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Radiation protection — Sealed radioactive sources — Leakage test methods

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Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9978 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Sub-Committee SC 2, *Radiation protection*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

Introduction

The use of sealed radioactive sources has become so widespread that standards to guide the user, manufacturer and regulatory agency are necessary. When establishing these standards, radiation protection is the prime consideration.

Leakage test methods for sealed radioactive sources were published in ISO/TR 4826[1] and the experience acquired since this date has permitted the elaboration of this International Standard.

1) ISO/TR 4826:1979, *Sealed radioactive sources — Leak test methods*

Radiation protection — Sealed radioactive sources — Leakage test methods

1 Scope

This International Standard specifies the different leakage test methods for sealed radioactive sources. It gives a comprehensive set of procedures using radioactive and non-radioactive means.

This International Standard applies to the following controls:

- quality control allowing validation of required tests for determining the classification of a prototype sealed radioactive source according to ISO 2919,
- production control of sealed radioactive sources;
- periodic inspections of the sealed radioactive sources performed at regular intervals, during the working life.

Annex A of this International Standard gives recommendations to guide the user in his choice of the most suitable method(s) according to control and source type.

It is recognized that there may be special circumstances where special tests, not described in this International Standard, may be required.

It is emphasized, however, that insofar as production, use, storage and transport of sealed radioactive sources are concerned, compliance with this International Standard is no substitute for complying with the requirements of the relevant IAEA regulations and other relevant national regulations.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2919:1980, *Sealed radioactive sources — Classification.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 sealed radioactive source: Radioactive material permanently sealed in one or several capsules and/or associated with a material to which it is closely bonded. This (these) capsule(s) and/or material shall be strong enough to maintain leak tightness of the sealed source under the conditions of use and wear for which it was designed.

NOTE 1 In the text of this International Standard, the term "sealed source" is used instead of "sealed radioactive source" for brevity.

3.2 leaktight: Term applied to sealed sources which, after undergoing leakage testing, meet the limiting values given in table 1.

3.3 capsule: Protective envelope, usually made of metal, used to prevent leakage of radioactive material.

3.4 dummy sealed source: Facsimile of a sealed source, the capsule of which has the same construction and is made with exactly the same materials as those of the sealed source that it represents but containing, in place of the radioactive material, a substance resembling it as closely as possible in physical and chemical properties.

3.5 simulated sealed source: Facsimile of a sealed source, the capsule of which has the same construction and is made with exactly the same materials as those of the sealed source that it represents. However it contains, in place of the radioactive material, a substance resembling it as closely as possible in physical and chemical properties and only radioactive material of tracer quantity.

NOTE 2 The tracer shall be in a form soluble in a solvent which does not attack the capsule and has the maximum activity compatible with its use in a containment enclosure.

3.6 model designation: Descriptive term or reference number to identify a specific sealed source design.

3.7 prototype sealed source: Original of a sealed source which serves as a pattern for the manufacture of all sealed sources identified by the same model designation.

3.8 quality control: Controls on a prototype sealed source which are necessary to establish the compliance of the sealed sources with ISO 2919, including the determination of the classification.

3.9 production control: Performance testing of a new sealed source before sealed sources of the same model designation are put into actual manufacture and use.

3.10 recurrent inspections: Particular controls performed at regular intervals in order to establish (both during storage and use) the leak tightness of the sealed source.

3.11 leakage: Transfer of radioactive material from the sealed source to the environment.

3.12 non-leachable: Term used to convey that the radioactive material in the form contained in the sealed source is virtually insoluble in water and is not convertible into dispersible products (see ISO 2919).

3.13 standard helium leakage rate: Helium leakage rate at an upstream pressure of 10^5 Pa $\pm$ 5×10^3 Pa and a downstream pressure of 10^3 Pa or less at a temperature of 296 K $\pm$ 7 K (23 °C $\pm$ 7 °C); in this International Standard the unit micro pascal cubic meter per second is used, taking into account the range of limiting values 1 $\mu Pa \cdot m^3 \cdot s^{-1}$ = 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ $\approx$ 10^{-5} $atm \cdot cm^3 \cdot s^{-1}$ $\approx$ $7,5 \times 10^{-3}$ lusec.

4 Requirements

The tests described in this International Standard shall be carried out by competent and qualified persons who have had appropriate training in radiation protection.

According to the control type and the sealed source type, at least one of the tests described in clauses 5 and 6 should be carried out [see annex A for the choice of the test(s)].

However, in the case where a special test, which is not described in this International Standard, is carried out (see clause 1), the user should be able to demonstrate that the applied method is at least as effective as the corresponding method(s) given in this International Standard.

It should be noted that it is often normal practice to carry out more than one type of leakage test and also to perform a final wipe test as a contamination check.

At the conclusion of the performed test(s), the sealed source shall be considered to be leaktight if it complies with the limiting values specified in table 1.

If there is no direct correspondance between the levels of measurement of the different methods, the results will depend upon measurement equipment and procedures.

A leakage rate of 10 $\mu Pa \cdot m^3 \cdot s^{-1}$ for non-leachable solid contents and a rate of 0,1 $\mu Pa \cdot m^3 \cdot s^{-1}$ for leachable solids, liquids and gases would, in most cases, be considered to be equivalent to the activity release limit of 2 kBq ($\approx$ 50 nCi) according to [12].

A further confirmation of the volumetric acceptance threshold is given by [2]. A leakage rate of 10^{-7} $atm \cdot cm^3 \cdot s^{-1}$ or less based on dry air at 298 K (25 °C) and for a pressure difference of 1 atm against a vacuum of 10^{-2} atm or less is considered to represent a loss of leaktightness, irrespective of the physical nature of the content.

Table 1 — Threshold detection values and limiting values for different test methods

Test method	Sub-clause	Threshold detection value	Limiting value: Non-leachable content	Limiting value: Leachable or gaseous content
		Activity, Bq	kBq	
Immersion test (hot liquid)	5.1.1	10 to 1	0,2	0,2
Immersion test (boiling liquid)	5.1.2	10 to 1	0,2	0,2
Immersion test with a liquid scintillator	5.1.3	10 to 1	0,2	0,2
Gaseous emanation test	5.2.1	4 to 0,4	—1)	0,2 (^{222}Rn/12 h)
Emanation test with a liquid scintillator	5.2.2	0,4 to 0,004	—1)	0,2 (^{222}Rn/12 h)
Wet wipe test	5.3.1	10 to 1	0,2	0,2
Dry wipe test	5.3.2	10 to 1	0,2	0,2
		Standard helium leakage rate, $\mu Pa{\cdot}m^3{\cdot}s^{-1}$		
Helium test	6.1.1	10^{-2} to 10^{-4}	1	10^{-2}
Helium pressurization test	6.1.2	1 to 10^{-2}	1	10^{-2}
Vacuum bubble test	6.2.1	1 2)	1	—3)
Hot-liquid bubble test	6.2.2	1 2)	1	—3)
Gas pressurization bubble test	6.2.3	1 2)	1	—3)
Liquid nitrogen bubble test	6.2.4	10^{-2} 2)	1	10^{-2}
		Mass gain of water, μg		
Water pressurization test	6.3	10	50	—3)

1) Unsuitable.
2) These detection limits are applicable only to single leaks under favourable visual conditions.
3) Not sensitive enough.

Prior to any testing, except in the case of recurrent inspections, the sealed source shall be thoroughly cleaned and shall undergo a thorough visual examination.

All equipment used for tests shall be suitably maintained and calibrated periodically.

Where applicable, the following parameters should be specified, whenever possible:

— pressure,

— temperature,

— proportionality factor between the volume of the sealed source and the volume of the test enclosure used for certain tests, as well as the volume of liquid used to cover the sealed source to be tested.

The wipe test should not be considered as a leakage test, except for some specific types of sources (e.g. sources with thin windows), for recurrent inspections and in cases where no other test is more suitable.

Wipe tests or liquid immersion test samples should, wherever possible, be checked immediately on basic contamination measuring equipment; for example, a Geiger counter to establish whether there is any gross contamination prior to final measurement on more sophisticated calibrated equipment.

5 Test methods by radioactive means

5.1 Immersion tests

5.1.1 Immersion test (hot liquid)

Immerse the sealed source in a liquid which does not attack the material of the outer surfaces of this source and which, under the conditions of this test, is considered effective for removal of all traces of radioactive materials present. Examples of such liquids include distilled water and weak detergent solutions or chelation agents and also slightly alkaline or acid solutions with concentrations of about 5 %. Heat the liquid to 323 K ± 5 K (50 °C ± 5 °C) and maintain it at that temperature for at least 4 h. Remove the sealed source and measure the activity of the liquid.

NOTE 3 An ultrasonic cleaning method can also be used. In this case, the immersion time in the liquid at 343 K ± 5 K (70 °C ± 5 °C) can be reduced to approximately 30 min.

5.1.2 Immersion test (boiling liquid)

Immerse the sealed source in a liquid which does not attack the material of the outer surfaces of the source and which, under the conditions of this test, is considered effective for removal of all traces of radioactive materials present. Boil for 10 min, allow to cool, then rinse the sealed source in a fresh batch of liquid. Repeat these operations twice, re-immersing the source in the original liquid. Remove the sealed source and measure the activity of the liquid.

5.1.3 Immersion test with a liquid scintillator

Immerse the sealed source for at least 3 h at room temperature in a liquid scintillator solution which does not attack the material of the outer surface of the source. Store away from light to avoid photoluminiscence. Remove the sealed source and measure the activity of the liquid by a liquid scintillation counting technique.

5.1.4 Immersion test at room temperature[2)]

Immerse the sealed source in a liquid which does not attack the material of the outer surfaces of this source and which, under the conditions of this test, is considered effective for removal of all traces of radioactive materials present.

Immerse the sealed source in a liquid at room temperature 293 K ± 5 K (20 °C ± 5 °C) and maintain it at this temperature for 24 h. Remove the sealed source and measure the activity of the liquid.

5.1.5 Approval criteria

The sealed source is considered to be leaktight if the activity detected does not exceed 0,2 kBq (≈ 5 nCi).

5.2 Gaseous emanation tests

5.2.1 Gaseous emanation test by absorption (for radium-226 sealed sources)

Place the sealed source in a small gas-tight container with a suitable absorbent, for example activated carbon, cotton or polyethylene, and leave it for at least 3 h. Remove the source and close the container. Immediately measure the activity of the absorbent.

5.2.2 Gaseous emanation test by immersion with a liquid scintillator (for radium-226 sealed sources)

Follow the procedure described in 5.1.3.

5.2.3 Gaseous emanation test (for krypton-85 sealed sources)

Maintain the sealed source under reduced pressure for 24 h. Analyse the content of the chamber for krypton-85 by a plastic scintillation counting technique. Repeat the test after at least 7 days.

5.2.4 Other gaseous emanation tests

Any other test methods which are equivalent to those described in 5.2.1 to 5.2.3 may be used.

5.2.5 Approval criteria

When the tests described in 5.2.1 and 5.2.2 are completed, the sealed source is considered to be leaktight if the activity detected does not exceed 0,2 kBq (≈ 5nCi) of radon for a collection time of 12 h. When the test period is shorter than 12 h, appropriate corrections shall be made.

When the tests described in 5.2.3 and 5.2.4 are completed, the sealed source is considered to be leaktight if the activity detected does not exceed 4 kBq/24 h (≈ 100 nCi/24 h).

2) This test may be useful where hot liquid tests are not practical, however the latter are recommended whenever possible since their use has been widely recognized for many years and also because they may be more effective.

5.3 Wipe tests

If a wipe test is used to determine leak tightness after mechanical or thermal prototype testing, the sealed sources to be tested shall be cleaned (decontaminated) prior to the tests.

When the wipe test is a means of leakage testing carried out at the manufacturing stage, the sealed source shall be cleaned prior to the test and a 7-day waiting period shall be observed before the test.

In the case of the wipe test methods, it is necessary to take into account the technique used, the instrumentation and the pressure applied, because the method used may not give sufficiently accurate reproducibility guarantees.

5.3.1 Wet wipe test

Wipe all the external surfaces of the sealed source thoroughly with a swab of filter paper or another suitable highly absorbant material, moistened with a liquid which will not attack the material of which the external surfaces of the sealed source are made and which, under the conditions of this test, has been demonstrated to be effective in removing any radioactive material present. Measure the activity of the swab.

5.3.2 Dry wipe test

This test can be used in situations where it may not be appropriate to use a wet swab, for example for high activity cobalt-60 sources or in some recurrent inspections.

To carry out the test, thoroughly rub all the external surfaces of the sealed source with a dry swab of filter paper and measure the activity of the swab.

5.3.3 Approval criteria

If the activity detected does not exceed 0,2 kBq (≈ 5nCi), the sealed source is considered to be leaktight.

NOTE 4 The important points concerning the use of wipe tests on accessible surfaces which are as close as possible to the sealed source and the need to consider radiation protection are noted [see clause 3, item b)].

6 Test methods by non-radioactive means

When non-radioactive procedures are used, a relationship between volumetric leakage rate and loss of radioactive material should be established. In practice, it is difficult to do this because of the wide range of forms of radioactive material used in sealed sources and also the different types of leaks.

The data given in this International Standard for the relationship between volumetric leakage rates and loss of radioactive material are based on values published in IAEA documents and, although they have not been confirmed absolutely by experimental work, volumetric leakage test methods have been used for many years and experience shows that they can be accepted as valid test methods.

Before conducting any of the tests described in 6.1 to 6.3, the sealed source should be thoroughly cleaned and dried.

For sealed sources with leachable or gaseous content, the helium test described in 6.1 can be used.

Ensure that there are no gross defects which might invalidate the results of the described test, for example by visual inspection or by a method less sensitive than that of the described test. For these tests to be valid, except that described in 6.3, the free volume inside the sealed source shall be greater than 0,1 cm^3. If this test is used for sealed sources with a free volume of less than 0,1 cm^3, the user shall be able to demonstrate the validity of the test[9].

Because of their lower detection limit, only those tests using helium (6.1) are applicable to sealed sources with leachable or gaseous contents.

6.1 Helium mass spectrometer leakage tests

6.1.1 Helium test

Place the sealed source containing helium in a suitable vacuum chamber, which is subsequently evacuated through a helium mass spectrometer. Evaluate the actual helium leakage rate in accordance with the recommendations of the manufacturer of the leakage test equipment.

Ensure that the free volume inside the sealed source contains a concentration of commercial grade helium of more than 5 %. The indicated helium leakage rate according to the previous evaluation divided by the concentration of helium in the free volume gives the actual standard helium leakage rate.

6.1.2 Helium pressurization test

Place the sealed source in a pressure chamber. Using helium, purge the chamber of air. Pressurize the chamber to a given helium pressure and maintain it for a given period. Depressurize the chamber, clean the sealed source by flushing it with dry nitrogen or rinsing it in a volatile fluorocarbon liquid, and transfer it to a suitable vacuum chamber. Measure the helium leakage rate as described in 6.1.1.

With the indicated helium leakage rate, Q, the actual standard helium leakage rate, L, can be evaluated by using the equation

$$Q = \frac{L^2 pt}{p_o^2 V} \qquad \ldots (1)$$

where $p_o = 1{,}01325 \cdot 10^5$ Pa.

NOTES

5 With helium pressure, p, in megapascals (in pratice between 0,5 MPa and 10 MPa) maintained for a conditioning time t, in hours, a delay time between pressurization and measurement of less than 10 min and taking into account the free volume, V, in cubic centimetres greater than 0,1 cm^3 inside the sealed source, convenient test parameters may be chosen and the test results evaluated using the following relationship:

$$Q = 0{,}35 \frac{L^2 pt}{V} \qquad \ldots (2)$$

where

Q is the indicated leakage rate ($\mu Pa \cdot m^3 \cdot s^{-1}$)

L is the actual standard helium leakage rate ($\mu Pa \cdot m^3 \cdot s^{-1}$) in the range between the limiting values of 1 $\mu Pa \cdot m^3 \cdot s^{-1}$ and 10^{-2} $\mu Pa \cdot m^3 \cdot s^{-1}$ $\left(L \leqslant 1{,}7\sqrt{QV/pt}\right)$.

6 Equation (2) is valid in the case of molecular flow through one or more leaks. In the case of a high percentage of viscous laminar flow, this equation leads to a moderate over-estimation of the actual standard helium leakage rate, but this factor only slightly influences the test result.

6.1.3 Approval criteria

When these tests are completed, the sealed source is considered to be leaktight if the actual standard helium leakage rate is less than 1 $\mu Pa \cdot m^3 \cdot s^{-1}$ for non-leachable contents and 10^{-2} $\mu Pa \cdot m^3 \cdot s^{-1}$ for leachable or gaseous contents (see table 1).

6.2 Bubble leakage tests

Bubble leakage tests rely on an increase in the internal pressure. Then gas from internal voids can penetrate any leaks and form visible bubbles in a liquid bath. For one particular leak, the rate of bubbling increases with a decrease in surface tension.

6.2.1 Vacuum bubble test

By using ethylene glycol, isopropyl alcohol, mineral or silicone oil, or water with a wetting agent as a leakage test fluid in a suitable vacuum chamber, lower the air content of the fluid by evacuating the chamber for at least 1 min. Re-establish atmospheric air pressure and submerge the sealed source completely to a depth of at least 5 cm below the fluid level. Reduce the absolute pressure in the chamber to between 15 kPa and 25 kPa. Observe any bubbles emanating from the sealed source for a period of at least 1 min.

6.2.2 Hot-liquid bubble test

Ensure that the sealed source is at ambient temperature. Immerse it at a depth at least 5 cm below water level in a water bath which is at a temperature between 363 K and 368 K (90 °C and 95 °C). Glycerine at 393 K to 423 K (120 °C to 150 °C) is an acceptable alternative to water. Observe any bubbles emanating from the sealed source for a period of at least 1 min; however a minimum period of 2 min is recommended whenever possible and particularly when capsules with large thermal mass and poor thermal conductivity are necessary.

6.2.3 Gas pressurization bubble test

Place the sealed source in a suitable pressure chamber of volume at least twice that of the source and at least five times the free volume inside the source. Pressurize the chamber with helium gas to at least 1 MPa and maintain it at that pressure for 15 min. Release the pressure, remove the sealed source from the chamber and immerse it 5 cm below the level of ethylene glycol, isopropyl alcohol, acetone or water containing a wetting agent in a bath. Observe for bubbles emanating from the sealed source over a period of at least 1 min.

6.2.4 Liquid nitrogen bubble test

Immerse the sealed source completely in liquid nitrogen for a period of 5 min, then transfer it to the test liquid (normally methanol). Observe for bubbles emanating from the sealed source over a period of at least 1 min.

6.2.5 Approval criteria

If no bubbles are observed at the end of the tests described in 6.2.1 to 6.2.4, the sealed source is considered to have a leakage rate of less than 1 $\mu Pa \cdot m^3 \cdot s^{-1}$ and to be leaktight only if the contents are non-leachable.

6.3 Water pressurization test

Determine the mass of the sealed source on a balance. Perform the experimental pressure test with water, wipe the sealed source dry and redetermine its mass on the same balance.

If the gain in mass is less than 50 µg, the sealed source is considered to be leaktight but only for non-leachable contents.

For this test to be valid, the calculated free volume within the sealed source has to be capable of holding at least five times as much water as the sensitivity of the mass measuring equipment. This test is applicable particularly for evaluating the external pressure test for classes 3, 4, 5 and 6 of ISO 2919.

Annex A
(normative)

Guidance for the choice of the tests to be carried out according to control and sealed source type

This annex provides a guide to assist in the selection of the most suitable tests for carrying out quality control, production control and recurrent inspections, taking into account the sealed source type (design, characteristics, etc).

Table A.1 is not fully comprehensive, however it covers a wide range and can act as a guide for many sealed source designs. It gives the preferred test and second choice test.

A.1 Leakage test for the production of sealed sources

The most appropriate leakage test for the production of sealed sources containing a radionuclide can be determined from table A.1, according to their particular source design and technology.

A.2 Leakage tests for prototype sealed sources

Leakage tests allowing validation of required tests for determining the classification of a prototype sealed source according to ISO 2919 may be carried out on

- prototype sealed sources with the nominal radioactive content,
- simulated sealed sources,
- dummy sealed sources.

For the last case, it will clearly be necessary to use a non-radioactive leakage test method.

The most appropriate leakage test will depend on the sealed source technology and design and can be determined from table A.1.

A.3 Recurrent inspections

It is obviously necessary to test sealed sources at regular intervals after they have been supplied by a manufacturer, to check that they have not developed a leak. In many countries there are statutory regulations which specify the frequency of tests. The time interval between tests may vary according to the sealed source type and design and also the working environment.

These tests are not necessarily the same as those which are appropriate during manufacture. It is important to take into account the utilization conditions of the sealed source and of any specific risks that it might encounter during its working life.

Thus, several conditions may be encountered in practice when considering recurrent tests.

a) The sealed source can only be tested on the site where it is used and it is practical to carry out a wipe test on the nearest accessible part. In this case, a wipe test (5.3) is chosen. A visual examination of the source should also be carried out if possible.

b) The source can only be tested on the site where it is used but direct access to the source is not possible or not desirable because of the unjustified exposure of persons carrying out the test, for example high activity teletherapy sources or other sources in sealed housings. In this case, wipe tests should be carried out on the nearest accessible part.

WARNING — If activity is found to be present, even if below the limiting value of 0,2 kBq[3] (≈ 5 nCi), action should be taken to establish whether this arises from source leakage. One procedure would be to repeat the tests at regular intervals to determine whether the activity detected is increasing.

3) The limiting value of 0,2 kBq is qualified by the statement that further tests shall be carried out if any activity is present.