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Replace GB 26721—2011

Arsenic trioxide

三氧化二砷

*（English Translation）*

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the People’s Republic of China

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Foreword

SAC/TC 243 is in charge of this English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

This document is drafted in accordance with the rules given in the GB/T 1.1-2020 *Directives for standardization—Part 1: Rules for the structure and drafting of standardizing documents*.

This document replaces the GB 26721-2011 *Arsenic trioxide*. In contrast, in addition to structural and editorial changes, the major technical changes are as follows:

1. Chemical composition are modified (see 5.1,3.2 of the 2011 edition);
2. Physical performance and appearance quality are combined into appearance quality(see 5.2,3.3 and 3.4 of the 2011 edition);
3. Whiteness rating are modified(see 5.2,3.3 of the 2011 edition);
4. Accompanying documents are modified(see 8.3,6.3 of the 2011 edition);
5. The contents of the order are modified(see Chapter 9,Chapter 7 of the 2011 edition);
6. Appendix A.6 are modified(see Appendix A.6, Appendix A.6 of the 2011 edition);
7. Appendix A.7 are modified(see Appendix A.7, Appendix A.7 of the 2011 edition);
8. The determination of antimony content in Appendix B is added（see Appendix B）;
9. The range of antimony measurements in Appendix B is added（see Appendix B）;
10. The method of preparing antimony standard solution of reagent B 3.1 is added (see Appendix B.3.1.6 and B.3.1.7);
11. Antimony concentration in the standard solution of Annex B 3.1 reagent is added（see Appendix B.3.1）;
12. The wavelength data of the antimony analysis line in Appendix B.4.1.2 is added（see Appendix B.4.1.2）;
13. The contents of Appendix B.7 are modified(see Appendix B.7, Appendix B.7 of the 2011 edition).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. The issuing body of this document shall not be held responsible for identifying any or all such patent rights.

This document is proposed and prepared by the National Technical Committee on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

The previous editions of this document are as follows:

—The first edition was issued in 2011 as GB/T 26721-2011;

—This is the first revision edition

Arsenic trioxide

Warning: Arsenic trioxide is a highly toxic product.Some of the inspection and testing processes specified in this document may lead to dangerous situations, and it is the responsibility of the user to take appropriate safety and health measures.

1 Scope

This document specifies the product classification, technical requirements, test methods, inspection rules and markings, packaging, transportation, storage, accompanying documents and order content of arsenic trioxide.

This document is applicable to the arsenic trioxide produced by wet and fire processes, which is mainly used in preservatives, pesticides, glass industry, ceramics, dyeing and weaving, pigments, medicine, tanning, fireworks, etc.

2 Normative references

The following documents are essential 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.

GB 190 *Packing symbol of dangerous goods*

GB/T 1605 *Sampling method for commodity pesticides*

GB/T 8170 *Rules of rounding off for numerical values & expression and judgement of limiting values*

GB 13690 *General rule for classification and hazard communication of chemicals*

3 Terms and definitions

There are no terms and definitions to be defined in this document.

4 Product classification

Arsenic trioxide is divided into three grades according to chemical composition: As2O3-1, As2O3-2, As2O3-3.

5 Technical requirements

5.1 Chemical compositions

The chemical compositions of Arsenic trioxide shall conform to the requirements in Table 1.

Table 1 Chemical compositions of Arsenic trioxide(%)

|  |  |  |  |
| --- | --- | --- | --- |
| Grade | As2O3-1 | As2O3-2 | As2O3-3 |
| Chemical compositions | As2O3,Min | 99.5 | 98.0 | 95.0 |
| Contents of impurity, Max | Cu | 0.005 | — | — |
| Zn | 0.001 | — | — |
| Fe | 0.002 | — | — |
| Pb | 0.001 | — | — |
| Bi | 0.01 | — | — |
| Sb | 0.20 | — | — |
| Note: The requirements for impurity elements not listed in Table 1,the supplier and buyer shall negotiate it.  |

5.2 Appearance quality

5.2.1 Arsenic trioxide should be a white or off-white powder or granule.

5.2.2 The particle size of arsenic trioxide and other composition requirements ,the supplier and buyer shall negotiate it.

5.2.3 The whiteness of arsenic trioxide shall conform to the requirements in Table 2.

Table 2 Whiteness of Arsenic trioxide

|  |  |  |  |
| --- | --- | --- | --- |
| Grade | As2O3-1 | As2O3-2 | As2O3-3 |
| Witeness,Min | ≥60 | ≥40 | — |

6 Test methods

6.1 The test of the arbitration analysis method of arsenic trioxide chemical compositions shall be determined by the supplier and the buyer through negotiation, and the test may also refer to Annex A and Annex B.

6.2 The whiteness of Arsenic trioxide is determined in accordance with Annex C, and the determination of moisture, particle size shall be negotiated by the supplier and buyer.

6.3 The appearance quality of arsenic trioxide shall be examined visually.

7 Inspection rules

7.1 Inspection and acceptance

7.1.1 Arsenic trioxide shall be tested by the supplier or a third party quality supervision department to ensure that the product quality meets the provisions of this document, and fill in the accompanying documents.

7.1.2 The buyer shall make quality inspection on received products. In case the inspection result does not conform to this document or the requirements of the purchase order, the buyer may notify the supplier within 90 days after receiving the products, and the supplier and the buyer shall negotiate to settle it. In case of arbitration, the sampling for arbitration shall be made jointly by the buyer and the supplier in the buyer’s site.

7.2 Lot

The products shall be submitted for acceptance in batches. Each batch shall consist of arsenic trioxide produced on the same batch of feeding, from the same production cycle and the same designation.

7.3 Inspection items

Each lot of arsenic trioxide should be inspected for chemical compositions, whiteness and appearance quality. The whiteness, moisture, particle size and other components can be determined if the buyer has requirements.

7.4 Sampling and sample preparation

7.4.1 The sampling method shall be determined according to GB/T 1605.

7.4.2 Sample preparation method: The sample was divided into 80g by the quarter method, and put into a dry, airtight container with closed light, and stored for later use.

7.5 Judgement of inspection results

7.5.1 The inspection results of rounding numerical and judgement shall be determined according to GB/T 8170.

7.5.2 If the test result of chemical compositions does not conform to the requirements of this document or the order, the batch of products shall be judged as unqualified.

7.5.3 If the measured result of appearance quality does not conform to the requirements of this document or the order, it shall be disqualified according to the barrel.

8 Marking, packaging, transportation, storage and accompanying documents

The marking, packaging, transportation, storage and accompanying documents of arsenic trioxide as specified in GB 190 and GB 13690.

8.1 Marking

8.1.1 This product should be marked on the outside of the packaging bucket.

8.1.2 Each barrel packaging should be indicated:

a) name and address of the manufacturer;

b) product name and brand;

c) batch number and weight;

d) document number.

8.2 Packaging, transportation and storage

8.2.1 Arsenic trioxide shall be packed in sealed iron barrels with the inner lining is not easy to fall off and with anti-corrosion paint or plastic packaging, the net weight per barrel can be 25 kg, 50 kg, 100 kg, 200 kg, or 250 kg. If any special requirements exist, the supplier and buyer shall negotiate it.

8.2.2 The transportation and storage of arsenic trioxide must be provided with safety conditions ,such as clean, moisture-proof, anti-corrosion, and breakage-proof.Do not contact with other pollutants to avoid product leakage and pollution.

8.3 Accompanying documents

Each batch of products shall be accompanied by an accompanying document indicating:

a) name and trademark, address, telephone or fax of the supplier;

b) product name and brand;

c) batch number；

d) net weight and number of packages；

e) analysis and inspection results and the seal of the technical supervision department;

f) document number；

g) date of manufacture.

9 Order contents

The order list of products listed in this document shall include the following information:

a) product name；

b) brand;

c) special requirements such as chemical compositions and appearance quality；

d) net weight and number of packages；

e) document number；

f) others.

A

（annex informative）

Determination of arsenic trioxide content Iodometry Iodine quantity method

A.1 Theory

Titration of As (III) to As(V) with iodine standard titration solution using starch as indicator in sodium hydroxide slightly alkaline solution.



A.2 Reagents or materials

A.2.1 Reagents

A.2.1.1 Potassium iodide

A.2.2 Sodium hydroxide solution (100 g/L)

A.2.3 Saturated sodium bicarbonate solution

A.2.4 Sulfuric acid (0.5 mol/L)

A.2.5 Sulfuric acid (1+9)

A.2.6 Phenolphthalein ethanol solution (10 g/L)

A.2.7 Starch solution (5 g/L)

A.2.8 Iodine standard titration solution [c (I2) = 0.062 mol/L]

a) Prepare a solution：Weigh 16g iodine tablets, place them in a 400mL beaker, mix with 50g potassium iodide, and dissolve them with 300mL water，strain in 1000mL volumetric bottle, dilute with water to scale and shake well. Leave for two weeks and calibrate.

b) Calibration：Weigh 0.30 grams（Accurate to 0.0001g）Arsenic trioxide（Purity of excellence，Containing As203，not less than 99.99%）Triplicate.The following procedures are carried out simultaneously with the test material determination according to A.4.2.3.The actual concentration of iodine standard titration solution was calculated according to Equation (A.2).



Formula:

*m*——Mass of arsenic trioxide in grams (g)；

$ω$——Mass fraction of arsenic trioxide in the reference material, expressed as a percentage (%);

*V*1——The volume of iodine standard titration solution consumed at calibration, in milliliters (mL)

0.0989——The mass of arsenic trioxide equivalent to 1.00 mL iodine standard titration solution [c (I2) = 1.00 mol/L] in grams per mole (g/mol).

When the relative error of the measured values was not more than 0.2 %, the average value was taken; otherwise, the calibration was re-calibrated.The actual concentration of iodine standard titration solution [c (I2)] should be between 0.062 mol/L and 0.063 mol/L，Otherwise, re-calibrate after adjustment.

A.3 Apparatus

Unit：millimeter

Figure A.1 Burette diagram

A.4 Testing procedure

A.4.1 Samples

Weigh the sample according to Table A.1, accurate to 0.0001 gram.

Table A.1 Sample weighing quality

|  |  |
| --- | --- |
| As2O3 mass fraction/% | Sample weighing quality/g |
| 94.00～97.00 | 0.305 |
| >97.00～99.70 | 0.300 |

A.4.2. Analysis procedure

A.4.2.1 Place the test sample (A.4.1) in a 500mL triangular beaker, add sodium hydroxide solution, heat at low temperature to dissolve until clear, rinse the cup wall with water, and cool to room temperature.

A.4.2.2 Add phenolphthalein ethanol solution dropwise, first neutralize with sulfuric acid (A.2.4) until the solution turns pale red, then neutralize with sulfuric acid (A.2.5) until the red color disappears.

A.4.2.3 Add 20 mL of saturated sodium bicarbonate solution and 2 mL of starch solution, and mix well. Titrate with iodine standard titration solution until light blue is the endpoint.

A.5 Test data processing

The mass fraction of arsenic trioxide ωAs2O3 was calculated according to Equation (A.3) :



Formula:

c——The actual concentration of iodine standard titration solution in moles per liter (mol/L);

V2——The volume of iodine standard solution consumed by the reagent, in milliliters (mL);

m0——The mass of the sample, in grams (g);

0.0989——The mass of arsenic oxide equivalent to 1.00mL iodine standard titration solution [c (I2) = 1.00mol/L]，The unit is grams per mole (g/mol);

ωSb——The mass fraction of antimony in the sample, expressed in percentage;

0.8125——The coefficient for converting antimony to arsenic trioxide.

The result is expressed to two significant digits.

A.6 Precision

A.6.1 Repeatability

In the range of the average given below, the absolute difference between the two independent test results obtained under the condition of repeatability does not exceed the repeatability limit (r), and the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is obtained by linear interpolation or extrapolation method according to the data in Table A.2.

Table A.2

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| As2O3 mass fraction/% | 94.31 | 95.68 | 98.20 | 99.62 |
| r/% | 0.19 | 0.24 | 0.36 | 0.40 |

A.6.2 Reproducibility

Within the average range given below, the absolute difference between the two independent test results obtained under the reproducibility condition does not exceed the reproducibility limit (R), and the reproducibility limit (R) does not exceed 5 %. The reproducibility limit (R) is obtained by linear interpolation or extrapolation method according to the data in Table A.3.

Table A.3

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| As2O3 mass fraction/% | 94.31 | 95.68 | 98.20 | 99.62 |
| R/% | 0.23 | 0.29 | 0.44 | 0.49 |

B

（annex informative）

Determination of copper, lead, zinc, iron, bismuth and antimony in arsenic trioxide Inductively coupled plasma atomic emission spectrometry

B.1 Theory

The samples were dissolved with hydrochloric acid and nitric acid. The contents of Cu, Pb, Zn, Fe, Bi and Sb were determined by inductively coupled plasma atomic emission spectrometry in acidic medium. The measurement range is shown in Table B.1.

Table B.1 Measuring range

|  |  |  |  |
| --- | --- | --- | --- |
| Element | Measuring range/% | Element | Measuring range/% |
| Cu | 0.0010%～0.010% | Fe | 0.0010%～0.010% |
| Pb | 0.0010%～0.010% | Bi | 0.010%～0.10% |
| Zn | 0.0010%～0.010% | Sb | 0.010%～1.00% |

B.2 Reagents or materials

B.2.1 Reagents

B.2.1.1 Hydrochloric acid (1+1)

B.2.1.2 Nitric acid (1+1)

B.2.1.3 Standard storage solutions of copper, lead, zinc, iron, and bismuth: Weigh 0.5000 g of copper (≥99.99 %), 0.5000 g of lead (≥99.99 %), 0.5000 g of zinc (≥99.99 %), 0.5000 g of iron (≥99.99 %), and 0.5000 g of bismuth (≥99.99 %) (all of superior purity) into a 400 mL beaker, cover with a watch glass, and gradually add 50 mL of nitric acid (B.2.1.2) in portions to completely dissolve at a low temperature. Cool to room temperature, transfer to a 500 mL volumetric flask, dilute to the mark with water, and mix well. This solution contains 1 mg of copper, 1 mg of lead, 1 mg of zinc, 1 mg of iron, and 1 mg of bismuth per mL.

B.2.1.4 Mixed standard solution A of copper, lead, zinc, iron, and bismuth: Pipette 10.00 mL of the standard storage solution of copper, lead, zinc, iron, and bismuth (B.2.1.3) into a 100 mL volumetric flask, add 10 mL of nitric acid (B.2.1.2), dilute to the mark with water, and mix well. This solution contains 100μg of copper, 100μg of lead, 100μg of zinc, 100μg of iron, and 100μg of bismuth per mL.

B.2.1.5 Mixed standard solution B of copper, lead, zinc, iron, and bismuth: Pipette 10.00 mL of the mixed standard solution of copper, lead, zinc, iron, and bismuth (B.2.1.4) into a 100 mL volumetric flask, add 10 mL of nitric acid (B.2.1.2), dilute to the mark with water, and mix well. This solution contains 10μg of copper, 10μg of lead, 10μg of zinc, 10μg of iron, and 10μg of bismuth per mL.

B.2.1.6 Standard storage solution of antimony: Weigh 0.5000 g of antimony (≥99.99 %) (of superior purity) into a 400 mL beaker, cover with a watch glass, gradually add 10 mL of nitric acid (B.2.1.2) and 30 mL of hydrochloric acid (B.2.1.1) to completely dissolve at a low temperature. Cool to room temperature, add an additional 200 mL of hydrochloric acid (B.2.1.1). Transfer to a 500 mL volumetric flask, dilute to the mark with water, and mix well. This solution contains 1 mg of antimony per mL.

B.2.1.7 Standard solution of antimony: Pipette 10.00 mL of the antimony standard storage solution (B.2.1.6) into a 100 mL volumetric flask, add 40 mL of hydrochloric acid (B.2.1.1), dilute to the mark with water, and mix well. This solution contains 100μg of antimony per mL.

B.2.1.8 Series of standard solutions for copper, lead, zinc, iron, bismuth, and antimony curves: Pipette 0.00 mL, 1.00 mL, 3.00 mL, 5.00 mL, 8.00 mL, and 10.00 mL of the mixed standard solution B of copper, lead, zinc, iron, and bismuth (B.2.1.5) into 100 mL volumetric flasks labeled 1 to 6. Pipette 0.00 mL, 1.00 mL, and 5.00 mL of the antimony standard solution (B.2.1.7) into flasks labeled 1 to 3, and 2.00 mL, 5.00 mL, and 10.00 mL of the antimony standard storage solution (B.2.1.6) into flasks labeled 4 to 6. Add 15 mL of hydrochloric acid (B.2.1.1) and 5 mL of nitric acid (B.2.1.2), dilute to the mark with water, and mix well. The concentrations of the working standard solutions of copper, lead, zinc, iron, bismuth, and antimony are listed in Table B.2.

Table B.2 Standard Solution Concentration

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Standard Solution Series No. | 1 | 2 | 3 | 4 | 5 | 6 |
| Cu、Pb、Zn、Fe、Bi/(μg/mL) | 0 | 0.1 | 0.3 | 0.5 | 0.8 | 1.0 |
| Sb/(μg/mL) | 0 | 1.0 | 5.0 | 20.0 | 50.0 | 100.0 |

B.2.2 Materials

High-purity Argon (purity greater than 99.99%).

B.3 Apparatus

B 3.1 Inductively coupled plasma atomic emission spectrometer

B 3.1.1 .Stability of instrument shown in Table B.3.

Table B.3 Plasma Spectrometer Measurement Conditions

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Item | Power (W) | Auxiliary Gas Flow Rate (L/min) |  Carrier Gas Pressure (MPa) | Pump Speed (r/min) | Integration Time (s) |
| Measurement Conditions | 1150 | 0.5 | 0.18 | 100 | 5～30 |

B 3.1.2 The recommended analysis lines for each element are shown in Table B.4.

Table B.4 Recommended analytical lines for each element

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Element | Cu | Pb | Zn | Fe | Bi | Sb |
| Analysis line /nm | 324.75 | 220.35 | 206.20 | 259.94 | 223.06 | 206.83 |

B.4 Testing procedure

B.4.1 Test material

Weigh the 1.0g sample，Accurate to 0.0001g.

Perform two independent measurements and take the average value.

B.4.2 Blank test

Make a blank test with the test material.

B.4.3 Sample Dissolution

B.4.3.1 Place the test sample (B.4.1) in a 150mL beaker, cover with a watch glass, moisten with water, add 20mL of hydrochloric acid (B.2.1.1), and 5mL of nitric acid (B.2.1.2) to dissolve completely at low temperature. Cool to room temperature.

B.4.3.2 Transfer the dissolved sample solution (B.4.3.1) into a 100mL volumetric flask, dilute to the mark with water, and mix well.

B.4.4 Working Curve

Introduce the working curve standard solutions (B.2.1.8) into the plasma spectrometer successively to measure the spectral line intensity of each element, and plot the working curves for each element. The linearity (r) of the working curve should be ≥0.999.

B.4.5 Measurement

Introduce the blank solution (B.4.2) and the sample solution (B.4.3.2) into the plasma spectrometer for measurement separately. The computer will automatically provide the concentration of each measured element in the sample.

B.5 Test data processing

Calculate the contents of copper, lead, zinc, iron, bismuth, and antimony separately according to formula (B.1), expressed as mass fraction wx, and the numerical value is expressed in %:



Formula:

ρ1----The mass concentration of the tested element in the test solution, in micrograms per milliliter (μg/mL);

ρ0----The mass concentration of the element to be measured in the blank test solution, in micrograms per milliliter (μg/mL);

V0----The volume of the sample solution, in milliliters (mL);

m0----The mass of the sample, in grams (g);

The results should be expressed to four decimal places.

B.6 Precision

B.6.1 Repeatability

In the range of the average given below, the absolute difference between the two independent test results obtained under the condition of repeatability does not exceed the repeatability limit (r), and the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is obtained by linear interpolation or extrapolation method according to the data in Table B.5.

Table B.5 repeatability limit

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Cu mass fraction/% | 0.0010 | 0.0025 | 0.0052 | 0.0089 |
| r/% | 0.0002 | 0.0003 | 0.0004 | 0.0005 |
| Pb mass fraction/% | 0.0010 | 0.0018 | 0.0051 | 0.0091 |
| r/% | 0.0003 | 0.0004 | 0.0005 | 0.0007 |
| Zn mass fraction/% | 0.0011 | 0.0021 | 0.00055 | 0.00094 |
| r/% | 0.0004 | 0.0005 | 0.0006 | 0.0008 |
| Fe mass fraction/% | 0.0010 | 0.0026 | 0.00053 | 0.0092 |
| r/% | 0.0003 | 0.0004 | 0.0005 | 0.0006 |
| Bi mass fraction/% | 0.011 | 0.025 | 0.053 | 0.098 |
| r/% | 0.001 | 0.002 | 0.004 | 0.008 |
| Sb mass fraction/% | 0.010 | 0.15 | 0.30 | 0.98 |
| r/% | 0.001 | 0.02 | 0.03 | 0.04 |

B.6.2 Reproducibility

Within the average range given below, the absolute difference between the two independent test results obtained under the reproducibility condition does not exceed the reproducibility limit (R), and the reproducibility limit (R) does not exceed 5%. The reproducibility limit (R) is obtained by linear interpolation or extrapolation method according to the data in Table B.6.

Table B.6 reproducibility limit

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Cumass fraction/% | 0.0010 | 0.0025 | 0.0052 | 0.0089 |
| R /% | 0.0003 | 0.0004 | 0.0006 | 0.0008 |
| Pbmass fraction/% | 0.0010 | 0.0018 | 0.0051 | 0.0091 |
| R /% | 0.0004 | 0.0005 | 0.0007 | 0.0009 |
| Znmass fraction/% | 0.0011 | 0.0021 | 0.00055 | 0.0094 |
| R /% | 0.0005 | 0.0006 | 0.0008 | 0.0009 |
| Fe mass fraction/% | 0.0010 | 0.0026 | 0.00053 | 0.0092 |
| R /% | 0.0004 | 0.0005 | 0.0007 | 0.0008 |
| Bi mass fraction/% | 0.011 | 0.025 | 0.053 | 0.098 |
| R /% | 0.002 | 0.003 | 0.006 | 0.010 |
| Sb mass fraction/% | 0.010 | 0.15 | 0.30 | 0.98 |
| R /% | 0.001 | 0.03 | 0.04 | 0.05 |

C

（annex informative）

Arsenic trioxide - Determination of whiteness Comparative method of whiteness meters

C.1 Theory

The arsenic trioxide sample was compared with the standard whiteboard to measure the whiteness of the sample.

C.2 Apparatus

Kate digital powder whiteness meter (C-100 type);

Measurement principle:Reflective index with photo-diode;

Resolution：0.1;

Refraction：GAP photo-diode;

Light source：Halogen lamp；

Blue light filter：The central wavelength was 440 nm.

C.3 Testing procedure

a) Open the upper cover of the instrument, confirm that the filter is a blue filter, and press the blue button.

b) Verify that the number on the back of the instrument is the same as the value on the standard whiteness plate, and can be adjusted manually.

c) The standard whiteness plate was mounted in the sample dish holder and the holder was inserted into the measurement chamber.

d) Connect the power supply, turn on the switch, after about 6 minutes, the "WAIT" light goes off, and the display displays the standard whiteness value. If the display value is different from the standard whiteness value, press the "SENS" key to adjust it automatically.

e) The sample plate holder filled with samples was inserted into the measurement chamber, and the built-in switch was activated to test the sample whiteness value. The sample whiteness value was measured twice in a row, and the AVERAGE result was obtained by pressing the "average" key.

f) After the determination, clean the sample dish and support with a vacuum cleaner and return them to their original places.