petridishes, water glasses, pipette, centrifuge tubes, glass rods, etc. and Marinelli beaker of 1 litre capacity for gamma ray spectrometry.

4.3 Standard Analytical Methods for Measurement of Radionuclide Content in Certain Matrices

4.3.1 For Drinking Water Samples

The methods given in the IS 14194 (Part I & II), 1994 shall be followed for the measurement of gross beta and gross alpha activities in the drinking water samples. The results should be expressed as Bq/l with uncertainty (1 σ).

4.3.2 Alternative Method for Measurement of Gross Beta Activity

(a) Reduce the volume of water from 500 ml to about 100 ml after acidification with 2 ml of HNO_3/HCl. Transfer the content completely into a leak proof plastic

container. Count for ^{137}Cs using NaI(Tl)/HPGe. The system should be calibrated with certified standards of the same geometry. (Activity A). OR

(b) Acidify 100 ml of water with 1 ml HNO_3. Add 2 mg of Cs carrier and stir with 25 mg of AMP for 30 minutes. Transfer the AMP to a tared 2.5 cm diameter aluminum planchet after washing once with distilled water. Count in a low background beta counter. (Activity A).

(c) For water with high dissolved solids (> 200 mg/l), follow procedure given in sections 6.2.1 to 6.2.4 of IS 14194(Part I), 1994. (Activity B). Gross beta activity is A + B.

4.3.3 For Food Items

Powdered and homogenised dry sample may be filled in a suitable pre-weighed geometry like plastic bottles, Marinelli beakers or any other container designed specifically for the purpose and subjected to gamma ray spectrometry using NaI(Tl) or HPGe. The weight of the sample transferred in that particular geometry should be noted. The system should be calibrated with certified standard source in similar matrix and similar geometry. ^{137}Cs activity in the samples should be expressed in Bq/kg.

4.3.4 For Soil, Flyash, Ores and other Environmental Samples for NORM

After sieving, preferably using a 150 micrometer mesh, the samples are transferred to a porcelain dish and dried in an oven at 110°C for an hour. This dried sample is transferred to a 250 ml plastic container, packed to its full volume and then sealed with an adhesive tape. This sealing is to ensure that all the daughter products of uranium and thorium and in particular radon and thoron daughters that would be formed thereafter would not escape. These prepared samples should be stored for 30 days to ensure equilibrium between radium and its short lived daughters. The net weight of the sample is to be determined before subjecting the sample to spectral analysis. (Note: The above guidelines may be given to the client or followed by the accredited laboratory.)

4.4 Qualified Manpower

The laboratory carrying out measurements of radionuclide content in commodities shall have at least the following team.

Laboratory Officer incharge: M.Sc. (Physics/Chemistry) or B.Sc with 5 years experience in similar work – 1 post

Laboratory Assistant: B.Sc. (Physics/Chemistry) – One or more (depending on the workload)

- At least one of the above two persons should be a chemist and
- At least one of the above two persons should have undergone minimum one month training in a similar analytical laboratory

Helper – X standard or equivalent with experience in chemical laboratory work.

5. Procedures for Issuance of Accreditation

5.1 Submission of Application

The laboratory undertaking measurement of radionuclide content in commodities shall apply to Chairman, AERB in the prescribed format as given in Annexe-2 for issuance of accreditation. The applicant may seek accreditation for:

(i) Gross alpha and gross beta measurement or

(ii) Gross gamma measurement or

(iii) Both (i) and (ii)

The application form shall be completed and signed by the authorised representative of the laboratory seeking accreditation. The following documents shall be submitted by the laboratory alongwith the application:

(a) Procedures for the measurement of radionuclide content in the samples and likely samples to be analysed in a year, laboratory workload, laboratory details such as facilities, layout, etc

(b) Staff and their bio-data alongwith the work responsibilities,

(c) Quality assurance programme, and

(d) Reports on participation in any intercomparison programmes.

5.2 Pre-accreditation On-site Assessment Visit

The application form shall be reviewed by the AERB Committee for the accreditation of laboratories for the measurement of radionuclide content in commodities and the laboratory may be notified of any additional information, which shall be supplied prior to on-site assessment visit. If the details given in the application form are found to be satisfactory, the AERB Committee shall conduct an on-site assessment visit. The assessment visit shall comprise of:

- A meeting with the management and supervisory personnel responsible for the laboratory activities and discussions with technical personnel to ensure their understanding of the procedures,
- Demonstration of performance testing,
- Examination of major equipment, apparatus and facilities for their appropriateness, capability, adherence to specifications, etc.
- Review of personnel records and job descriptions of key personnel, competency of all the staff members, calibration procedures and test reports (if any) and quality assurance programme.

The Committee shall submit its assessment report to Chairman, AERB.

5.3 Performance Testing

After meeting all the requirements indicated during the on-site assessment visit, the laboratory would be asked to demonstrate satisfactory performance as per the procedure given in this booklet. The results shall be submitted to the AERB Committee for evaluation and the laboratory shall not be permitted to change or void the reported values after they are released. Simply passing a performance test after multiple attempts may not qualify as satisfactory proficiency.

5.4 Quality Assurance Programme

The laboratory undertaking the measurement of radionuclide content in commodities shall have a well-documented programme for quality assurance procedures to be

followed for the measurement of radionuclide content to demonstrate compliance and satisfactory performance. The quality assurance programme shall comprise of routine maintenance of the equipment, background check, calibration of measurement systems with radionuclide standards. The validity of analytical results should be checked by an internal programme, which incorporates the use of blind duplicates, blanks and standards. The laboratory shall maintain adequate backup equipment or systems for key processing steps to be used in the event of failure of a system. Continuity of quality service is a commitment of the laboratory. If it fails to provide the committed service at any point of time, the accreditation shall stand void. The International System (SI) units and symbols shall be used for presentation of the results of the analysis. Some standard units of presentation are: water and milk in Bq/l, foods in Bq/kg fresh mass, etc.

6. Conditions of Accreditation

Based on the information submitted by the laboratory through the application, report on the on-site assessment visit, actions taken by the laboratory to correct any deficiencies and the results of the performance tests, the AERB Committee shall recommend to Chairman, AERB for issuance of accreditation for the laboratory to undertake measurement of radionuclide content in the commodities. Each accreditation shall be valid for a period of three years subject to meeting the terms and conditions of the same. The accredited laboratory shall not partly or fully subcontract the testing of commodities to any other laboratory. Application for renewal should be submitted at least six months before the expiry of the accreditation. Renewal of accreditation may be granted depending upon the results of periodic quality assurance tests and the performance of the laboratory. During the period of validity of the accreditation, the laboratory shall be responsible for maintaining compliance pertaining to the availability of qualified and trained manpower, adherence to operational procedures as outlined in this booklet and quality assurance programme. In case of any modifications or changes in any of these stipulations, the laboratory shall inform the AERB Committee immediately. The Committee shall review the status of the laboratory in the light of the information submitted and give its recommendations to Chairman, AERB.

7. Post Accreditation Requirements

7.1 Documentation and Record-keeping

The laboratory shall have documents on the operational procedures followed for measurement of radionuclide content in commodities, details of the equipment used, quality assurance programme, organisational set-up and responsibilities of persons working in the laboratory. These documents shall be readily accessible to any member of the laboratory and also during the visit of the AERB Committee. These documents should be reviewed and updated in the light of any modifications or changes.

7.2 Intercomparison Exercises

It is important that the laboratory participates in intercomparison exercises with other accredited laboratories undertaking similar analysis at national as well as international levels. This will help to detect systematic errors if any arising due to improper calibration or calculation. Samples used in such comparisons should be, as far as possible, similar in composition and concentration to the samples to be analysed on a routine basis.

7.3 Training Programme for Staff

Any new staff member should receive practical training for the assigned responsibility and the details of the staff training programme should be intimated to the AERB Committee. Periodic refresher training programme should be conducted for all the staff members of the laboratory.

8. References

1. Measurement of Radionuclides in Food and the Environment, A Guidebook, Technical Report Series No. 295, IAEA, Vienna, 1989.
2. Radionuclides in Environmental Samples – Methods of Estimation, Part I Gross Beta Activity Measurement, IS 14194 (Part I), 1994.
3. Radionuclides in Environmental Samples – Methods of Estimation, Part I Gross Alpha Activity Measurement, IS 14194 (Part II), 1994.
4. Environmental Radiological Laboratory Procedure Manual (1998), Complied by A.G. Hedge, L.N. Sharma, S. Chandramouli, D.D. Rao, V. Matkar, P.C. Verma and M.P. Rajan and reviewed by K. Rudran, M.A.R. Iyengar, R.P. Gurg, and M. Sundaram, Health Physics Division, BARC.

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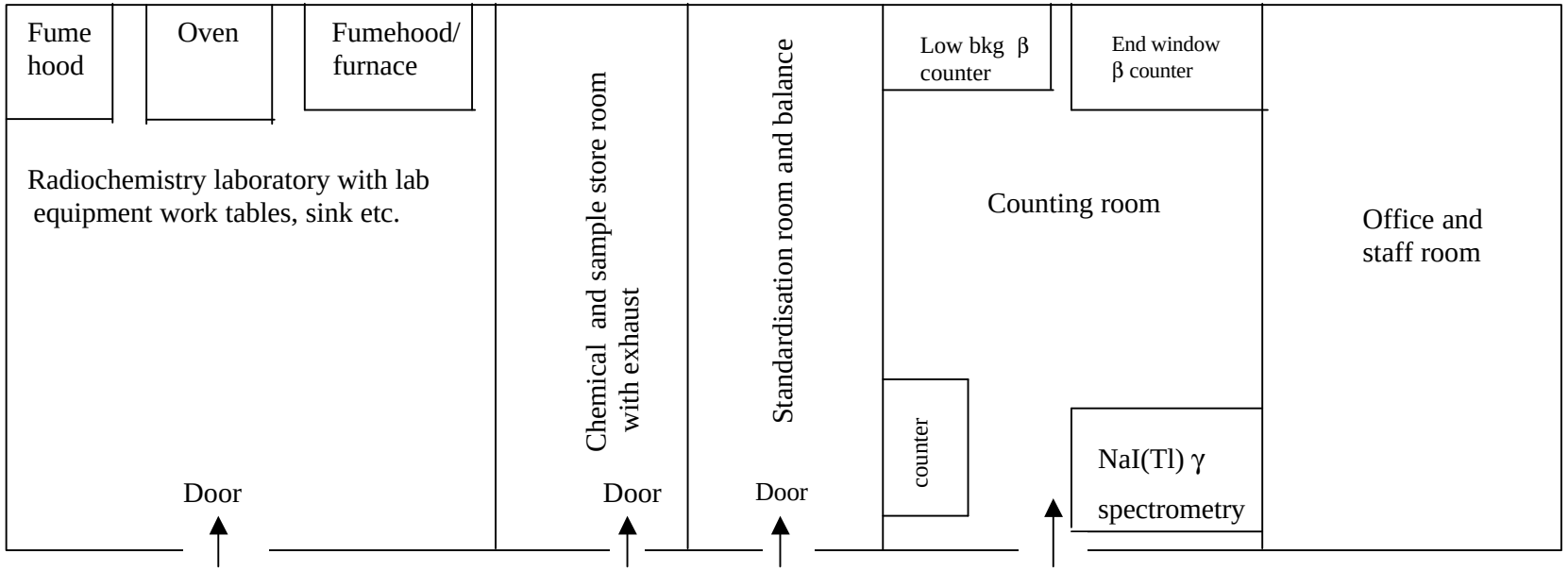
Layout of a Typical Laboratory for Measurement of Radionuclide Content in Commodities



ANNEXE-1

ANNEXE – 2

FORMAT OF APPLICATION FORM FOR ISSUANCE OF ACCREDITATION TO LABORATORY FOR UNDERTAKING MEASUREMENT OF RADIONUCLIDE CONTENT IN COMMODITIES

1. Name, Designation and Official Address of the applicant (with Tel. Nos., FAX No. and e-mail address) :

2. Name and Address of Head of the Institution :

3. Commodities to be analysed for radionuclide content content :

4. Facility for gross alpha measurement (Please provide details) :

5. Facility for gross beta measurement (Please provide details) :

6. Facility for gamma measurement (Please provide details) :

7. Radioactivity standards for calibration :

8. Analytical procedures to be followed (Brief description in a Annex) :

9. Details of Staff involved in the measurements

Name and designation	Educational Qualification	Training	Experience

10. List of documents submitted along with the application:

11. Please enclose a sketch of the laboratory, indicating the location(s) of radioanalysis room, counting room, storage for samples, chemicals, etc.

12. Any other relevant information:

I hereby certify that all the statements made above are correct to the best of my knowledge and belief.

Place:

Signature:

Date: **Name (Applicant)**

Signature:
Name (Head of the Institution)

Seal of the Institution

Mailing Address:

To
Head, Radiological Safety Division
Atomic Energy Regulatory Board
Niyamak Bhavan, Anushaktinagar
Mumbai – 400 094.

ANNEXE-3

FORMAT OF A TYPICAL RADIOACTIVITY TEST CERTIFICATE ISSUED BY THE ACCREDITED LABORATORY

RADIOACTIVITY TEST CERTIFICATE

Certificate Number:

Date of Issue:

This has reference to the request from the institution M/s. ---------- vide their letter No. ---------- for measurement of radionuclide content in the ---------- samples submitted by them. The results of analysis are as follows:

1. Name and address of the laboratory which has carried the analysis:

2. Accreditation No. of laboratory:

3. Method of analysis:

4. Results of gross alpha measurement analysis:

5. Results of gross beta measurement analysis:

6. Results of gamma spectrometry:

Place:

Signature & Name
of the Officer-in-charge of the Laboratory:

Date:

Seal of the Institution