BS EN 62554:2011

BSI Standards Publication

Sample preparation for measurement of mercury level in fluorescent lamps

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National foreword

This British Standard is the UK implementation of EN 62554:2011. It is identical to IEC 62554:2011.

The UK participation in its preparation was entrusted by Technical Committee CPL/34, Lamps and Related Equipment, to Subcommittee CPL/34/1, Electric lamps.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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EN 62554

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Sample preparation for measurement of mercury level in fluorescent lamps
(IEC 62554:2011)

Préparation des échantillons en vue de la mesure du niveau de mercure dans les lampes fluorescentes
(CEI 62554:2011)

Vorbereitung des Prüfmusters zur Messung des Quecksilbergehalts in Leuchtstofflampen
(IEC 62554:2011)

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Foreword

The text of document 34A/1484/FDIS, future edition 1 of IEC 62554, prepared by SC 34A, "Lamps", of IEC TC 34, "Lamps and related equipment" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62554:2011.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2012-06-23
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2014-09-23

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Endorsement notice

The text of the International Standard IEC 62554:2011 was approved by CENELEC as a European Standard without any modification.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

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.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
ISO/IEC 17025	2005	General requirements for the competence of testing and calibration laboratories	EN ISO/IEC 17025	2005
IEC 62321	2008	Electrotechnical products - Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)	EN 62321	2009
ISO 3696	1987	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	1995

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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.

The holder of this patent right has assured the IEC that he/she is willing to negotiate licences free of charge with applicants throughout the world. In this respect, the statement of the holder of this patent right is registered with IEC. Information may be obtained from:

General Electric Company

Appliance Park AP35-1002, Louisville, KY, 40225-0001, US

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ISO (www.iso.org/patents) and IEC (http://patents.iec.ch) maintain on-line data bases of patents relevant to their standards. Users are encouraged to consult the data bases for the most up to date information concerning patents.

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

SAMPLE PREPARATION FOR MEASUREMENT OF MERCURY LEVEL IN FLUORESCENT LAMPS

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 CCFL for backlighting) containing 0,1 mg mercury or more. The intended resolution of the methods described in this standard 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.

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)*

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.

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.

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.

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.

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 light output of the lamp will drop significantly and the discharge colour will typically turn pink. 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.

5.4.1.3 Sample preparation of linear fluorescent lamps with cold spotting

Sample containers shall be as follows.

– Use 250 ml or 500 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 250 ml, 500 ml , 1 000 ml or 2 000 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 fragment retention cover, if any.

b) Mark discharge tube in a non-destructive manner for first sectioning. Mark 12 cm from the labelled end for the initial cut; mark 6 cm on both sides of the cold spot.

c) Collect the free mercury with cold spotting – see 5.4.1.1 – until mercury starvation is verified.

d) Remove lamp from cooler. Keep lamp horizontal until sectioning.

e) Place the lamp on cutting table covered by bench coat – with the plastic side up, toward the lamp.

f) 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.

g) Score and break the lamp at the remaining two marks. Place cold spot section (12 cm) immediately into the first container. Close the first container. Shake the 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 the 5.5.2 sample digestion immediately.

h) Next, separate discharge tube from its plastic and metallic surrounds. Cut associated lead wires as close to the glass seal as possible. Only the discharge tube will be used for mercury level measurement.

i) 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. Use the minimum possible number of sections allowing the parts to fit into the third container.

j) Section the ends of the discharge tube using hot rod or wire at scores marked. Score and break tip offs and check for metal parts. Crush tip offs with pliers into the second container. 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 end portions – inclusive of metal parts in them – of the discharge tube into the second container and close the second container.

k) Section the remaining discharge tube using hot rod or wire at scores marked in step i).

l) Place discharge tube sections into the third container.

m) Check bench coat for material chips. Any material on bench coat shall be placed into the third container. Then close the third container.

n) Shake the third container allowing the discharge tube to crush. Allow 5 min for the floating dust to settle before opening.

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

5.4.2 Sample preparation of fluorescent lamps by non-cold-spot (sectioning) methods

Sample containers shall be as follows.

– 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 first container.

– Use 125 ml wide mouth screw-capped plastic bottle for end portions of discharge tube as second 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) Separate discharge tube from its plastic and metallic surrounds. Cut associated lead wires as close to the glass seal as possible. Only the discharge tube will be used for mercury level measurement.

c) Clean the discharge tube with chemical wipe.

d) Place the lamp on cutting table covered by bench coat – with the plastic side up, toward the lamp.

e) 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. Use the minimum possible number of sections allowing the parts to fit into the first container.

f) Select a tip off that does not contain metal part. Score and break it allowing the discharge tube to fill with air slowly that no fluorescent powder coating of the tube is blown off. Break tip off with pliers into the second container.

g) Score and break all tip offs and check for metal parts. Break tip-offs with pliers into the second container.

h) Cut lead wire containing ends of the discharge tube at the score using hot rod or hot wire.

i) 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.

j) Section the discharge tube using hot rod or wire at scores marked in step e).

k) Place discharge tube sections into the first container.

l) Check bench coat for material chips. Any material on bench coat shall be placed into the first container. Then close the first container.

m) Shake the first container allowing the discharge tube 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.

5.4.3 Sample preparation of fluorescent lamps by non-cold-spot (crushing) methods

The sample preparation shall be executed according to the following procedure.

a) Separate discharge tube from any plastic surrounds or metallic ends. Cut lead wires as close to the glass seal as possible. Only the discharge tube will be used for mercury measurement.

b) Clean the discharge tube with a chemical wipe to remove any dust particles.

c) Using pliers, break the tip off at one end of the fluorescent lamp, allowing the discharge tube to fill with air. Place broken tip into plastic bag.

d) Squirt a small amount (approximately 3 ml) of deionised water into the fluorescent lamp to wet the phosphor powder inside the lamp. This will prevent the loss of any mercury contained in the dry phosphor coating when the bulb is broken into pieces.

e) If the fluorescent lamp is of a small compact type (single capped fluorescent multi limbed), place the whole lamp into a wide mouthed thick plastic bag. Fold over the open end to create a temporary seal and using a mallet carefully break the lamp into small pieces hitting the outside of the bag.

f) If the fluorescent lamp is of a linear type, place the first portion in to a wide mouth thick plastic bag. Carefully using a mallet break the lamp hitting the outside of the bag while feeding the non-broken end into the bag until all the linear lamp is in the bag. Fold over the open end to create a temporary seal and using a mallet carefully break the lamp into small pieces hitting the outside of the bag.

g) Empty the contents of the plastic bag into a suitable sized first container of 5.4.2.

h) Rinse the contents of the plastic bag using a small amount of deionised water. This can be achieved by cutting the sealed end of the plastic bag off and rinsing the inside straight into the container with the broken lamp.

Samples are ready for digestion. Proceed to sample digestion immediately according to 5.5.2. If any metals remain after digestion according to 5.5.2 these are to be dissolved using step 5.5.3.

5.4.4 Nitric acid rinse method for linear fluorescent lamps

Sample containers shall be as follows.

– Use 50 ml or 100 ml plastic sample beaker for end portions of discharge tube as first container.

– Use 250 ml plastic sample beaker as second container.

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

a) Separate discharge tube from its fragment retention cover, if any.

b) Separate discharge tube from its plastic and metallic surrounds (including end caps). Cut associated lead wires as close to the glass seal as possible. Only the discharge tube will be used for mercury level measurement.

c) Carefully break the tip-off, crush and collect it into the first container. Inject a volume of concentrated nitric acid 1/30th of the lamps interior volume using a pipette or an injection syringe having no attached needle. Alternatively use the following method for the injection of nitric acid. Place a plastic tubing over the tip-off. Place the other end of the tubing into a vessel containing the appropriate volume of nitric acid. Carefully break the tip-off inside the tubing with a pair of needle nose pliers. The less than atmosphere pressure inside the lamp will draw the acid into the lamp.

NOTE 1 An appropriate example of plastic tubing is a 30 cm piece of Tygon or PVC tubing with 4,8 mm inner and 7,9 mm outer diameter.

d) Holding the lamp in a near horizontal orientation, rotate the lamp such that the acid contacts all interior surfaces. Place the lamp in a vertical orientation for 15 min. Repeat this procedure a minimum of three times.

e) Remove the open tip-off end of the lamp (approximately 2 cm) using a diamond pen or hot wire and place the 2 cm section including the coil mount into the first container. Decant the concentrated nitric acid from the lamp into the second container.

f) Wash the interior of the lamp with water and decant into the second container. Wash the interior of the lamp a minimum of five times.

g) Remove the other end of the lamp (approximately 2 cm) using a diamond pen or hot wire. Crush tip off with pliers into the first container and place the 2 cm section including the coil mount also into the first container. Add an appropriate volume of concentrated nitric acid and allow to stand for at least 15 min.

h) Decant the concentrated nitric acid from the first container into the second container and wash the first container a minimum of three times with water and decant into the second container.

i) Transfer all glass components from the first container to the second container, leaving the metallic components.

NOTE 2 It is important that the majority of glass is removed from the first container as this may influence the results of the metal digestion using HF (see 5.5.3).

j) Process the first container in accordance with 5.5.3 digestion of metal samples.

k) Process the second container in accordance with 5.5.2 b) digestion of glass samples.

5.4.5 Direct mercury measurement

This method is applicable for small diameter fluorescent lamps (e.g. EEFL, CCFL).

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

a) Cut associated lead wires as close to the glass seal as possible. Remove the external electrodes of EEFL. Only the discharge tube will be used for mercury level measurement (see Note 1).

b) Clean the discharge tube with a chemical wipe.

c) Make scratches and section discharge tube near the both ends. Then section discharge tube remainder into 100 mm segments. Place discharge tube segments on a quartz boat (see Note 2).

Sample is ready for analysis with electrothermal vaporization atomic absorption spectrometry.

NOTE 1 Any solder adhered to the lead may cause contamination in the measurement section because it has a low boiling point and contains paste. Completely remove any oil from the surface as it may cause contamination in the measurement section.

NOTE 2 Carefully break the sample just above the boat, so that the mercury does not scatter.

5.4.6 Sample preparation of other fluorescent lamps

For any other differently shaped fluorescent lamps follow 5.4.1.2 if lamp is self-ballasted or 5.4.1.3 if not self-ballasted.

5.5 Sample digestion

5.5.1 Ambient conditions

The sample digestion shall be executed at room temperature.

5.5.2 Glass samples (in 250 ml, 500 ml, 1 000 ml or 2 000 ml container)

Samples prepared according to Subclause 5.4.1.2 h), 5.4.1.2 q) , 5.4.1.3 g), 5.4.1.3 n), 5.4.2. k), 5.4.3 h) and 5.4.4 k).

The following recipe applies to samples in 250 ml container. For samples in 500 ml, 1 000 ml or 2 000 ml container, use appropriate multiple (2x, 4x, 8x) amount of each listed ingredient. Make sure that acid mixture covers crushed material.

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

a) Add 25 ml concentrated nitric acid. Add 10 ml water and swirl to mix.

b) Add 0,25 ml of 5 % potassium permanganate and allow to stand for 16 h (overnight) in a well-ventilated fume cupboard.

NOTE To accelerate reactions, heating solution up to 80 degree centigrade on a hot plate is also allowed.

If any metal remains after digestion these are to be dissolved using step 5.5.3.

5.5.3 Metal samples (in 125 ml container)

Samples prepared according to Subclause 5.4.1.2 m), 5.4.1.3 j), 5.4.2.i) and 5.4.4.j).

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

a) Add 3 ml concentrated hydrochloric acid and 1 ml concentrated nitric acid.

b) If dissolution is incomplete except tungsten coils, add 2 ml HF.

When all metals (not necessarily glass materials) are dissolved, add 20 ml nitric acid. Add 10 ml water and swirl to mix.

c) Add 0,25 ml of 5 % potassium permanganate and let stand for 16 h (overnight) in a well-ventilated fume cupboard.

NOTE To accelerate reactions, heating solution up to 80 degree centigrade on a hot plate is also allowed.

5.6 Filtering

Filter all digested samples through a medium retention filter into the same 250 ml (500 ml, 1 000 ml or 2 000 ml) volumetric filter flask and dilute with deionised water to the mark on the flask. The filter packs are never reused.

6 Measurement

6.1 Blank test

Before sample is treated, a blank test should be performed in order to confirm that the blank value has no influence on the sample measurement value.

6.2 Data reporting

Measurements should be repeated three times on each of the extracted solutions. The reported values should be the average and 95 % confidence interval of the average.

Amount of mercury measured in accordance with this standard should be expressed with 2 significant digits.

6.3 Analysis

The analytical test procedure shall comply with the requirements of Clause 7 of IEC 62321.

For 5.4.5 sample preparation method, the electrothermal vaporization atomic absorption spectrometry method is applicable (see Annex A).

Annex A
(informative)

Electrothermal vaporization atomic absorption spectrometry (EVAAS) method

A.1 Electrothermal vaporization atomic absorption spectrometer

The mercury vapour generator vaporizes mercury from the sample by heating broken pieces of a lamp. This mercury vapour is then introduced into the atomic absorption spectrometer to measure the total mercury quantity. The atomic absorption spectrometer should be stable and linear across the measuring range. The controller monitors the ultraviolet absorbance of mercury introduced into the spectrometer, and controls the temperature of the generator so that the absorbance does not exceed the linear range of the spectrometer. The integrator sums the ultraviolet absorbance signal during the entire heating period. Figure A.1 shows a block diagram of the EVAAS test. Figure A.2 illustrates an example of an EVAAS test apparatus layout.

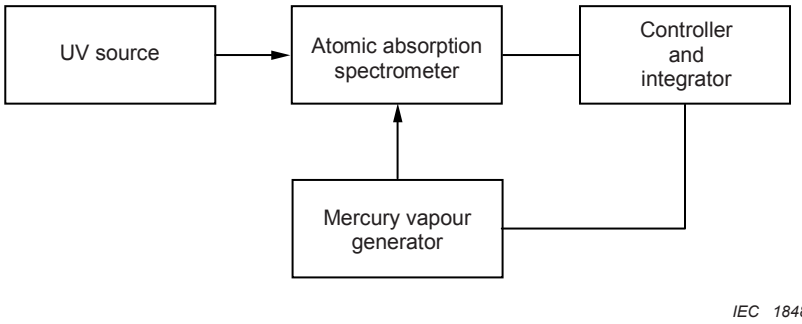
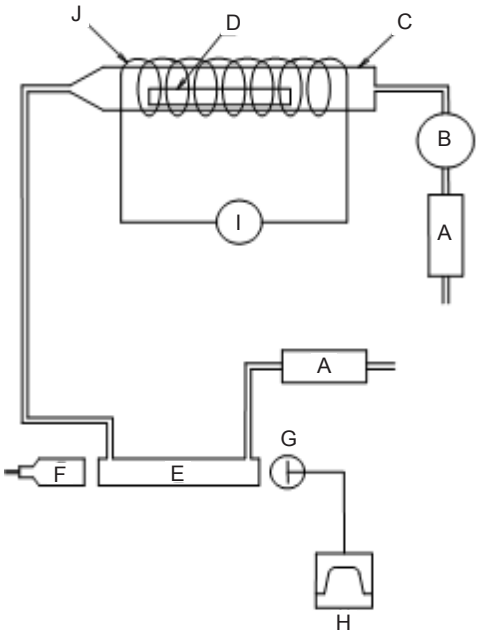


Figure A.1 – Configuration of the electrothermal vaporization atomic absorption spectrometry testing apparatus



Key

A	Mercury removing device	F	Mercury lamp
B	Air pump	G	Atomic absorption detector
C	Quartz heating tube	H	Integrator
D	Quartz boat	I	Power unit with controller
E	Quartz absorption cell	J	Heater

Figure A.2 – An example of the electrothermal vaporization atomic absorption spectrometer test apparatus layout

A.2 Reagents

The following reagents shall be used.

a) Water: Ion-exchange water or distilled water should be used throughout this procedure.

b) Mercury acetate standard solutions: Mercury acetate of more than 99 % purity is dissolved in water to make standard solutions.
The mercury acetate standard solution should be a certified reference material or should be traceable to it.

c) Granular or powdered activated alumina having particle size range from 40 μm to 2 000 μm should be used.

A.3 Measurement

A.3.1 Sample measurement

Insert the boat into (see 5.4.5) the quartz heating tube of the electrothermal vaporization atomic absorption spectrometer, and start the generator (heater), controller and integrator to initiate measurement. Integration of the ultraviolet absorbance signal from the atomic absorption spectrometer should begin as soon as heating starts. Control the heater while monitoring the concentration of mercury being generated (see Note in A.3.2). Keep the temperature at 240 °C or higher, and continue heating and integrating until no more mercury is generated.

A.3.2 Calibration curve

The calibration curve of the apparatus should be linear over the measuring range from 0,01 mg to 20 mg. Use a mercury acetate standard solution to make calibration curves. Place a layer of activated alumina on the boat and drop the appropriate quantity of standard solution onto the activated alumina with a micropipette. Start measuring as soon as the boat is inserted into the quartz heating tube in the electrothermal vaporization atomic absorption spectrometer, and keep the temperature at least 360 °C. Make a calibration curve from the relation between the amounts of mercury vaporized from the standard solution and the measured integration of the ultra violet absorbance signal. The amount of mercury in the lamp sample is estimated from the calibration curve.

NOTE Occasionally, samples may suddenly generate an amount of mercury that exceeds the concentration measurement range of the atomic absorption spectrometer. If this does occur, the measurement results may be biased toward underestimation of the total mercury quantity.

Annex B
(informative)

Information on the cold spotting method

B.1 General description of mercury collection by the cold spotting method on both single- and double capped lamps

B.1.1 General

A cold spot is a certain area on a fluorescent lamp which is cooled to approximately 0 °C.

Mercury will condense at the coldest place in the discharge chamber.

After the process has reached completion, virtually no free mercury is left in the lamp and consequently no UV radiation is emitted. The light output of the lamp becomes very dim and typically pink in colour. This state of the lamp is called ‘dark burning’. When this ‘dark burning’ state is observed, nearly all of the free mercury is condensed at the cold spot.

B.1.2 Double-capped fluorescent lamp

For double-capped lamps, the cold spot is created by a cooling system. This cooling system circulates a mixture of water and ethanol at approximately 0 °C through a glass cell. This cell has a round shape that fits tightly around the discharge tube.

B.1.3 Single-capped fluorescent lamp

For a single-capped lamp, the cold spot is created by using a copper rod, which is tightly applied to the lamp surface. The copper rod in turn is connected to the same or identical cell and cooling system as is used with double-capped lamps (see B.1.2).

B.1.4 General

Usually the middle of the double-capped tube is selected for the site to condense the mercury.

For a single-capped lamp the middle of one of the legs is selected.

The lamp shall be operated on a suitable control gear while condensing the free mercury at the cold spot.

Operating the lamp during mercury collection causes large temperature difference between the cold spot and the rest of the lamp, thereby speeding collection rate.

The size of the cold spot area should be relative to the size of the lamp. In a typical 120 cm fluorescent lamp, the cold spot is made approximately 10 cm long. For smaller lamps, the cold spot area will necessarily be reduced. The glass segment eventually removed shall be greater than the cold spot size.

B.1.5 Liquid nitrogen treatment of a cold spot

Although it is only necessary to keep the cold spot cool once the condensation process is completed, the collection may be preserved by means of liquid nitrogen.

This is done by tightening cotton wool around the cold spot surface and then soaking the cotton wool with liquid nitrogen for 10 min. After this treatment, the lamp is prepared to cut open.

B.1.6 Extracting the cold spot segment

It is best to condense the mercury to a cold spot away from the electrode regions. This allows extraction of the cold spot segment with comfortable margins between the electrode regions and the cold spot centre. After a small crack is made in the glass discharge tube and it fills with air, the remainder of the discharge tube may be readily segmented.

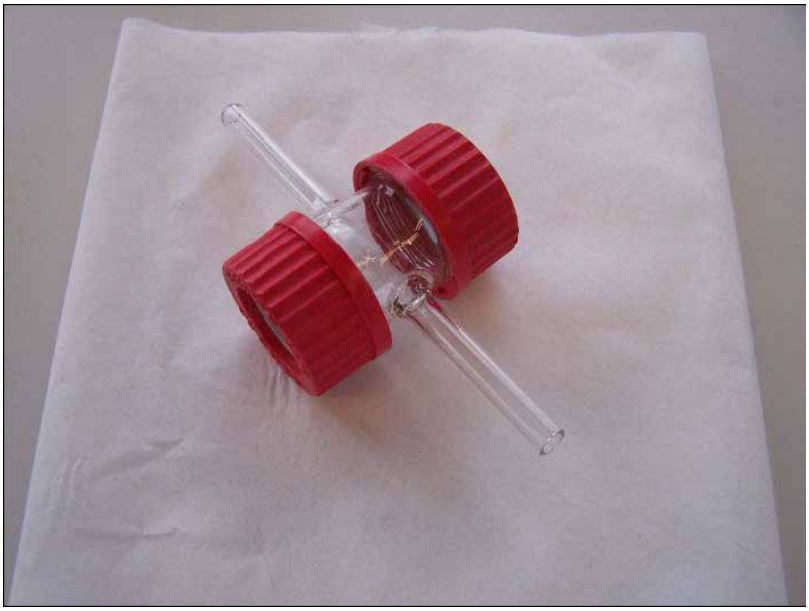
B.2 Detailed procedure for condensation of free mercury to the cold spot

B.2.1 Double-capped lamp

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

a) Measure the length of the fluorescent lamp and mark the middle of the lamp.

b) Place the glass cell (see Figure B.1) on the mark in the middle of the lamp. The glass cell fits completely and tight around the fluorescent lamp.



IEC 1850/11

Figure B.1 – Example of glass cell arrangement

c) Connect the glass cell to the cooling instrument using plastic hosing. The cooling instrument supplies a continuous flow of a water - ethanol mixture at approximately 0 °C through the hosing to the glass cell.

d) Next, transfer the lamp, with the glass cell attached, to a burning rack.

e) Attach the cooling instrument and secure such that there is a continuous flow of water - ethanol in the glass cell.

f) Let the lamp burn with properly selected control gear, and condensation of the free mercury to the cold spot will start.

g) When the lamp burns “dark”, the cooling instrument is switched off and the glass cell is removed.

h) The complete lamp is dried on the outside using a cotton towel and at once the cold spot area is wrapped with cotton wool and soaked with liquid nitrogen for 10 min (see B.1.5).

i) Next the lamp is transferred into a fume-hood.

j) The cold spot segment is removed by cutting the lamp open on both sides of the cold spot. To ensure the maximum mercury retention, open the lamp as far away as possible from the cold

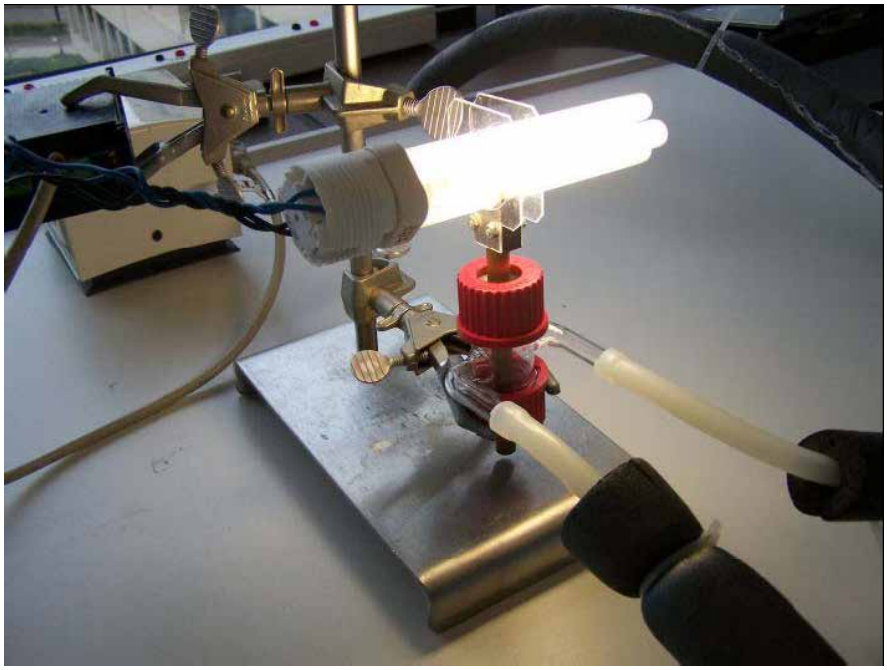
spot. This cutting takes place by means of an appropriate cutting tool. First, at both sides a scratch is made and the glass envelope is cracked, then once the pressure in the lamp is equal to the outside pressure, the lamp can be opened. The cold spot glass segment is transferred to a container for further analysis.

B.2.2 Single-capped lamp

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

a) Mark the middle of one leg of the lamp.

b) Place the cooling device (rod + glass cell, see Figure B.2) on the mark of the lamp.



IEC 1851/11

Figure B.2 – Example of cooling device arrangement

c) Put the lamp with the cooling device on a stand.

d) Connect the glass cell of the cooling device to the cooling instrument using plastic hosing. The cooling instrument supplies a continuous flow of a water - ethanol mixture at approximately 0 °C through the hosing to the glass cell.

e) Attach the cooling instrument and take care such that there is a continuous flow of water - ethanol in the glass cell.

f) Let the lamp burn with properly selected control gear and condensation of the free mercury to the cold spot will start.

g) When the lamp burns “dark”, the cooling instrument is switched off and the glass cell is removed from the cooling device.

h) The cooling device, still in contact with the lamp, is put into liquid nitrogen for 5 min.

i) Next the lamp is transferred into a fume-hood.

j) The cold spot segment is removed by cutting the lamp open on both sides of the cold spot. To ensure the maximum mercury retention, open the lamp is as far away as possible from the cold spot. This cutting takes place by means of an appropriate cutting tool. First, at both side a scratch is made and the glass envelope is cracked, then once the pressure in the lamp is equal to the outside pressure, the lamp can be opened. The cold spot glass segment has to be transferred to a container for further analysis.

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