IEC 62554

Edition 1.2 2025-02
CONSOLIDATED VERSION

INTERNATIONAL STANDARD

Sample preparation for measurement of mercury level in fluorescent lamps and low-pressure mercury UV radiation sources

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IEC 62554:2011-08+AMD1:2017-10+AMD2:2025-02 CSV(en)

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 29.140.30

ISBN 978-2-8327-0224-6

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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SAMPLE PREPARATION FOR MEASUREMENT OF MERCURY LEVEL IN FLUORESCENT LAMPS AND LOW-PRESSURE MERCURY UV RADIATION SOURCES

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This consolidated version of the official IEC Standard and its amendment has been prepared for user convenience.

IEC 62554 edition 1.2 contains the first edition (2011-08) [documents 34A/1484/FDIS and 34A/1502/RVD], its amendment 1 (2017-10) [documents 34A/1997/CDV and 34A/2028/RVC] and its amendment 2 (2025-02) [documents 34A/2398/CDV and 34A/2427/RVC].

In this Redline version, a vertical line in the margin shows where the technical content is modified by amendments 1 and 2. Additions are in green text, deletions are in strikethrough red text. A separate Final version with all changes accepted is available in this publication.

International Standard IEC 62554 has been prepared by subcommittee 34A: Lamps, of IEC technical committee 34: Lamps and related equipment.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this document and its amendments will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

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INTRODUCTION

The International Electrotechnical Commission (IEC) draws attention to the fact that it is claimed that compliance with this document may involve the use of a patent concerning Cold spotting given in 5.4.1.

IEC takes no position concerning the evidence, validity and scope of this patent right.

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According to IEC SMB 136/7 decision, the technical committee decided to remove designation of a reference method.

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INTRODUCTION to Amendment 1

IEC 62554:2011

IEC 62554 specifies the method of sample preparation for the measurement of mercury level in fluorescent lamps. It refers to IEC 62321:2008 for the technique for determining the amount of mercury.

In the meantime it has been found that for fluorescent lamps, some of the techniques specified in IEC 62321 can lead to inaccurate and misleading results and in addition this standard has been split into several parts.

In bilateral discussions between members of subcommittee 34A and technical committee 111, it was agreed to update the relevant part of IEC 62321 and the reference made to it in IEC 62554. Amendment 1 to IEC 62321-4 has now been published (IEC 62321-4:2013/AMD1:2017).

SAMPLE PREPARATION FOR MEASUREMENT OF MERCURY LEVEL IN FLUORESCENT LAMPS AND LOW-PRESSURE MERCURY UV RADIATION SOURCES

1 Scope

This International Standard specifies sample preparation methods for determining mercury levels in new tubular fluorescent lamps (including single capped, double capped, self-ballasted and cold cathode fluorescent lamp (CCFL) for backlighting) and new low-pressure mercury UV radiation sources, containing 0,1 mg mercury or more. The intended resolution of the methods described in this ~~standard~~ document is of the order of 5 %.

Mercury level measurement of spent lamps is excluded, as during lamp operation, mercury gradually diffuses into the glass wall and reacts with the glass materials. The test method of this standard does not recover mercury that is diffused into or reacted with or otherwise incorporated irreversibly with the glass wall of discharge tubes.

This standard does not contain information on measurement. Measurement is specified in IEC 62321.

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2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

~~IEC 62321:2008, *Electrotechnical products – Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)*~~

IEC 62321-4:2013, *Determination of certain substances in electrotechnical products – Part 4: Mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS*
IEC 62321-4:2013/AMD1:2017

ISO 3696:1987, *Water for analytical laboratory use – Specification and test methods*

3 Terms and definitions

For the purposes of the present document, the following terms and definitions apply.

3.1
new lamp
a lamp that has not been energized since manufacture

3.2
cold cathode fluorescent lamp (CCFL) for backlighting
small diameter fluorescent lamp having cold cathode in the lamp, in which most of light is emitted by the excitation of phosphors coated in discharge tube and used as backlight in LCD

3.3
external electrode fluorescent lamp (EEFL) for backlighting
small diameter fluorescent lamp having cold cathode attached outside the lamp, in which most of light is emitted by the excitation of phosphors coated in discharge tube and used as backlighting in LCD

EEFL is a subtype in CCFL lamp group.

4 General

Mercury in fluorescent lamps exists in the following states:

a) vapour in a lamp;

b) liquid metal;

c) compound;

d) alloy.

There is a wide variety of mercury dosing solutions including appearance and placement of mercury dispensing devices and also composition and structure of those devices. Although some of the lamps are dosed with amalgam or solid mercury alloy, there are also many fluorescent lamps dosed with liquid mercury.

Amalgam dosed lamps often have device(s) that act as an auxiliary amalgam. Form and location of these devices vary widely as well.

The introduction of a cold spot (see Annex B) minimizes the loss of mercury in the vapour state when the discharge tube is opened. With the lamp operating, the cold spot will condense all the mercury in the discharge, allowing superior control for mercury recovery.

The procedure in Clause 5 below includes a method to collect liquid mercury, mercury compounds and alloys and amalgams.

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The total amount of mercury is determined by measuring the amount of liquid mercury, mercury compounds and alloys and amalgam.

The amount of mercury is calculated from the measured mercury concentration, the volume of the filtered solution and the dilution factor.

Where "fluorescent lamp(s)" and "lamp(s)" are mentioned in Clause 5 and in Annex B of this document, also "low-pressure mercury UV radiation source(s)" shall be read.

5 Procedure for collecting mercury from a fluorescent lamp

5.1 General

For test arrangement and ambient conditions, relevant parts of ISO/IEC 17025:2005 shall be followed.

WARNING – Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices, to avoid pollution of the environment and to ensure compliance with any national regulatory conditions.

5.2 Reagents

The following reagents shall be used:

– water: Grade 1, as specified in ISO 3696;
– the mass fraction of mercury in the following reagents shall be below 1×10^{-9};
– potassium permanganate 5 % aqueous solution (m/v);
– nitric acid, concentrated 65 %;
– hydrochloric acid, concentrated 37 %;
– hydrofluoric acid, concentrated 40 %.

5.3 Chemical lab ware

Chemical lab ware shall be verified to be mercury non-absorbing.

Chemical lab ware shall be as follows:

– disposable vacuum filter pack with medium retention filter;
– disposable wide mouth screw-capped plastic bottles: 125 ml, 250 ml, 500 ml, 1 000 ml, 2 000 ml;
– disposable wide mouth sturdy plastic bag nominal 500 ml, 1 000 ml;
– beaker 50 ml, 100 ml, 125 ml, 250 ml, 500 ml;
– volumetric flasks: 50 ml, 100 ml, 250 ml, 500 ml;
– micropipettes;
– dispensers;
– bench coat: sheet of plastic lined laboratory bench paper.

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NOTE The plastic bag may be clear polyethylene or similar chemical and acid resistant material nominally 0,01 mm or thicker. The 1 000 ml bag would be approximately 200 mm × 300 mm. Sometimes known as a “blender or stomacher bag” they are available from biological laboratory suppliers. Bag size may be adjusted to suit availability and lamp size being tested.

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5.4 Sample preparation

Sample preparation process shall be a continuous operation without excessive hold time.

5.4.1 Cold spotting methods

5.4.1.1 General

Cold spotting is a method for condensing free mercury in a localized position (see Annex B).

The mercury localization occurs while the low-pressure discharge lamp is “ON” under normal operating conditions while a small area (the cold spot) of the discharge tube is maintained at a low temperature. During the cold spotting process, no heavy end blackening should be observed.

When the free mercury is fully condensed, the total light output of the lamp will drop significantly and the discharge colour will ~~typically turn pink~~ change throughout the lamp. The process of free mercury localization (cold ~~-~~spotting) is then completed.

NOTE Mercury collection with cold spot below 0 °C and operating with the normal control gear of the lamp may take several days.

5.4.1.2 Sample preparation of self-ballasted and single capped compact fluorescent multi limbed lamps with cold-spotting

Discharge tube cutting operations shall be carried out above the wide mouth screw capped plastic bottle to minimize the risk of material loss.

Sample containers shall be as follows.

– Use 250 ml wide mouth screw-capped plastic bottle for cold spot section as first container.
– Use 125 ml wide mouth screw-capped plastic bottle for end portions of discharge tube as second container.
– Use 500 ml or 1 000 ml wide mouth screw-capped plastic bottle for glass parts of discharge tube, depending on which one fits better to the discharge tube dimensions under test as third container.

The sample preparation shall be executed according to the process steps listed below.

a) Separate discharge tube from its outer bulb, if any.
b) Clean the discharge tube with chemical wipe.
c) Mark discharge tube in a non-destructive manner for first sectioning. Mark 3 cm on both sides of the cold spot.
d) Collect the free mercury with cold spotting – see 5.4.1.1 – until mercury starvation is verified.
e) Remove lamp from cooler. Keep lamp in same position as it was during cold spotting until sectioning.
f) Place the lamp on cutting table covered by bench coat – with the plastic side up, toward the lamp.
g) Score and break the discharge tube at the first mark allowing the arc tube to fill with air slowly so that no fluorescent powder coating of the tube is blown off.
h) Break the lamp fully at the first mark. Score and break the lamp at the other mark around the cold spot. Place cold spot section (6 cm) immediately into the first container. Close the container. Shake first container allowing the discharge tube section to crush. Keep the first container in crushed ice until digestion. Allow 5 min for the floating dust to settle before continuing. Proceed to 5.5.2 sample digestion with the first container immediately.
i) Next, separate discharge tube from plastic surrounds and associated electronics, if any. Cut associated lead wires as close to the glass seal as possible. Only the discharge tube will be used for mercury level measurement.
j) Score and break all tip offs and check for metal parts. Crush tip offs with pliers into the second container.
k) Score both of the lead wire containing ends of the discharge tube approximately 7 mm from the end of the tube. Pre-score discharge tube for sectioning, step n) below. Use the minimum possible number of sections allowing the parts to fit into the third container.
l) Cut lead wire containing ends of the discharge tube at the score using hot rod or hot wire.
m) Check end portions for any hollow glass objects and crush them gently with pliers into the second container. Carefully avoid touching the content of hollow glass objects with the pliers. Place the removed end portions – inclusive of metal parts in them – of the discharge tube into the second container and close the container.
n) Section the discharge tube using hot rod or wire at scores marked in step k) above.
o) Place discharge tube section(s) into the third container.
p) Check bench coat for material chips. Any material on bench coat shall be placed into the third container. Then, close the third container.
q) Shake the third container allowing the discharge tube sections(s) to crush. Allow 5 min for the floating dust to settle before continuing.

Samples are ready for digestion. Proceed to 5.5 sample digestion immediately.

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