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Fireworks — Test methods for determination of specific chemical substances —

Part 9: Mercury content by hydride generation atomic fluorescence spectrometry

Artifices de divertissement — Méthodes d'essai pour la détermination de substances chimiques spécifiques —

Partie 9: Teneur en mercure par spectrométrie de fluorescence atomique par génération d'hydrures

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Foreword

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This document was prepared by Technical Committee ISO/TC *264*, *Fireworks*.

A list of all the parts in the ISO 22863 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fireworks — Test methods for determination of specific chemical substances —

Part 9: Mercury content by hydride generation atomic fluorescence spectrometry

1 Scope

This document specifies the test method for the determination of the mercury content in pyrotechnic compositions by hydride generation atomic fluorescence spectrometry.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 22863-1, *Fireworks — Test methods for determination of specific chemical substances — Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22863-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle of the method

The sample is heated and digested in a boiling water bath using a nitric acid - hydrochloric acid mixed reagent. In the acidic medium, the mercury in the sample is reduced to atomic mercury by potassium borohydride and then loaded into the atomic fluorescence photometer by a carrier gas (argon). Under the irradiation of a mercury hollow cathode lamp, the mercury atoms will emit fluorescence with a characteristic wavelength when they transit from high energy state to ground state. The fluorescence intensity is proportional to the mercury concentration in the liquid to be measured and is quantitatively compared with mercury standard solutions.

5 Reagents

5.1 Hydrochloric acid (GR)

5.2 Nitric acid (GR)

5.3 Potassium dichromate (GR)

5.4 Sodium hydroxide (GR)

5.5 Potassium borohydride (AR)

5.6 Nitric acid solution (volume fraction 5 %):

Take 50 ml of nitric acid (5.2) with a pipette and dilute it to 1 000 ml with water.

5.7 Nitric diluted solution of potassium dichromate (0,5 g/l):

Weigh and dissolve 0,5 g of potassium dichromate (5.3) in 1 000 ml of nitric acid solution (5.6).

5.8 Hydrochloric acid-nitric acid mixed reagent [(1 + 1) aqua regia]:

Mix 150 ml of hydrochloric acid (5.1) with 50 ml of nitric acid (5.2) and then dilute it with water to double.

5.9 Sodium hydroxide solution (mass fraction 0,2 %):

Weigh and dissolve 1,0 g of sodium hydroxide (5.4) in 500 ml of water.

5.10 Potassium borohydride solution (mass fraction 2 %):

Weigh and dissolve 10,0 g of potassium borohydride (5.5) in 500 ml of sodium hydroxide solution (5.9).

5.11 Mercury standard solution (1 000 mg/l)

5.12 Mercury standard intermediate solution (1 µg/ml):

Take 100 µl of mercury standard solution (5.11) with a pipette, dilute it to 100 ml by adding nitric diluted potassium dichromate solution (5.7), shake well.

5.13 Mercury standard use solution (20 µg/l):

Take 1 ml of mercury standard intermediate solution (5.12) with a pipette and dilute it to 50 ml by adding nitric diluted potassium dichromate solution (5.7), shake well.

5.14 Preparation of mercury standard working curve solutions:

Separately place 0,0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml, 5,0 ml mercury standard use solution (5.13) in a 50 ml volumetric flask (6.5), dilute with the nitric diluted potassium dichromate solution (5.7) to 50 ml so that the concentrations of the mercury standard working curve solutions are 0,0 µg/l, 0,2 µg/l, 0,4 µg/l, 0,8 µg/l, 1,2 µg/l and 2,0 µg/l. Shake well.

6 Apparatus

6.1 Agate mortar

6.2 80 mesh standard sample sieve

6.3 Water bath

6.4 Atomic fluorescence photometer: equipped with a mercury hollow cathode lamp

6.5 Volumetric flasks (50 ml)

6.6 Capped test tubes: volume 100 ml

6.7 Filter paper

6.8 Analytical balance, accurate to 0,0001 g

7 Test procedure

7.1 Sample pre-treatment, digestion and preparation of the solution to be tested

Firstly, the sample is crushed in the agate mortar (6.1) and then sieved with a 80-mesh standard sample sieve (6.2). The sieved sample powder is weighed to 0,2 g using the analytical balance (6.8) and placed in the 100 ml capped test tube (6.6), add 2 ml of water, shake to mix.

Then 15 ml of the hydrochloric acid - nitric acid mixed reagent (5.8) is added. Shake well and place it for 2 hours in the boiling water bath (6.3), and then take it out from the water bath and let it cool for a short while. Add nitric diluted solution of potassium dichromate (5.7) to 100 ml and shake well.

Filter the solution through a filter paper (6.7) and then place it on the atomic fluorescence photometer (6.4). Perform the test.

At the same time, a blank test shall be carried out. Prepare a blank test solution by mixing 2 ml of water with 15 ml of the hydrochloric acid - nitric acid mixed reagent (5.8). Shake well and place it for 2 hours in the boiling water bath (6.3), and then take it out from the water bath and let it cool for a short while. Add nitric diluted solution of potassium dichromate (5.7) to 100 ml and shake well. Place it on the atomic fluorescence photometer (6.4). Perform the blank test.

7.2 Test conditions

The operative conditions of the atomic fluorescence spectrometer (6.4) shall be set to the appropriate settings to obtain the best performance.

For instance, the following requirements shall apply to the atomic fluorescence photometer where appropriate:

Negative high pressure/voltage: 270 V; lamp current: 30 mA; furnace height: 10 mm; carrier gas flow: 500 ml/min; shielding flow: 1 000 ml/min; reading mode: peak area; measurement method: standard curve method.

Instrument precision requirements: the blank test solution is measured several times, fluorescence intensity range of not more than 5.

8 Calculations

Calculate the mercury concentration by using Formula (1):

$$W(Hg)=\frac{(p-p_0)\cdot V}{1000m} \quad (1)$$

where

$W(Hg)$ is the content of mercury in the sample, mg/kg or µg/g.

p is the concentration of the test solution measured on the atomic fluorescence photometer, µg/l.

p_0 is the concentration of the reagent blank measured on the atomic fluorescence photometer, µg/l

V is the volume of the sample after the digestion process is completed, ml

m is the mass of the sample, g

1 000 is the coefficient of convert "ml" to "l".

9 Accuracy

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 20 % of the arithmetic mean.

Accuracy improvement can be obtained using the standard addition method (See Annex A)

10 Other

When the sample is 0,2 g and the volume is 100 ml, the detection limit is 0,03 mg/kg and the limit of quantification is 0,09 mg/kg.

11 Test report

The test report shall include at least the following information:

— name and address of the testing laboratory;

— date of issue;

— reference to this document, i.e. ISO 22863-9:2021;

— necessary description of the sample and how it was obtained according to ISO 22863-1;

— the identification of qualitative analysis and quantitative analysis;

— results of the analysis;

— any anomaly that occurred while performing the tests.

Annex A
(informative)

Standard addition method

A.1 General

This second method eliminates the “matrix” effects that result from the digestion process where other ions corresponding to other compounds than mercury ones may have been formed and remain in the digested sample solution to be tested. Such ions are likely to have an impact on the spectrometric records.

It may be used to improve the accuracy of measurements.

A.2 Sample size

Take one 0,5 g sample, using the analytical balance (6.8)

Duplicate the sample.

A.3 General requirement

The analysis of the two samples shall be carried out immediately one after the other.

For error correction, a blank test shall be carried out in parallel with a mercury-free blank solution.

A.4 Test procedure

A.4.1 Digestion process

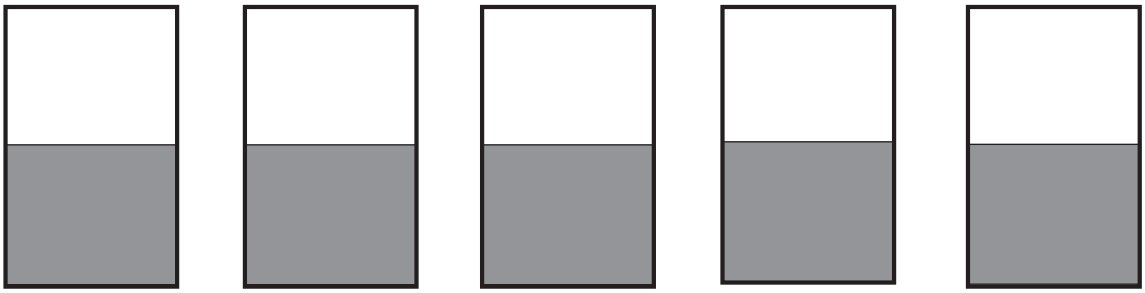
The same digestion process as described in (7.1) shall be carried out to obtain the digested sample solution that is to be diluted and tested according to A.4.2 to A.4.4.

The mentioned quantities of each of the acids to be used shall be multiplied by 2,5 to take into account the larger sample size as given in (A.2).

A.4.2 Dilution of the digested sample solution

Prepare 100 ml of a diluted solution of the standard diluted solution of mercury (5.13) to a concentration of 10,0 µg/l.

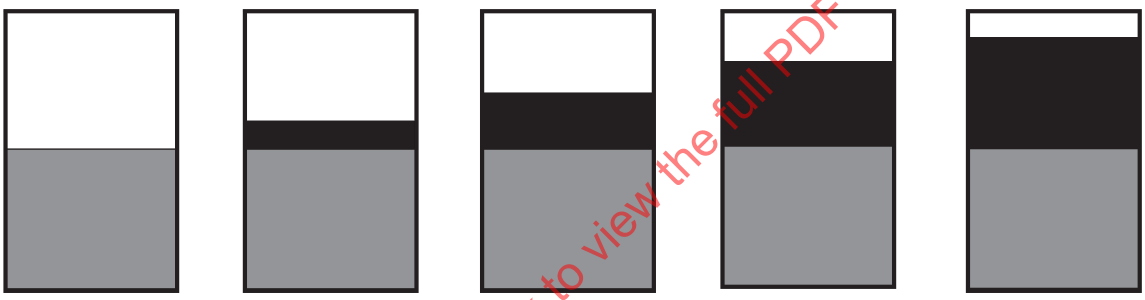
Pour 50 ml of the digested sample solution in each of a set of 100 ml flasks that shall be numbered from 1 to 5.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

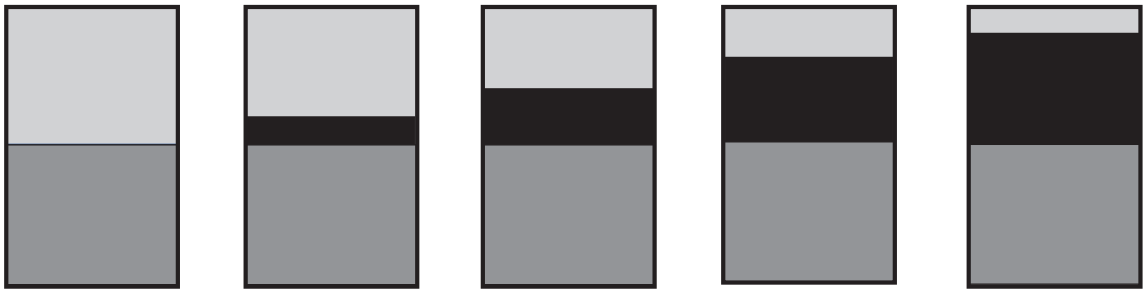
Add carefully 10 ml of the above 10,0 µg/l diluted solution of mercury in flask Nr 2, 20 ml of the same solution in flask Nr 3, 30 ml in flask Nr 4 and 40 ml in flask Nr 5.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

Add water in all flasks 1 to 5 up to the 100 ml graduation and mix minutely.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

The dosing of the digested sample solution and of the 10,0 µg/l diluted solution of mercury in each of the flasks shall be done as accurately as possible by using the best laboratory practices.

A.4.3 Measurement

Set up the atomic fluorescence photometer (6.4) to the optimized working conditions according to its manufacturer's instructions.

Perform the test according to manufacturer's instructions and record the absorbances for each of the solutions of flasks Nr 1 to 5.

A.4.4 Calculations

Calculate the concentration of added 10,0 µg/l diluted solution of mercury for each flask as measured after dilution.

Plot recorded absorbance values vs. corresponding calculated concentrations of added 10,0 µg/l diluted solution of mercury. The plotted points should be approximatively aligned.

Determine the equation of the regression line which is the closest from all points. Most spreadsheet software that are currently available have such capability.

Using the equation of the regression line, calculate the value where the regression line intercepts the x-axis. That value represents the concentration of mercury in flask Nr 1 and shall be multiplied by 2 to obtain the actual concentration C of the digested sample solution.