भारतीय मानक
Indian Standard

IS 14194 (Part 2) : 2022

पर्यावरण नमूनों में रेडियोन्युक्लाइड — आंकलन की पद्धतियाँ

भाग 2 कुलअल्फा सक्रियता मापन

(दूसरा पुनरीक्षण)

Radionuclides in Environmental Samples — Methods of Estimation

Part 2 Gross Alpha Activity Measurement

(Second Revision)

ICS 13.020.40; 13.280

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FOREWORD

This Indian Standard (Part 2) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Nuclear Energy for Peaceful Applications Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1994 for measurement of gross alpha activity in the environmental samples. While formulating standard, it was kept in view that long lived gross alpha activity of air filter sample can be used to detect any air contamination especially Plutonium (Pu). In case of water samples, gross activity determined by evaporation of an aliquot can indicate the trend of contamination especially for the waters receiving wastes from fuel reprocessing plant.

This standard was subsequently revised in 2013 in view of the technological advancement in this field. In first revision, modifications were made in sample size, activity measurement procedure and in calculations. Requirement of calibration was also incorporated in the standard.

This second revision has been undertaken in order to upgrade the test method procedure as per the current practice followed by the industries. In this revision, the following changes have been made:

- Principle of the test method has been elaborated;
- Efficiency of the alpha counting system has been prescribed;
- Standard source for calibration ^{239}Pu has been prescribed;
- Limit for low and high total dissolve solid (TDS) in water sample has been prescribed; and
- Test method procedure for water sample has been modified.

All laboratories carrying out analysis or measurement of radioactivity in commodities shall be duly certified by Atomic Energy Regulatory Board, Govt of India.

This standard is being published in five parts. Other parts of this standard are:

Part 1 Gross beta activity

Part 3 Uranium

Part 4 Radium

Part 5 Sampling

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

RADIONUCLIDES IN ENVIRONMENTAL SAMPLES — METHODS OF ESTIMATION PART 2 GROSS ALPHA ACTIVITY MEASUREMENT

(Second Revision)

1 SCOPE

This standard (Part 2) prescribes the method of test for measurement of gross alpha activity in the environmental samples (particularly air and water).

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
264 : 2005	Nitric acid (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water-Specification (*third revision*)
10332 : 1982	Hydrofluoric acid, aqueous

3 TERMINOLOGY

For the purpose of this standard the following terms shall apply.

3.1Activity

The number of spontaneous nuclear disintegrations occurring in a certain quantity (weight/volume) of material during a specified time interval, divided by that time interval. It is expressed in Becquerel (Bq), where 1 Bq = 1 disintegration/second. Earlier unit of activity was Curie (Ci), where 1 Ci = 3.7 x 10^{10} disintegrations/second.

3.2 Nuclide

A species of atom characterized by its mass number, atomic number and nuclear energy state, provided that the mean life in that state is long enough to be observable.

3.3 Radionuclide

An element, with a certain mass number and atomic number, which undergoes spontaneous disintegration in a measurable period of time.

4 PRINCIPLE

Alpha particle incident on silver activated zinc sulphide [ZnS(Ag)] powder spend their energy completely in raising the valence band electrons into conduction band. The electron from the excited state returns to the ground state, either directly or through activator sites. The loss of energy appears as visible/ultraviolet (UV) light when they reach the ground state. Cathode of multiplier tube (PMT) is positioned in such a way that it absorbs full light energy and emits primary electron. PMT multiplies the primary electron and develops a current signal at the output of anode which is amplified and saved to register it in a counter.

5 APPARATUS

5.1 Alpha Counting System

Comprising of a ZnS (Ag) detector, with sample holder in close proximity of the detector, is capable of housing stainless steel planchets, of 2.5 cm diameter. The background of the system is about 0.002 counts per seconds (cps) and the efficiency for the plated ^{239}Pu source is 25-30 percent. The efficiency is largely influenced by thickness of sample. Minimum detectable activity (MDA) for unit volume of sample is 8.9 mBq for a counting time of 5 000 s. The system shall be so sensitive and capable to detect gross α radioactivity below 0.1 Bq/l in the drinking water sample when measured as per the described method.

5.2 Efficiency of the Alpha Counting System

An electroplated standard source of ^{239}Pu is counted on the ZnS (Ag) detector for 3 600 s. Efficiency of the detector is obtained by the following formula:

$$\text{Efficiency (\%)} = \frac{\text{Counts per second x 100}}{\text{Disntegration per second}}$$

5.3 Stainless Steel Planchet — of 0.5 mm thick and 25 mm in diameter.

5.4. Infrared Lamp — of 500 W

6. REAGENTS

6.1 Fe Carrier

A carrier of 5mg/ml is to be prepared by adding 1.45 g of ferric chloride in 100 ml of ultrapure water.

6.2 Ba Carrier

A carrier of 2mg/ml is to be prepared by adding 0.2 g of barium sulphate in 100 ml of ultrapure water.

6.3Alpha Calibration Standard Source — electroplated source of ^{239}Pu

7 PROCEDURE

7.1 Air Filter for Long Lived Alpha Activity

7.1.1 Collect air particulate sample on Millipore or glass fibre or Whatman No. 40 filter paper of 25 mm diameter placed in the air sampler. Filter about 5 m^3 of air. Carefully transfer the filter paper along with particulate matter to α counting system and count the α activity after allowing the short lived radon daughter products to decay. Calculate the activity per cubic metre of air based on the flow rate (m^3/s) and time of filtration.

7.2 Water Samples with Low Dissolved Solids (< 100 mg/l)

a. The collected water sample, present in a pre-conditioned polyethylene bottle, is properly labelled with a code including the following details: Sample code, date of collection, sampling location and sample type. Prior to sample processing the water sample is passed through 0.45 µm filter using vacuum filtration assembly, to remove the suspended solids. Thereafter the total dissolved solids (TDS) is measured in the filtered water sample. The residue is discarded. The filtered sample, in a glass beaker, is further processed for gross alpha measurement.

b. Evaporate 100 ml sample to 10 ml in a clean glass beaker at 100 °C on a hotplate. Sputtering of liquid and overheating are to be avoided.

c. Add nearly 10 ml of pre-concentrated sample on a stainless steel planchet, with an increment of

2ml at a time using a graduated pipette.

d. Completely evaporate the liquid on a new, background counted stainless steel planchet under infrared lamp.

e. Heat the planchet to a red hot condition.

f. Count the cooled planchet for gross alpha in an alpha counting system.

7.3 Water Samples with High Dissolved Solids (≥ 100 mg/l)

a. Take 1 liter of filtered water sample.

b. Acidify with 2 ml conc nitric acid.

c. Add 5 mg/ml of Fe carrier and 2 mg/ml of Ba carrier.

d. Add ammonia and 50 percent ammonium sulphate (to precipitate ferric hydroxide [$Fe(OH)_3$] and barium sulphate (to precipitate radium barium sulphate). Ammonia is added dropwise to ensure complete precipitation.

e. Centrifuge and transfer the precipitate on new, background counted stainless steel planchet of 2.5 cm diameter (the precipitate thickness should not be more than 10 mg/cm^2).

f. Evaporate it under an infrared (IR) lamp and heat the planchet to a red hot condition in flame.

g. Count in ZnS (Ag) based alpha counting system.

8 ACTIVITY MEASURMENT

8.1 The alpha counting system is switched on for at least an hour before measurement, for stabilization.

8.2 Take a 10 000 s background with a blank stainless steel planchet.

8.3 Determine the efficiency of the counter using the standard reference source.

8.4 The sample, on stainless steel planchet, is counted on the alpha counting system for 5 000 s. This includes background of the alpha counter.

9 CALCULATION

9.1 Calculate the gross alpha activity (A) of the sample (Bq/unit volume) as follows:

$$\text{A (Bq/l)} = \frac{\left(\frac{S}{t_S} - \frac{B}{t_B}\right) \pm \text{SD x } 100}{\text{E(\%) x V}}$$

where

S = Total alpha counts due to sample plus background,

t_S and t_B = Counting times of sample and background, in seconds,

B = Background counts,

SD is the stadard deviation

$$= \sqrt{\left(\frac{S}{t_S}\right)^2 + \left(\frac{B}{t_B}\right)^2}$$

E = Efficiency (%) of the alpha counting system, and

V = Volume of water sample, in l.

10 PRECAUTION

Precaution should be taken that the precipitate load on stainless steel planchet should not be more than 10 mg/cm^2 to avoid alpha absorption.

NOTES

1 Analytical Reagent (AR) grade chemicals and reagents shall be used.

2 Ultrapure water shall be used for preparation of stock standards and reagents.

3 Glassware to be used for sample processing and storage are to be soaked in 10% nitric acid followed by rinsing with ultrapure water before use, to prevent sorption of elements on the walls of the container.

4 Analyse duplicate samples, reagent/process blanks and analytical blanks, known standards during the process for quality control.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Nuclear Materials for Peaceful Applications Sectional Committee, CHD 30

Organization	*Representative(s)*
Bhabha Atomic Research Centre (Health Safety & Environment Group), Mumbai	DR D. K. ASWAL (***Chairperson***) DR B. K. SAPRA (*Alternate*)
Atomic Energy Regulatory Board, Mumbai	SHRI S. K. PAWAR
Atomic Mineral Directorate, Hyderabad	DR T. S. SUNIL KUMAR
Bhabha Atomic Research Centre (Radiochemistry & Isotope Group), Mumbai	DR H. J. PANT
Bhabha Atomic Research Centre (Nuclear Fuels Group) Mumbai	DR S. K. JHA
Bharat Heavy Electricals Limited, Trichy	SHRI A. SUNDARARAJAN SHRI M. ARUN KUMAR (*Alternate*)
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Indian Institute of Technology, Kanpur	NOMINATION AWAITED
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Uranium Corporation of India Ltd, Jharkhand	NOMINATION AWAITED
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BIS Directorate General	SHRI A. K. LAL, SCIENTIST 'E' AND HEAD (CHD) [REPRESENTING DIRECTOR GENERAL (Ex-officio)]

Member Secretary
SHRI PUSHPENDRA KUMAR
Scientist 'B' (CHD), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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